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Advanced Characterization and Modeling of Coatings and Inter- faces for next-Generation Power Semiconductor Devices

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Abstract

This doctoral thesis investigates several challenges introduced by the industrial transition toward 300 mm wafer manufacturing for next-generation power devices, with particular focus on Insulated Gate Bipolar Transistors (IGBTs). The increase in wafer size has led to an intensification of mechanical deformation phenomena, which can compromise the structural integrity and reliability of the device, as well as to the introduction of new materials and process steps that generate additional manufacturing challenges. The research addresses these issues through a multiscale strategy that combines numerical modeling and experimental characterization of thin films and interfaces.

In the first part of the study, a finite element analysis (FEA) methodology was developed to accurately predict wafer warpage induced by thin-film deposition during selected IGBT manufacturing steps. Such mechanical deformation can assume asymmetric shapes, such as saddle-like curvature, driven by the presence of a regular pattern of high-aspect-ratio trenches covering the entire wafer surface. By comparing a “discrete trench method” with a homogenization approach, it was demonstrated that the inclusion of intrinsic residual stresses is essential to achieve good agreement between simulations and experimental data. These models aim to provide a predictive tool of significant interest for the optimization of industrial design and to reduce costly and time-consuming empirical approaches.

In parallel, the study focused on the mechanical properties of IGBT coating materials, namely the silicon nitride (SiN_x) passivation layer and polybenzoxazole (PBO), a dielectric polymer overcoat. A systematic characterization of three PECVD-synthesized SiN_x thin films was carried out, investigating how stoichiometric variations affect fracture toughness and residual stress development. In addition, adhesion at the interface between silicon nitride and PBO was studied as a function of substrate (SiN_x) surface treatments and environmental parameters. The results highlight the critical role of surface treatments and ambient humidity in delamination mechanisms, providing guidelines for improving the long-term reliability of protective coatings.

In conclusion, this thesis proposes an integrated approach that combines materials science and process engineering to address key challenges associated with the transition to 300 mm wafers, tailored to specific industrial needs in the microelectronics sector.

Sommario

Il presente lavoro di tesi di dottorato analizza alcune sfide critiche introdotte dalla transizione industriale verso la produzione su wafer da 300 mm per i dispositivi di potenza di nuova generazione, con particolare riferimento agli Insulated Gate Bipolar Transistors (IGBT). L'aumento delle dimensioni dei wafer ha infatti portato a un'intensificazione dei fenomeni di deformazione meccanica, che possono compromettere l'integrità strutturale e l'affidabilità dei dispositivi, e all'implemento di nuovi materiali e step di produzione da cui possono originare ulteriori sfide procedurali. La ricerca affronta queste problematiche attraverso una strategia multi-scala che combina modellazione numerica e caratterizzazione sperimentale di film sottili e interfacce.

Nella prima parte della ricerca, è stata sviluppata una metodologia di simulazione agli elementi finiti (FEA) per predire accuratamente l'imbarcamento dei wafer, indotto dalla deposizione di film sottili in alcuni step di produzione dell'IGBT. Tale deformazione meccanica assume infatti forme asimmetriche, ad esempio a sella, indotte dalla presenza di un pattern regolare di trincee con rapporti di forma piuttosto elevati che ricopre l'intera superficie dei wafer. Attraverso il confronto tra un "metodo a trincee discrete" e un metodo di omogeneizzazione, è stato dimostrato come l'inclusione degli stress residui intrinseci sia essenziale per la fedeltà del modello rispetto ai dati sperimentali. Questi modelli cercano di introdurre uno strumento predittivo di grande interesse per l'ottimizzazione del design industriale e la riduzione di approcci empirici costosi in termini di tempo e denaro.

Parallelamente, lo studio si è concentrato sulle proprietà meccaniche dei materiali di rivestimento dell'IGBT, ovvero il film di passivazione di nitruro di silicio (SiN_x) e il polybenzoxazolo (PBO), un polimero dielettrico di ricoprimento. È stata condotta una caratterizzazione sistematica di tre film sottili di SiN_x , depositati tramite PECVD, analizzando come le variazioni stechiometriche influenzino la tenacità a frattura e lo sviluppo dello stress residuo. Inoltre, è stata indagata l'adesione all'interfaccia tra il nitruro di silicio e il PBO al variare dei trattamenti superficiali del substrato (il SiN_x) e dei parametri ambientali. I risultati evidenziano il ruolo determinante dei trattamenti superficiali e dell'umidità ambientale nei meccanismi di delaminazione, fornendo alcune linee guida per migliorare l'affidabilità a lungo termine dei coating protettivi.

In conclusione, questa tesi propone un approccio integrato che unisce scienza dei materiali e ingegneria di processo, per offrire soluzioni ad alcune problematiche legate alla transizione verso i 300 mm dei wafer applicate a specifiche necessità aziendali nel settore della microelettronica.

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Introduction

Since the inception of the integrated circuit in the late 50s and the subsequent rapid development of microelectronics between 1960 and 1970, the semiconductor industry has been driven by a relentless pursuit of device miniaturization. This trend was famously formalized by Gordon Moore in 1965, who observed that the number of transistors on a microchip doubles approximately every two years, leading to exponential increases in computational power and energy efficiency. This empirical observation, widely known as Moore's Law, has been a guiding principle for semiconductor research and development for over five decades.

However, as technology nodes have progressed into the deep sub-micron and nanometer regimes, the physical and economic challenges associated with further miniaturization have become increasingly formidable. Quantum mechanical effects, short-channel effects, variability, and heat dissipation issues impose fundamental limits on transistor scaling. Moreover, the escalating costs of fabrication facilities and process complexity have raised questions about the sustainability of Moore's Law as a driver of innovation. Consequently, the semiconductor industry is witnessing a paradigm shift. The focus is gradually moving away from aggressive device scaling towards alternative strategies for performance enhancement, including architectural innovation, heterogeneous integration, novel materials, and process optimization.

Microelectronic devices are fabricated starting from a thin disc of a semiconductor material - typically silicon - called wafer, which allows for several chips or integrated circuits to be produced simultaneously during processing. The wafer acts both as a support for subsequent grown structures and as a part of each device itself, undergoing processes which locally modify its electrical or morphological properties. Increasing its size has emerged as a key approach to improving manufacturing efficiency and reducing cost per chip, thereby extending the economic viability of semiconductor production.

The transition from 200 mm to 300 mm diameter silicon wafers represents a significant milestone in semiconductor manufacturing. Increasing wafer size offers substantial advantages, foremost among them the ability to produce a greater number of devices per

wafer, thereby improving throughput and reducing unit costs. This scaling up aligns with industry goals to maintain competitiveness and meet the growing demand for advanced electronic components. However, the enlargement of the wafer area introduces a new set of engineering challenges that must be addressed to ensure device performance and reliability. Larger wafers are inherently more susceptible to mechanical deformation phenomena such as warpage, which can arise from intrinsic stresses in thin films, thermal gradients, and heterogeneous material properties. Warpage manifests as curvature or distortion of the wafer surface, potentially leading to alignment issues, yield loss, and mechanical failures during processing. While this issue was not critical for 200 mm diameter wafers, it can instead lead to serious damage and production blocking points for the enlarged wafers, even for well-known devices whose production was perfectly consolidated.

Within this frame, the present research is focused on a particular device called Insulated Gate Bipolar Transistor (IGBT), which represent by years a cornerstone for power electronics applications. The IGBT combines the advantages of metal-oxide-semiconductor field-effect transistors (MOSFETs) and bipolar junction transistors (BJTs), offering low conduction losses, high switching speeds, and the ability to withstand high voltages. These properties make IGBTs indispensable in sectors such as electric vehicles, renewable energy systems, industrial motor drives, and power conversion units. Despite the maturity gained by its long history in the semiconductor industry, the IGBT continues to benefit from ongoing research and the transition to 300 mm wafers has prompted renewed efforts in material selection, surface and interface engineering, and process-induced stress management to maintain device integrity at larger scales.

These priorities directly motivate my doctoral research lines in three main topics:

- Production of a modeling tool for prediction and understanding warpage phenomena in trench patterns on 300 mm wafers during three critical IGBT steps of production.
- Toughness evaluation of sub-micrometric PECVD SiN passivation layers of different stoichiometries.
- Assessment and ranking of interfacial adhesion between a novel organic coating, called Polybenzoxazole (PBO), and silicon nitride (SiN) passivation layers with different stoichiometries and surface treatments.

These three lines of research investigate the challenges related to the 300 mm transition at three different levels, going from the full-wafer scale to the single film investigation, ending up at the interfaces.

The subsequent chapters will delve into modeling approaches, experimental characteriza-

tion techniques, and interface engineering strategies developed throughout this doctoral research to address these challenges. The chapter list is composed as follows:

Chapter 1 - Warpage Phenomena and Objectives: the basic concepts framing warpage phenomena are reported, delving into main definitions, causes and characterization methods, and modeling approaches. By linking these concepts with 300 mm transition the main goals of this research will be stated, and then further developed in chapters 3, 4 and 5.

Chapter 2 - Methodologies and Experimental Setup: after the overview of the scientific framework and the definition of the objectives of this thesis work, the experimental methods employed in this research are reported. The chapter starts with a description of the adopted modeling tools and approaches, followed by the synthesis and characterization of all the material exploited for the experimental research.

Chapter 3 - Modeling the Trench-IGBT Warpage: this chapter reports the development and results of the first line of research regarding warpage prediction by FEA in trench-patterned IGBTs during key processing steps. These models support the first stages of the wafer processing, lightening the dependency from the trial & error approaches and reducing cost and time consumption.

Chapter 4 - Composition-Dependent Mechanical Behavior of Silicon Nitride Thin Film: the second line of research is discussed in this chapter, which follows the investigation of mechanical toughness of silicon nitride films by PECVD with varying stoichiometry. The results obtained support the recipe selection for this kind of passivation layers in function of the properties necessary for the specific role they must perform in the device.

Chapter 5 - In-Situ vs Ex-Situ Indentation for Adhesion Evaluation of Nitride/Organic interfaces: this chapter describes the evaluation of adhesion between organic PBO coatings and silicon nitride layers under different treatments and environmental conditions. The research focused on the effect that the substrate's surface treatments have on the adhesion with PBO, and the different behavior observed while testing the ambient conditions in-situ or ex-situ.

Chapter 6 - Conclusions and Prospectives: a summary of findings and recommendations for process optimization is reported in this last conclusive chapter, which focus also on the future prospectives of this thesis research.

The original content of this Ph.D. thesis has actively contributed to the publication of the following works:

- Applied Sciences: *In Situ vs. Ex Situ Indentation for Adhesion Evaluation of Nitride/Polymer Interfaces: A Comparative Study Under Controlled Ambient Conditions.* (<https://doi.org/10.3390/app16052485>)
- Thin Solid Films: *Investigating composition-dependent fracture toughness in silicon-rich silicon nitride layers.* (<https://doi.org/10.1016/j.tsf.2026.140898>)
- EuroSimE 2025 Proceedings: *Finite Element Analysis of the Asymmetric Warpage induced by the Oxidation Process in Trench Insulated Gate Bipolar Transistor (Trench IGBT) 12" Si patterned Wafers* (<https://hdl.handle.net/11311/1296637>)
- Application Note 1550 (Bruker website): *Adhesion Testing of Photosensitive Insulators to Passivation Layers Under Controlled Humidity (Application Note: Adhesion Testing of Photosensitive Insulators to Passivation Layers Under Controlled Humidity | Bruker)*

Moreover, the results achieved in this work were also presented at several international conferences, reported via Poster or Oral Presentation by myself:

Comsol Conference 2023 (Munich, Germany, 2023) – poster contribution: “*Predictive Simulations of Warpage Phenomena on Arbitrarily Patterned Silicon Wafers*”

MRS Spring Meeting & Exhibit 2024 (Seattle, USA, 2024) – poster contribution: “*Investigation of Amorphous Silicon Nanoparticles dispersed in Silicon Nitride Passivation Layer for Semiconductor Industry*”

Nanobrücken 2025 (Halle, Germany, 2025) – poster contribution: “*Investigating the Effect of Nanoparticles on Toughness in Silicon Nitride Thin Films*” & oral presentation: “*Moisture dependence of Polyimide - Silicon Nitride interfaces: comparison between In-Situ and Ex-Situ indentation-induced delamination*”

EuroSimE 2025 (Utrecht, Netherlands, 2025) – oral presentation: “*Finite Element Analysis of the Asymmetric Warpage induced by the Oxidation Process in Trench Insulated Gate Bipolar Transistor (Trench IGBT) 12" Si patterned Wafers*”

Finally, this project was carried out in collaboration with the company ST Microelectronics, which partially funded the research work and provided most of the materials and characterization tools used.

1 | Warpage Phenomena and Objectives

The evolution of semiconductor manufacturing, particularly in the context of increasing wafer diameters and complex device architectures, has brought to the surface the critical challenge of wafer warpage. Within this framework, warpage refers to the out-of-plane deformation of the processed wafer, manifested as deviations from the ideal flat and planar geometry required for precise lithographic alignment, bonding, and subsequent processing steps.

In section 1.1 of this chapter, a specific description of what is warpage will be provided, with considerations of its main geometrical definitions and the methodologies commonly adopted for measuring it. Section 1.2 will explore the causes of warpage formation, describing the main components and effects of residual stress, the classical and innovative methods to measure it, and the most commonly used approaches to reduce the accumulation of stress and, thus, the magnitude of warpage. However, Section 1.3 offers an outlook on the numerical approaches that can be used to simulate warpage phenomena, giving the opportunity of manipulating, or even predicting, the outcome of specific production steps on the final deformation of the wafer. Finally, section 1.4 reports the three main objectives of this thesis, describing the correlation with the 300 mm transition challenge for each of them.

1.1. Description of Warpage

The increase in wafer diameter from 200 mm to 300 mm significantly amplifies the mechanical and thermal challenges related to semiconductor manufacturing [1]. Larger wafers are inherently more susceptible to deformation due to their increased surface area and reduced structural rigidity, but also due to increased accumulation of residual stress across the expanded substrate and film layers [2, 3]. The larger wafer area intensifies the impact of stress gradients through the thickness of the deposited films and across the wafer surface: variations in deposition conditions, temperature gradients, and material prop-

erties manifest more prominently, resulting in spatially heterogeneous stress fields and contributing to non-uniform deformations [4]. Moreover, the dense patterning and large aspect ratio characteristics of certain devices, such as the trench structures in insulated gate bipolar transistors (IGBTs) [5], further exacerbate this effect, inducing directional variations in stress and leading to saddle-shaped warpage that becomes more pronounced in larger wafers [6].

From a reliability point of view, warpage influences mechanical stability and even electrical characteristics [7]. Non-planarity can hinder wafer handling and processing in several ways, also causing production shutdown in the most critical cases [8]. During photolithography, it can affect the depth of focus during exposure, resulting in blurred or distorted features that compromise device functionality. In micro-electromechanical systems (MEMS) and resonators, warpage-induced stiffness variations alter resonance frequencies, degrading sensor accuracy and performance [7]. Warpage also interferes with bonding processes, such as wafer-to-wafer or die-to-substrate bonding, by causing uneven contact and increasing the risk of void formation. In packaging, warpage complicates die attachment, flip-chip assembly, and wire bonding operations [1].

Zooming into a single device architecture, warpage can lead to more localized mechanical failures, such as cracking and delamination, which greatly reduce the device life expectancy [3, 9, 10]. Cracking typically occurs when the tensile stresses within a brittle film exceed its fracture strength. In most of these cases, the functionality of such films is no longer effective: electrical current leaks through passivation layers, and humidity and external pollutants pass through protective films. Another significant failure mechanism associated with warpage is represented by delamination, the separation of a thin film from its underlying substrate or adjacent layers, which becomes critical especially after device singulation [1]. Delamination arises when the interfacial adhesion is insufficient to withstand the shear and normal stresses induced by mismatched thermal expansion, intrinsic film stresses, or external mechanical loads. In multilayer stacks, weak adhesion can lead to delamination under thermal cycling or mechanical stress, creating voids and moisture pathways that degrade device performance and longevity.

The combination of these issues has led to the development of countless new technical challenges that still keep the topic of the 300 mm transition active today. In Figure 1.1 it is reported how the size of the wafers and individual devices has evolved with relative consistency over the years [4]; however, the size of the wafer seems to have reached a blocking point. In the work of Pettinato et al. [2], it can be noted that in the early years of 2005 this technological phase was thought to be nearing completion and that the adoption of 450 mm diameter wafers was imminent, yet even today there are no companies

employing such sizes on an industrial scale.

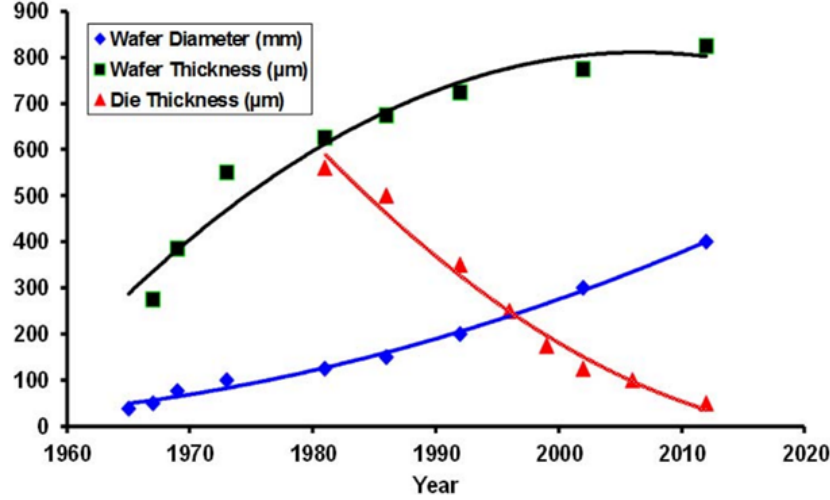


Figure 1.1: trends of the last six decades for wafer thickness, wafer diameter, and die thickness. Image taken from Savastiouk et.al. [11].

This indicates how the enlargement of wafer sizes can generate non-negligible issues ranging from the macroscale, like tool replacement and wafer handling and processing, to more localised failure modes such as cracking and delamination. Effective management of these phenomena through material engineering, process control, and integrated metrology is essential for the advancement of semiconductor manufacturing.

1.1.1. Geometric Definition of Warpage

As wafer sizes increase and device architecture becomes more complex, quantifying univocally wafer shape deviations is essential to ensure compatibility and alignment along the integrated production process. As defined by Semiconductor Equipment and Materials International (SEMI), the geometric state of a wafer can be characterized by two main parameters that describe its deviation from an ideal flat disc, known as *bow* and *warp*, each capturing different aspects of wafer deformation [1, 7].

Bow is defined by the SEMI MF534 as the displacement of the median surface of the wafer at its centre relative to a reference plane, indicating whether the wafer exhibits a convex or concave curvature. Warp is defined by the SEMI MF657 as the difference between the maximum and minimum deviations of the wafer's median surface from a best-fit plane, capturing more complex deformation profiles [1]. These parameters are schematically illustrated in Figure 1.2

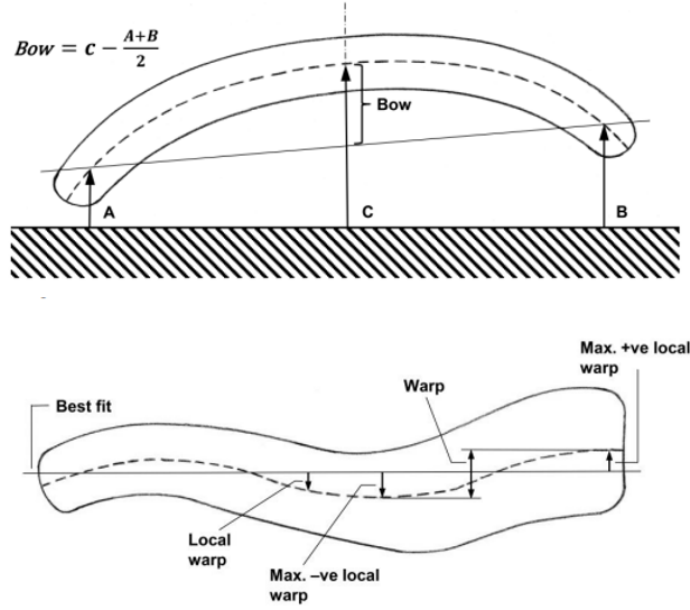


Figure 1.2: definition of bow (top side) and warp (bottom side) as defined by the SEMI standard. Image taken from Marks et.al. [4].

When the deformed morphology exhibits a spherical or ellipsoidal shape, the warp value is equal to the bow value, which is the most used descriptor. Thus, warp value is generally used only for more complex shapes [1].

Considering the functional surface on the top side of the wafer, numerous researchers in the field assume the orientation of the warpage deformation to be positive for a concave, smiling, or sad shape or negative for a convex, or crying, shape [8, 12, 13]. The general threshold considered for 300 mm wafers sets a warpage upper limit at 0.2 mm, beyond which handling issues start to seriously compromise process integration, while 0.1 mm is the value generally adopted to ensure high yield of the process [1, 8].

1.1.2. Measuring Warpage

Although several contact-mode measurements can be exploited to evaluate wafer warpage, such as stylus-based and ruler-based methods [8], in this section we will focus on the principal non-contact measurement methodologies employed in the semiconductor field. Such techniques can be categorized into four main groups: moiré-based methods, digital image correlation, laser scanning methods, and optical interferometry [8]; each of these groups will be described more in detail in the following paragraphs. All the main metrology

techniques used in the field of semiconductors are reported in Figure 1.3.

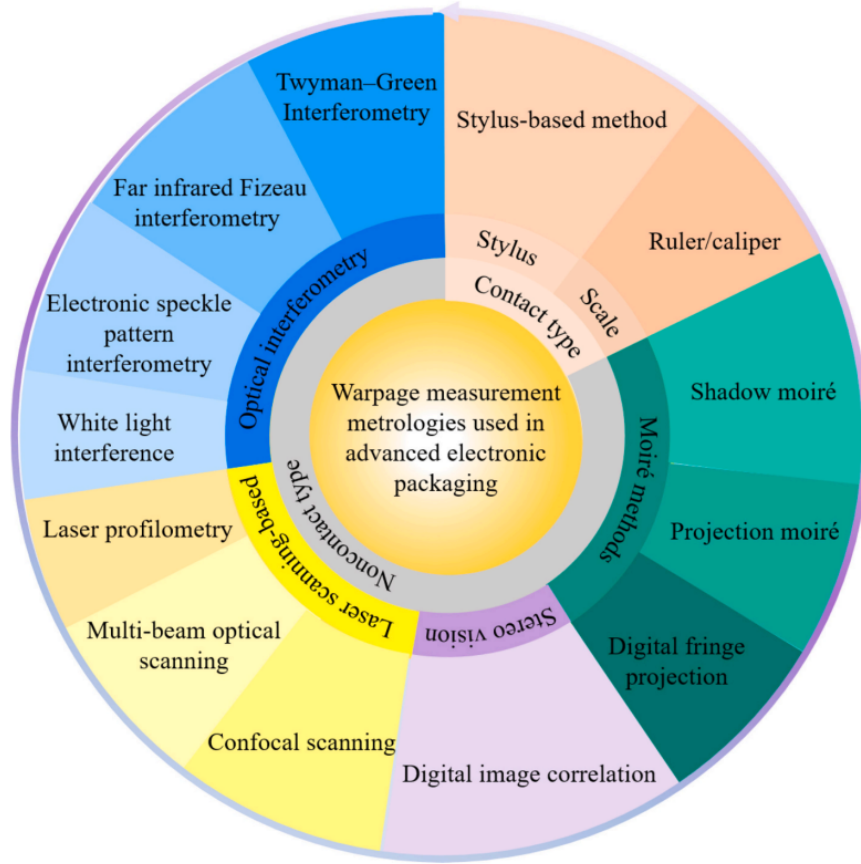


Figure 1.3: collection and classification of the measurement methodologies employed in the semiconductor field for warpage measurement. The non-contact methodologies are largely the most adopted due to their non-invasive nature. Image taken from Sun et.al. [8].

Moiré-based techniques are among the earliest and most widely adopted methods for measuring warpage and rely on interference patterns generated by projecting gratings onto the surface of the wafer [1]. The resulting moiré fringes correspond to contours of equal displacement, enabling the reconstruction of the surface topography with high sensitivity. The shadow moiré method [14] involves projecting a grating onto the sample and capturing the fringe pattern with a camera. It offers full-field, real-time measurement capabilities with vertical resolution on the order of micrometers or better. However, shadow moiré requires close proximity of the grating to the sample surface, which can be challenging with non-planar or discontinuous surfaces. Projection moiré and Digital Fringe Projection (DFP) improve upon shadow moiré by projecting structured light patterns and analyzing phase shifts to reconstruct 3D surface profiles [15, 16]. DFP is particularly suited for

complex geometries, but requires careful calibration and is sensitive to environmental noise. Moiré methods are widely used in industry due to their balance of precision, speed, and ease of use [1]. They are especially applied for packaging steps, to monitor warpage during molding, curing, and thermal cycling, providing valuable data for process optimization [8].

Digital Image Correlation (DIC) is a non-contact optical technique that tracks the displacement of a speckle pattern applied to the sample surface [8]. Using stereo vision, DIC computes the displacement and deformation of the full-field with high spatial and temporal resolution. It is well-suited for dynamic and temperature-dependent warpage measurements, enabling in-situ monitoring during thermal cycling or mechanical loading [17, 18]. Advances in DIC include 3D systems that improve accuracy and flexibility in the field of view. However, DIC requires the surface of the analysed wafer to be prepared with speckle patterns, which can affect the accuracy of the measurement [8]. It is sensitive to environmental conditions and requires significant computational resources for data processing. Integration of DIC with finite element analysis has enhanced the correlation between experimental measurements and simulations, aiding in model validation and process control [18].

A schematic representation of the two main Moiré-based systems and the digital image correlation system is reported in Figure 1.4.

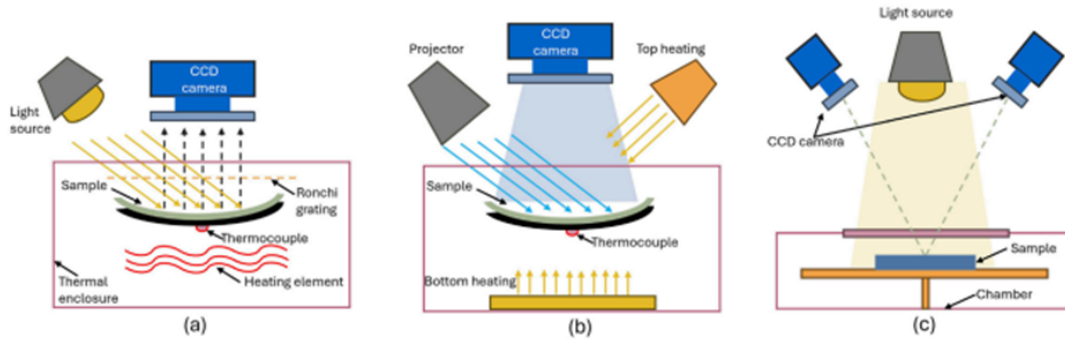


Figure 1.4: a schematic representation of Shadow Moiré setup (a), Digital fringe projection Setup Set (b), and DIC setup (c). Image taken from Praful et.al. [1].

Laser scanning techniques are widely adopted in industry for their non-contact nature, high accuracy, and compatibility with automated wafer handling [19]. The method utilizes focused laser beams to probe the wafer surface profile through triangulation of single-beam systems, which measure surface height by detecting changes in reflected laser position or angle (Figure 1.5). Precise single-beam scanning is time-consuming for full wafer map-

ping; thus, Multi-Beam Laser Scanning (MBLS) employs arrays of laser beams to simultaneously capture data over larger areas and increase throughput, enhancing speed and stability [20]. However, these systems face challenges in wafer edge scanning and require complex optical setups and data processing [8]. Confocal Laser Scanning Microscopy is an improved laser scanning technique that offers high-resolution 3D surface reconstruction by focusing light at specific depths and filtering out-of-focus signals [21]. This method excels at measuring complex topographies and discontinuous surfaces, but is slower and limited in field of view.

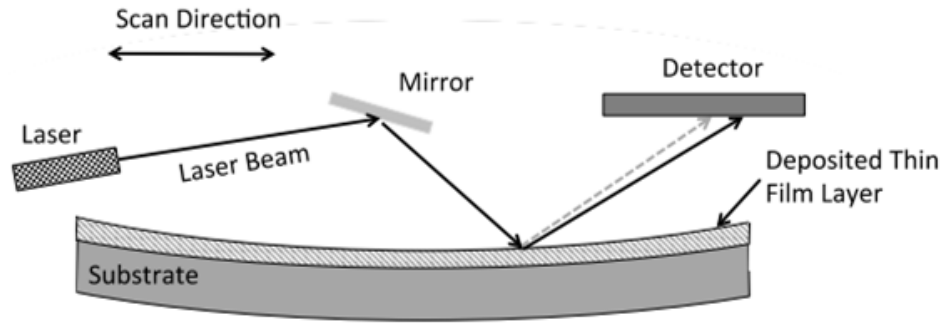


Figure 1.5: schematic representation of the basic setup for the laser scanning technique. By employing multiple lasers simultaneously (MBLS technique), it is possible to increase speed and stability of the full wafer area measurement. Image taken from Huff et.al. [9].

Finally, optical interferometry techniques exploit the interference of coherent light waves to detect minute surface displacements. Methods such as Twyman-Green interferometry (TG), far-infrared interferometry (FIFI) and electronic speckle pattern interferometry (ESPI) achieve sub-micrometer resolution [8]. TG interferometry offers high sensitivity but requires smooth, reflective surfaces and stable environments [22]. FIFI extends measurement capability to less reflective surfaces, but involves complex configurations [23]. ESPI provides high-precision real-time measurements, but is sensitive to environmental disturbances and requires sophisticated data analysis [24]. An emerging technique in this group is Patterned Wafer Geometry (PWG) metrology, which addresses the challenges of measuring patterned wafers during processing. Unlike traditional metrology tools that measure bare wafers, PWG can characterize wafers with complex surface patterns and film stacks, providing critical insights into process-induced distortions affecting lithographic overlay and focus [25]. PWG metrology employs dual interferometric measurements of the wafer's front and back surfaces, generating high-resolution height maps. A key challenge is the "false topography" effect, where transparent films cause the measured surface to differ from the true surface [25, 26]. This issue can be mitigated by applying opaque coatings or

by modelling corrections. PWG analysis enables computation of thickness maps and wafer shape, facilitating the assessment of local and global distortions, and allows calculation of In-Plane Distortion (IPD) metrics that correlate strongly with lithographic overlay errors. These capabilities make PWG a powerful diagnostic tool for pinpointing the process steps causing distortion [25]. Moreover, applications of PWG metrology include monitoring process-induced focus and overlay problems, optimizing thermal processes such as rapid thermal annealing (RTA), and diagnosing equipment-induced distortions. PWG enables rapid, high-density wafer mapping, supporting efficient process control and yield improvement; a schematic representation of a system based on a dual-Fizeau configuration is reported in Figure 1.6. This tool, utilized by STMicroelectronics in the factory of Agrate, also possesses a wafer holding system that allows one to measure warpage while the wafer stands vertically, minimizing the gravitational effect [27].

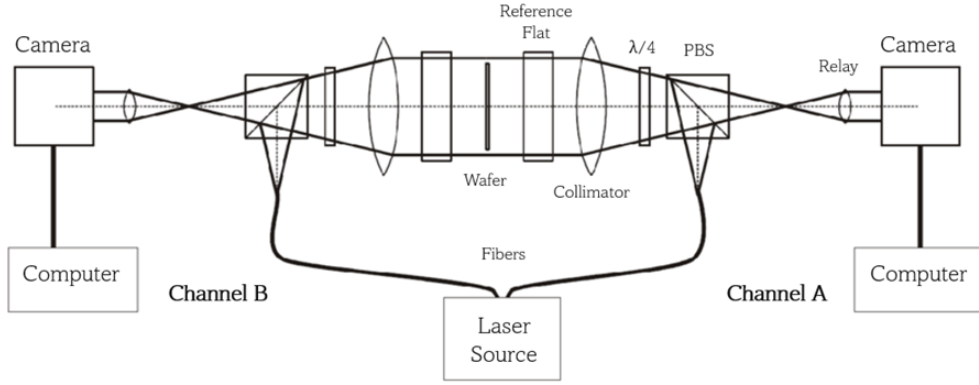


Figure 1.6: schematic representation of the PWG tool, showing the dual-Fizeau interferometers at $\lambda = 635$ nm which allow the measurement of the front and back surfaces of the wafer simultaneously. Image taken from Brunner et.al. [25].

The selection of measurement techniques depends on factors such as wafer thickness, deformation magnitude, processing stage, and required spatial and temporal resolution. Accurate characterization of bow and warp is essential for process control and for mitigating the adverse effects of wafer deformation on subsequent manufacturing steps.

1.2. Origin of Warpage: Residual Stress

The phenomenon of wafer warpage arises primarily from the accumulation of residual stresses within the wafer and its constituent layers, defined as states of stresses present in the absence of any other externally applied forces [28]. The residual stresses developed by the deposited layers are formed by several components that can be broadly categorized

into intrinsic and extrinsic, each arising from distinct physical phenomena and processing conditions. Understanding the origins, characteristics, and implications of these residual stresses is essential for the design and fabrication of advanced semiconductor devices, especially as the industry transitions to larger wafer sizes and more complex architectures.

1.2.1. Intrinsic Stresses

Intrinsic stresses originate from the film deposition process itself and the microstructural features of the deposited material [28]. They are inherent to the film and arise even in the absence of external environmental influences. The sources of intrinsic stresses are multiple and often interrelated, encompassing microstructural and atomic-scale phenomena [3, 29].

One of the primary causes of intrinsic stress is the lattice mismatch between the deposited film and the substrate (see Figure 1.7). When a film is grown on a substrate with a differing lattice constant, the atomic bonds at the interface are strained to accommodate the mismatch, resulting in compressive or tensile stresses within the film [30]. For example, a silicon film grown on a silicon substrate with identical lattice parameters exhibits minimal intrinsic stress from lattice mismatch, whereas films of different materials or polycrystalline films can develop significant stresses.

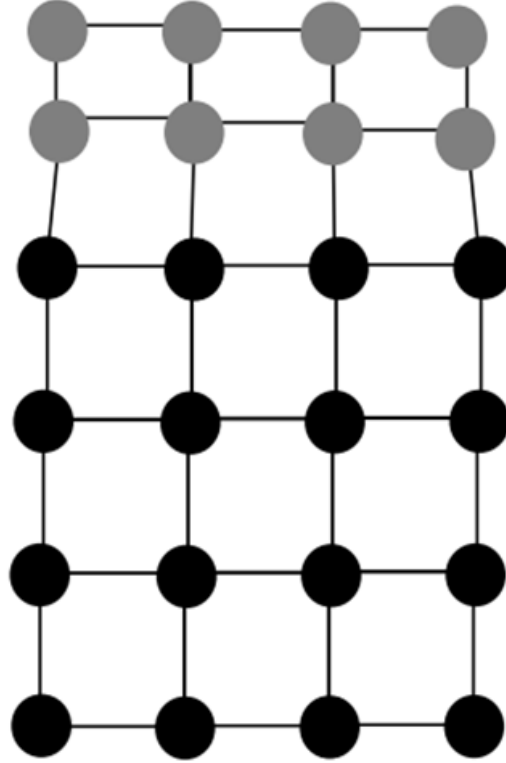


Figure 1.7: Schematic illustration of a strained thin-film layer epitaxially grown on a crystalline substrate. The reported case would induce a tensile intrinsic stress (positive sign, smiling shape). Image taken from Huff et.al. [9].

In addition to lattice mismatch, intrinsic stresses arise from microstructural imperfections such as grain boundaries, voids, dislocations, and impurities incorporated during deposition or doping [31]. These defects disrupt the equilibrium atomic arrangement, leading to localized strains and overall film stress. The microstructure of the film, including grain size and orientation, is heavily influenced by deposition parameters such as temperature, pressure, and the chemical environment [9, 10]. Furthermore, deposition techniques involving energetic particle bombardment, such as sputtering or plasma-enhanced processes, can induce intrinsic stresses by atomic peening [32]. This phenomenon occurs when high-energy ions impact the surface of the growing film, causing densification and compressive stress buildup. In contrast, films deposited under low-energy conditions may exhibit tensile stresses due to porous microstructures or incomplete densification [9].

1.2.2. Extrinsic Stresses

Extrinsic stresses are induced by external environmental factors and subsequent processing steps that alter the film and substrate after deposition [28]. These stresses are not intrinsic

to film formation, but are the result of interactions with the surrounding environment or thermal history. For this reason, the transition to larger wafer diameters has led to stronger extrinsic components of stress, since larger wafers are more susceptible to non-uniform temperature gradients, different cooling rates, and gravitational loads [29].

A dominant source of residual stress leading to warpage is the mismatch in thermal expansion coefficients (CTE) between the silicon substrate and the various deposited films or encapsulating materials [1, 28]. During semiconductor processing, wafers undergo multiple thermal cycles, including high-temperature deposition, annealing, and cooling. If the deposited film and substrate have differing CTEs, differential contraction or expansion occurs upon cooling, inducing biaxial stresses within the film and substrate, as represented in Figure 1.8. The cumulative effect of these mismatches across multiple layers and interfaces produces complex stress distributions that lead to strong wafer deformation. The magnitude and direction of warpage depend on the relative thicknesses, elastic moduli, and thermal properties of the constituent layers, as well as the processing temperature profiles [28].

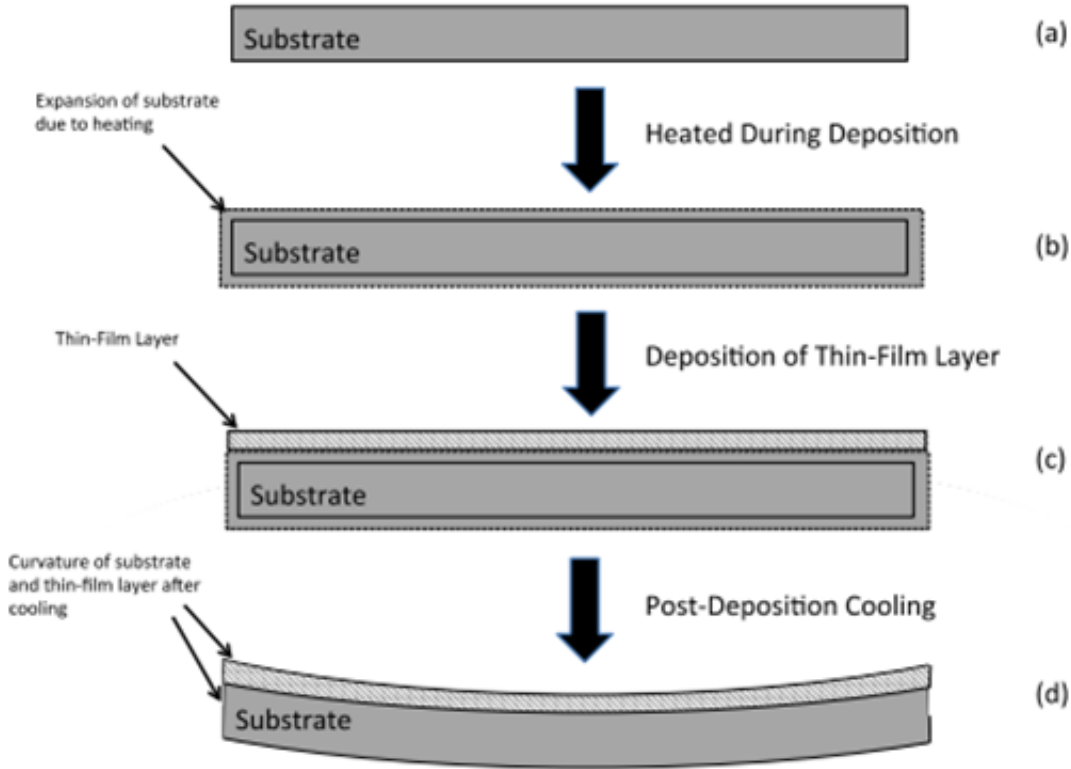


Figure 1.8: an illustration of the warpage induced by the extrinsic stress generated by a thin-film layer deposited onto a wafer substrate. The starting free wafer (a) undergoes a thermal budget during the film deposition which causes it to expand isotopically (b). The newly deposited layer is then deposited onto the expanded substrate (c). Once the substrate and the film obtain an equilibrium state at room temperature (d), the film, having a higher CTE, will attempt to contract more than the substrate inducing a in a state of tensile stress (positive sign, smiling shape). Image taken from Huff et.al. [9].

In addition to thermal stresses, environmental factors, such as moisture absorption, can also cause extrinsic stress formation, especially when an organic layer with a high hygroscopic capacity is involved [1]. Certain polymeric films and encapsulants absorb water vapor, leading to swelling and stress development. Chemical reactions, oxidation, and aging processes further modify the stress state over time, potentially affecting the reliability of the device. Moreover, mechanical handling and processing steps - such as wafer thinning, grinding, and dicing - can introduce localized extrinsic stresses that contribute to the overall residual stress profile [4]. These stresses can be spatially non-uniform and time-dependent, complicating their characterization and control.

1.2.3. Residual Stress Measurement Techniques

Various methodologies have been developed for measuring the stress of deposited films, ranging from global wafer-scale measurements to localized assessments using specialized tests and advanced analytical tools. Anyway, such techniques are possible today thanks to theoretical concepts developed more than a century ago by George J. Stoney, who in 1909 formulated his famous equation linking the residual stress (σ_r) of a thin film deposited on a substrate to the curvature (k) of the system [33].

In their work [28], Freund and Suresh report a clear derivation of the Stoney equation from an energy method starting from a bulk disk-shaped substrate of thickness t_s and radius R and a film membrane of thickness t_f and radius a . A cylindrical (r, θ, z) -coordinate system is introduced ($z = 0$ on the upper surface of the substrate), and the film is initially detached from the substrate and subjected to a force f (with a physical force dimension / length) that induces an elastic mismatch strain that maintains the radius a equal to the radius R (see Figure 1.9). The goal of the demonstration is to estimate the curvature of the substrate induced by the stress associated with the incompatible strain when the film is brought in contact with the substrate surface and bonded to it, and the external means of maintaining the initial film tension is released.

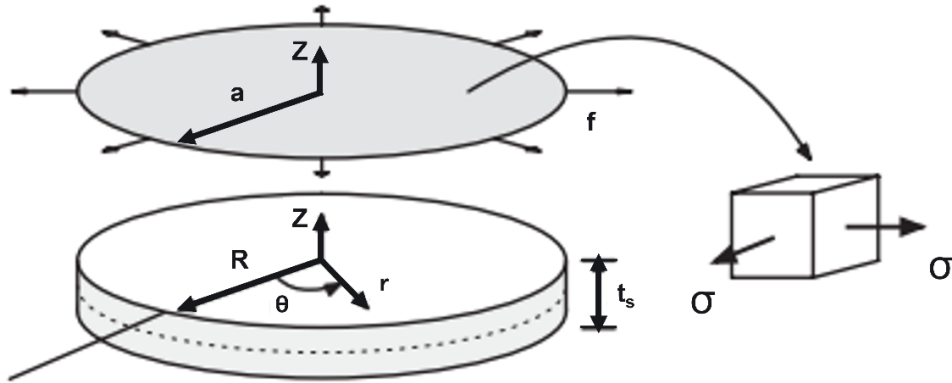


Figure 1.9: initially film and substrate are separated, but a distributed force f is acting on the film edge so that its radius, considering the imposed strain, is equal to R . This loading gives rise to an equi-biaxial state of stress at each material point in the film, as illustrated on the right side. Image taken from Freund et.al. [28].

To analyze the system deformation, several assumptions are made:

- The film and substrate are considered uniform in thickness, and the aspect ratio of the system is considered $t_f \ll t_s \ll R$.

- All components of the displacement gradient are considered very small in the substrate, and the linear theory of elasticity can be applied (strains and rotations are infinitesimal).
- The substrate deforms following the Kirchhoff thin plate theory, considering the normal stress component σ_z equal to zero everywhere (the film stress states are only in-plane isotropic).
- The deformation is axially symmetric, so that $\varepsilon_{r\theta} = 0$ and all fields are independent of θ (curvature components are equiaxial and twist components are zero).
- The curvature of the substrate surface k is spatially uniform.

As a result, the deformed shape of the substrate surface is spherical, and such approximations are valid as long as we are considering quasi-isotropic materials and small deformations.

In these cases, the strain energy density at any point of the substrate is expressible in terms of the non-zero strain components such as:

$$U(r, z) = \frac{\mu_s}{1 - \nu_s} [\varepsilon_{rr}^2 + \varepsilon_{\theta\theta}^2 + 2\nu_s \varepsilon_{rr} \varepsilon_{\theta\theta}] \quad (1.1)$$

where $\mu_s = \frac{1}{2} \frac{E_s}{1 + \nu_s}$ is the elastic shear modulus of the substrate and E_s and ν_s its elastic modulus and Poisson ratio, respectively. For small deflections, the normal in-plane strain is $\varepsilon_{\theta\theta} = \varepsilon_{rr} = \varepsilon_0 - kz$, where ε_0 represents the extensional strain of the substrate surface and kz denotes the extensional strain due to the bending at any position z of the substrate. Thus, substituting in equation (1.1) we obtain:

$$U(r, z) = \frac{E_s}{1 - \nu_s} (\varepsilon_0 - kz)^2 \quad (1.2)$$

The total potential energy in the substrate for the deformed system, following relaxation of the external agent, is:

$$V(\varepsilon_0, k) = 2\pi \int_0^{t_s} \int_0^R U(r, z) r \, dz \, dr + 2\pi f u_r(R, t_s) R \quad (1.3)$$

Within the class of permissible deformation, the equilibrium deformation of the substrate surface, represented by ε_0 and k , is the one that renders the total potential energy V stationary, thus when $\frac{\partial V}{\partial \varepsilon_0} = 0$ and $\frac{\partial V}{\partial k} = 0$. The results are then:

$$\varepsilon_0 = -\frac{f(1 - \nu_s)}{E_s t_s} \quad (1.4)$$

and,

$$k = \frac{6f(1 - \nu_s)}{E_s t_s^2} \quad (1.5)$$

Where the curvature k has the same sign as the force f . Finally, if we consider the reduced modulus $E_r^s = \frac{E_s}{1 - \nu_s}$, and that the stress in the film can be described as $\sigma_f = \frac{f}{t_f}$, the expression (1.5) can be written in function of the stress as:

$$\sigma_f = \frac{E_r^s}{6} \frac{k t_s^2}{t_f} \quad (1.6)$$

Although expression (1.5) is the original Stoney Equation, several variations have been developed from equation (1.6) to consider stress formation in cases of non-isotropic and/or non-elastic materials, multi-layered stacks and other evolutions to allow the description of more complex systems [9, 33, 34]. However, most modern metrology systems implement the methods described in section 1.1.1 to measure the curvature before and after a film deposition and evaluate directly the residual stress by implementing the standard Stoney equation.

These systems are widely used for fast and non-destructive screenings, but have some limitations that must be faced when more specific characterizations are required [9]. Primarily, they provide only an average stress value throughout the substrate and cannot resolve local variations in residual stress, which often arise due to inhomogeneities in processing conditions such as reactive species concentration, gas flow, plasma power, and temperature fluctuations throughout the wafer [8]. Additionally, they assume that the elastic properties of the thin film, particularly the Young's modulus, are known and uniform, although these properties can also vary with processing, although less than stress. Finally, while the most innovative scanning technique accounts for the presence of stress gradients through the film thickness in its overall measurement, it is not able to separate or quantify such gradients. This is a critical limitation because a film may have a zero average residual stress if the stress varies symmetrically through thickness but still exhibits significant stress gradients that strongly influence mechanical behavior and device performance [9, 31]. Therefore, a comprehensive characterization should include both average stress and stress gradient assessments.

For these reasons, other techniques are employed to obtain more localized and detailed information on residual stresses. A list of brief descriptions of such methods is reported in the work of Huff et al. [9]:

- **Buckling Beam Structures:** arrays of doubly clamped beams with varying lengths

are fabricated from the film of interest. When compressive stress exceeds a critical threshold, the beams buckle out-of the plane. Identifying the critical buckling length can calculate the compressive residual stress. This method is limited to compressive stresses and requires careful design to avoid stiction and measurement uncertainties.

- **Guckel Rings:** these ring-shaped structures convert tensile film stress into compressive stress in a central beam, which buckles upon reaching a critical stress level. Guckel rings enable measurement of tensile stresses, but share limitations similar to those of buckling beams.
- **Pointer and Strain-Based Structures:** these designs allow the film to deform freely, with deflections measured optically or via microscopy. The pointer structure, for example, uses offset supports to amplify strain-induced displacements, enabling the measurement of both tensile and compressive stresses. However, the small magnitude of displacements often necessitates high-resolution imaging techniques, such as scanning electron microscopy.
- **Resonator Structures:** fixed-fixed beam resonators exhibit shifts in resonance frequency proportional to the residual stress. This method provides high sensitivity and accuracy, which is particularly useful for MEMS devices.
- **Bulge Test:** membrane structures subjected to uniform pressure loading provide stress measurements through deflection analysis. The bulge test can assess both tensile and compressive stresses but requires complex sample preparation.

1.2.4. Tailoring the Residual Stress Behavior

The combined effect of intrinsic and extrinsic stresses, modulated by film thickness, elastic moduli, and substrate constraints, determines the overall deformation profile of the wafer [1]. Managing and controlling the distribution of residual stresses is therefore critical to maintain wafer flatness and minimize warpage-induced manufacturing challenges [3].

Residual stresses within thin films are highly sensitive to the parameters that govern their deposition. As previously reported, Huff et al. [9] emphasizes that factors such as deposition temperature, chamber pressure, gas composition, and plasma power critically influence the magnitude and sign of residual stresses. For example, chemical vapor deposition (CVD) processes exhibit distinct regimes in which deposition kinetics and film microstructure vary with temperature and pressure [32, 35]. Low-pressure CVD (LPCVD) often produces films with high intrinsic tensile stresses, while plasma-enhanced CVD allows greater flexibility in tailoring stress through plasma parameters [36]. Incorporation

of dopants, gas flow ratios, and substrate bias further modulate film properties, allowing partial control over stress states [31, 36]. Similarly, physical vapor deposition (PVD) techniques, including evaporation and sputtering, present complex dependencies on process conditions. Sputtering parameters such as target power, gas pressure, and substrate bias affect film density, microstructure, and consequently residual stress [37]. The interplay of these variables results in a broad spectrum of achievable stress states, ranging from compressive to tensile.

To address the challenges posed by this strong variability in residual stress distribution, several mitigation strategies have been developed, focusing on material engineering, process optimization, and advanced deposition techniques. The most common among such techniques are reported below [9]:

- **Post-Deposition Thermal Treatments:** thermal annealing remains a cornerstone approach to relieving residual stresses in thin films. Annealing at elevated temperatures facilitates atomic diffusion and structural relaxation, enabling partial or complete stress relaxation [38]. The effectiveness of annealing depends on the temperature, duration, and ambient atmosphere, with rapid thermal treatments (RTT) offering advantages in minimizing thermal budgets while achieving significant stress reduction [39, 40]. However, the application of high temperature anneals is not possible beyond certain steps of production, such as metallization, and must therefore be balanced against potential adverse effects on other device layers, dopant profiles, and overall process integration [9]. Careful optimization is required to maximize stress relief without compromising the functionality of the device.
- **Stoichiometric Modification of Films:** adjusting the chemical composition of the layers in use provides a powerful means to tailor residual stresses, especially for materials synthesized by CVD, where it is fairly easy to modify precursor flux ratios, pressure, and other deposition parameters [41]. This compositional adjustment enables moderate control over mechanical robustness and stress distribution [42]. However, it often also involves changes to other aspects, such as the conductive properties, which can impose serious limitations on certain types of materials.
- **Multilayer Stack Design:** composite film structures comprising alternating layers with opposing stress states can effectively balance overall residual stresses. By designing multilayer stacks where tensile and compressive stresses counteract, manufacturers achieve reduced net stress and minimized warpage [9, 43]. This approach, while increasing process complexity and potentially affecting other material properties, offers a practical pathway to managing stress in advanced device architectures.

- **Ion-Assisted Deposition Techniques:** ion beam-assisted deposition (IBAD) and related methods introduce energetic ions during film growth to modify microstructure, density, and stress [44]. IBAD enables independent control over film properties by adjusting the ion energy and flux, promoting denser films with tailored stress states. This technique enhances film adhesion, reduces porosity, and mitigates moisture sensitivity, contributing to improved device reliability, and can also modify the conductivity of the material depending on the types of ions introduced. IBAD and similar approaches represent cutting-edge strategies for stress control, particularly relevant in the fabrication of complex multilayer films for advanced packaging [9].

1.3. Numerical Approaches for Warpage Modeling

To find the solution to general field problems, such as elasticity, fluid flow, or heat transfer problems, several common methods have been developed in the last century and can be classified as presented in Figure 1.10.

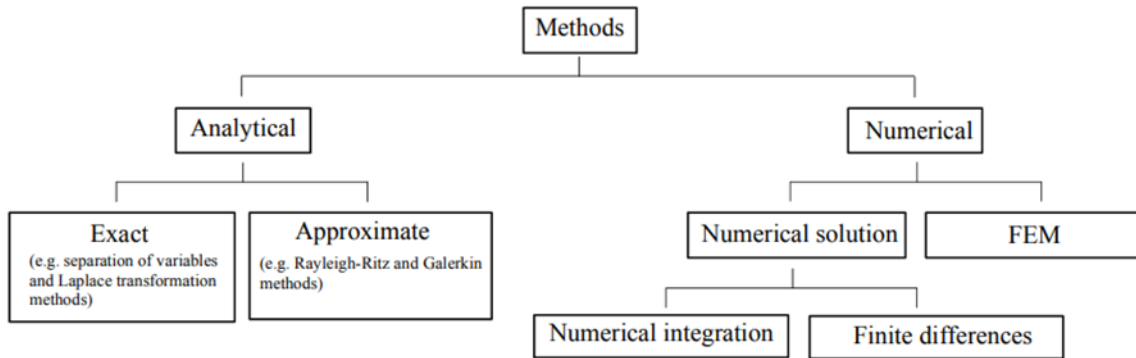


Figure 1.10: classification of common methods to resolve general field problems. Image taken from Barkanov et.al. [45].

Although analytical models were the first to be developed, the rise of numerical models originated in the early twentieth century, from the pioneering work of Ritz [46] on the approximate solution of problems in the mechanics of deformable solids, then further optimized by Courant [47], whose linear functions defined over triangular regions were used as effective boundary conditions. Within the field of numerical methods, Finite Element Analysis (FEA) emerged only in the late 1960s, when Clough [48] utilized the mathematical basis of Ritz and Courant on large scales due to the possibility to use computers for the big volume of required computations. In his work, Clough was the first to introduce the term “finite element”, paving the way for a revolution in the way complex

structural and mechanical problems are approached and solved in the field of engineering.

Today, FEA provides a versatile numerical framework for approximating solutions to boundary value problems that are otherwise analytically intractable. Its widespread adoption in various fields, including civil, mechanical, aerospace, and, increasingly, semiconductor manufacturing, attests to its robustness and adaptability [49].

1.3.1. Fundamentals of FEA

At its core, FEA transforms a continuous physical domain into a discrete system composed of interconnected elements, each governed by simplified equations that approximate the behavior of the material and structure within that region [49]. These elements have the same dimensionality as the simulated model (commonly 2D or 3D), but in any case, it is assumed that they are connected in a finite number of nodal points whose displacement determines the field of displacements of each finite element [45], as represented in Figure 1.11. This gives the possibility to write a system of equilibrium equations for the element assembly (i.e. the full model) as was done for a calculation of a shaft system.

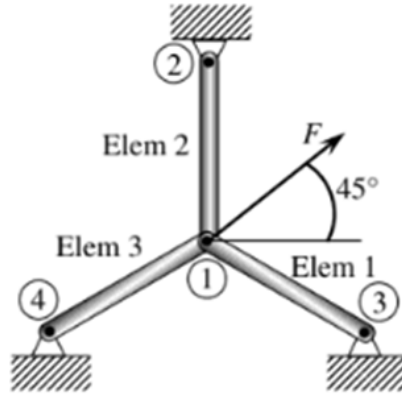


Figure 1.11: representation of element contributions to total nodal force in a simple system.

This discretization process reduces partial differential equations, which describe the continuum mechanics of the system, to a large but finite set of algebraic equations. These equations relate nodal displacements to applied forces through the system's stiffness matrix, allowing the calculation of deformation and stress distributions under various loading and boundary conditions. For these reasons, the practical implementation of FEA is heavily dependent on computational resources, as the size and complexity of the resulting algebraic systems can be immense, especially for three-dimensional problems with fine

meshes [13, 50, 51]. Advances in computer hardware and software have made it feasible to routinely perform detailed simulations of large-scale structures, including semiconductor wafers with multilayered stacks and intricate device patterns [45]. In this framework, several FEA software packages have been developed, such as ANSYS, Abaqus, and COMSOL Multiphysics, providing comprehensive environments for model creation, solution, and post-processing. These tools include extensive libraries of element types - ranging from simple truss and beam elements to sophisticated shell and solid elements - allowing users to tailor models to the specific physics and geometry of their problem.

Despite its strong potential, FEA still has limitations. The precision of simulations depends critically on the quality of the input data, including material properties, boundary conditions, and loading scenarios [52, 53]. Inaccurate or incomplete data can lead to erroneous results, which may go unnoticed without careful validation. Therefore, it is essential to complement FEA with analytical solutions, experimental data, and engineering judgment to develop a comprehensive understanding of the problem. Elastic modulus, coefficient of thermal expansion (CTE), and residual stresses are the most impactful parameters to consider when implementing a thermo-mechanical simulation, and in some cases there is no possibility of having direct measurements of such values [54]. This happens because of the lack of advanced characterization instrumentation, which could be needed for overcoming physical barriers like thin thicknesses, or the high variability of the processes in use for synthesis or thermal treatments in the semiconductor industry, which hinders the possibility of adequately characterizing the material in use for implementing simulations.

1.3.2. Challenges and Strategies for Warpage Modeling

Analytical models, such as the classical Stoney equation, provide a foundational approach to relate film stress to wafer curvature. However, these models assume a uniform stress distribution, isotropic materials, and simple geometries, limiting their applicability to complex multilayer and patterned structures typical of modern devices. To overcome these limitations, numerical methods are becoming the standard for simulating, predicting, and ultimately mitigate warpage, enabling enhanced manufacturing yield and device reliability [1].

The modeling process for warpage investigation begins with the construction of a geometric representation of the wafer and its constituent layers and patterns. The full model will then be composed of several domains that identify the distinct materials or areas that must be subjected to different external inputs or described with different mechanical

properties. Each domain is discretized into finite elements, with careful attention to mesh density and element quality, particularly in regions of high stress gradients such as near trenches or interfaces [55], which could vary strongly from one case study to the other due to the large variability of the devices on the market. When possible, material properties are assigned on the basis of experimental characterization; otherwise such values can be inserted from material libraries already present inside the simulation software or found in the literature. Following this approach, it is also possible to take advantage of simulation as a predictive tool [54], looking for a specific result based on the property of fictitious material ‘ that could then become the target for subsequent synthesis. Boundary conditions that reflect the wafer support during processing and thermal loading profiles can also be incorporated [56]. The simulation proceeds by solving the equilibrium equations, often employing iterative or direct solvers optimized for sparse, symmetric, and banded matrices typical of FEA systems.

The specific challenge of modeling wafer warpage is inherently complex due to the following factors [1]:

- **Multiscale Complexity:** wafer assemblies encompass length scales ranging from nanometer-thick films to 300 mm wafer diameters, with intricate patterning such as trenches and vias. Capturing this multiscale nature in a single FEA model requires advanced meshing strategies and computational resources.
- **Material Heterogeneity:** the wafer stack includes diverse materials with varying mechanical and thermal properties, often exhibiting anisotropy and nonlinear behavior. Accurate representation of these properties can be critical.
- **Process Dependency:** warpage evolves dynamically through sequential fabrication steps each altering the stress state. Modeling must incorporate these process sequences with appropriate temporal resolution, becoming increasingly complex while the production line gets closer to its end.

To address these challenges, recent advances in FEA methods include multi-scale modeling techniques that bridge microstructural material behavior with wafer-scale deformation, or methods such as homogenization that simplify complex patterned layers into equivalent anisotropic materials, reducing computational costs while preserving accuracy [1].

Following, a brief list of such approaches is reported, while a dedicated sub-section will describe the homogenization approach more in depth, since such technique is of particular interest for the work of this PhD project.

- **Thermal-Mechanical Coupling and Boundary Conditions:** accurate warpage

simulation requires detailed thermo-mechanical coupling. Temperature gradients during processing greatly influence deformation modes, inducing differential expansion and contraction, which are the main drivers of warpage. FEA models can incorporate realistic thermal profiles obtained from process simulations or experimental measurements, while boundary conditions can be used to reflect wafer support, clamping, and gravity aspects [56]. For example, gravity-induced sagging becomes significant for thin, large-diameter wafers and must be included to accurately predict asymmetric warpage [52].

- **Sequential Process Modeling with Element Birth and Death:** An effective approach to simulate the stepwise evolution of warpage is the element birth and death technique [1]. This method sequentially activates or deactivates domains corresponding to layers or process steps, mimicking the actual manufacturing sequence. For example, a time-dependent simulation can give a first result at t_1 before the deposition of a specific layer and a second result at t_2 immediately after such a deposition. This approach can also be utilized in steady-state simulations imposing a sequence of activation – simulating new deposited materials – and deactivations – simulating material removal, like etching processes – of specific domains (thus, “birth” and “death”). This technique allows for the capture of transient stress redistribution and warpage evolution along the sequence of actions, also allowing the variation of process-specific parameters such as temperature profiles, curing kinetics, and mechanical boundary conditions [57].
- **Incorporation of Viscoelasticity and Cure Kinetics:** polymers such as EMC exhibit viscoelastic behavior and undergo chemical curing reactions during processing, which significantly influence warpage. Unlike purely elastic materials, viscoelastic materials exhibit time-dependent stress relaxation, which complicates the stress evolution. Advanced FEA models incorporate coupled thermo-chemical-mechanical analyses, where the degree of cure (DOC) of the polymer is modeled alongside temperature and mechanical fields. The curing kinetics are often described by empirical models such as the Kamal model [58], which relates heat release to the progression of polymerization. Viscoelastic properties are represented using Prony series or generalized Maxwell models, capturing relaxation behavior over time and temperature [58]. These models allow simulation of stress build-up during curing and subsequent relaxation during cooling, providing realistic warpage predictions [53].
- **Parametric and Sensitivity Analyses:** FEA enables systematic parametric studies to identify dominant factors affecting warpage. Parameters such as material properties (elastic modulus, CTE), layer thicknesses, pattern geometries, and process temperatures can be varied to assess their impact [59]. Sensitivity analyses guide material selection

and process adjustments to minimize warpage. For example, increasing the thickness or stiffness of carrier wafers, optimizing the curing time of molding compound, or adjusting the layout of the devices can substantially reduce deformation [1].

- **Emerging Machine Learning and Artificial Intelligence Approaches:** the computational intensity of detailed FEA models motivates the integration of machine learning (ML) and artificial intelligence (AI) techniques [1, 13]. ML models trained on extensive FEA simulation datasets can rapidly predict warpage for new designs and process conditions. Hybrid approaches combine physics-based models with data-driven corrections, enhancing accuracy beyond classical analytical formulas such as Stoney’s equation. Convolutional neural networks (CNNs) have been applied to map complex geometric features to warpage outcomes, while physics-informed neural networks embed governing equations to improve interpretability [13]. Despite their promise, AI/ML models require comprehensive training datasets and careful validation to ensure generalizability across diverse packaging scenarios.

1.3.3. Homogenization Approach

Homogenization involves replacing a complex patterned region with an equivalent “effective” material layer whose mechanical properties represent the averaged behavior of the heterogeneous structure. This approach significantly reduces model complexity while preserving accuracy in predicting global warpage and is commonly used to model periodic structures, as happens in composite materials, laminated plates, and other systems that can be described by the repetition of a unit cell in one or more directions [54, 60].

The homogenization process begins with the identification of such unit cells, commonly known as a Representative Volume Element (RVE), which have to be selected as a small but statistically representative sample of the heterogeneous macrostructure. The RVE contains the essential features of the pattern, including material phases and thermomechanical properties, geometrical arrangement, and interfaces [12, 61]. Then, continuity boundary conditions are applied to the RVE to simulate the periodic constraints of the pattern, and the homogenization approach starts with the application of various tensile, compressive, and shear test loadings on each of its faces. By computing the thermomechanical response of the RVE to such loadings, the related equivalent thermomechanical material properties can be obtained through solutions of the constitutive Hooke’s Law, which, after being determined at the scale of the RVE, allows the approximate representation of a whole pattern as an equivalent homogeneous layer with effective homogenized properties [62].

To briefly report such an approach analytically, let us determine the equivalent homogenized properties of a multilayered thin film structure, as reported in Figure 1.12, to replace it with a single effective layer with the resulting homogenized properties [63, 64].

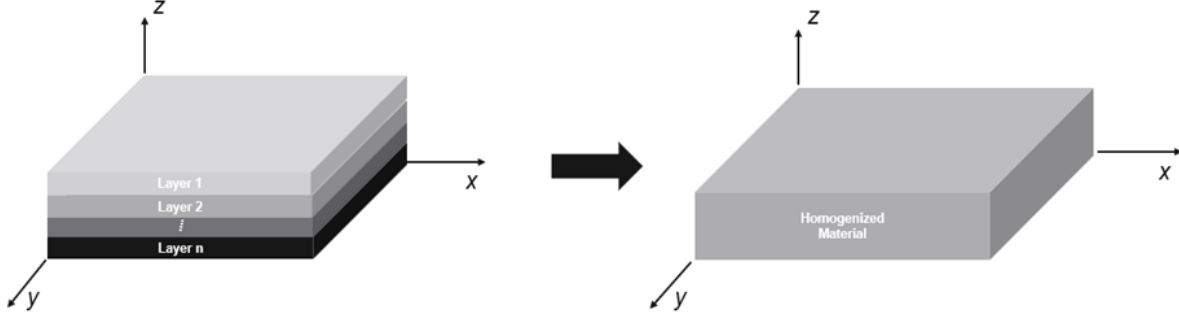


Figure 1.12: schematic representation of a multilayered RVE composed of n films individuated by a k number (on the left), and the corresponding homogenized orthotropic material.

To face this problem, we consider an RVE assumed to be much smaller than the overall structure and composed of n layers parallel to the (x, y) plane, while the z axis is perpendicular to the layers and describes the thickness of the stack. Each material composing any layer k is considered anisotropic to face the most general case, thus inherently solving the problem also for isotropic layers. We will then consider that this RVE is under uniform state of stress, and further assume, based on continuity of displacement at the layer's interfaces, that the in-plane strains parallel to the layers are uniform and equal to the corresponding strains in the homogenized material; then, we average the corresponding stresses:

$$\begin{bmatrix} \varepsilon_{xx}(k) \\ \varepsilon_{yy}(k) \\ \varepsilon_{xy}(k) \end{bmatrix} = \begin{bmatrix} \bar{\varepsilon}_{xx} \\ \bar{\varepsilon}_{yy} \\ \bar{\varepsilon}_{xy} \end{bmatrix} \quad (1.7)$$

$$\begin{bmatrix} \bar{\sigma}_{xx} \\ \bar{\sigma}_{yy} \\ \bar{\sigma}_{xy} \end{bmatrix} = \sum_{k=1}^n \frac{V^{(k)}}{V} \begin{bmatrix} \sigma_{xx}(k) \\ \sigma_{yy}(k) \\ \sigma_{xy}(k) \end{bmatrix} \quad (1.8)$$

Where k is the layer number and $\varepsilon_{ii}(k)$ and $\sigma_{ii}(k)$ the corresponding strain and stress component, respectively; $\bar{\varepsilon}_{ii}$ and $\bar{\sigma}_{ii}$ the strain and stress components, respectively, of the homogenized material, and $V^{(k)}$ and V the volume of the specific material k and of the complete RVE, respectively.

In parallel, based on the continuity of stress at the layer interfaces, it is assumed that the

out-of-plane stresses, which are perpendicular to the layers, are uniform and equal to the corresponding stresses in the homogenized material, and we average the corresponding strains:

$$\begin{bmatrix} \sigma_{yz}(k) \\ \sigma_{xz}(k) \\ \sigma_{zz}(k) \end{bmatrix} = \begin{bmatrix} \bar{\sigma}_{yz} \\ \bar{\sigma}_{xz} \\ \bar{\sigma}_{zz} \end{bmatrix} \quad (1.9)$$

$$\begin{bmatrix} \bar{\varepsilon}_{yz} \\ \bar{\varepsilon}_{xz} \\ \bar{\varepsilon}_{zz} \end{bmatrix} = \sum_{k=1}^n \frac{V^{(k)}}{V} \begin{bmatrix} \varepsilon_{yz}(k) \\ \varepsilon_{xz}(k) \\ \varepsilon_{zz}(k) \end{bmatrix} \quad (1.10)$$

These assumptions, mixed with continuity boundary conditions, satisfy both the equilibrium conditions at the interfaces of each layer and the compatibility conditions of the material [63]. Thus, it is possible to determine, knowing the generalized Hooke's Law in each layer, the elastic constants of an equivalent homogeneous anisotropic material, also considering the extrinsic stress imposed by an eventual thermal budget. The constitutive law in the presence of thermal contributions is given for each layer k by:

$$\begin{bmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ \varepsilon_{zz} \\ \varepsilon_{yz} \\ \varepsilon_{xz} \\ \varepsilon_{xy} \end{bmatrix}^{(k)} = \begin{bmatrix} S_{xyz} \end{bmatrix}^{(k)} \begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{yz} \\ \sigma_{xz} \\ \sigma_{xy} \end{bmatrix}^{(k)} + \begin{bmatrix} \alpha_x \Delta T \\ \alpha_y \Delta T \\ \alpha_z \Delta T \\ 0 \\ 0 \\ 0 \end{bmatrix}^{(k)} \quad (1.11)$$

Where

$$\begin{bmatrix} S_{xyz} \end{bmatrix}^{(k)} = \begin{bmatrix} \frac{1}{E_{xx}} & -\frac{\nu_{yx}}{E_{yy}} & -\frac{\nu_{zx}}{E_{zz}} & 0 & 0 & 0 \\ -\frac{\nu_{xy}}{E_{xx}} & \frac{1}{E_{yy}} & -\frac{\nu_{zy}}{E_{zz}} & 0 & 0 & 0 \\ -\frac{\nu_{xz}}{E_{xx}} & -\frac{\nu_{yz}}{E_{yy}} & \frac{1}{E_{zz}} & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{2G_{yz}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{2G_{xz}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{2G_{xy}} \end{bmatrix}^{(k)} \quad (1.12)$$

Where $left[S_{xyz}right]^{(k)}$ is the compliance matrix of layer k , where E and ν are Young's modulus and Poisson's coefficient, respectively, G is the shear modulus, α is the coefficient of thermal expansion, and ΔT is the temperature change.

Since in the assumed assumptions the transverse shear strains and the in-plane strains

do not interact with each other, it is possible to rearrange Hooke's Law (1.11) by adding permutation matrices that allow you to automatically separate the in-plane and out-of-plane components:

$$\begin{bmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ \varepsilon_{xy} \\ \varepsilon_{yz} \\ \varepsilon_{xz} \\ \varepsilon_{zz} \end{bmatrix}^{(k)} = [P_1] [S_{xyz}]^{(k)} [P_1]^{-1} \begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{xy} \\ \sigma_{yz} \\ \sigma_{xz} \\ \sigma_{zz} \end{bmatrix}^{(k)} + [P_2] \begin{bmatrix} \alpha_x \Delta T \\ \alpha_y \Delta T \\ 0 \\ 0 \\ 0 \\ \alpha_z \Delta T \end{bmatrix}^{(k)} \quad (1.13)$$

Dove:

$$[P_1] = \begin{bmatrix} 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 \\ 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 \end{bmatrix} \quad [P_2] = \begin{bmatrix} 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 \end{bmatrix} \quad (1.14)$$

Therefore, Hooke's Law can be rewritten as:

$$\begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \end{bmatrix}^{(k)} = \begin{bmatrix} M_{11} & M_{12} \\ M_{21} & M_{22} \end{bmatrix}^{(k)} \begin{bmatrix} \sigma_1 \\ \sigma_2 \end{bmatrix}^{(k)} + \begin{bmatrix} B_1 \\ B_2 \end{bmatrix}^{(k)} \quad (1.15)$$

where:

$$\begin{aligned} \varepsilon_1^{(k)} &= \begin{bmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ \varepsilon_{xy} \end{bmatrix}^{(k)} & \sigma_1^{(k)} &= \begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{xy} \end{bmatrix}^{(k)} & B_1^{(k)} &= \begin{bmatrix} \alpha_x \Delta T \\ \alpha_y \Delta T \\ 0 \end{bmatrix}^{(k)} \\ \varepsilon_2^{(k)} &= \begin{bmatrix} \varepsilon_{yz} \\ \varepsilon_{xz} \\ \varepsilon_{zz} \end{bmatrix}^{(k)} & \sigma_2^{(k)} &= \begin{bmatrix} \sigma_{yz} \\ \sigma_{xz} \\ \sigma_{zz} \end{bmatrix}^{(k)} & B_2^{(k)} &= \begin{bmatrix} 0 \\ 0 \\ \alpha_z \Delta T \end{bmatrix}^{(k)} \end{aligned} \quad (1.16)$$

At this point, the terms M_{11} , M_{12} , M_{21} and M_{22} —resulting from the permutation of the compliance matrix—are known assuming that the mechanical properties of each layer

k are known. For the same reason, the terms B_1 and B_2 are also known, as well as the thermal variation ΔT . Note that this approach, like any FEA approach, is not applicable without knowing the thermo-mechanical properties of the material under study.

The remaining goal is to transform the expression (1.15), valid for each layer k , into a new form dependent on homogenized deformations, homogenized compliance matrix, stresses, and secondary thermal terms, so that it can be used for the entire thin film stack:

$$\begin{bmatrix} \bar{\varepsilon}_1 \\ \bar{\varepsilon}_2 \end{bmatrix} = \begin{bmatrix} \bar{A}_{11} & \bar{A}_{12} \\ \bar{A}_{21} & \bar{A}_{22} \end{bmatrix} \begin{bmatrix} \bar{\sigma}_1 \\ \bar{\sigma}_2 \end{bmatrix} + \begin{bmatrix} \bar{C}_1 \\ \bar{C}_2 \end{bmatrix} \quad (1.17)$$

By developing the above expression considering the assumptions made for the analytical approach (equations 1.8 and 1.10), one obtains, after basic algebraic operations, the determination of the homogenized compliance matrix and the homogenized secondary thermal term:

$$\begin{aligned} \bar{A}_{11} &= \left[\sum_{k=1}^N \frac{V^{(k)}}{V} \left(M_{11}^{(k)} \right)^{-1} \right]^{-1} \\ \bar{A}_{22} &= \sum_{k=1}^N \frac{V^{(k)}}{V} \left(M_{22}^{(k)} - M_{21}^{(k)} \left(M_{11}^{(k)} \right)^{-1} M_{12}^{(k)} \right) \\ \bar{A}_{12} &= \left[\sum_{k=1}^N \frac{V^{(k)}}{V} \left(M_{11}^{(k)} \right)^{-1} \right]^{-1} \left[\sum_{k=1}^N \frac{V^{(k)}}{V} \left(M_{11}^{(k)} \right)^{-1} M_{12}^{(k)} \right] \\ \bar{A}_{21} &= \bar{A}_{12}^T \\ \bar{C}_1 &= \left[\sum_{k=1}^N \frac{V^{(k)}}{V} \left(M_{11}^{(k)} \right)^{-1} \right]^{-1} \left[\sum_{k=1}^N \frac{V^{(k)}}{V} \left(M_{11}^{(k)} \right)^{-1} B_1^{(k)} \right] \\ \bar{C}_2 &= \sum_{k=1}^N \frac{V^{(k)}}{V} \left(B_2^{(k)} - M_{21}^{(k)} \left(M_{11}^{(k)} \right)^{-1} B_1^{(k)} \right) \end{aligned} \quad (1.18)$$

Once the terms $\bar{A}_{11}$, $\bar{A}_{12}$, $\bar{A}_{21}$, $\bar{A}_{22}$, $\bar{C}_1$ and $\bar{C}_2$ have been determined, it is possible, using the permutation matrices $\text{left}[P_1\text{right}]$ and $\text{left}[P_2\text{right}]$ (equation 1.14), to trace the expression of the original compliance matrix $\text{left}[S_{xyz}\text{right}]$, which in

this case will refer to the homogenized material. The same can be done for the original thermal term corresponding to homogenized thermal expansion coefficients.

Today, various dedicated software implement homogenization in a relatively automated way once the RVE has been modeled by the user, making this approach accessible to researchers. By employing homogenization, the complexity of patterned regions in wafer-level simulations can be addressed in many situations by simply identifying an effective repeating cell. For example, considering a trench pattern, the RVE consists of a unit cell that contains a single trench and the surrounding silicon and dielectric materials. The boundary conditions applied to the RVE simulate the periodic constraints of the pattern, and the computational power required for the simulation is drastically reduced by replacing thousands of trenches with a homogenized regular layer.

1.4. Objectives of this Thesis

In the context of addressing the warpage challenges associated with the transition to 300 mm wafers, this research focuses on facing three parallel lines of research related to specific issues that appeared in the development of an Insulated Gate Bipolar transistor device (IGBT) [5]. Even for this well-known technology, the shift to 300 mm wafer processing has catalyzed renewed efforts in material selection, surface and interface engineering, and the management of process-induced stresses to ensure device reliability and integrity at larger wafer scales. The three objectives of this thesis have been identified as follows:

- Modeling of trench-IGBT Warpage during a specific critical step of production by comparing die-to-wafer and homogenization approaches.
- Investigate composition-dependent mechanical behavior of silicon nitride thin films as passivation layers.
- Evaluate the adhesion of nitride/organic interfaces in controlled ambient through nanoindentation testing In-Situ and Ex-Situ.

A detailed description of each objective is given below.

At wafer level, the most obvious consequence of the 300 mm transition is the increasing of saddle-shaped anisotropic warpage [50, 65]. In IGBT this phenomenon emerges already at early stages, when depositing on a patterned structure of the active area composed by thousands of aligned trenches with strong geometric aspect ratios, which - mixed with thermo-elastic and intrinsic stresses of the thin films added during processing - leads to the formation of asymmetric warpage with opposite curvatures along and across the trench

direction. Recently, warpage phenomena have been extensively investigated through finite element analysis (FEA) [66–68], but the everchanging shape, patterns, and materials used in the different devices of the industry lead to the constant need for new tailored approaches. Thus, one objective of this project was to develop an ad-hoc finite-element model calibrated on the warpage results observed in real wafers during the production of the IGBT in the three most critical steps – Gate Oxide Deposition, Trench Poly-Filling, and Body Annealing – to use it for predictive purposes in future applications with similar patterns.

At the film scale, the 300 mm transition has intensified the need to lower the stresses produced to keep warpage under control, resulting in further reduction of their thickness where possible. Silicon nitride (SiN_x) produced by plasma-enhanced chemical vapor deposition (PECVD) is widely used in passivation layers due to its excellent properties of chemical inertness, electrical insulation, and thermal stability, also tunable by varying its stoichiometry during deposition [69, 70]. However, its fracture resistance remains a critical weakness for their long-term reliability [71] and its evaluation at sub-micrometer thickness is difficult to perform due to the failure of the classical toughness analysis due to the influence of the so-called “substrate effect” [72, 73]. Several micromechanical approaches (micro-pillars, cantilevers, chevrons) exist but involve complex preparation and equipment [74]. Thus, a particularly practical alternative was a nanoindentation based method developed by Xia et al. [75], has been used to develop and implement a standardized approach to assess fracture toughness (K_{ic}) on a sub-micrometric SiN_x film with increasing silicon over-stoichiometry to assess the dependency of mechanical properties on silicon content.

Finally, at the interface scale, the need to replace or modify selected materials following the 300 mm transition has led to the development of new combinations of layers whose mutual adhesion must be qualified. In IGBT, the transition led to the introduction of a new polymeric protective layer called Polybenzoxazole (PBO) — a photo-sensible organic coating that acts as a moisture barrier and mechanical buffer [76] — which is deposited on the SiN_x passivation layer. The PBO/ SiN interaction is a critical determinant of long-term reliability, and its baseline adhesion is often too high for classical techniques such as peeling or four-point bending [77]. An alternative is nanoindentation-based delamination testing, which takes advantage of the presence of a discontinuity in the load-penetration curve to identify the critical delamination load [78, 79]. This value is not directly related to the adhesion energy of the interface, but under similar testing conditions it allows for a comparative study between different interfaces in the analysis. Moreover, it is well known that the main causes of degradation at the interface between polymeric and inorganic sub-

strates are mainly environmental factors such as temperature and humidity [80–82]. Thus, the third objective of this project was to assess the best interface, in terms of adhesion, between PBO and different SiN substrates – both in terms of stoichiometry and surface treatments – under different ambient conditions controlled during the nanoindentation testing (in-situ analysis) or before them via ambient aging (ex-situ analysis).

Taken together, these three research lines and objectives highlight the sequence of challenges that arise following the 300 mm transition, starting from the increased warpage due to the larger surfaces of the wafer and the related need to understand the origins and contingency methods of this phenomenon, moving to the necessity of achieving greater control over the mechanical properties of the films to help calibrate such macro effects, and concluding with the need to properly monitor the new interfaces that are created as a result of such changes.

2 | Methodologies and Experimental Setup

This chapter presents the methodologies and experimental framework employed throughout this doctoral research. The overarching objective is to elucidate the mechanical and interfacial challenges that arise during the industrial transition to 300 mm wafer-scale manufacturing of Insulated Gate Bipolar Transistors (IGBTs), a pivotal device class in power electronics. The research is structured along three complementary lines of inquiry - warpage modeling, thin-film toughness evaluation, and interfacial adhesion characterization - each addressing critical aspects of device reliability and manufacturability.

To establish a coherent understanding of these investigations, section 2.1 introduces the detailed structure of the IGBT device, highlighting the specific regions of interest that serve as focal points for the experimental and modeling efforts. Subsequently, the synthesis and preparation of the materials involved in each research line are described in section 2.2, including thermal treatments, surface modifications, and patterning protocols necessary to obtain the samples to be characterized. Finally, in section 2.3 such characterization techniques are outlined, emphasizing their roles in probing mechanical properties, interfacial adhesion, and microstructural features necessary to bring forward all the three lines of research of this thesis.

2.1. The IGBT Structure

The IGBT device analyzed in this research is based on a trench-gate design, which enhances the performance of the device by optimizing the electric field distribution and reducing conduction losses. This device must indeed withstand voltages above 600 V and currents around hundreds of Amperes, values certainly higher than those supported by most devices. For these reasons, IGBT falls into the group of technologies known as power devices, specifically designed to support very high voltages and currents. The trench-gate structure, which is the main structural characteristic of the IGBT, is used primarily for these purposes.

In their work, Iwamuro et al. [5] developed an in-depth review on the history and current uses of IGBT, which the reader can consult for a more detailed understanding of this technology.

2.1.1. Device Architecture Overview

The transition to 300 mm diameters allows an increase in productivity by having, at the end of the manufacturing process of each wafer, a greater number of devices in the same processing time. In particular, the gain obtained increases the larger the size of the individual device (for simple geometric reasons), and in the case of the IGBT, of medium-large size, the transition allowed for a strong increase, passing from less than 300 devices (or dies) for 200 mm to more than 600 devices for 300 mm.

Each device on the wafer is identical to the others and is produced in parallel following a series of production steps that are maintained aligned by a specific set of masks. The untouched silicon lines surrounding each die are called “scribe lines”: these are the areas where the sawing step is carried out at the end of the device growth process allowing their singularization. However, before sawing these areas also contain the lithographic alignment structures and metrological structures to monitor and carry out parametric tests of the processes.

The peripheral areas of each individual device are instead known as Ring and Mesh, which acts as physical and insulating barriers to prevent current leakage between the devices. Due to the privacy policy of STMicroelectronics - which provided the majority of the material tested during this project - it is not possible to discuss the geometry of a single device in detail, but it is sufficient to know that each IGBT device occupies an almost-squared area of about 1 cm².

Since the device area is not perfectly squared, a shift of the mask set with respect to the center of the wafer has been implemented to maximize the dies allocation within the operative area of the wafer, as shown in Figure 2.1

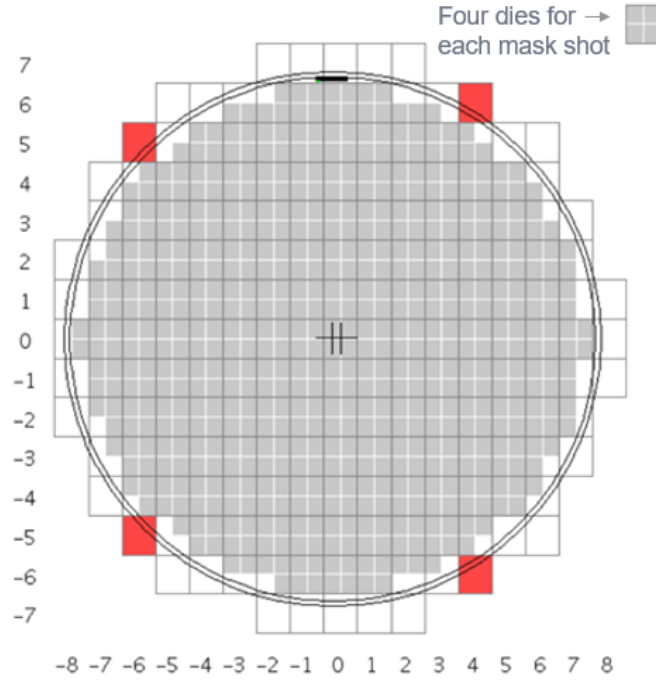


Figure 2.1: wafer mapping with “free shift” of the mask to maximize the device allocation onto the pristine silicon wafer. With this approach the 300 mm wafer can support up to 604 identic IGBT devices. With a wafer diameter of 200 mm the maximum number of dies was less than 300.

The crystalline silicon wafer serves as the fundamental semiconductor substrate for the growth of the devices and part of its thickness is doped to participate in their electrical operations. This means that the starting wafer for the production of a power device such as the IGBT must have very low values and tight ranges of impurities and doping concentrations.

The 200 mm wafers for this device were produced by STMicroelectronics using the float zone (FZ) method [83, 84] due to the high precision of this wafer growing method compared to the most widely adopted Czochralski (CZ) method [85, 86]. After the transition to 300 mm the FZ method was not possible anymore and the optimization of the CZ for the required ranges at this scale was a big challenge to withstand by itself for the company. However, the wafer fabrication challenge was solved prior to the beginning of this project, and it is not related to warpage.

Figure 2.2 schematically represents a cross-section of the device, where it is possible to distinguish the upper region, generally known as the “back end of line” (BEOL), from the buried region, instead known as the “front end of line” (FEOL). While the former contains

the metallization needed for the external electrical interconnects and the protective layers above them, the latter contains the architecture that allows the electrical performances of the devices and their edge terminations, and is often referred to as the “active area”. The two regions are separated by the pre-metal dielectric (PMD) stack, which is a series of oxides and dielectric layers that have the role of completely isolating the active area both electrically and physically from external contributions, and incorporating the vias connecting the upper metal plates to the core of the device.

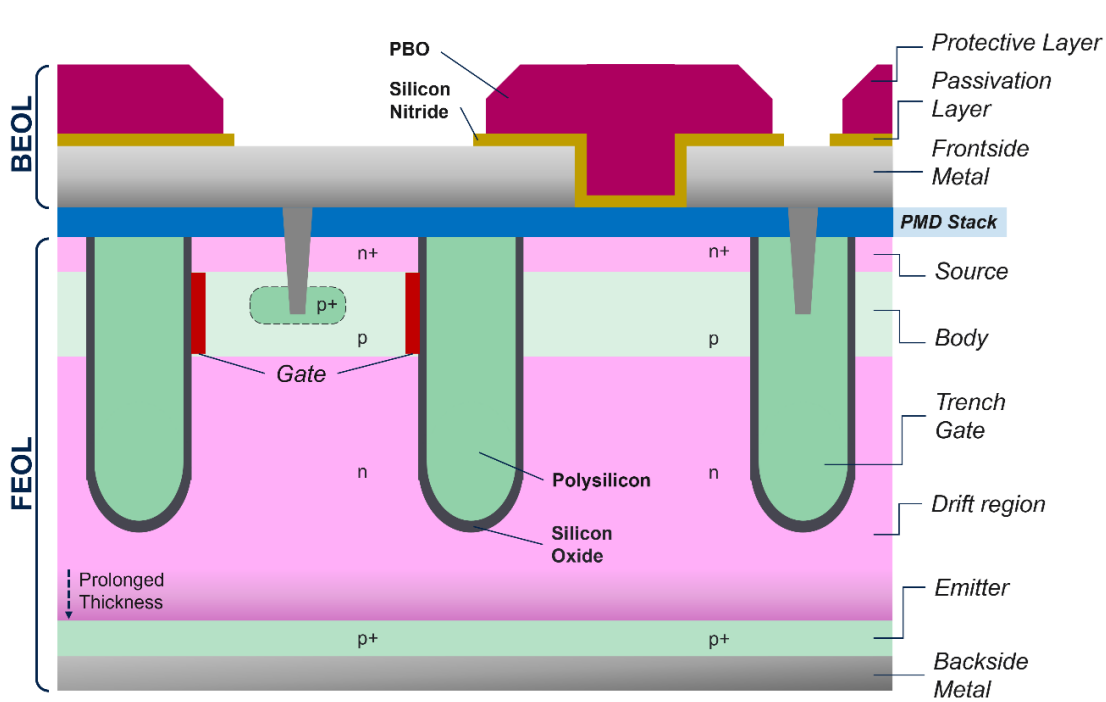


Figure 2.2: cross-sectional schematic of a trench-gate IGBT illustrating both BEOL and FEOL architectures. The figure highlights the FEOL region, comprising the silicon substrate with n-type drift region, p-type body, highly doped n^+ source and p^+ emitter, polysilicon-filled trench gates insulated by silicon oxide, and the backside metal contact. The BEOL region includes the frontside metal interconnects, silicon nitride passivation layer, and the PBO protective coating.

The FEOL is composed of the first sequence of operations performed on the pristine wafer to create the active area. For the device under study, an array of vertical parallel trenches with strong aspect ratios is initially etched. These trenches are subsequently oxidized to form a thin silicon oxide layer following their profile and then filled with polysilicon to form the gate electrodes, which control the device switching behavior. Applying a voltage to this gate above a certain threshold modulates the channel conductivity by inducing

an inversion layer in the p-type body adjacent to the trench walls, allowing electrons to flow from the source into the drift region. Simultaneously, holes are injected from the p^+ buffer layer into the drift region, facilitating bipolar conduction. This dual carrier injection reduces the on-state voltage drop of the device compared to unipolar devices, improving efficiency and allowing the withstand of very strong voltages and currents, as required for power devices.

When the gate voltage is removed or reduced below the threshold, the inversion channel disappears, interrupting the electron flow. The device then blocks current flow through the drift region, with the depletion region extending to sustain the applied voltage. All these functions are possible because the silicon remaining around the trenches undergoes a series of doping procedures which lead to the formation of the drift, body, source and emitter areas that constitute the support for the minority (h^+) and majority (e^-) carrier flows. The functions of such areas are:

- The lightly doped n-type drift region extends vertically through the majority of the substrate thickness to support the high blocking voltages that appear when the device is in the “off” state. Its low doping concentration is embedded during the creation of pristine silicon wafers and ensures that the electric field is distributed over a wide area, preventing premature breakdown.
- Embedded within the drift region, the p-type body is subsequently manufactured to permit the formation of the channel when the trench gate is charged.
- Within the body, highly doped source regions of n^+ are formed near the surface, acting as electron injection sites during conduction.
- Finally, a heavily doped p^+ emitter is responsible for injecting charge carriers into the base region, which act as the main switch of the device.

The precise doping profiles, trench geometry and material interfaces of the FEOL structure critically influence the electrical performance, switching speed, and reliability of the IGBT, but within this thesis only the trench pattern - from which warpage issues arise - will be treated in detail.

Focusing on the BEOL region, situated on the topside of the device, the two most external layers - i.e. silicon nitride passivation layer and polybenzoxazole (PBO) organic protective layer - are a secondary source of challenges emerging after the 300 mm transition. In summary, the BEOL region is essential for translating the electrical functionality of the FEOL active device into a robust, manufacturable, and reliable semiconductor product, ensuring electrical connectivity, protection of the core device structures, and contributions

to the mechanical stability of the full device.

To conclude, considering the full device structure, two principal regions have been identified as critical for the investigations undertaken in this thesis. Although the active area corresponds to the focal point of the warpage investigation, the contact area addresses the more experimental lines of research focused on the characterization of the SiN films and the SiN-PBO interface.

2.1.2. Front End of Line

The FEOL represents the region where the electrical operations of the device take place. Within this region, each single IGBT device contains specialized structures dedicated to particular functions, such as emitter and base contacts or terminations, but the vast majority of the volume is occupied by the trench-gate pattern necessary to sustain the high voltages and currents that the IGBT must undertake. The operations performed in this area are then transmitted to the BEOL via specific contacts both terminating inside the polysilicon-filled trenches and the silicon strips between them. However, for the purposes of this thesis, we will focus exclusively on the device architecture achieved after trench filling and the subsequent body annealing steps, as these are the processing stages that generate the maximum peak of wafer warpage and will be taken into consideration in Chapter 3.

Figure 2.3 reports, on the left (a), a TEM cross-section of the device at this stage, where it is possible to observe three silicon strips - lined by the silicon oxide layer - and the trenches filled with polysilicon between them. Without considering the borders and small functional portions, it is possible to describe the trench pattern of the active area referring to the sketch reported on the right (b) as a unitary cell repeated with a step p , known as the “pitch”.

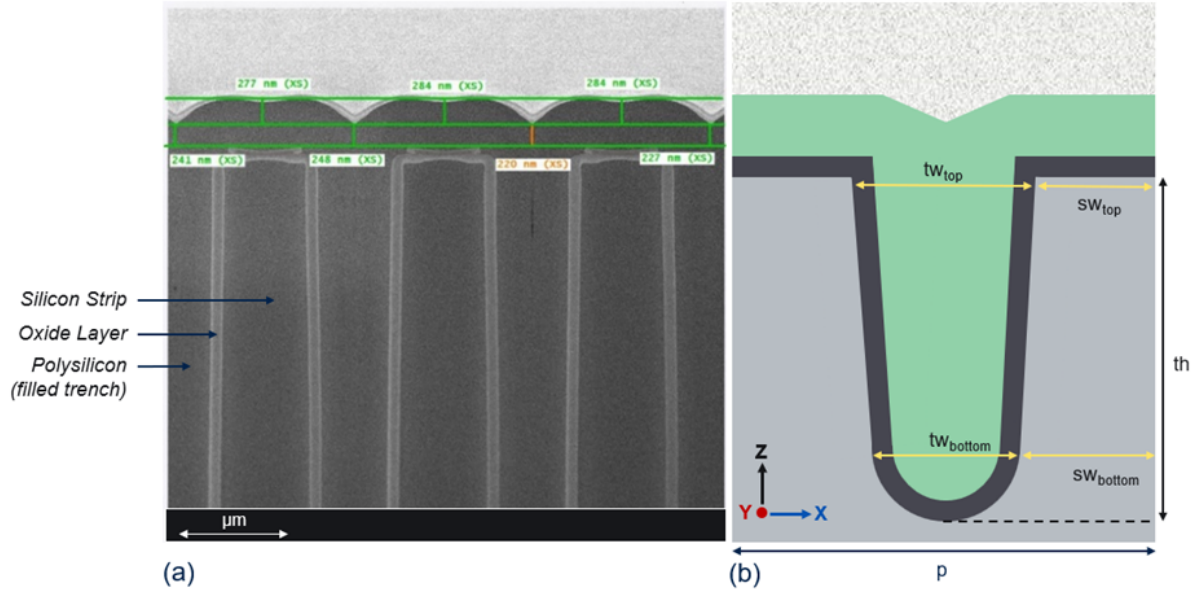


Figure 2.3: TEM cross-section of the of the uppermost zone of the active area after trench polysilicon filling step (a) and schematic representation of a single trench observed in cross-section (aspect ratio not respected) (b).

Each device features a total of 6250 identical trenches aligned with themselves - interspersed with the same number of silicon strips - and running for all the device length, i.e. approximately 1 cm. Due to the strong depth of etching required to create the pattern, the side walls of each trench are slightly tilted with an angle of 89.5° ; this effect is commonly known as “tapering” and leads to the obvious formation of a different width of the trenches if measured at their top or bottom. Table 2.1 reports the average dimensions of the main trench features in the production stage reported in Figure 2.3.

Table 2.1: Main features of the trench pattern referring to figure 2.3 (b), where p is the pitch, th is the trench height, tw and sw are the trench and strip width, respectively, both measured at the top and the bottom of the trench.

p	th	tw_{top}	tw_{bottom}	sw_{top}	sw_{bottom}
1600 nm	5000 nm	836 nm	744 nm	382 nm	428 nm

The design of the trench was dictated by operational limits, but the strong aspect ratio they feature between the three dimensions is the main origin of the asymmetric warpage issues encountered over the 300 mm transition. Indeed, the pattern features a scale in the order of the nanometers on the width (X), the μm on the height (Z), and the cm on length (Y), and it is thus difficult to be represented on large scale maintaining a high

level of details, as should be necessary for thermo-mechanical simulation. This topic will be discussed further in chapter 3.

Once the pattern has been created the silicon oxide layer is grown following the profile of the trenches, which are subsequently filled with the polysilicon. Thus, the materials of interest for this project within the active area are three: the crystalline silicon (silicon [100]) of the wafer, the silicon oxide, and the polycrystalline silicon.

The silicon oxide layer is obtained by a series of etchings and thermal growth which concludes with a 110 nm thick film lining the trenches. This silicon oxide must act as a high-quality electrical insulator between the gate electrode and the silicon substrate; thus, it is deposited mainly controlling its electrical properties.

Polysilicon is used to fill the etched trenches, forming the gate electrodes that modulate the conductivity of the underlying silicon. The trenches are filled with three layers of polysilicon produced by CVD with the following sequence:

- First layer: 150 nm thick p-doped amorphous polysilicon deposited at 520°C, followed by an in-situ annealing at 620°C and a subsequent rapid thermal treatment at 1050°C.
- Second layer: 130 nm thick p-doped amorphous polysilicon deposited at 520°C, followed by an in-situ annealing at 620°C.
- Third layer: 250 nm thick p-doped amorphous polysilicon deposited at 520°C.

The subsequent deposition, mixed with ad-hoc thermal treatments, is a strategy known to reduce the stress formation and thus the warpage issue, which would have been unmanageable for a single-deposition filling. However, this approach also requires performing a high temperature annealing to permit a final interdiffusion between the three layers. This treatment is based on the “body annealing”, performed at 1100°C to form the body region within the silicon strips, but also indirectly solves the issue related to trench filling with three depositions.

In section 2.2 more specifics on material synthesis will be shared, while chapter 3, related to the warpage modeling line, will focus on this region to develop predictive finite element models that capture the thermo-mechanical behavior of the patterned wafer.

2.1.3. Back End of Line

Within the scope of this thesis the features of interest of the BEOL region are the environmental protection and mechanical stability given by the silicon nitride and PBO couple. The silicon nitride Layer, produced by plasma-enhanced chemical vapor deposi-

tion (PECVD), serves multiple functions: it electrically insulates the metal wiring, protects the underlying structures from contamination and moisture ingress, and acts as a mechanical barrier against physical damage during handling and packaging. Above the passivation layer, an organic protective coating, such as PBO is often applied that acts as a moisture barrier and mechanical buffer, absorbing stresses induced by thermal cycling and mechanical loading.

When the device was produced on the 200 mm wafers a specific silicon nitride was used, supporting a polyimide (PI) organic coating that, in the last years, has fallen into disuse. The transition to 300 mm wafers has prompted new integration challenges, but it also created the opportunity to replace obsolete materials. These changes were made according to three principles:

- Better performance.
- Lower environmental impact.
- Lower costs.

Polybenzoxazole spin-coated films were introduced to replace PI in the new production site due to their increased qualities with respect to all three requests outlined above. In particular, the absence of PFAS makes PBO a highly appreciated material in terms of environmental impact.

For what regards silicon nitride, the integration challenges was mainly driven by the fact that the IGBT production after the 300 mm transition was displaced in a different manufacturing factory. While the old site was used to exploit a stoichiometric “standard” silicon nitride, the new site production tool was better suited for depositing PECVD “silicon-rich” silicon nitride layers, i.e., SiN films deposited in the over stoichiometry of silicon.

Therefore, this production relocation from one manufacturing site to another has created the need to understand the real differences between a standard and a silicon-rich nitride, both with regard to its passivating properties and its adhesion with the newly adopted polymer layer. The two experimental research lines, described in chapters 4 and 5, address these two issues, respectively.

2.2. Material Synthesis and Fabrication

All material characterized in this thesis has been synthesized on 300 mm full wafer by STMicroelectronics, considering all the subsequent processing steps required to build up

the device up to the point of interest for the research. The real IGBT production process has been followed up to the trench filling step for the research on predictive simulation methods, while all the tests for the two experimental projects - regarding SiN toughness and SiN/PBO adhesion - have been carried out on ad-hoc flat films, still deposited by STMicroelectronics on 300 mm wafers and subsequently cut on samples of reduced dimensions to perform subsequent thermal treatments and/or specific characterization by me in the laboratories of the company or the Department of Energy of Politecnico di Milano university. Further details on the tested samples will be reported at the beginning of chapters 3, 4 and 5.

Despite the possible presence of numerous intermediate process steps (e.g. surface cleanings), all the materials of interest for this thesis have been synthesized by CVD, thermal oxidation, or spin coating techniques and further prepared for characterization with etching procedures and/or thermal treatments. Etching procedures have also been exploited for substrate preparation (e.g. the forming of the trench pattern). Due to STMicroelectronics privacy policy, some of the synthesis parameters will not be shared, especially if not directly involved in this thesis research. Table 2.2 reports the relationship between the synthesis processes and the objectives in which they have been utilized.

Table 2.2: Summary of the synthesis processes employed for the three objectives of this thesis: “Modeling of the trench-IGBT Warpage during specific critical step of production comparing die-to-wafer and homogenization approaches” (1); “Investigate composition-dependent mechanical behavior of silicon nitride thin films as passivation layers” (2); “Evaluate the adhesion of nitride/organic interfaces in controlled ambientes through nanoindentation testing In-Situ and Ex-Situ” (3).

Objective	CVD	Thermal Oxidation	Spin Coating	Etching Techniques	Thermal Treatments
1) Simulations	✓	✓	–	✓	✓
2) Toughness	✓	–	–	✓	✓*
3) Adhesion	✓	–	✓	–	✓

* *Procedures carried out firsthand by the author.*

2.2.1. Chemical Vapor Deposition

Chemical Vapor Deposition (CVD) constitutes a fundamental thin-film deposition methodology extensively utilized in semiconductor device fabrication. This technique enables the formation of high-quality, uniform and conformal films essential for the electrical, mechanical, and chemical performance of advanced microelectronic devices [87]. The CVD

process involves in-vacuum chemical reactions of volatile precursor gasses in the vicinity of a heated substrate, resulting in the deposition of solid material layers with precise control over thickness, composition, and microstructure.

Several CVD variants have been developed to address specific material requirements and processing constraints [88]. Plasma-Enhanced CVD (PECVD) introduces plasma energy to activate chemical reactions at significantly lower substrate temperatures, often below 400 °C. This capability is particularly advantageous when processing temperature-sensitive substrates and has led this method to be widely used in the semiconductor field [89]. Moreover, PECVD allows for enhanced flexibility in tuning film composition and mechanical properties, including residual stress, which, as described in chapter 1, is crucial for managing wafer warpage and device reliability. A schematic representation of a typical PECVD system is reported in Figure 2.4.

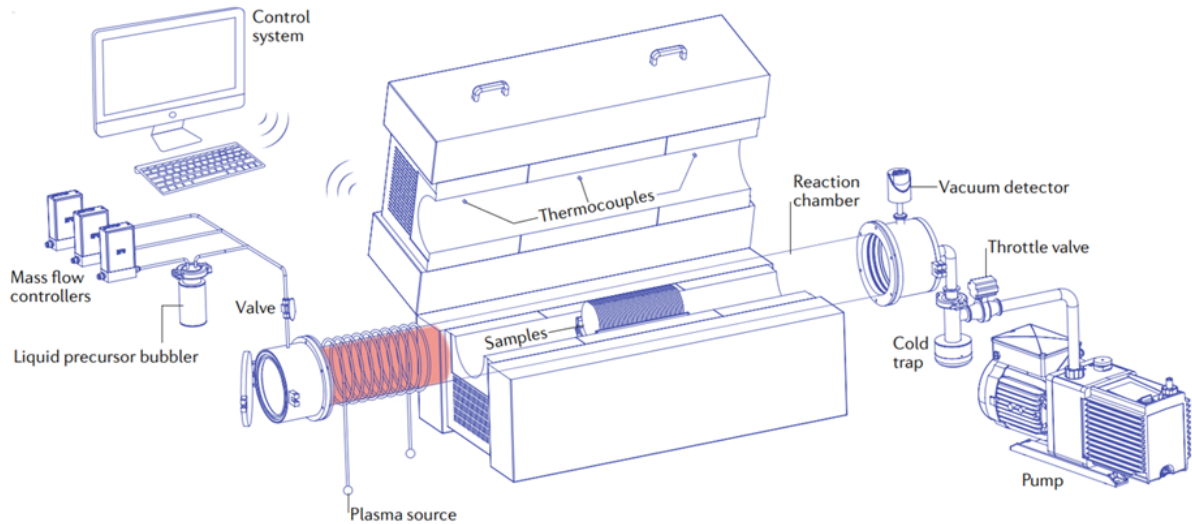


Figure 2.4: schematic representation of a horizontal plasma-enhanced chemical vapor deposition system, including the precursory gas delivery system and the plasma source on the left, the reaction chamber and heating control at the center, and the vacuum system on the right. STMicroelectronics tools are equipped with a reactor enabled to fit up to 25 wafers of 300 mm diameters - called a “lot” - on which the CVD films can grow simultaneously. Image taken from Sun et.al. [87].

The underlying mechanism of all CVD techniques encompasses several sequential stages, detailed in Figure 2.5. Initially, gaseous precursors are transported through the reactor environment to the substrate surface by diffusion into the boundary layer: a thin region of gas adjacent to the substrate surface where the transport of reactive precursor species is

dominated by molecular diffusion rather than bulk convection. This layer acts as a transition zone between the bulk gas flow and the substrate surface because the concentration of precursor gasses decreases from the bulk value to near zero at the substrate surface due to their gradual consumption by surface chemical reactions - such as pyrolysis, reduction, or oxidation - that lead to the formation of the solid film. Concurrently, reaction by-products originating by gas-phase reactions or desorbed after solid-state reactions on the substrate are evacuated from the chamber.

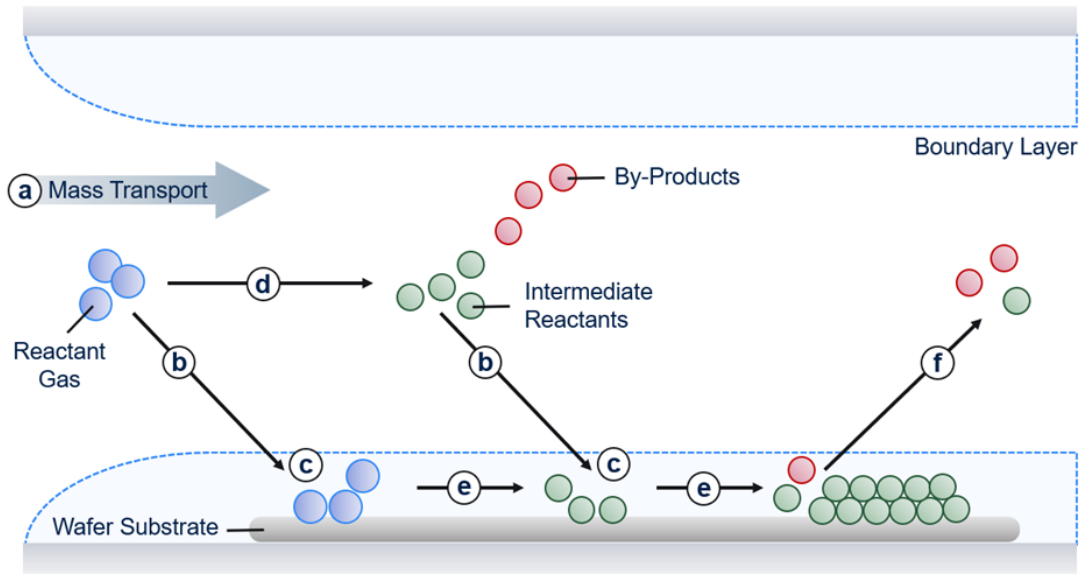


Figure 2.5: schematic steps of a typical CVD process. Reactant gases are initially transported into the reactor (a) and then pulled through two possible routes: directly diffusing through the boundary layer (b) and be adsorbed onto the substrate (c) or forming intermediate reactants and by-products via gas-phase reactions (d) and being deposited onto the substrate by diffusion (b) and adsorption (c). Surface diffusion and heterogeneous reactions (e) take place on the surface of the wafer substrate before the formation of thin films or coatings. Finally, by-products and unreacted species are desorbed from the surface and forced out of the reactor (f).

The deposition kinetics and film characteristics are highly sensitive to the thickness and properties of the boundary layer, which, in turn, is dependent on the process parameters, including substrate temperature, chamber pressure, precursor flow rates, and reactor design. These characteristics permit fine-tuning the physical and mechanical properties of the deposited films, but also make the techniques highly sensitive to external factors and fluctuations [88].

Specific Instrumentation

Within the fabrication of trench-gate IGBT, the material of interest for this thesis synthesized by CVD is the polysilicon used for the trench filling during the FEOL and the silicon nitride as the passivation layer during the BEOL.

In the first case, the objective was to completely fill the trench with polysilicon, minimizing the production of warpage and slip lines. To do so, the best approach was to perform subsequent depositions of three layers of 150 nm, 130 nm, and 250 nm thicknesses. Regardless of the type of subsequent thermal treatments - discussed in section 2.2.4 - the three depositions were performed with the same tool and parameters. An Applied Materials Producer PECVD tool was used, with a deposition temperature of 520°C and a Silane gas flux (SiH_4) of 1200 sccm, imposing pressure in the chamber of 0.5 Torr. A 2×10^{20} atm/cm^3 phosphorus doping was obtained by introducing a controlled flux of phosphine (PH_3) inside the chamber during deposition.

For silicon nitride deposition, a Lam Research Vector tool was used. The investigation was carried out on three different SiN_x films with nominal Si/N At% ratios of 0.8, 1.1 and 1.3, obtained by varying the precursor gas mixture, composed of silane (SiH_4) and ammonia (NH_3). The three films were renamed standard (St), silicon-rich 4 (SR4) and silicon-rich 5 (SR5), respectively: while the first film was the material originally used for the device – an almost-stoichiometric silicon nitride – the two second films were deposited with an over-stoichiometry of silicon to investigate the effect of silicon enrichment on SiN_x mechanical properties. Table 2.3 reports the deposition specifics for the three films.

Table 2.3: PECVD parameters for the SiN_x films deposition. The deposition times cannot be shared due to STM privacy policy, but the values have been varied to obtain different film thicknesses, as will be discussed in Chapter 4.

	RF Power (W)	SiH_4 (sccm)	NH_3 (sccm)	Si/N At% nominal ratio	Temperature (°C)	Pressure (Torr)
SiN St	1175	890	555	0.8	380	4.3
SiN SR4	1270	1400	350	1.1	380	4.3
SiN SR5	1270	1750	350	1.3	380	4.3

2.2.2. Thermal Oxidation

Thermal oxidation is a cornerstone process in semiconductor manufacturing, widely employed to grow high-quality silicon dioxide (SiO_2) layers on silicon substrates. This technique fundamentally differs from classical deposition methods, as it relies on a chemical reaction between the silicon wafer surface and an ambient oxidization at elevated temperatures, resulting in the formation of an oxide layer that is integral to device functionality [90, 91].

The process is typically conducted in a controlled furnace environment in which silicon wafers are exposed to dry oxygen (O_2), water vapor, or a mixture of both. Dry oxidation involves the reaction of silicon with pure oxygen at temperatures that generally range from 900°C to 1100°C. This method produces thin, dense, and electrically superior oxide films characterized by excellent interface quality and minimal defect densities, making it the preferred choice for insulating layers [90, 92]. In contrast, wet oxidation utilizes steam as the oxidizing species, which significantly accelerates the oxidation rate due to the higher diffusivity and reactivity of water molecules compared to oxygen. This accelerated growth is advantageous for the formation of thicker oxide layers in shorter process times, such as for field oxides or isolation regions, although at the expense of slightly reduced film density and electrical robustness compared to dry oxides [93]. To optimize the trade-offs between growth rate and film quality, diluted dry oxidation processes are employed, where dry oxygen is mixed with controlled amounts of water vapor.

The growth mechanism of thermal oxidation is governed by the diffusion of oxidizing species through the already-existing oxide layer spontaneously formed on the silicon surface, under which successive silicon atoms react to form silicon dioxide. This reaction results in a volumetric expansion - approximately 2.27 times the volume of the consumed silicon - that introduces mechanical stresses within the oxide film and at the silicon-oxide interface [94]. Process parameters such as temperature, ambient composition, pressure, and time are meticulously controlled to tailor oxide thickness and properties to specific device requirements. The resulting oxide layers exhibit uniform thickness, chemical stability, and excellent dielectric characteristics, making them an indispensable component of modern semiconductor devices.

Specific Instrumentation

Following trench etching into the pristine wafer, two sequential thermal oxidation processes are performed to create a 110 nm thick silicon oxide layer lining the trench pattern, both in the STM facility using a Centrotherm Oxidation Furnace. The first, called “Sacrificial Oxidation”, is a thick deposit that is subsequently removed and made to round the edges of the trench (both at the bottom of the trenches and at the top of the silicon strips).

The second oxidation is performed to deposit the film of interest for this thesis. The film is grown in a dry oxygen environment diluted with water vapor at 1050°C with a long temperature ramp to enhance stress relief.

2.2.3. Spin Coating

Spin-coating is a widely utilized thin-film deposition technique, particularly effective in applying uniform layers of liquid materials onto flat substrates. This method is widely used in semiconductor fabrication and microelectronic packaging to deposit photoresists, polymers, and other organic or inorganic coatings with controlled thickness and excellent uniformity [95].

The spin-coating process involves dispensing a liquid precursor or solution onto the center of a substrate, which is then rapidly rotated at high speeds, typically ranging from hundreds to several thousands of revolutions per minute (rpm). The centrifugal force generated during spinning spreads the liquid radially across the substrate surface, while excess material is expelled beyond the substrate edges. The final thickness of the film is mainly governed by the spin speed, the spin duration, and the viscosity of the liquid [96]. A representative schematic is reported in Figure 2.6.

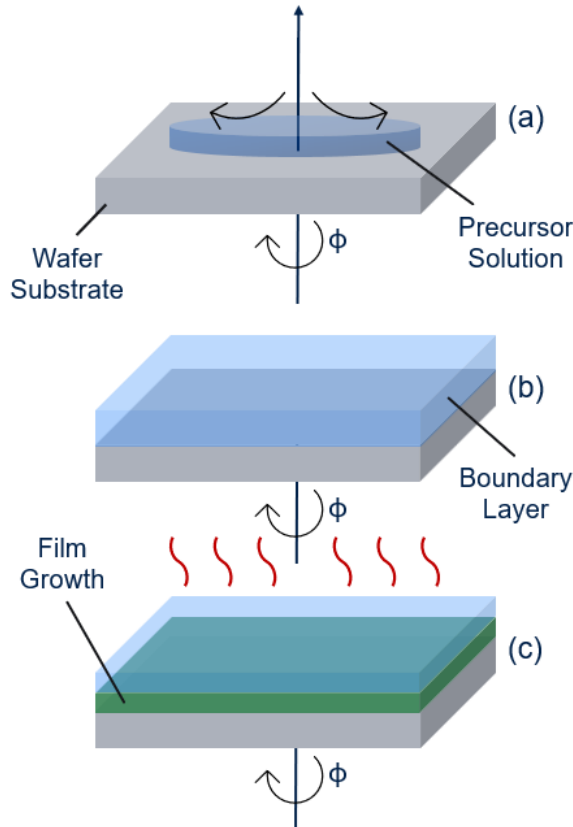


Figure 2.6: the precursor solution is dispersed on a room temperature or pre-heated substrate (a). When the solution reaches the spin speed ϕ of the sample, a hydrodynamic boundary layer is formed (b) and, with solvent evaporation, the film gradually starts to grow (c).

Moreover, as the substrate spins, solvent evaporation occurs concurrently, leading to the solidification of the deposited film. The evaporation rate, the ambient conditions (temperature, humidity) and the properties of the solvent secondarily influence the morphology of the film and the uniformity of the thickness [97]. Finally, post-deposition thermal treatments or curing steps are often applied to the film to induce hardening or cross-linking, respectively, depending on the nature of the material.

Compared to other liquid-to-solid deposition techniques, spin-coating offers several advantages, including simplicity, rapid processing, low material consumption, and the ability to produce highly uniform films over large areas, such as the case of 300 mm diameter wafers. It is particularly suited for coatings that require precise thickness control in the sub-micrometer to micrometer range. However, the technique is generally limited to planar substrates and may face challenges in coating substrates with complex topographies or structures with high aspect ratio [95].

Specific Instrumentation

In the context of this research, spin-coating has been employed to deposit the polybenzoxazole (PBO) polymeric film as the most external protective layer. At this stage, the metallization and passivation steps have already been performed, creating an almost flat substrate for the PBO deposition.

A Laurell WS-650 Series Spin Coater tool has been used to perform the deposition, setting an initial spin speed of 500 rpm, then increased to 3000 rpm to obtain a final film thickness of approximately 9 μm .

2.2.4. Etching Technique

Etching is a fundamental process in semiconductor fabrication and microelectronics, employing a process that selectively removes material from the substrate or thin films to define device structures with high precision. Etching techniques are broadly classified into two categories: wet etching and dry etching, each offering distinct advantages depending on the material system, feature size, and process requirements.

Wet etching involves the use of liquid chemical solutions that react with the surface of the material to dissolve it. This method is typically isotropic, that is, it removes material uniformly in all directions, which can limit its applicability for high-resolution patterning [98]. However, wet etching is widely used for processes requiring high selectivity and smooth surface finishes, such as the removal of sacrificial layers or bulk substrate thinning. The choice of etchant chemistry is critical and depends on the material to be etched.

Dry etching, on the other hand, utilizes plasma or reactive ion species to remove material through physical sputtering, chemical reaction, or a combination of the two [99, 100]. Techniques such as Reactive Ion Etching (RIE), inductively coupled plasma etching (ICP), and Deep Reactive Ion Etching (DRIE) provide high anisotropy and excellent control over feature dimensions, making them indispensable for fabricating high-aspect ratio structures like trenches and vias in modern devices. Dry etching processes allow for precise pattern transfer with minimal undercutting even for materials generally inert like ceramics [101].

Specific Instrumentation

For what concerns this research, a dry etching technique has been employed to define the trench structure in the FEOL and to prepare the passivation silicon nitride films in the BEOL for the application of the method proposed by Xia et al. for toughness measurements (for further details, see Chapter 4). Although for the first case, trench patterning was a process step already consolidated in STMicroelectronics for IGBT production, partial removal of SiN_x layers required the development of a sequence of customized etching recipes developed in collaboration with the etching team of the company, based in Crolles (FR).

For both cases a LAM Kiyo EX tool was used employing a fluorine-based plasma with CF_4 gas mixed with Oxygen and Argon as precursors.

2.2.5. Thermal Treatments

Thermal treatments are essential post-deposition processes widely employed in semiconductor manufacturing to modify material properties, relieve residual stresses, activate dopants, and improve film quality. These treatments involve controlled heating of the substrate and the deposited layers under specific temperature profiles and ambient conditions.

The primary objectives of thermal treatments include [102]:

- **Stress Relaxation:** Thin films deposited by techniques such as PECVD often contain intrinsic residual stresses that can lead to wafer warpage or mechanical failure. Thermal annealing allows the atomic rearrangement and relaxation of these stresses, thus improving mechanical stability and reducing deformation [103].
- **Dopant Activation and Diffusion:** In doped semiconductor layers, thermal treatments activate implanted or in-situ dopants by enabling their incorporation into the crystal lattice and promoting uniform distribution. This step is critical to achieving the desired electrical characteristics.

- **Film Densification and Crystallization:** Amorphous or porous films may undergo densification or crystallization upon annealing, enhancing their electrical, optical, and mechanical properties. For example, silicon nitride films can become denser and less permeable after thermal treatment, improving passivation performance [104].
- **Interface Improvement:** Thermal treatments can enhance adhesion and chemical bonding at interfaces between different layers, reducing the likelihood of delamination and improving the device reliability. Such treatments are commonly referred to as “curing”.

Conventional thermal treatments are generally performed in controlled atmosphere furnaces, allowing uniform heating of multiple wafers simultaneously. Temperatures and durations vary depending on the requirements of the material and the process. A short-duration, high-temperature process that rapidly heats wafers is generally referred to as Rapid Thermal Annealing (RTA), performed to limit thermal budget and diffusion, however, provoking crystallization, stress release or other morphological changes [105]. Process parameters such as temperature ramp rates, soak times, ambient gas composition (e.g., nitrogen, forming gas, oxygen), and cooling rates are also carefully optimized to balance material improvements with device integrity [106].

Specific Instrumentation

Several thermal treatments have been performed during this research. For what concerns FEOL processes, the first two polysilicon layers deposited to fill the trenches undergo an annealing process at 650°C for 1 hour. Only the first layer also undergoes an RTA at 1050°C for 1 minute to improve the tensile contribution to compensate for the compressive contribution obtained during the second layer deposition. These treatments are primarily used to reduce the global stress distribution during trench filling. Subsequently, to close the trench structure build-up procedure, another high temperature thermal treatment, called “Body Annealing”, is performed at 1100°C for 2 hours of dwell time. This treatment is made to diffuse the dopant boron species in the silicon bulk up to a depth of about 0.5 μm to create the body region of the IGBT device and is one of the production steps simulated for the warpage investigation described in chapter 3. All treatments described above have been performed in the STM facility with a Centrotherm Multipurpose Batch Furnace in Nitrogen ambient.

For BEOL procedures, several treatments have been performed for various reasons. The silicon nitride toughness investigation required two types of thermal treatments, both conducted in the university Energy Department laboratories with a Lenton Muffle furnace (ECF type). This apparatus allows annealing in atmosphere of small dimension

samples (not full wafers) with a maximum operating temperature of 1200°C controlled through a PID controller (Lenton type 3216) which can regulate the heating rate up to 5°C/minute. Concerning the protective layer of PBO, the film must undergo a curing treatment at 320°C for 60 minutes; this treatment is performed in the STM facility with a Blue M Electric Multi-Chamber Oven immediately after film deposition by spin coating to completely solidify the PBO through the initialization of the oxazolation process (further discussed in chapter 5).

2.3. Material Characterization Techniques

In this research, a multidisciplinary suite of analytical techniques has been employed to investigate the structural, chemical, mechanical, and environmental attributes of the materials synthesized as described above. The following sections detail the principal techniques utilized, including spectroscopic methods for chemical and electronic structure analysis, mechanical testing to evaluate film toughness and adhesion, environmental control tools to simulate operational conditions, and advanced microscopy techniques for morphological and structural examination.

Such characterizations have been performed by me in both the laboratories of STMicroelectronics and the Department of Energy of the Politecnico di Milan University, with the exception of XPS and TEM analysis which have been executed by STM technicians. Table 2.4 reports the relationship between the characterization methods and the objectives in which they have been utilized.

Table 2.4: Summary of the characterization methods employed for the three objectives of this thesis: “Modeling of the trench-IGBT Warpage during specific critical step of production comparing die-to-wafer and homogenization approaches” (1); “Investigate composition-dependent mechanical behavior of silicon nitride thin films as passivation layers” (2); “Evaluate the adhesion of nitride/organic interfaces in controlled ambientes through nanoindentation testing In-Situ and Ex-Situ” (3).

Objective	Raman	XPS	Nanoindentation	Ambient Control	SEM	TEM	AFM	DM	OP
1) Simulations	–	–	✓*	–	✓	✓	–	–	✓
2) Toughness	✓*	✓	✓*	–	–	✓	✓*	–	–
3) Adhesion	–	✓	✓*	✓*	✓	–	✓*	✓*	✓

* Procedures carried out firsthand by the author.

2.3.1. Raman and Photoluminescence Spectroscopy

Raman Spectroscopy

Raman spectroscopy is a non-destructive vibrational spectroscopic technique based on the inelastic scattering of monochromatic light, typically from a laser source, by the vibrational modes of molecules or crystal lattices. When monochromatic photons interact with a material, most scatter elastically (Rayleigh scattering), maintaining their original energy. However, a small fraction undergoes inelastic scattering, exchanging energy with vibrational states in the material, resulting in a shift in photon energy known as the Raman effect [107, 108].

In a typical Raman setup, the sample is illuminated by a laser with frequency ν_L . The scattered light is collected and passed through a monochromator that filters out the intense Rayleigh scattered light, allowing only the shifted Raman scattered photons to be analyzed. The resulting Raman spectrum plots the scattered light intensity as a function of the Raman shift ($\Delta\nu$), defined as the difference in wavenumbers (cm^{-1}) between incident and scattered photons. Quantum mechanically, Raman scattering can be viewed as an interaction between photons and phonons - the quantized lattice vibrations - where phonons are either created (Stokes) or annihilated (anti-Stokes), with conservation of energy and momentum governing the process. Thus, the spectral region with lower energy than the laser line corresponds to Stokes scattering, which is more intense and commonly analyzed, while the higher energy anti-Stokes scattering is weaker (See Figure 2.7) [109].

From a physical point of view, the Raman effect arises because the incident electromagnetic field induces a time-dependent dipole moment in the material, related to the electronic polarizability tensor α . Vibrations modulate this polarizability, and only vibrational modes that cause a change in α are Raman active [110]. This effect is thus different from photon absorption, as it does not require the photon energy to be equal or higher than a present energy level, but instead excites the electrons to temporary "virtual states" that rapidly collapse. If the electrons return in a state of equal energy as the starting one, the photon scattering is elastic (Rayleigh), otherwise it is inelastic. If the energy of the incoming photon is such that the electron can reach a real electronic state, the Raman is called resonant, and the signal intensity gets highly enhanced.

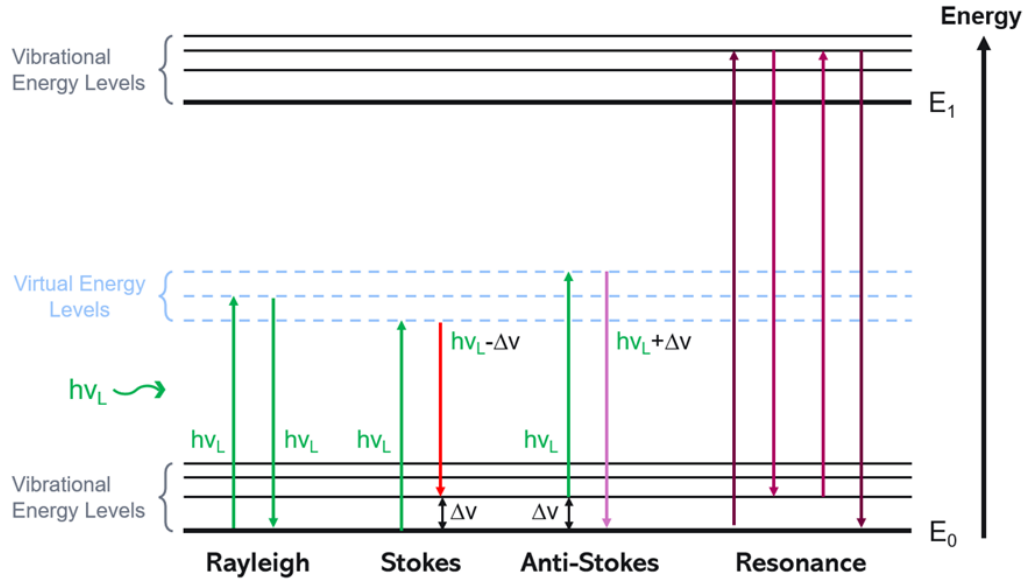


Figure 2.7: energy diagram showing the transitions involved during Rayleigh, Stokes, Anti-Stokes and Resonance Raman scattering, where E_0 and E_1 are the ground and excited electronic states, respectively, $h\nu_L$ is the laser energy and $\Delta\nu$ the Raman Shift.

Raman spectroscopy is particularly sensitive to crystal structure, phase composition, and defect states, thus being largely used in the field of semiconductors [111]. In crystalline materials, sharp Raman peaks correspond to well-defined phonon modes, while amorphous or nanocrystalline materials exhibit broader bands due to relaxed selection rules. This technique is widely used to identify material phases, assess crystallinity, and detect sub-stoichiometric or defect-induced changes in thin films and nanostructures.

Photoluminescence (PL) Spectroscopy

Photoluminescence spectroscopy is a non-destructive technique that probes the electronic structure of materials by measuring the spontaneous emission of light following optical excitation [112]. When a material is illuminated with photons of energy greater than its bandgap, electron-hole pairs are generated. These carriers quickly thermalize to the conduction band minimum and valence band maximum, respectively, before recombining in a radiative or non-radiative way [113]. For the former case, during recombination a photon is emitted with energy dependent on the characteristic electronic transitions; for the latter case there is no photon emission. Such processes are schematically reported in Figure 2.8.

Radiative recombination occurs freely across the bandgap or mediates through stable energy levels inside the bandgap, created by defects or excitons. For the semiconductor

industry, where heavy doping is often applied, it is common to encounter materials that show the band-tail effect: the large amount of impurities create an equal number of energy levels so similar to create new bands that extend the valence and conduction bands of the material, also changing its Density of States (DOS) [114]. The same effect has also been observed in disordered materials, such as amorphous silicon or hydrogenated silicon nitride, where the large amount of defect states leads to tail band formation [115–117].

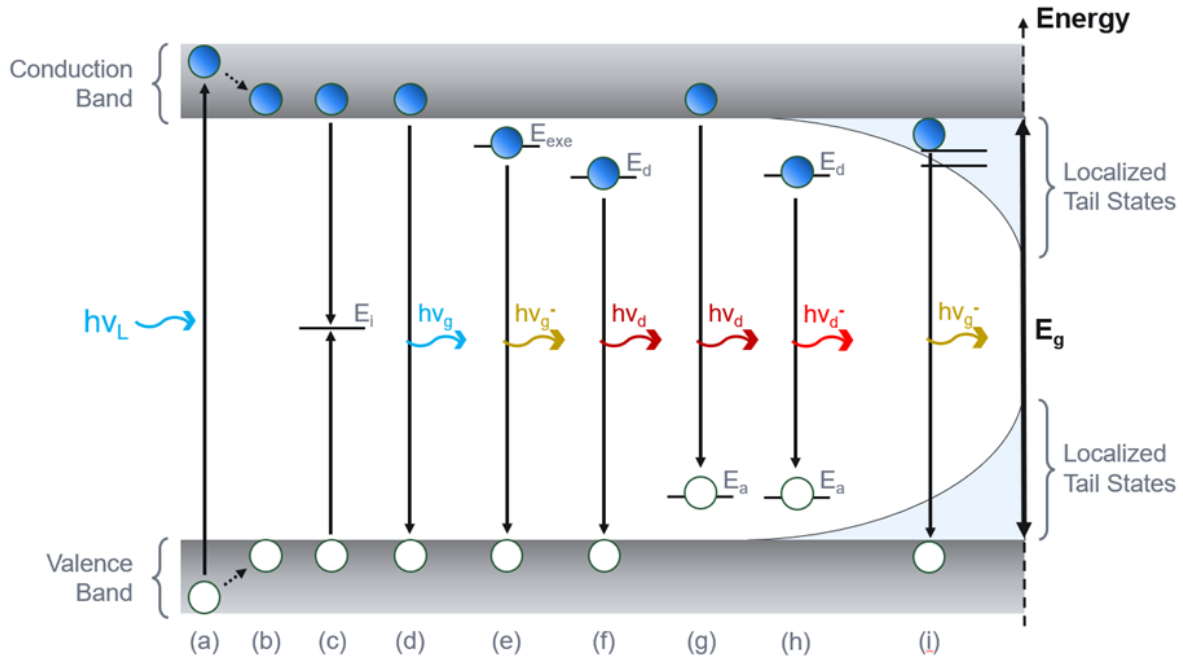


Figure 2.8: schematic representation of the most common optical transition in solids: (a) photoexcitation by incoming photon with energy $h\nu_L$ higher than the energy gap (E_g); (b) non-radiative thermalization of photo-excited charges which moves near the band edge; (c) non-radiative recombination via an intermediate state; (d) radiative recombination across the full bandgap; (e) radiative recombination mediated by an exciton state; (f-h) radiative recombination mediated by a donor level, an acceptor level, or both, respectively; (i) radiative recombination mediated by tail states.

The PL effect is exploited to reveal transitions involving localized states within the bandgap - such as donor or acceptor levels introduced by doping or defects - which manifest as emission peaks at energies below the fundamental bandgap. The relative intensities and energies of these peaks provide insight into the types of impurities, the densities of defects, and the quality of the interface [118]. Moreover, PL can also be observed in the presence of small inhomogeneities spread throughout the characterized material, where energy quantization can create new stable energy levels that promote photoluminescence

signals [119, 120]. Finally, advanced PL techniques, such as time-resolved photoluminescence (TRPL), enable the study of carrier dynamics by measuring the temporal decay of emission following pulsed excitation [113], while performing PL at cryogenic temperatures enhances spectral resolution and allows probing of subtle electronic features [121].

For these reasons, photoluminescence spectroscopy is widely employed to characterize surfaces and interfaces, detect defects, and evaluate the effects of processing conditions on material quality. Its versatility, sensitivity, and non-destructive nature make it an invaluable tool in semiconductor materials research. However, it must be noticed that for materials with indirect bandgap such as crystalline silicon, radiative recombination is phonon-assisted and less probable, often resulting in weaker PL signals.

Specific Instrumentation

For the analysis performed in this thesis, a Renishaw InVia micro-Raman spectrometer has been used, equipped with an Ar^+ gas green laser ($\lambda = 514 \text{ nm}$). Spectra have been acquired by a $1800 \text{ grooves mm}^{-1}$ grating, a super-notch filter (cutoff at 100 cm^{-1}) and a Peltier-cooled CCD camera, allowing a spectral resolution of about 3 cm^{-1} . With the same instrument, photoluminescence spectroscopy was performed using a different laser wavelength ($\lambda = 457 \text{ nm}$) and a $2400 \text{ grooves mm}^{-1}$ grating. In both cases, the laser power on the samples was kept below 1 mW to avoid thermal modification.

Raman and PL spectroscopy has been used to characterize SiN_x in search of signals related to amorphous silicon dispersed inside the silicon nitride matrix, as described in detail in Chapter 4.

2.3.2. X-Ray Photoelectron Spectroscopy (XPS)

X-Ray Photoelectron Spectroscopy (XPS) is a powerful surface-sensitive analytical technique widely used to investigate the elemental composition, chemical states, and electronic structure of materials. XPS provides quantitative and chemical information about the first few nanometers of a sample's surface, which makes it particularly valuable for characterizing thin films, interfaces, and surface treatments in semiconductor devices [122, 123].

The principle of XPS is based on the photoelectric effect: the sample surface is irradiated with monochromatic X-rays, and the incident photons excite core-level electrons from atoms in the surface layers, causing their emission with kinetic energies characteristic of their binding energies within the material. By measuring the kinetic energy and the number of emitted electrons, the binding energy spectrum is obtained, which is unique for each element and sensitive to its chemical environment. Indeed, XPS spectra consist of

peaks corresponding to specific core electron orbitals of elements present on the surface. The position of these peaks reveals the state of oxidation, chemical bonding and local electronic structure, allowing differentiation between, for example, silicon in elemental form, silicon oxide or silicon nitride [124]. Quantitative analysis is achieved by integrating peak areas and applying sensitivity factors, allowing the determination of elemental concentrations typically down to parts per thousand.

Specific Instrumentation

XPS analysis made in this research have been performed by STMicronics specialists on my requests; while I was able to attend the measurement, I was not the direct operator of these measures. The analysis was performed with a PHI5000 Versaprobe III (ULVAC-PHI) with a photon energy of 280 eV, equipped with an Ar sputtering gun to perform in-depth profile analysis up to 10 μm of acquisition depth.

XPS has been utilized to characterize the chemical states and surface composition of SiN_x passivation layers both for investigation of the mechanical properties of silicon nitride and the adhesion of PBO on different silicon nitride substrates.

2.3.3. Nanoindentation

Nanoindentation is a widely utilized technique for characterizing the mechanical properties of thin films and small volumes of materials at micro-nanoscale depths [125]. It provides quantitative measurements of several mechanical properties, such as hardness, elastic modulus, and, with specialized protocols, fracture toughness or interfacial adhesion. Its high spatial resolution and minimal sample preparation make it particularly valuable for investigating thin films used in advanced semiconductor devices and packaging [126, 127].

The fundamental principle of nanoindentation involves pressing a hard, well-defined indenter tip into the sample surface under controlled load or displacement while continuously recording the applied force and penetration depth. The resulting curve of load (P) versus displacement (h) reflects the response of the material to deformation, allowing the extraction of mechanical properties. Figure 2.9 shows a schematic representation of a classical indentation test and a relative load-displacement curve.

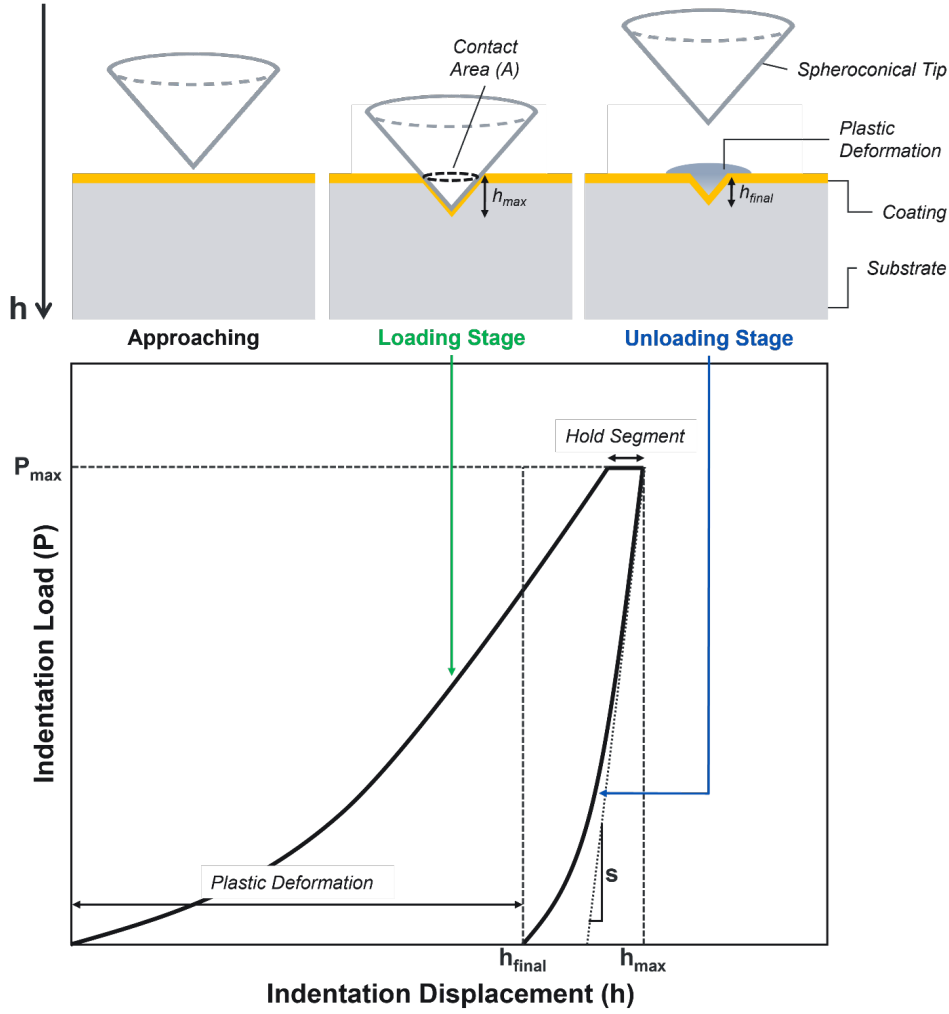


Figure 2.9: on the top is reported an illustration of the nanoindentation process on a sample cross-section, highlighting the approaching of the tip toward the sample surface, the loading stage where the tip penetrates the material, and the unloading stage where the tip retracts leaving a residual indentation profile related to the remaining plastic deformation. On the bottom is reported the related representative load-displacement curve, where h_{final} and h_{max} are the final and maximum indentation displacement, respectively, p_{max} is the maximum load, and s is the contact stiffness.

The nanoindentation load-displacement data is commonly analyzed using the Oliver-Pharr method to estimate the elastic modulus (E) from the relation between the stiffness of the load (s) and the contact area between the tip and the target material during the test (A) [126, 128]. Such a contact area is evaluated as

$$A = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + \dots + C_n h_c^{2-\frac{1}{n}} \quad (\text{Eq 2.1})$$

where h_c is the contact depth of the indentation and C_0 to C_n are coefficients based on the calibration of the indentation equipment. This area function is expressed as a polynomial, with the dominant contribution typically arising from the first few terms, whereas remaining terms are typically considered negligible.

Moreover, it is common to express Young's modulus considering its dependence on both the material and the indenter properties through the reduced modulus (E_r), evaluated as:

$$\frac{1}{E_r} = \frac{1 - \nu_c^2}{E_c} + \frac{1 - \nu_i^2}{E_i} \quad (\text{Eq 2.2})$$

where E_c and ν_c are the Young's modulus and Poisson's ratio of the coating, and E_i and ν_i are the Young's modulus and Poisson's ratio of the indenter. Finally, the contact stiffness s , defined as the slope of the initial segment of the unloading stage ($\frac{dP}{dh}$), is equal to [128]:

$$s = \frac{dP}{dh} = 2\sqrt{\pi}E_r\sqrt{A} \quad (\text{Eq 2.3})$$

Thus, by measuring A and s from the experiment and knowing the mechanical properties of the tip, it is possible to evaluate the reduced modulus of the coating.

The hardness (H) is another critical property evaluated from nanoindentation and represents the resistance of a material to plastic deformation. Its definition is more straightforward, being equal to:

$$H = \frac{P_{\max}}{A} \quad (\text{Eq 2.4})$$

where $P_{\max}$ is the maximum load reached during the indentation test.

A third important property typically measured with indentation is the toughness of the fracture (K_{ic}), i.e., the resistance of the material to propagation of cracks under stress [129]. This value is particularly important for brittle thin films, where cracks can significantly impact reliability, and it is classically evaluated using a single load-unload indentation test performed with a sharp tip - typically a cube corner - to generate highly localized stresses and control crack formations. Applying a stress that exceeds the film's fracture threshold, the radial cracks start and spread outward from the indentation site with scales and geometries dependent on the applied load, the properties of the film and the interaction of the underlying substrate. The resulting crack lengths are measured after indentation - typically using SEM -, and the fracture toughness is then determined by the Lawn-Evan-Marshall (LEM) equation [130]:

$$K_{ic} = \beta \left(\frac{E}{H} \right)^{1/2} \frac{P_{\max}}{c^{3/2}} \quad (\text{Eq 2.5})$$

where β is an empirical constant based on the geometry of the indenter tip, and c is the measured crack length from the center of indentation.

Different indenter tips are used to vary the spatial distribution of the stress in the volume of the tested material: sharp tips, such as Vickers, provide a localized deformation ideal for thin films, while blunter tips, such as Berkovich or spheroconical, reduce the stress concentration, which is better suited for fragile or viscoelastic materials [131]. Choosing the right tip ensures accurate and reliable mechanical measurements, but a general rule suggests that indentations should not reach depths greater than 10% of the film thickness in order to minimize substrate influence [132]. The “substrate effect” is a common challenge for thin-film indentation, where even the 10% rule could not be enough to avoid interference of the stressed area with the substrate, leading to results related to mixed film-substrate properties [133].

Indeed, Zak et al. [134] critically reassess the 10% rule guideline, demonstrating that while it holds reasonably well for hardness measurements, the elastic modulus is significantly more sensitive to substrate effects and requires indentation depths often less than 1–2% of the film thickness for accurate assessment. This discrepancy arises because hardness is governed primarily by the plastic deformation zone beneath the indenter, which is relatively localized within the film when shallow indents are used. In contrast, the elastic deformation zone extends much deeper, often penetrating into the substrate, influencing the measured elastic modulus that results and consequently the stiffness of the fracture (see Figure 2.10) [134].

Other factors such as temperature, the elasto-plastic properties of polymers, or strong doping can influence the methodology to use to correctly perform nanoindentation [135]. In addition, surface roughness and tip geometry critically affect measurement accuracy, particularly at very shallow indentation depths. Films with higher surface roughness exhibit greater scatter and potential underestimation of mechanical properties. Therefore, ensuring a smooth surface and sharp and well-calibrated indenter tips is essential for reliable data [136].

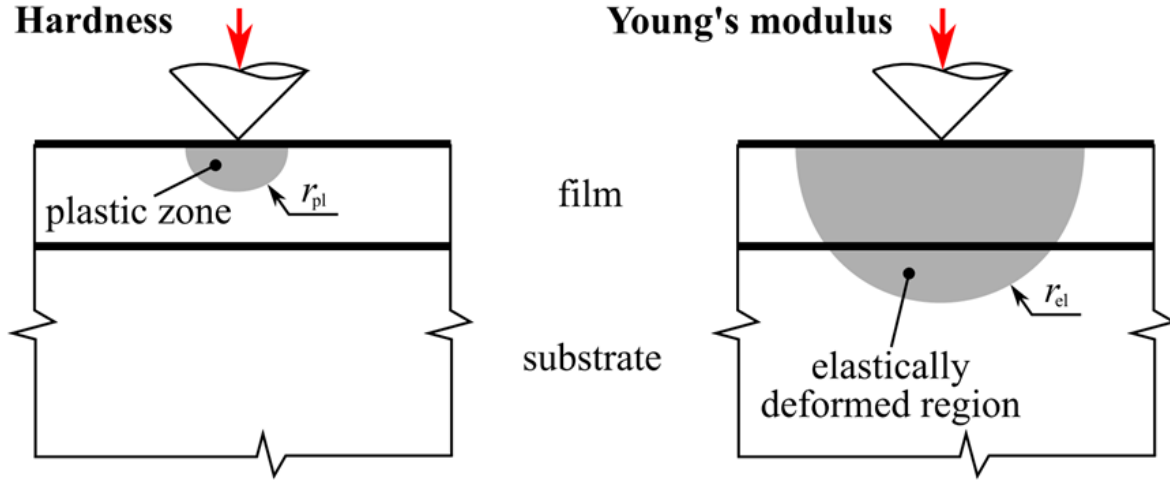


Figure 2.10: schematic of the difference between plastic deformation (hardness measurement) and elastic deformation (Young's modulus measurement). Image taken from Zak et.al. [134].

Specific Instrumentation

The mechanical characterization performed in this thesis was carried out employing a TriboScan TI300 nanoindenter using various tips for the different lines of research. The hardness and reduced modulus E_r were determined with a Berkovich tip using the well-established Oliver–Pharr method by restricting the indentation depths to a range of 8–15% of the thickness of the film and exploiting a specific analysis software, called Intrinsic Thin Film [137], which implements a methodology by Li & Vlassak [138] to exclude the effect of the substrate for a thin mono-layer deposited on a bulk substrate. Thus, it was possible to characterize these two values even for sub-micrometric films used both in the development of a predictive model for warpage simulations and the investigation of the mechanical properties of SiN_x .

The software was not suitable for toughness evaluation; thus, the method proposed by Xia et al. [75] was utilized for the accurate determination of the fracture toughness; in this case a Vickers tip was used (for further details in chapter 4).

Investigating the adhesion properties of PBO has instead been performed using a spherical tip with a radius of $20\text{ }\mu\text{m}$. In this case, two different tips have been used with standard or prolonged shaft to allow the implementation of the Humid|xSol module for ambient control (see section 2.3.4).

2.3.4. Tools for Ambient Control

Controlling environmental conditions such as temperature and humidity is crucial in materials characterization to ensure accurate and reproducible results, as these factors significantly affect material properties and device reliability [139].

Classic climatic chambers provide stable and uniform control of temperature and relative humidity within an enclosed space, enabling ex-situ aging, conditioning, and stress testing of samples over extended periods. These chambers typically cover broad temperature ranges and allow precise humidity adjustment, making them suitable for simulating various environmental conditions.

However, for real-time mechanical testing techniques such as nanoindentation, where direct access to the sample is required, in-situ ambient control systems are employed. These specialized modules integrate with nanoindenters to regulate temperature and humidity directly on the sample surface during testing. They enable rapid thermal cycling and precise humidity control while minimizing thermal drift and mechanical noise, allowing a dynamic assessment of material behavior under realistic operating conditions [140].

Specific Instrumentation

In this research, both classic climatic chambers and in-situ environmental control systems have been utilized to investigate the effects of temperature and humidity on mechanical properties and adhesion, providing comprehensive insight into material performance in different environmental scenarios.

The climatic chamber used has been a LIB DHPV-04_PV-04 with a maximum temperature and relative humidity capacity of 85°C and 85%, respectively. For in-situ testing, the Bruker xSol-Humidity module has been used with the TriboScan TI300 nanoindenter (described in Section 2.3.3) with a maximum temperature and relative humidity capacity of 75°C and 75%, respectively. Figure 2.11 reports a picture of the indenter set-up with the xSol-Humidity module.

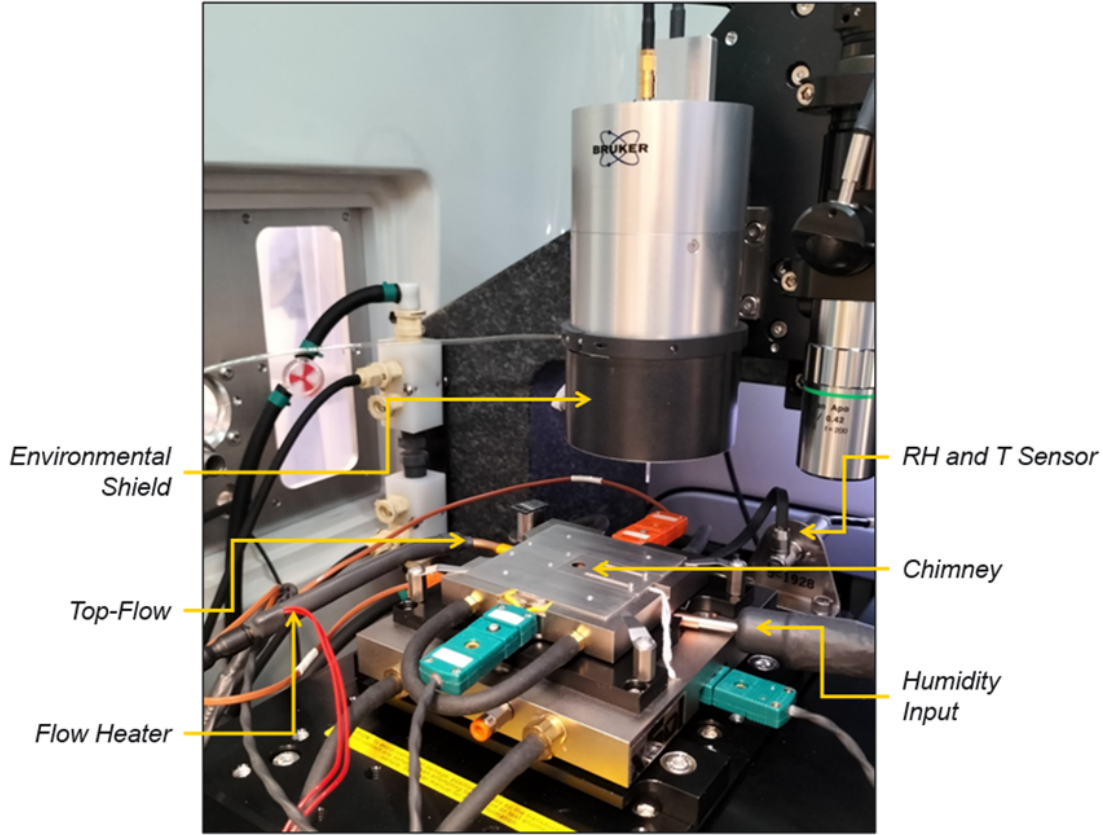


Figure 2.11: picture of the nanoindenter set-up with mounted xSol|Humidity module. In order to perform in-situ measurement with controlled environment several parts must be in function: the indenter transducer - equipped with a tip with elongated shaft - must be protected by the “enviro-shield”; the tip enters the chamber where the sample is allocated through the chimney, and the opening shielded by the external environment by an N_2 top flow (heated for test in temperatures higher than 50°C); through the humidity input a controlled vaporous H_2O flux permits to control the humidity, which is monitored, with the temperature, by a sensor positioned inside the chamber.

2.3.5. Microscopy Characterization Tools

Microscopy techniques are essential in materials science and semiconductor research for revealing surface morphology, microstructure, and nanoscale features that influence device performance and reliability. These tools provide complementary information across different length scales, from micrometers to atomic resolution, enabling detailed analysis of thin films, interfaces, and patterned structures. This section introduces the microscopy methods employed in this research to perform surface and cross-sectional inspections, highlighting their principles, capabilities, and typical applications.

2.3.5.1. Scanning Electron Microscopy (SEM)

SEM is a versatile imaging technique that uses a focused electron beam - typically with energies ranging from 0.5 to 30 keV - scanned across the sample surface to generate high-resolution images. When primary electrons interact with the sample, they produce various signals, including backscattered electrons (BSE), secondary electrons (SE) and characteristic X-rays (see Figure 2.12). BSE imaging offers compositional contrast due to atomic number differences, while SE imaging provides detailed topographic contrast with a spatial resolution of a few nanometers [141].

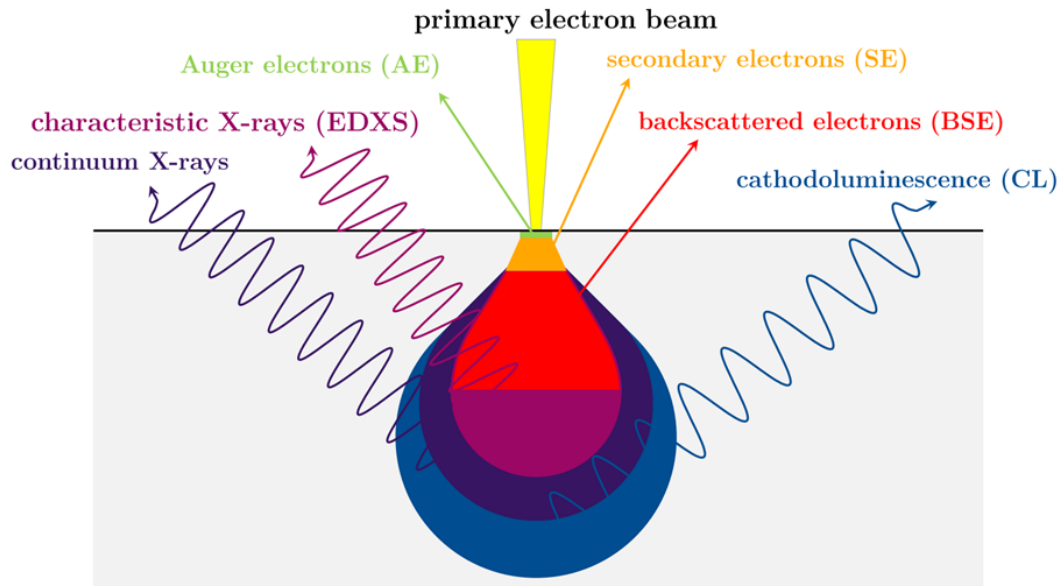


Figure 2.12: schematics of the interaction depth profile between the incident electron beam and the sample surface.

Specifically, BSE are produced by elastic scattering events between primary electrons and atomic nuclei. Due to the big difference between the electron and nuclei masses, the former lose little energy in these events. Since the backscatter cross-section is proportional to Z^2 , the higher the atomic number of the specimen, the more BSE are produced. For this reason, BSE can provide qualitative information about the sample composition. Primary electrons can also interact inelastically with the sample, i.e. with electrons bound in atoms. If the energy transferred is sufficient, SE are generated. Generally, SEs have a kinetic energy lower than 50 eV, but they need a higher energy than the material work function to escape from the surface of the sample and reach the detector. For this reason, typically, only the SE generated in the first nm from the surface can be detected. Other signals come from the sample surface upon the interaction with the primary beam, such

as X-rays, Auger electrons, catholuminescence and others, analyzed for different types of characterization not considered in this work.

Finally, Focused Ion Beam (FIB) systems are often integrated with SEM instruments, providing complementary capabilities for site-specific sample preparation and high-resolution imaging [138]. FIB employs a finely focused beam of ions - typically gallium ions - to mill, sputter, or deposit material with nanometer precision. This technique enables the preparation of precise patterning or electron-transparent cross sections subsequently used for TEM analysis (see next section).

Specific Instrumentation

The SEM employed for this thesis is a high-resolution Field Emission-SEM Zeiss Supra 40 with accelerating voltage capacity ranging from 1 to 30 kV (in this work, 3 kV were typically used). The sample holder is connected with five software-controlled motors (x, y, z, rotation, and tilt) and the images reported were obtained by acquiring SE electrons. The tool is also equipped with a Ga-based FIB system. Although the majority of the SEM inspections have been performed by me, FIB milling procedures have been performed by STMicronics specialists.

In this thesis, SEM has been employed mainly to examine surface morphology in cross-section to confirm the correct geometry obtained after etching for the application of the Xia method required for the evaluation of SiN_x . Cross-sectional SEM imaging, often combined with FIB milling, provides detailed views of layer thicknesses, interfaces, and structural features critical to correlating mechanical behavior with microstructure.

2.3.5.2. Transmission Electron Microscopy (TEM)

TEM provides atomic-scale imaging by transmitting a high-energy electron beam (typically 80–300 keV) through an ultrathin sample revealing detailed internal microstructure, crystallography, defects, and interfaces with sub-nanometer resolution ($\simeq 0.1$ nm) [142].

TEM imaging modes include bright-field, dark-field, and high-resolution TEM (HRTEM), each providing unique contrast mechanisms sensitive to crystallography, defects, and interfaces. Selected Area Electron Diffraction (SAED) patterns obtained in TEM reveal crystal structure and orientation, while scanning TEM (STEM) combined with spectroscopic techniques such as Electron Energy Loss Spectroscopy (EELS) or EDX enables nanoscale chemical analysis.

Specific Instrumentation

All TEM measurements were performed by STMicroelectronics specialists with my supervision on electron transparent lamellae with a Thermo Fisher Themis Z aberration-corrected STEM, operating at 300 kV acceleration voltage, equipped with an electron gun monochromator. Bright-field TEM (BF-TEM) micrographs were acquired for morphological analysis, while dark-field TEM (DF-TEM) images were acquired for microstructural analysis and determination of crystal dimensions. All lamellae for TEM characterization were prepared using a Thermo ScientificTM HeliosTM 5 UC dual beam, which combines an Elstar electron column with a Tomahawk HT Focused Ion Beam (FIB). This preparation was made through in-situ extraction method with a tungsten micromanipulator system. To avoid the silicon amorphization final cleaning at 5 kV was applied.

TEM has been instrumental in this work in characterizing the crystallinity and defect density of the silicon nitride passivation layers, providing critical insight for the investigation of the mechanical properties of SiN_x. Moreover, several TEM cross-sections have been used to obtain specific dimensions of the trench pattern analyzed for warpage simulations.

2.3.5.3. Atomic Force Microscopy (AFM)

AFM is a scanning probe technique that measures surface topography by detecting forces between a sharp tip (with radius 10 nm) mounted on a flexible cantilever and the sample surface. The operating modes include contact, tapping, and non-contact, each suited to different sample types and measurement objectives [143].

AFM achieves vertical resolution on the order of sub-nanometers and lateral resolution down to tens of nanometers, enabling detailed surface roughness quantification, morphological analysis, and mechanical property mapping at the nanoscale. Advanced AFM techniques can measure local stiffness, adhesion, and viscoelasticity through force spectroscopy.

Specific Instrumentation

The AFM measurements were conducted using a Bruker Dimension IconXR instrument equipped with a Nanoscope V controller, operating in Peak Force Tapping Mode in ambient air. A silicon cantilever with an Adama AD 2.8-AS tip (spring constant 2 N/m) was used to scan a 5 $\mu\text{m} \times 5 \mu\text{m}$ area with a resolution of 512 $\times$ 512 points at a scan rate of 0.5 Hz and a maximum force setpoint of 5 nN. The system provides nanometric spatial resolution, with X, Y, and Z calibrations accurate within $\pm 3\%$, and Z-axis non-linearity controlled below 0.5%, ensuring high precision and reliability in topographical

and mechanical surface characterization.

In this research, AFM has been used in non-contact mode to quantify the surface roughness of passivation

2.3.5.4. Digital Microscopy (DM)

Digital microscopy employs optical lenses coupled with high-resolution digital cameras and image processing software to capture magnified images of sample surfaces. Although limited by optical diffraction to lateral resolutions typically above 200 nm, digital microscopes offer rapid, large-area imaging with adjustable magnification and depth of field.

These systems facilitate quick inspection of surface defects, pattern fidelity, and macro-scale uniformity on wafers and device surfaces. Digital microscopy is user-friendly and suitable for routine quality control and process monitoring.

Specific Instrumentation

In this research a KEYENCE VHX-7000 / 7000N digital microscope was used, featuring motorized zoom lenses with magnifications from 20× to 6000× and a 3.19-megapixel CMOS camera. The system provides high-resolution, real-time visualization on a 27-inch UHD monitor, and is equipped with multiple illumination modes and a motorized stage that enables precise focusing, large-area imaging via image stitching, and quantitative surface measurements.

In this work, digital microscopy has complemented higher-resolution techniques by providing overview images of wafer surfaces and patterned areas, enabling efficient defect localization and morphological assessment. Its main use has been the measurement of crack extensions during the application of the Xia method for the evaluation of the toughness of SiN_x .

2.3.5.5. Optical Profilometer (OP)

Optical profilometry is a non-contact technique that measures surface topography by analyzing the interference or reflection of light from the sample surface. Techniques such as white-light interferometry and confocal microscopy provide vertical resolution on the order of nanometers and lateral resolution in the micrometer range [144].

White-light interferometry exploits the interference between light reflected from the sample surface and a reference beam to rapidly reconstruct three-dimensional surface profiles over large areas. Confocal microscopy uses spatial filtering to obtain depth-resolved images

with high vertical resolution and is particularly effective for measuring step heights and surface roughness. Optical profilometers enable quantitative mapping of surface roughness, thickness uniformity, and film morphology without damaging the sample.

Specific Instrumentation

Optical profilometry was performed using a Sensofar S-NEOX system operating in Extended Phase Shift Interferometry mode. The instrument employs a white light source and a 50×DI (Double-beam interference) high-magnification objective lens. The use of white light interferometry ensures accurate vertical resolution and minimizes sample damage, making it well suited for delicate thin-film structures.

In this research, optical profilometry was employed to monitor process-induced surface deformations that contribute to wafer warpage and to measure the extension of the delaminated areas during SiN/PBO adhesion analysis.

3 | Modeling the Trench-IGBT Warpage

This chapter addresses the first objective of this thesis: modeling trench-IGBT warpage during specific critical steps of production, comparing die-to-wafer and homogenization approaches.

The chapter begins with a brief explanation of the three most critical IGBT production steps for what concern wafer warpage - Gate Oxide Deposition, Trench Poly-Filling, and Body Annealing - and report of the related experimental values used to calibrate the simulations, also supported by a set of auxiliary flat wafers to better characterize the materials involved without the presence of the trench pattern, which of course makes the analysis more complex (section 3.1). Following, section 3.2 addresses the fundamental role of intrinsic stresses for warpage evolution and reports a procedure of stress-decoupling which I developed starting from the study of the flat auxiliary wafers. Section 3.3 reports the dual approach developed to solve the scaling problem of full-wafer simulations, which combines a classical finite element model, based on a discrete description of the device, with a model based on the homogenization method. The simulation results are then presented and compared with experimental data in Section 3.4. While good agreement is achieved for almost each considered process step, the model fails to accurately reproduce the warpage behavior observed at the trench closure stage during poly-filling, revealing unresolved challenges. Consequently, this section concludes with the following guidelines for further developments to improve model fidelity and predictive capability, while Section 3.5 reports a summary of the major information treated in this chapter and a condensed list of key takeaways.

3.1. Experimental Procedures

An accurate prediction of wafer warpage in trench-patterned IGBT devices is fundamentally dependent on the availability of reliable and representative experimental data. This section delineates the key experimental targets that serve as benchmarks for the validation

and calibration of the finite element models developed in this thesis.

The primary metric of interest of this research is the wafer bow, which is used to quantify the out-of-plane deformation of the wafer surface, while the warp value is not considered. In the first part of this Section the experimental bow data of the real patterned wafers are reported, providing a set of discrete values used as targets toward which the simulations will be set to converge. In the second part I instead describe the set of flat wafers used to support the simulation models as well as to allow the direct characterization, through nanoindentation, of the mechanical properties of the films involved.

3.1.1. Patterned Wafers

As reported in Chapter 2, the study of the warpage is ascribed to the initial process steps of the FEOL procedures. Following the very first step applied to the pristine wafer, i.e., the trench etching, the three consecutive steps of Gate Oxide Deposition, trench poly-filing, and Body Annealing were those that led to the greatest warpage development. The main information of such steps are reported below:

- **Gate Oxide Deposition:** this step involves the growth of a 110 nm thick silicon oxide layer lining the trench sidewalls and bottom surfaces. Film growth is carried out at 1050°C through thermal oxidation, which is known to typically induce strong residual stresses. The resulting stress distribution is spatially heterogeneous due to the geometry of the trench pattern, leading to a starting asymmetric warpage.
- **Trench Poly-Filling:** this step entails the sequential deposition of three polysilicon layers to fill the etched trenches and form the gate electrodes. It is characterized by complex stress evolution, as each polysilicon layer possesses distinct intrinsic stress components influenced by the deposition parameters and subsequent thermal treatments.
 - First layer (Poly A): 150 nm thick p-doped amorphous polysilicon deposited at 520°C, followed by an in-situ annealing at 620°C (1 hour) and a subsequent rapid thermal treatment at 1050°C (1 minute).
 - Second layer (Poly B): 130 nm thick p-doped amorphous polysilicon deposited at 520°C, followed by an in-situ annealing at 620°C (1 hour).
 - Third layer (Poly C): 250 nm thick p-doped amorphous polysilicon deposited at 520°C without further thermal treatments.
- **Body Annealing:** this step involves a high-temperature thermal treatment performed at 1100°C for 2 hours of dwell time after trench filling to activate the dopants and

form the body region of the device. This annealing step further modifies the stress state through thermal expansion and relaxation phenomena, affecting the final warpage profile.

For each of these production steps the wafer profile was measured along two perpendicular directions set as the X and Y coordinates, where X is the direction perpendicular to the trenches and Y is the direction parallel to them. All images presented in this chapter will use a blue color code for the X profile (direction across trenches) and a red color code for the Y profile (direction along trenches). The profiles of a large number of wafers ($\simeq 200$) were measured in both directions through PWG optical interferometry during wafer processing. Such measurements were performed before the Gate Oxide Deposition step (initial state) and after each of the three steps of interest, in order to evaluate the bow variation apported. Since the Trench Poly-Filling step is operated through the consecutive deposition of three polysilicon layers, an intermediate point of evaluation is taken after the second polysilicon layer (Poly B); thus, a total of four sets of values for experimental bow targets have been analyzed. The statistical analysis process carried out on the experimental data is reported in Figure 3.1, where only a representative sub-set of all experimental values has been represented, specifically, a single lot of 25 wafers. The variability between the results of each lot of 25 wafers has always been found to be less than 8% on both profiles.

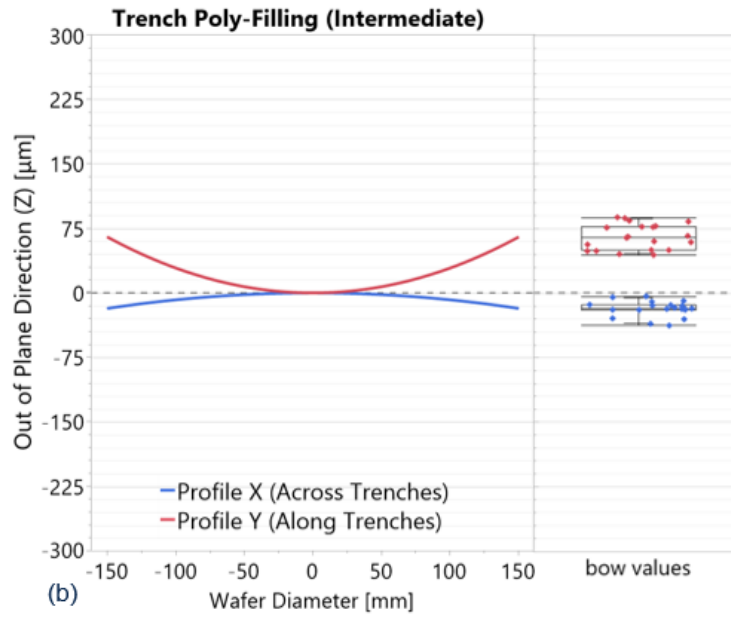
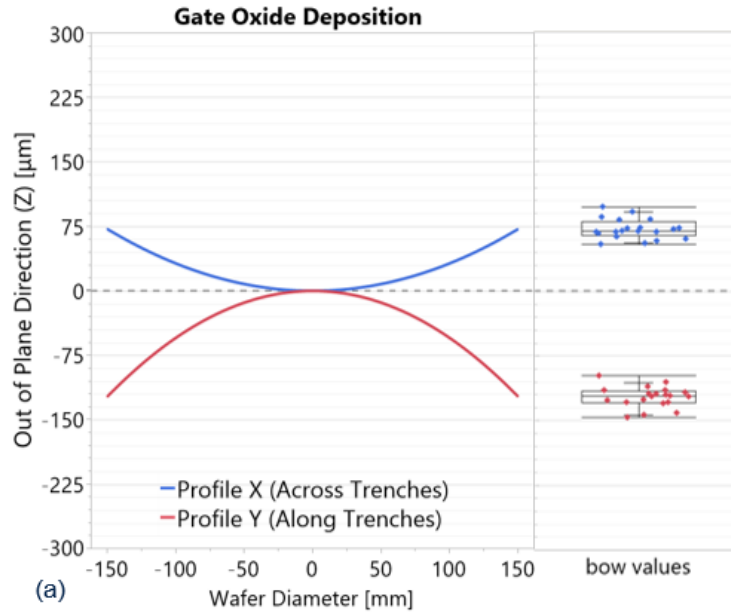
From the measured profiles, the extrapolation of the wafer bow values is immediate, being defined as the vertical displacement difference between the wafer center and its edge along the axis normal to the wafer surface. The mean values of all 200 experimental wafers evaluated for each step of interest along the X and Y directions are reported in Table 3.1. The extremes of the box plots (whiskers) obtained by analyzing the delta bow values of each wafer have been considered as the limits for the “targets” of the simulations.

Table 3.1: Average (mean) bow values along X and Y profiles for the four process steps of interest. In parenthesis are reported the extremes of the box plot, considered in this work as the acceptable targets for the simulations results.

Step of Production	Profile X (Across Trenches) [μm]	Profile Y (Along Trenches) [μm]
Gate Oxide Deposition	+72 [97.8 51.5]	-123 [-97.9 -138.3]
Trench Poly-Filling (Intermediate)	-18 [-3.9 -37.8]	+65 [87.9 44.2]
Trench Poly-Filling (Complete)	+122 [145.5 110.2]	+20 [48.8 3.3]
Body Annealing	-284 [-262.4 -298.1]	-141 [-129.6 -154.1]

The observed bow values range from approximately 10 μm to 300 μm depending on the process step, highlighting the substantial deformation induced by the fabrication sequence.

Furthermore, the presence of asymmetric warpage is evident in each of the steps analyzed, with the formation of a saddle-shaped deformation of the wafer in the case of the Gate Oxide Step and the intermediate Trench Poly-Filling Step. These types of deformation are the most dangerous for the proper handling of wafers during device growth operations and constitute the main reason for this research and the need to develop predictive models to prevent them.



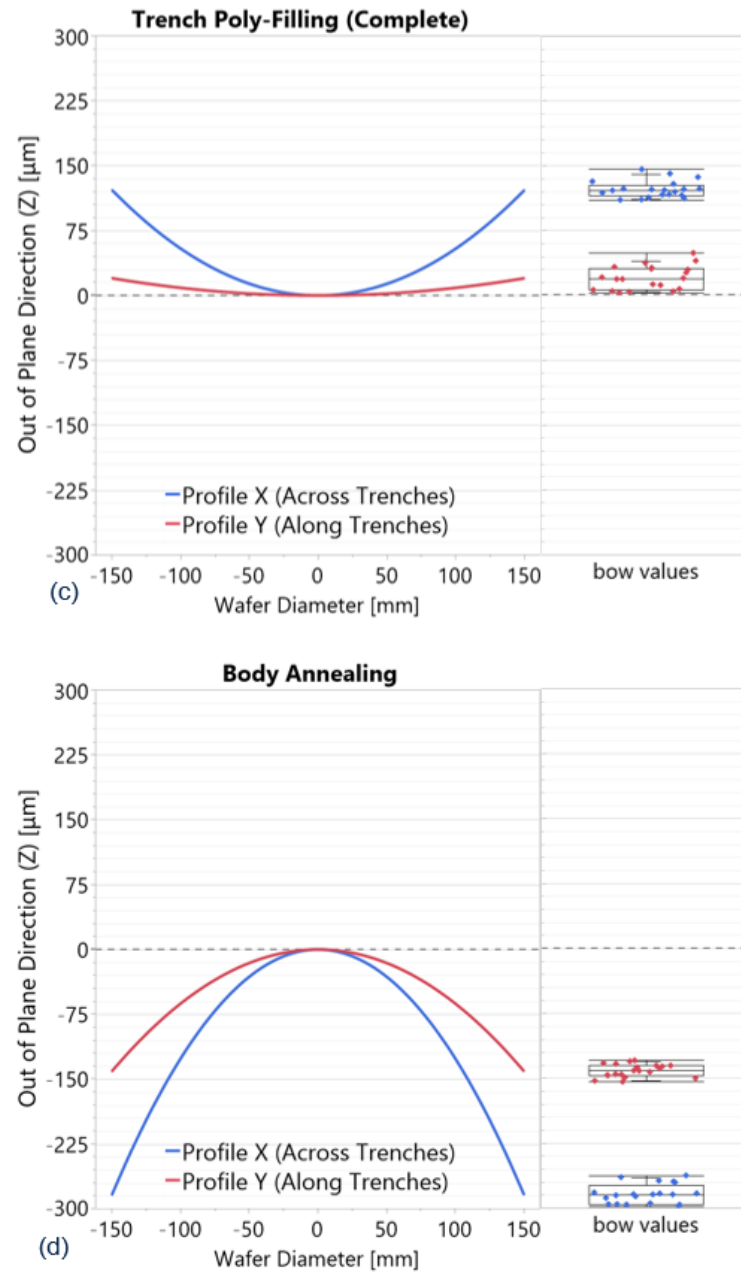


Figure 3.1: warpage profile along X (blue) and Y (red) direction for the average bow obtained by the analysis of a lot of 25 wafers for the Gate Oxide Deposition step (a), Trench Poly-Filling step after Poly B (b) and Poly C (d), and Body Annealing step (d). Each image shows on the right the delta bow values - compared to the previous step - for each individual wafer of the lot, and their resulting box plot. Due to the anisotropic nature of the trench pattern, warpage is direction-dependent and always exhibit asymmetrical warpage. For the case of the Gate Oxide deposition (a) and the Trench Poly-Filling step (intermediate) (b) the deformed state exhibits a saddle shape, individuated by the different direction of the profiles in the two directions.

The experimental data serve as quantitative targets to calibrate the model parameters and assess their predictive accuracy. It is important to note that the experimental warpage measurements inherently include contributions from all residual stress sources, both intrinsic and extrinsic; consequently, the modeling effort must incorporate a comprehensive representation of these factors to achieve meaningful agreement.

In summary, the experimental targets defined in this section establish the foundation for the subsequent modeling work. The direction-dependent bow values measured at the Gate Oxide Deposition, trench poly-setting, and Body Annealing steps constitute the benchmarks against which the dual finite element modeling strategies will be evaluated. Achieving close agreement with these targets is essential for validating the model's ability to capture the anisotropic warpage behavior that trench-patterned IGBT assumes when produced on 300mm wafers.

3.2. Flat Wafers

To support the simulation procedures, it was necessary to produce a set of 300-mm wafers deposited with a stack of the same materials involved in the patterned wafers but with a flat morphology. Each material involved in the simulations - namely, silicon oxide and polysilicon A, B, and C - has been deposited following the same stack present inside the trenches, thus considering the real substrate present at any deposition stage. This procedure led to the deposition of four different wafers, as reported in Figure 3.2.

Each deposited film was fabricated under identical deposition conditions as in the IGBT production procedure. However, the absence of patterning on the surface allowed both the direct characterization through nanoindentation of the four materials involved and the application of the Stoney Equation, which is strictly valid in the case of planar geometries within the linear regime. Moreover, each wafer has been subjected to a thermal treatment identical to that of the Body Annealing step, allowing for a second set of measurements.

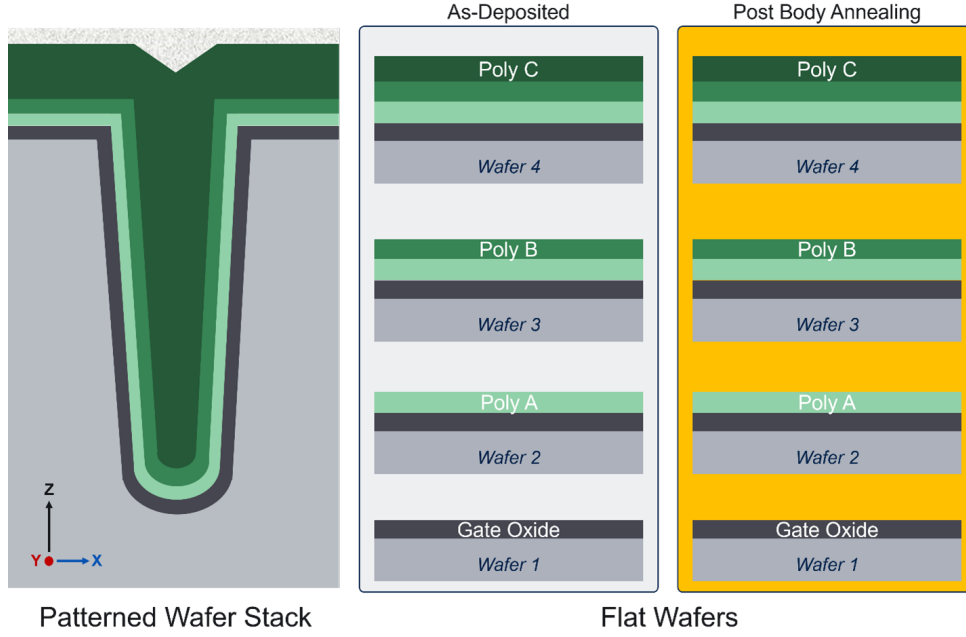


Figure 3.2: schematic representation of the set of four flat wafers deposited for supporting the simulation procedure. The flat wafers have been fabricated for every consecutive step of deposition involved in the three critical steps of production for warpage issue. Albeit the lack of trench patterning, each material has been deposited with the exact same deposition properties and on the same substrate. This would permit simpler evaluation of the mechanical properties of the materials involved and application of the Stoney's Equation to assess residual stresses. After a thermal treatment equal to the one implemented during the Body Annealing step such characterizations are repeated.

To evaluate the residual stress of the film deposited on a stack of materials, rather than a single bulk substrate, a modified version of the Stoney Equation has been used, reported in Equation 3.1:

$$\sigma_i = \frac{1}{6} \frac{E_{Si}}{1 - \nu_{Si}} \left(\sum_{k=1}^{i-1} t_k \right)^2 \frac{t_i}{\left(\frac{1}{R_i} - \frac{1}{R_{i-1}} \right)} \quad (3.1)$$

where σ represents the residual stress in the deposited film, E_{Si} and ν_{Si} are Young's modulus and Poisson's ratio of the silicon substrate, R is the radius of curvature, and t indicates the thickness of the involved materials which are identified by the subscript i (where $i = 0$ indicates the bulk substrate). To evaluate the radius of curvature R it is possible to exploit a direct relation between the bow and the curvature [145], which, in the case of a strong scale ratio between R and the diameter of the curved surface d (which

in our case is the wafer diameter), can be simplified as reported in Equation 3.2:

$$\text{bow} = R \left(1 - \cos \frac{d}{2R} \right) \rightarrow \text{bow} = \frac{d^2}{8R} \quad (3.2)$$

Using these formulas along with the previously exposed experimental procedure, a set of experimental values for each material has been obtained, reported in Table 3.2 along with other descriptor parameters related to the geometry of the system or assumed from the literature.

Table 3.2: Summary of major parameters used for thermo-mechanical simulation for each material involved before and after body annealing, where t is the film thickness, Max T is the maximum temperature considered for the thermal budgets imposed during the simulations, CTE is the coefficient of thermal expansion, E is the elastic modulus, ν is the Poisson's Ratio. The lines "As-Dep" consider the in-situ annealing carried out during the polysilicon deposition processes. Finally, the residual stress developed by each single film, evaluated using the modified Stoney's Equation (Eq 3.1) is reported.

Step of Production	t (nm)	Max T (°C)	CTE (1/°C)	E (GPa)	ν	Residual Stress (MPa)
Gate Oxide (Wafer 1) As-Dep	110	1050	5×10^{-7}	45	0.17	-352
Gate Oxide Body Ann.	110	1100	5×10^{-7}	45	0.17	-262
Poly A (Wafer 2) As-Dep	150	620 (+RTA)	2.5×10^{-6}	129	0.22	139
Poly A Body Ann.	150	1100	2.5×10^{-6}	129	0.22	12
Poly B (Wafer 3) As-Dep	130	620	2.8×10^{-6}	121	0.22	403
Poly B Body Ann.	130	1100	2.5×10^{-6}	129	0.22	-151
Poly C (Wafer 4) As-Dep	250	520	3.2×10^{-6}	80	0.22	-358
Poly C Body Ann.	250	1100	2.5×10^{-6}	129	0.22	-309

Such values have been used to describe the thermo-mechanical properties of each material involved in the simulations presented in the following sections for both flat and patterned wafers. While the values of thickness and temperature are nominal, those of Young's modulus and residual stress are experimental, the former evaluated through nanoindentation by specialized engineers at STMicroelectronics and the latter through the use of equation 3.1 following profilometries carried out with the PWG tool. The results are generally in line with the data found in the literature, even if the mechanical properties of polysilicon are known to vary greatly with different deposition parameters and after thermal treatments [146–148]. The greater difference between polysilicon A and polysilicon B and polysilicon C is explained by the different thermal treatments they have undergone. Specifically, the formers have been partially crystallized by the same in-situ annealing at 620°C, while the latter is left amorphous. In addition, Poly A also underwent rapid

thermal annealing (RTA) at 1050°C, which completed crystallization.

Due to the very thin thicknesses and the lack of adequate experimental tools at our disposal, it was impossible to obtain experimental data regarding the CTE; therefore, we relied on values typically used in STMicroelectronics for materials often involved in thermo-mechanical simulations such as silicon oxide or polysilicon. To reflect the different degree of crystallinity, we defined the CTEs of the three polysilicon before the Body Annealing step - which basically makes them identical to each other and fully crystalline - differently. Such values are strongly in line with values identified in the literature both for silicon oxide [149, 150] and polysilicon, where it is known that with an increasing degree of crystallinity, the CTE decreases [151, 152].

It should be noted that Poly B and Poly C are the two materials that have undergone the greatest variation in the CTE and elastic modulus parameters, as their morphology changes more radically from amorphous to crystalline (especially for Poly C, which after deposition did not undergo any types of annealing). The RTA experienced by Poly A is not directly considered in the simulations, but its effect is accounted for through the mechanical properties used to describe the material, which are the same as those of the crystalline polysilicon - obtained following body annealing also for polysilicon B and C - even during the steps before the thermal treatment.

3.3. Introduction of the Intrinsic Stresses

Despite the valuable insights gained from the initial application of the developed finite element models, the preliminary simulation results for the wafer bow during the Gate Oxide Deposition step did not reach the experimental target values, as will be discussed in Section 3.4. This discrepancy was traced back to a critical omission in the modeling approach: the neglect of the intrinsic stress component. Indeed, the initial implementation of the models considered the CTE mismatch among the materials involved to be the only driving force behind the warpage. However, as discussed in Chapter 1, the total residual stress in a deposited film is composed of multiple components, the principal ones being the extrinsic thermal stress and the intrinsic stress generated during film growth.

The first simulations did not account for the intrinsic stress effects. Due to the complexity of simulating the time-dependent development of intrinsic stress during film deposition, it was not feasible to model its evolution directly. Instead, it became necessary to determine the intrinsic stress values through a secondary study and then manually introduce them into finite element models within the “Initial Stress and Strain” module present in COM-SOL Multiphysics. However, directly characterizing intrinsic stress in thin film stacks

deposited on patterned substrates presents significant operational challenges.

This section describes the methodology used to decouple the total residual stress of the materials of interest into its thermal and intrinsic components. This stress decoupling approach enabled the isolation and quantification of intrinsic stress, which was then incorporated into the simulations.

3.3.1. Decoupling of the Residual Stress

Residual stress developing during thin film growth arises from multiple sources, including thermal mismatch and intrinsic growth mechanisms, but also etching procedures, doping and implantations, or other structural changes. The definition of residual stress and the theoretical foundations of this approach - developed by the author to solve a problem encountered in simulations - are presented in Section 1.2 of Chapter 1 of this thesis. Since the production step of interest only involves very low quantities of dopants, in this study, all the components of the residual stress were considered negligible or non-influential to warpage, except for the thermal and intrinsic components. Thus, the residual stress of each film present in the simulations has been considered as the sum of their intrinsic stress and thermal stress components.

The deformation resulting from thermal stress - originating from the mismatch of CTEs between the substrate and deposited films - is effectively captured by the finite element models developed described later in this chapter. In this case, the sources of discrepancies between real and simulated thermal stress are mainly related to the lack of accuracy in describing the properties of the material or the geometric fidelity of the model. The former aspect has been mitigated through mechanical characterization on the auxiliary flat wafers but remains a source of potential error of the study, as often happens for thermo-mechanical simulations. The latter aspect has been carefully faced by studying multiple kinds of geometries to test the sensibility of the results on the shape of the built models.

In contrast, intrinsic stress is not inherently considered in static thermo-mechanical simulations performed in COMSOL, which do not take in consideration the deposition process or its time-dependent nature, but only evaluate distinct blocks (model domains) related to the materials interacting between themselves through perfect interfaces. The development of intrinsic stress depends largely on the differences in microstructure and morphology between the substrate and the grown film, factors that require explicit modeling.

For this reason, to evaluate the intrinsic stress component, auxiliary flat wafers with uniform thin film stacks were manufactured and the residual stress of each film was estimated

with good precision before and after the body annealing step using the modified Stoney's equation after evaluating their curvature (Equation 3.1). Such results are shown in Table 3.2. Subsequently, the same flat wafers were modeled in COMSOL, and their deformation was simulated considering only the thermal budget as input. This procedure permitted to gain experimentally the residual stress component and by simulations the thermal stress component of each material involved. Assuming all other stress components to be negligible, the intrinsic stress component was estimated for each material before and after the body annealing step simply by subtracting the thermal component from the residual stress, as schematically represented in Figure 3.3.

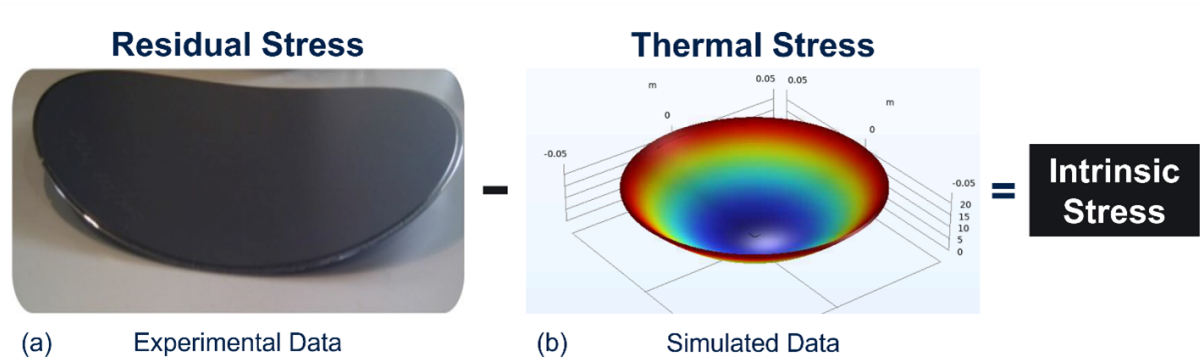


Figure 3.3: the stress decoupling procedure is performed starting from auxiliary wafers on which flat films of the material of interest are deposited with the same procedures implemented on the patterned wafers (a). After measuring the curvatures for these wafers through PWG profilometry the residual stress apported by the last film can be accurately measured applying the Stoney's Equation (Equation 3.1). A simple simulation of an identical wafers with the same stack of flat film is performed in COMSOL to obtain the curvature of the deformed wafer, from which the thermal stress in evaluated applying the same Stoney's Equation (b). The difference between the two values is assumed to be the intrinsic stress.

The total residual stress of each material and the related intrinsic and thermal contributions obtained by the stress decoupling procedure - necessary for accurate simulation of each of the process steps of interest - are reported in Table 3.3.

Table 3.3: Stress value for each material involved in the simulations. The intrinsic stress values have been calculated following the stress decoupling procedure.

Depositions	Residual Stress (Experimental Value) [MPa]	Thermal Stress (Simulation Value) [MPa]	Intrinsic Stress [MPa]
Gate Oxide As-Dep	-352	-120	-241
Gate Oxide Body Ann.	-262	-123	-139
Poly A As-Dep	139	126	13
Poly A Body Ann.	12	201	-189
Poly B As-Dep	403	70	333
Poly B Body Ann.	-151	-14	-137
Poly C As-Dep	-358	33	-391
Poly C Body Ann.	-309	-164	-145

3.4. Dual Approach: Discrete Trenches & Homogenization

One of the primary challenges in modeling wafer warpage for trench-patterned IGBT devices arises from the significant scale disparity between the overall wafer dimensions and the fine features of the device pattern. Although the wafer diameter extends to 300 mm, critical device structures, such as trenches, are characterized by lateral dimensions on the order of hundreds of nanometers and depths of a few microns. This multiscale nature imposes substantial computational burdens when attempting to simulate the entire wafer in full geometric detail.

To address this issue, two complementary finite element models were developed using COMSOL Multiphysics, based on classical FEA or the homogenization method, each refined progressively to simplify and reduce computational cost without compromising predictive accuracy. All simulations have been developed by employing the Solid Mechanics physics interface to model the thermo-mechanical behavior of the trench-patterned IGBT wafers. The materials were assumed to behave as linear elastic solids, characterized by isotropic or orthotropic elastic properties appropriate for silicon, silicon oxide, polysilicon, and homogenized layers. The simulations were conducted under time-independent (steady-state) conditions, imposing an initial temperature equal to the highest temperature seen by the material, thus the deposition or the in-situ annealing temperatures, where the stress-free condition is obtained. The final deformation state is measured by simulation after cooling from stress-free to room temperature (22°C). The thermal loading of each simulation was therefore applied as a delta temperature corresponding to this cooling process, with no transient or dynamic effects considered. Boundary conditions included symmetry constraints on appropriate faces of the model to reduce computational domain size, and fixed constraints to prevent rigid body motion. Gravity and other ex-

ternal forces were neglected, since the PWG tool used to characterize target bow values measures the wafers placed in a vertical position to minimize such effects (as described in Section 1.1.2). Thus, the dominant deformation mechanism was considered to arise only from residual stresses induced by thermal expansion mismatch and intrinsic film stresses.

The two approaches are described in detail later and complemented with a series of insights extrapolated from their synergic combined applications.

3.4.1. Discrete Trenches Method (DTM)

A classical and intuitive approach to warpage simulation involves identifying a representative portion of the full device to be modeled in detail and subsequently extrapolating the results to the full wafer scale. In this work such an approach was possible thanks to the strong repeatability of the trench pattern, which, at the step of interest for the simulations, is almost perfect within the border of each single device. Indeed, the scribe lines separating the IGBT devices that fill the wafer surface unfortunately break this repeatability. However, the approach proposed here considers such scribe lines as mildly impacting the full-wafer deformation state and is not considered.

Following this assumption, the “Discrete Trenches Method” (DTM) involves the construction of a finite element model incorporating the detailed geometry of the trenches within a small, millimeter-scale cell extracted from a general center portion of the device. The DTM simulates the thermo-mechanical behavior of this cell, capturing localized stress distributions and deformation patterns induced by the trench architecture, and then virtually extends the obtained bow deformation to a full-wafer dimension, considering a fixed curvature. Figure 3.4 reports two snapshots from the software interface showing the full cell and a magnification of the trenches, and a schematic representation of the concept permitting the bow extension to the full-wafer scale.

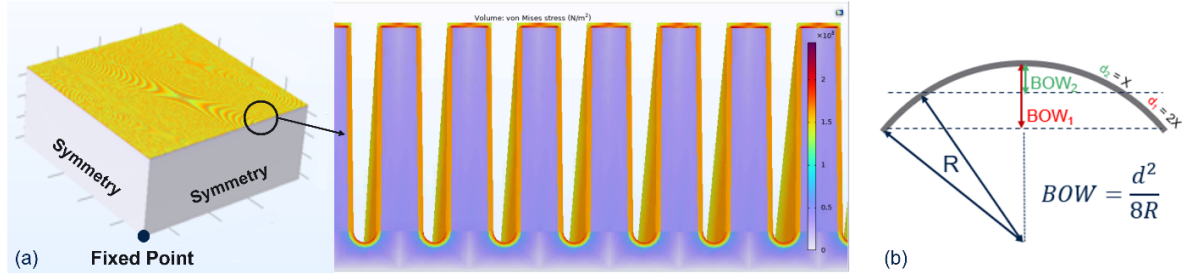


Figure 3.4: simulations with the DTM are performed on a small portion of a single device, built considering the full wafer thickness (770 μm) and a squared 2x2 mm area containing a total of 1250 trenches (a). Once the curvature of the simulated cell is known it is possible to calculate the wafer-scale bow considering the approximation of constant curvature (b).

The DTM model developed for this work consists of a 2×2 mm square region containing 1250 discrete trenches, modeled with realistic trench profiles including tapering and depth consistent with SEM and TEM cross-section observations made on real fabricated devices. The full wafer thickness (770 μm) is included to accurately represent bending and warpage effects. Boundary conditions impose symmetry on the lateral faces adjacent to the origin and free surfaces elsewhere, with a fixed vertex at the origin to eliminate all the remaining rigid body motion. Thermal loading simulates the cooling from the gate oxide deposition temperature to room temperature, accounting for the mismatch in CTEs between silicon and silicon oxide.

To further simplify the description of the trenches, aiming for faster simulations limiting the computational cost, the same approach has also been tested implementing squared trenches, thus removing the sidewalls tapering and the rounded bottom of the trenches. The two types of trench used in the DTM are reported in Figure 3.5. These parallel simulations using the same approach with a different description of the trenches also allowed us to verify how wafer-scale warpage is impacted by the trench geometry, as will be discussed in Section 3.4.

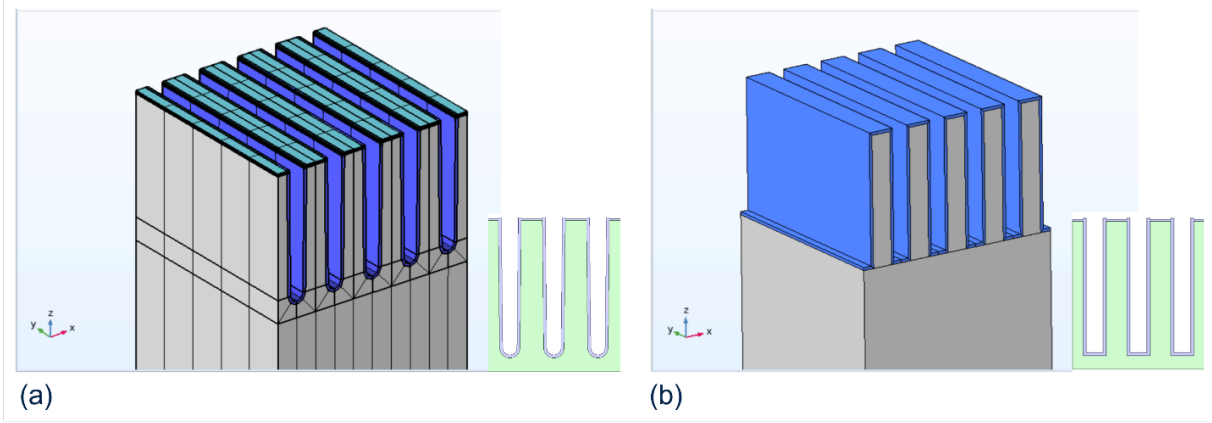


Figure 3.5: the trenches in the DTM are described in two different ways, the first are more realistic, with rounded bottom and tilted sidewalls (a), the second are more simplified, with squared angles at the top and the bottom of each trench (b).

Despite its detailed representation and physical fidelity, the DTM has intrinsic limitations. The computational cost restricts the model to a small portion of the wafer, necessitating assumptions to scale the local curvature to the full 300 mm wafer. Such assumptions are summarized below:

- Continuous Trenches: scribe lines, and secondary device layout variations are not considered and are assumed to be mildly impacting the global deformation.
- Constant curvature: the curvature of the simulated cell is considered representative of the entire wafer.
- Lack of Circular Geometry: virtually extending a squared cell to the full-wafer scale neglects the circular wafer geometry, instead giving the curvature of a virtual squared slab with a side of 300 mm.

These simplifications may introduce inaccuracies, but their impact on wafer-level warpage has been investigated, as reported in Section 3.4.

3.4.2. Homogenized Trenches Method (HTM)

To overcome the limitations inherent in the DTM, particularly those related to computational expense and scale-up assumptions, the Homogenized Trenches Method (HTM) was developed. This approach leverages finite element-based homogenization techniques to replace the complex trench pattern within the active area of the device by an equivalent orthotropic homogeneous layer whose effective thermo-mechanical properties are derived from a Representative Volume Element (RVE) based on the structure of the trenches, as

visually represented in Figure 3.6.

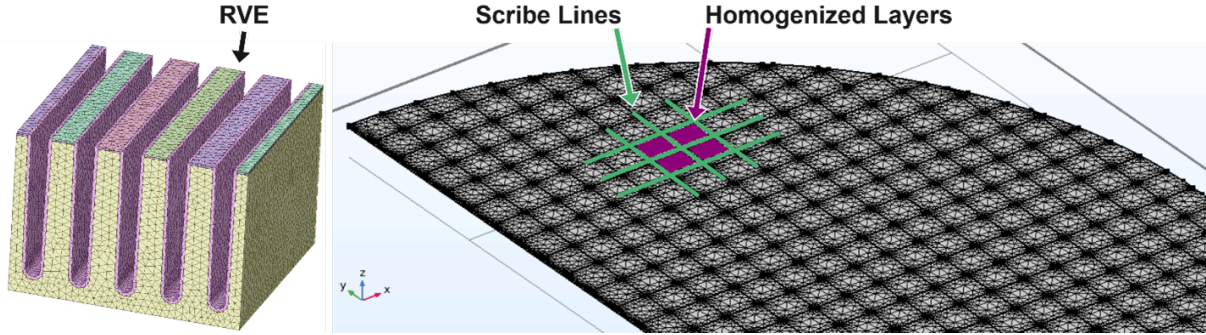


Figure 3.6: HTM approach is based on the use of an orthotropic material, obtained from the homogenization of a selected RVE. The obtained material is exploited in place of the trenched layer on the top-side of each device (examples in purple), with a thickness equal to that of the RVE. This permits to perform wafer-scale simulations considering scribe lines (examples in green), or circular shape of the wafer.

The homogenization process was implemented using ANSYS Material Designer to compute the effective elastic moduli, Poisson ratios, and coefficients of thermal expansion of the various RVE along each production step of interest, while the thermo-mechanical simulations were again performed with COMSOL Multiphysics. Several RVE configurations were considered to assess the impact of the thickness of the underlying silicon and the geometry of the trench on the homogenized properties and are reported in Figure 3.7.

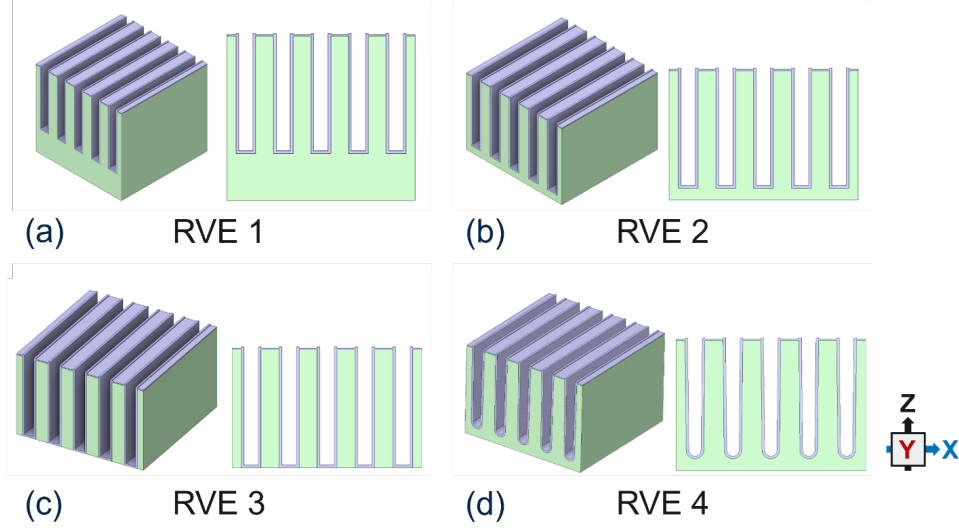


Figure 3.7: illustration of the different RVEs used for FE homogenization process in the HTM. Different bottom silicon thicknesses are implemented to assess its impact on homogenization results: $2\text{ }\mu\text{m}$ for RVE 1 (a), $0.5\text{ }\mu\text{m}$ for RVE 2 (b) and no underneath silicon thickness for RVE 3 (c). RVE 4 is a variant of RVE 2 with a realistic trench representation.

These RVEs, each containing five trenches, were developed in search of the cell that best represents a repetitive unit for the patterned “film” covering the top-side of the wafer during the production step of interest (the remaining portion of the models is completely occupied by bulk crystalline silicon). This investigation was carried out by varying the thickness of silicon below the trenches ($2\text{ }\mu\text{m}$, $0.5\text{ }\mu\text{m}$, or none) and the shape of the trench, reported as realistic or simplified (rounded or squared profiles, respectively).

The resulting orthotropic material properties were imported into COMSOL to perform wafer-scale structural analyses. For each RVE, the related properties were used to model a homogenized layer with the same thickness as the relative RVE to represent the patterned film. Each film was modeled on top of a bulk crystalline silicon substrate with the shape of a quarter of a disk of 150 mm radius. A symmetry boundary condition on the two straight sides allowed one to simulate the full 300 mm wafer. Moreover, the homogenized film was cut by a silicon grid of the same thickness that represents the scribe lines, thus separating the film in individual portions of the same dimension of a single device. Such an obtained wafer-scale macro-model has been tested in two distinct variations depending on how the shape of each device has been represented. The first macro-model was simplified to consider each IGBT having a squared footprint with a side of 1 cm , thus incorporating each homogenized layer in a regular silicon grid representing the scribe lines. The second

macro-model featured a more realistic device footprint, preserving the actual device shape and layout. Both macro-models are reported in Figure 3.8.

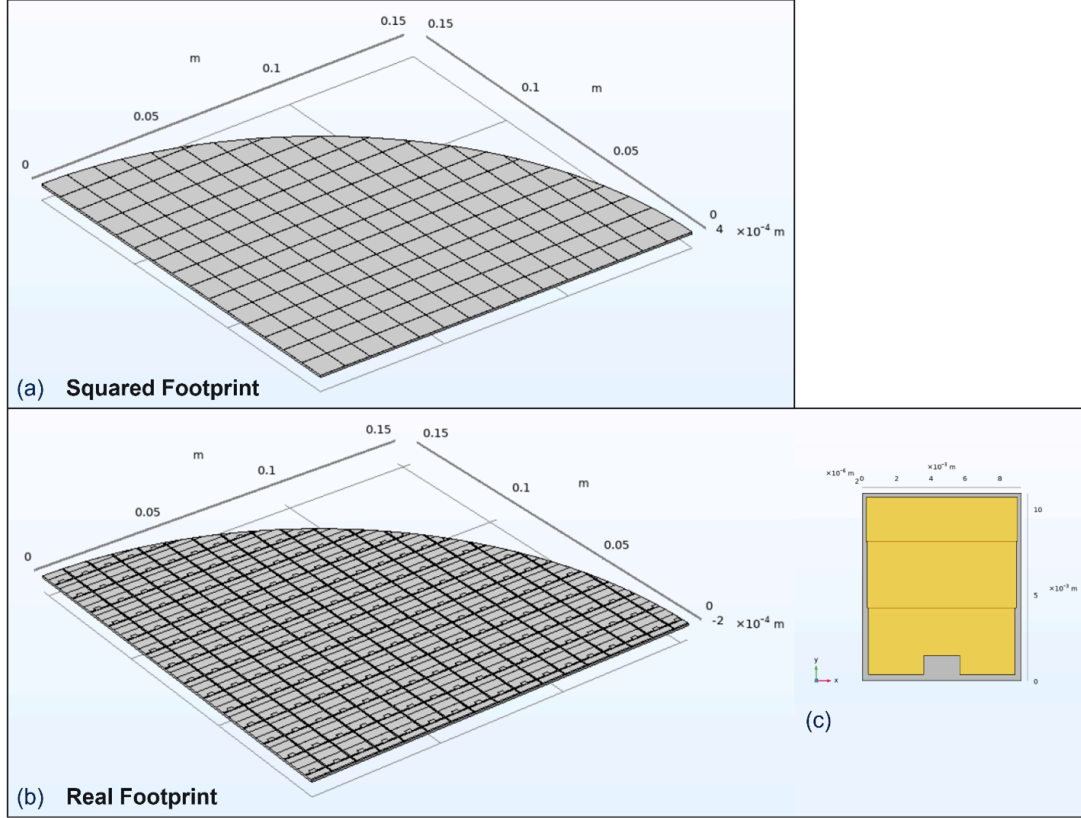


Figure 3.8: the homogenized layer must be integrated into a macro-model, which in this study was developed in two variants. The first version implemented a simplified squared footprint (a), and a second more realistic version implemented a real device footprint (b). The latter is reported on the right, with the yellow portion representing the homogenized layer (and thus the patterned area) and the grey portion the silicon scribe lines (c).

Similarly to the DTM, initial HTM simulations of the gate oxide step produced warpage predictions that fell outside the experimental target ranges. This outcome highlighted the need to incorporate additional optimization to further validate the homogenization approach, as discussed in Section 3.4.

3.4.3. Synergies of the Dual Approach

The complementary nature of the *Discrete Trenches Method* (DTM) and the *Homogenized Trenches Method* (HTM) approaches has proven instrumental in advancing the understanding of the underlying mechanics of warpage.

Each method presents distinct advantages and limitations that, when considered together, provide a comprehensive framework for warpage simulation. The DTM offers a detailed representation of the trench geometry and allows explicit assignment of localized material properties and intrinsic stresses. This flexible description facilitates the investigation of local stress concentrations and anisotropic deformation patterns. However, its computational cost restricts the model size to a small wafer portion and necessitates simplifying assumptions - such as constant curvature and neglect of scribe lines - for extrapolation to the full wafer scale. These simplifications, while necessary, could potentially introduce inaccuracies.

In contrast, the HTM significantly reduces computational complexity by replacing the intricate trench pattern with an equivalent homogeneous orthotropic layer derived from selected RVE analyzes. This enables full-wafer simulations that incorporate realistic device footprints and wafer geometries, including scribe lines. Nevertheless, the homogenization process inherently averages local variations and precludes the direct incorporation of intrinsic stresses associated with individual layers, limiting its resolution at the microscale. A summary of the pros and cons of the two approaches is reported in Table 3.4.

Table 3.4: Synthesis of pros and cons of the two models developed in this thesis – HTM and DTM – to simulate wafer warpage during critical steps of IGBT production.

			Pros	Cons
Discrete	Trenches	Method	- Presence of separate domains for each involved material or portion of the device. - Permits detailed analysis of stress field distributions.	- Limited to small areas due to large computational costs. - Impossible to consider scribe lines or other detailed structures. - Die-to-Wafer scale-up considering constant curvature.
Homogenized		Trenches	- Description of large areas without increasing computational costs. - Possibility to integrate macro-models with varying level of detail.	- Requires an optimization study of the RVE. - 2D homogenization is not automatic on every software (Ansys is needed). - Not possible to directly insert an external stress (intrinsic).
		Method (HTM)		

3.5. Results

This section presents the main simulation results obtained using both the Discrete Trenches Method (DTM) and the Homogenized Trenches Method (HTM), initially for the Gate Oxide Deposition step, used as benchmark for optimizing the models, and subsequently for the following process steps, performed exclusively using the DTM. Indeed, the HTM model has only been tested in the first step of the process due to its applicability limitations caused by the difficulty of incorporating the contribution related to intrinsic stress, as will be described below.

Although most results show good agreement with the experimental target values, the polysilicon C deposition step - corresponding to the complete closure of the trench - introduces a new deformation contribution that is evidently not captured by the proposed models. Therefore, this section concludes with identified directions for potential developments aimed at resolving the trench closure issue.

3.5.1. Gate Oxide Deposition

The gate oxide deposition step served as the primary platform for model development and validation. Being the first step in the process sequence and the least computationally demanding, it allowed for extensive verification tests and provided the most reliable experimental data for comparison.

Figure 3.9 reports the principal simulation results obtained for this step, illustrating the warpage profiles predicted by both the DTM and HTM models alongside the experimental measurements.

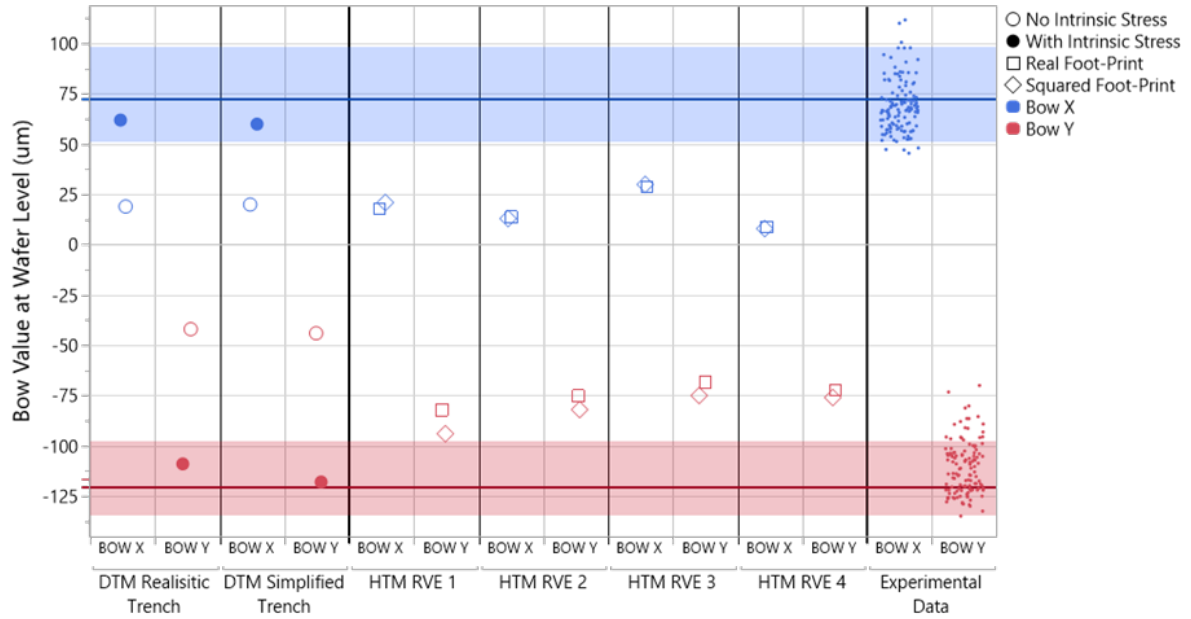


Figure 3.9: bow values for the Gate Oxide Deposition step along the X (blue) and Y (red) profiles for DTM and HTM simulation results in all the tested variants. On the right the experimental values taken from the curvatures of real wafers at the Gate Oxide Deposition step are reported. Related to the experimental values, the colored bands indicates the limit of the simulation targets, as obtained by the statistical analysis of the experimental data, while the colored solid lines indicates the mean value.

The results show that the introduction of the silicon oxide intrinsic stress in the DTM model - evaluated in Section 3.2 to be equal to the -241 MPa tensile stress - is the only factor that allows the simulation values to match the experimental ones, with both bow along the X and Y profiles falling within the acceptable target bands, thus paving the way for possible sensitivity tests or other types of predictive simulations. Specifically, the simulation with realistic trenches gives a bow X and Y of 62 μm and -109 μm respectively, while the simulation with simplified trenches gives a bow X and Y of 60 μm and -118 μm respectively. Such results indicate a small dependency of the wafer warpage on the geometric fidelity of the trench representation.

A noteworthy aspect of the DTM simulation is the careful treatment of intrinsic stress incorporation. Since intrinsic stress values were estimated using Stoney's equation, they represent planar isotropic stresses acting only within the film plane, without out-of-plane components. However, in a patterned film, such as trench oxide, the in-plane directions vary depending on the film portion under consideration. Specifically, film sections deposited on the bottom of the trench or the top of the silicon lines consider the X and Y

directions as in-plane, whereas film sections on the trench sidewalls consider the Y and Z directions as in-plane. By decomposing the total deposited film domain into these distinct planar sections, it was possible to assign the corresponding intrinsic stress components in the plane separately, thereby more accurately representing the stress state within the patterned structure.

Considering the HTM results, it can be observed that the results are generally in line with that obtained for the DTM without intrinsic stress, especially for the values on the X profiles, while on the Y profiles the values lean slightly better toward the target band. Comparing the HTM results between them, it is possible to notice that the description of the more realistic macro-model does not significantly change the bow results compared to those obtained with the simplified macro-model. The same applies to the more or less realistic description of the trenches within the RVEs, since the results of RVE 2 and RVE 4 are very similar to each other; this is in line with the results observed for the DTM. Finally, the decreasing thickness of silicon beneath the trenches (i.e. going from RVE 1 to RVE 3) seems to have an overall tensile effect on the Y profile, reducing the absolute bow value, while it does not affect sensibly the bow X until reaching the full absence of silicon beneath the trenches for RVE 3, which shows a slightly better result for the profile X. The latter RVE 3 was then selected as the best among the four variants - even if by slightly better performances - due to the minor average distance from both target bands and the major similarity with the DTM results.

Although the HTM failed to achieve the target due to the impossibility to consider intrinsic stresses, the approach was used as a benchmark to assess the impact of the DTM simplifications. The simulation results obtained through the HTM indicate that the geometric and boundary condition simplifications assumed in the DTM exert negligible influence on the overall warpage predictions. Figure 3.10 reports the flow of simplifications.

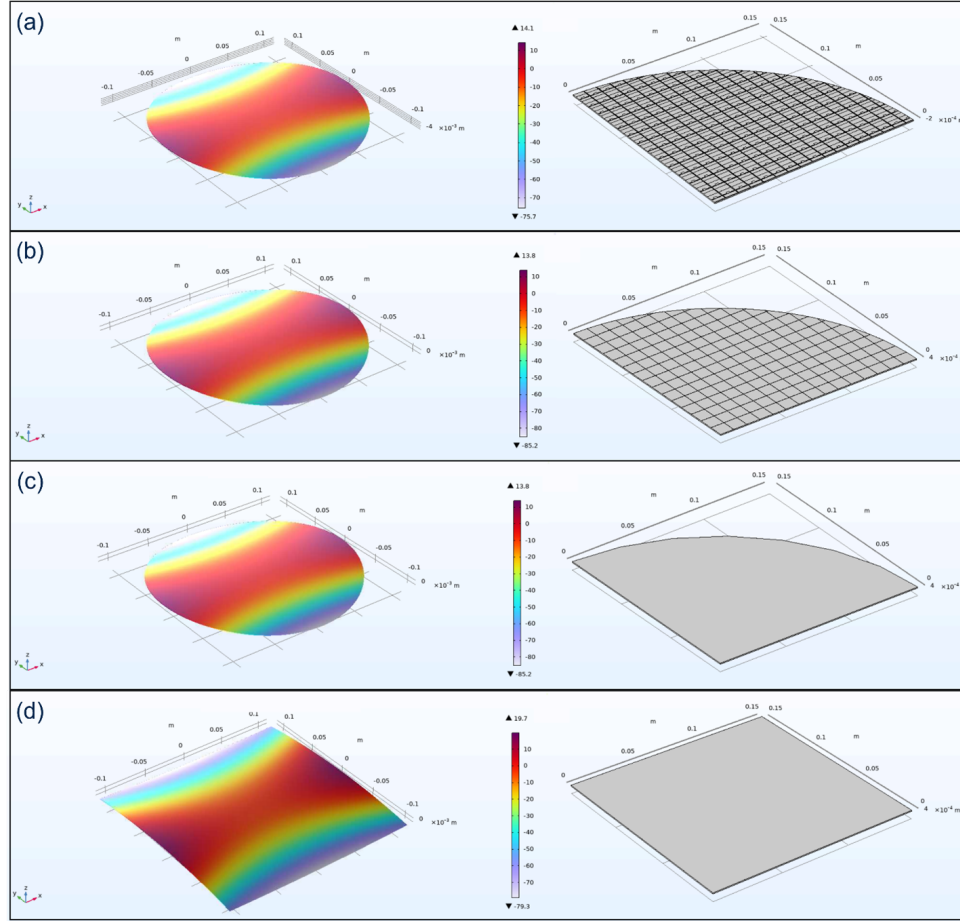


Figure 3.10: homogenization method allows simulations at wafer scale to explore further simplification of the models. From a realistic macro-model (a), it has been possible to simplify the structure using squared device shape (b), then further eliminating the presence of the scribe lines (c), and the circular shape of the wafer to describe a flat squared plate. The results showed almost no variation of the warpage results along the X and Y profiles, proving the simplifications implemented in the DTM to be unimpactful.

This sequence of simplifications not only proves the possibility of implementing simplified trenches and device footprint geometries, but also indicates the absent, or low, impact of scribe lines and wafer circular geometry. Moreover, by experimental observations it has been possible to assess that the real patterned wafer curvature along both the X and Y directions varies smoothly and continuously across the wafer surface. This continuity implies that the constant curvature assumption underlying the DTM is reasonable and does not severely compromise the model accuracy. Figure 3.11 reports the experimental bow profiles along the X and Y directions of a full wafer lot (25 wafers) at the Gate Oxide Deposition step, showing no major curvature variation for any of the profiles.

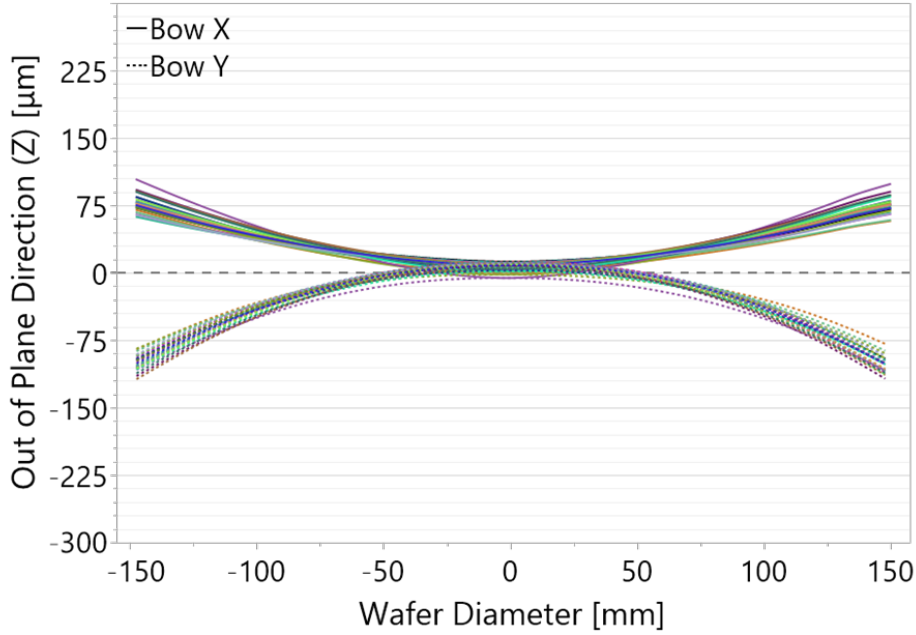


Figure 3.11: X and Y profiles (solid and dashed lines, respectively) from experimental measurements of 25 real wafer at the Gate Oxide Deposition step of IGBT production. The profiles along both directions show no major variation of curvature.

Given these validations, the DTM is assumed to be a valid approach which serves as a reliable tool for conducting sensitivity analyzes to quantify the influence of various mechanical properties on warpage.

Investigations focusing on silicon oxide mechanical parameters, such as the coefficient of thermal expansion or the intrinsic stress, or geometrical factors like the thickness of the silicon lines interspacing the trenches - a fundamental design parameter called Critical Dimension (CD) - reveal differential impacts on warpage along the two principal directions. Figure 3.12 shows the results of these sensitivity tests performed in the Gate Oxide Deposition step.

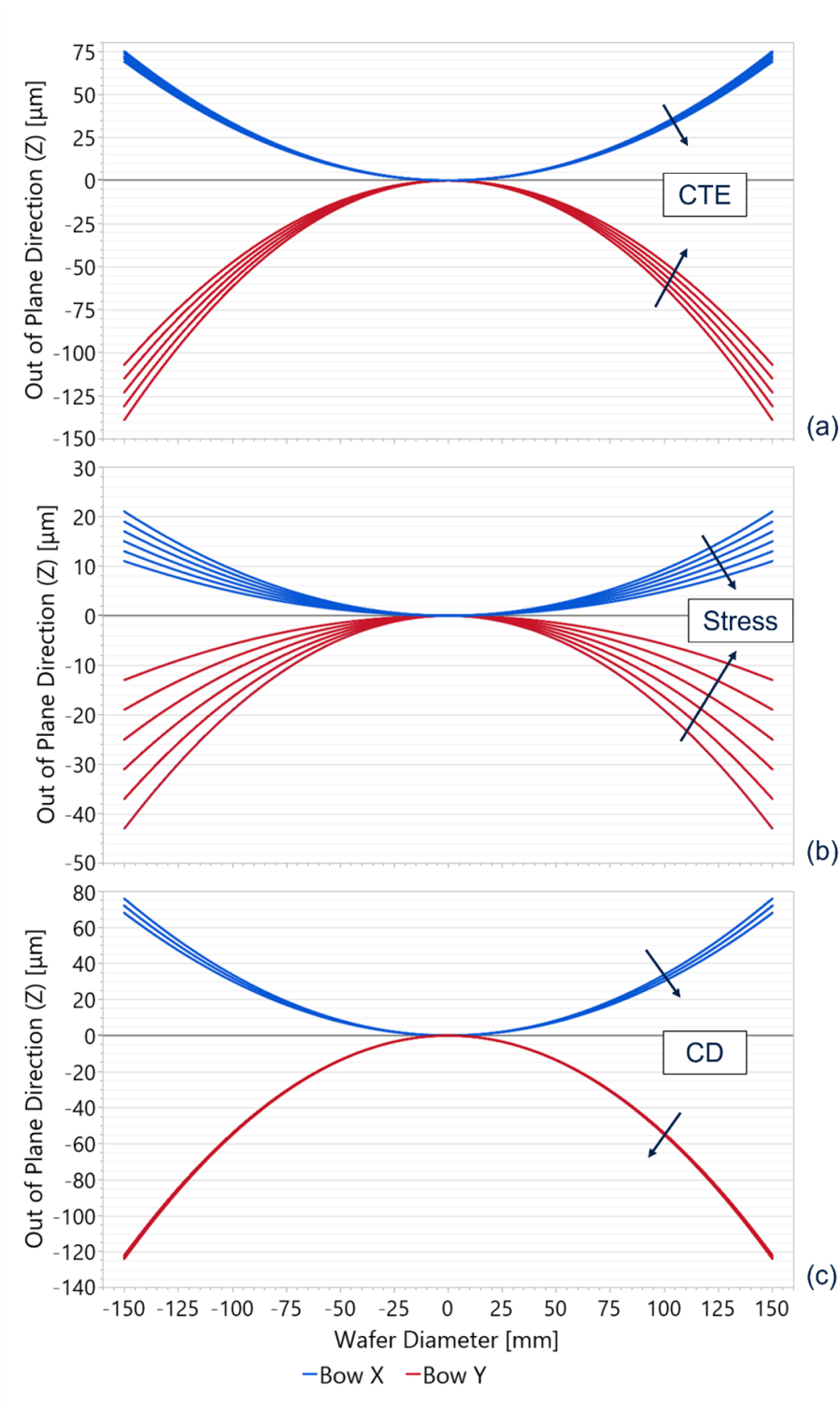


Figure 3.12: Sensibility test on varying parameters of the simulations for the Gate Oxide Deposition step: (a) varying CTE from $4e^{-7} \text{ 1/}^\circ\text{C}$ to $6e^{-7} \text{ 1/}^\circ\text{C}$ every $0,5e^{-7} \text{ 1/}^\circ\text{C}$; (b) varying silicon oxide intrinsic stress from 0 MPa to 100 MPa every 20 MPa; (c) varying trench CD as 540 nm, 600 nm (base value) and 660 nm.

This kind of analysis can be performed for every simulation parameter to evaluate the effect on warpage in a predictive way. However, in order to do so, it is necessary to have a starting point representative of the real physical state of the wafer and to completely validate the model with a large set of experimental data to certify the simulations for different variants of depositions and geometries.

In summary, the synergy between the DTM and the HTM provides a robust and complementary modeling framework. The HTM validates the simplifying assumptions of the DTM, while the detailed representation of the DTM enables nuanced parametric studies. Together, they form a strengthened approach for simulating and understanding wafer warpage in trench-patterned IGBT devices; however, their validity must be verified for the following step of production.

3.5.2. Following Steps of Production

The subsequent process steps - namely, the Trench Poly-Filling (intermediate and Complete) and the Body Annealing - were simulated exclusively using the DTM. Specifically, the reported results have been obtained with simplified trench geometry, which have been proven to impact the simulation in a negligible way. The results are summarized in Figure 3.13.

Simulations for these steps generally align well with the experimental warpage targets, confirming the robustness of the model and the reliability of the thermo-mechanical properties assigned to the films. However, the polysilicon C deposition step, which completes the trench filling, is a notable exception. In this case, the model fails to reproduce the expected bow values along the X direction, showing a significant deviation from the experimental data.

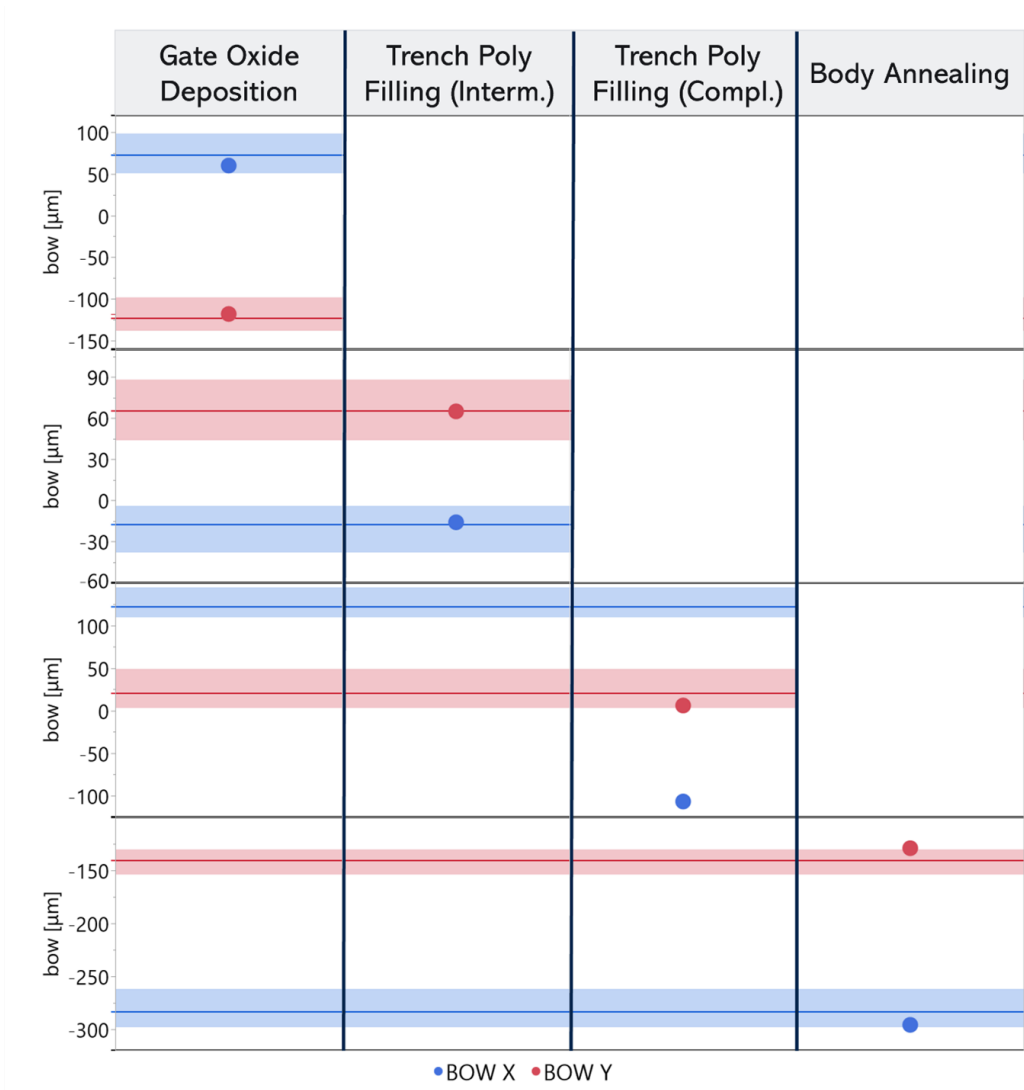


Figure 3.13: simulation bow results along X (blue) and Y (red) profiles are reported for the three deposition step (plus the intermediate point of the Trench Poly-Filling) in a comparison with the experimental data as reported in Table 3.1, with the solid colored lines reporting the mean value and the colored band the acceptable target range. All simulations are in target except for the one after complete trench poly filling, indicating a contribution to the warpage by the trench total closing not considered by the simulations.

The good agreement observed in the other steps indicates that the model effectively captures the principal deformation mechanisms and that the estimated material properties are accurate enough. The pronounced discrepancy between the intermediate and final trench filling steps cannot be explained solely by the addition of the polysilicon C layer. This layer maintains its nominal thickness of 250 nm only on the wafer surface, where it should apport a uniform tensile stress across both the X and Y directions, tending

to reduce the absolute bow values. Inside the trenches, the remaining free space along the X direction after previous depositions is instead only 54 nm. This indicates that the volume of poly C inside the trenches is relatively small compared to the other materials present. Some of these materials, such as the gate oxide, exhibit intrinsic stresses of similar magnitude but have not caused such an intense change in the wafer warpage.

Therefore, the observed error is attributed to the emergence of a new deformation contribution that arises specifically at the trench closure, which the current models are unable to capture. This phenomenon likely results from an abrupt increase in the overall stiffness of the system when the trench transitions from partially filled to fully closed. Before closure, the X direction is significantly more compliant than the Y direction due to the trench geometry; after closure, the constraints in both directions become comparable, fundamentally altering the warpage behavior.

Notable mention goes to the simulation of the Body Annealing step, which has been performed differently from the others considering the three polysilicon layers as merged in a single “crystallized” poly domain. In fact, this effect is intended by body annealing, which is performed to also crystallize the polysilicon C and to induce interdiffusion of layers. As shown in Table 3.2 the three layers after body annealing report indeed the same experimental mechanical properties. Similarly, the calculated intrinsic stresses, reported in Table 3.3, are similar between the three layers after the annealing and an average of -157 MPa has been considered for the united domain in the simulation. The bow results fall in the experimental target, even if with the greatest error between the four simulated steps (except for bow X in Complete Trench Poly-Filling), equal to 4.2% and 8.5% for bow X and bow Y, respectively, from the experimental mean values.

3.5.3. Trench Closing Effect

To investigate the origin of the error associated with trench closure, a parametric sweep simulation with DTM was performed, varying the thickness of the polysilicon C layer incrementally on both sides of the trench from 3 to 27 nm. Being the remaining space inside the trenches after polysilicon B deposition of 54 nm, a double thickness of 27 nm on the two sides of the trenches leads to their exact closing. Polysilicon C thickness has been varied only along the profile of the side walls and bottom of the trench, while the thickness above the trenches was maintained at 250 nm. This approach was selected to overcome the challenge of modeling a parametric sweep along two different scales for the areas inside and above the trenches. A representative scheme of the trench closure simulation (not at real scale and with only three of the nine simulated steps) and the bow

results along the X and Y profiles are reported in Figure 3.14. The quantitative results of the simulation are reported in table 3.5.

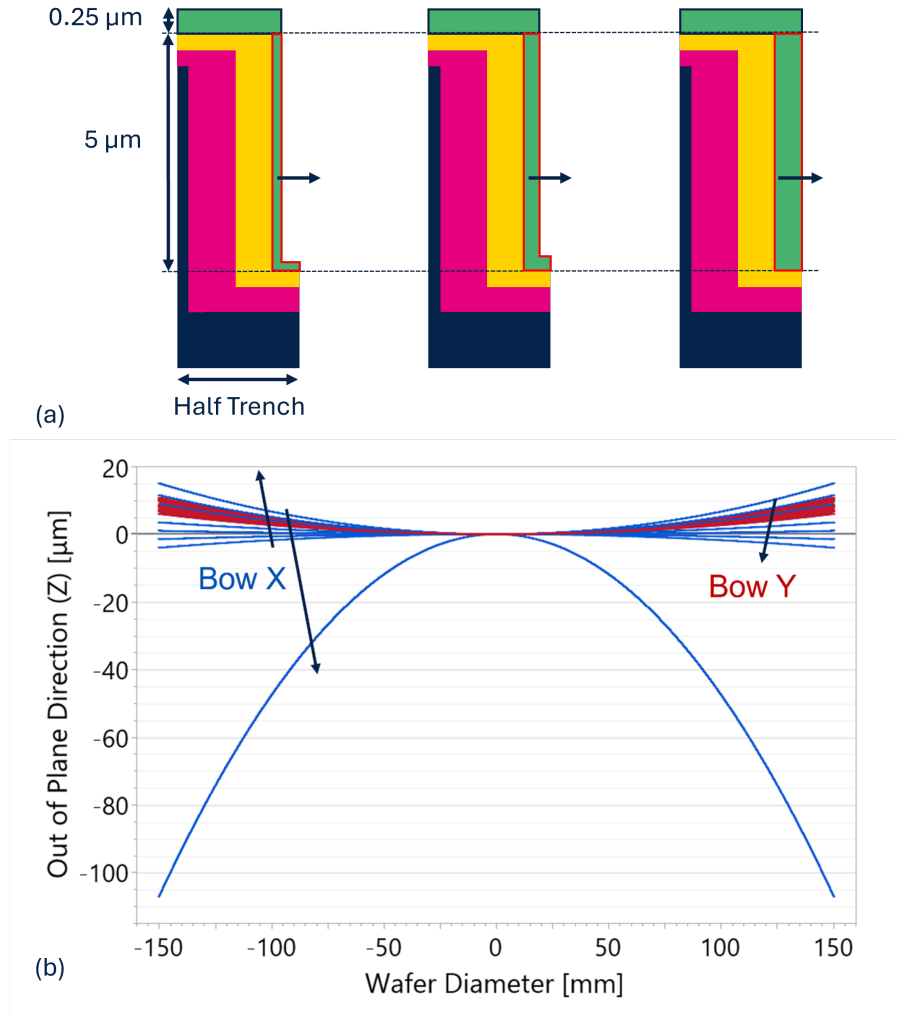


Figure 3.14: schematic representation of the trench closing simulation (a) and bow results along X and Y profiles for each tested Polysilicon C thickness up to total trench closing (b). Thickness above the trenches was maintained fixed at 250 nm. While bow Y is mildly influenced with a linear tensile trend by the thickness variation, bow X shows a particular evolution, starting at $-3.9 \mu\text{m}$ for a thickness of 3 nm and rising up linearly to $15.1 \mu\text{m}$ for a thickness of 24 nm, to then drop abruptly at a bow of $-108.3 \mu\text{m}$ with the total closing of the trench.

These data reveal that bow values in both both X and Y directions change approximately linearly with increasing polysilicon thickness until the trench is fully closed. At this point, a sudden discontinuity in bow X occurs, breaking the previously observed linear trend with an abrupt jump in the opposite direction. Specifically, bow Y varies from $10.8 \mu\text{m}$ to 5.9

Table 3.5: Bow X and bow y results of the trench closing simulation, relative to bow profiles reported in Figure 3.14. Considering a simultaneous growth of the film on both sides of the trench, 27 nm of poly C thickness comports a full closure of the 54 nm gap remaining in the trench after the deposition of Poly A and B.

t_{pC} (nm)	bow X (μm)	bow Y wafer (μm)
3	-3.9	10.8
6	-1.6	10.1
9	0.9	9.6
12	3.3	9.1
15	5.9	8.4
18	9.2	7.7
21	11.4	7.2
24	15.1	6.6
27	-108.3	5.9

μm varying the polysilicon C thickness from 3 nm to 27 nm, while bow X varies from -3.9 μm to 15.1 μm varying the polysilicon C thickness from 3 nm to 24 nm and then changes to -108.3 μm for the total closure of the trench with the polysilicon C thickness reaching 27 nm.

This sharp change in the X-direction bow indicates a numerical discontinuity unlikely to be as abrupt in reality, providing further evidence that the current model lacks some of the corrective mechanisms needed to accurately simulate the trench closure phenomenon. At a Poly C thickness of 27 nm the trench is already geometrically closed, so no significant overlap between opposite sidewalls should occur apart from thermal expansion effects. In the present simulations, however, no explicit contact physics has been activated; therefore, the model allows the domains to interpenetrate numerically once the nominal closure is reached, effectively enforcing an unrealistically compact profile along the X direction and causing a sudden change in the global stiffness and stress-transfer paths. In contrast, this change in stiffness and load path is not perceived along the Y direction, whose bow remains weakly affected by the variation in the thickness of poly C and by the complete closure of the trench. This further suggests that the observed discontinuity is mainly linked to the trench geometry and to the way its closure is numerically handled. A more robust description of the closure process, including an explicit contact formulation and a finer load stepping around the trench-filling condition, would likely mitigate these artificial jumps and yield a smoother, more physical evolution of the simulated bow.

Unfortunately, the aspect related to this discontinuity has not been resolved in the currently presented project. However, the DTM model has proven to be overall effective

in correctly simulating the thermo-mechanical behavior of the systems studied for most situations.

Clearly, further studies are necessary to fully understand the phenomena that occur at the complete closure of the trench and consequently to be able to correctly simulate this process step and be fully predictive.

3.5.4. Effective CTE

The decoupling of residual stress effectively overcomes the limitation related to the absence of intrinsic stress contribution in the initial models. However, this manual insertion is only feasible within the discrete trench method (DTM), where individual material domains remain distinct and can be assigned to specific stress states.

In the homogenized tunnel method, this approach is not possible within the RVE due to structural limitations imposed by the Ansys software used to carry out the homogenization processes. Moreover, the homogenization process merges multiple material domains into a single orthotropic equivalent layer, eliminating the possibility of assigning intrinsic stresses to individual constituents after the procedure. Consequently, an alternative method was developed - first presented during the EuroSimE conference [153] - to incorporate intrinsic stress effects within the HTM framework by definition of an effective coefficient of thermal expansion (CTE_{eff}). This varied CTE allows one to “brute-force” the wafer deformation knowing the curvature variation provided by the effect of the missing intrinsic stress.

This approach has been implemented only for the Gate Oxide Deposition, considering the simulation results obtained with homogenization by the RVE 3 - the one giving the best results between the four tested - and the squared footprint for the macro-model, as reported in Figure 3.9. Instead of considering the auxiliary flat wafers for the decoupling of the stress, to implement this method, we directly consider the difference between the curvatures of the wafer obtained with this simulation and those evaluated experimentally. This difference, which is considerable as the error between the simulation and the real case, is theoretically given by the missing contribution of the intrinsic stress: this hypothesis is considered valid thanks to the results of the DTM after the introduction of the intrinsic stress, which are in target with experimental values. The approach thus develops following the process reported below:

1. Using RVE 3, the deformation of the patterned wafer in the Gate Oxide Deposition step is simulated using HTM (the result is reported in Figure 3.9). The Δk along the X and Y profiles of the wafer is measured between such results and the experimental data. The values obtained are $\Delta k_X = 0.003733 \text{ m}^{-1}$ and $\Delta k_Y = -0.004266 \text{ m}^{-1}$.

2. The HTM simulation is repeated, imposing the stress-free condition. This is obtained setting each of the i components of the CTE vector (α_x , α_y and α_z) of the homogenized layer equal to the isotropic CTE value of crystalline silicon (α_{Si}).
3. A parametric study of the same HTM simulation is conducted by varying each i component of the CTE of the homogenized layer while holding the other two fixed (thus equal to α_{Si}). For each variation, the resulting curvature of the wafer-scale model along the X and Y profiles is computed as a function of the difference between the varying α_i and α_{Si} (a deviation from the stress-free condition). These results are reported in Figure 3.15.
4. In Figure 3.15 the linear relations between the curvatures and the three values α_i are studied. Although the wafer curvatures vary with the varying components α_x and α_y , the component α_z (the out-of-plane component) is confirmed not to be relevant to the wafer curvature along both profiles. The slopes of the curves obtained are reported in Table 3.6.

Table 3.6: Values of the coefficients A to F [$^{\circ}\text{C}/\text{m}$] determined by the linear best fit of the curves reported in Figure 3.16.

Profile	Coeff.	Value
Profile X	A	-153.7
	B	+2535
	C	~ 0
Profile Y	D	+2693
	E	-111.5
	F	~ 0

From this investigation, we can conclude that the curvatures of the simulated wafer have a linear dependence on the quantities $\alpha_x - \alpha_{Si}$, $\alpha_y - \alpha_{Si}$, and $\alpha_z - \alpha_{Si}$, which can be written as:

$$\begin{cases} K_X = A(\alpha_X - \alpha_{Si}) + B(\alpha_Y - \alpha_{Si}) + C(\alpha_Z - \alpha_{Si}) \\ K_Y = D(\alpha_X - \alpha_{Si}) + E(\alpha_Y - \alpha_{Si}) + F(\alpha_Z - \alpha_{Si}) \end{cases} \quad (3.3)$$

Knowing the coefficients A to F , and the values of Δk_X and Δk_Y calculated in point 1, such relations are exploited to calculate the related $\Delta\alpha_x$ and $\Delta\alpha_y$, that is, the variation that must be applied to the X and Y components of the CTE originally used for silicon oxide to obtain bow results in line with the experimental ones. The values obtained are $\Delta\alpha_x = -3.1 \times 10^{-5}$ and $\Delta\alpha_y = 0.83 \times 10^{-5}$.

5. Adding the two $\Delta\alpha_x$ and $\Delta\alpha_y$ to the initially considered isotropic CTE of silicon oxide, the final CTE_{eff} is calculated. The last HTM simulation is performed imposing the newly obtained CTE_{eff} on the homogenized layer to check the results, which gives bow values along the two X and Y profiles equal to +79 μm and -108 μm , respectively, showing an error of -8% and +4% with respect to the mean experimental target (reported in Table 3.1), as reported in Figure 3.16.

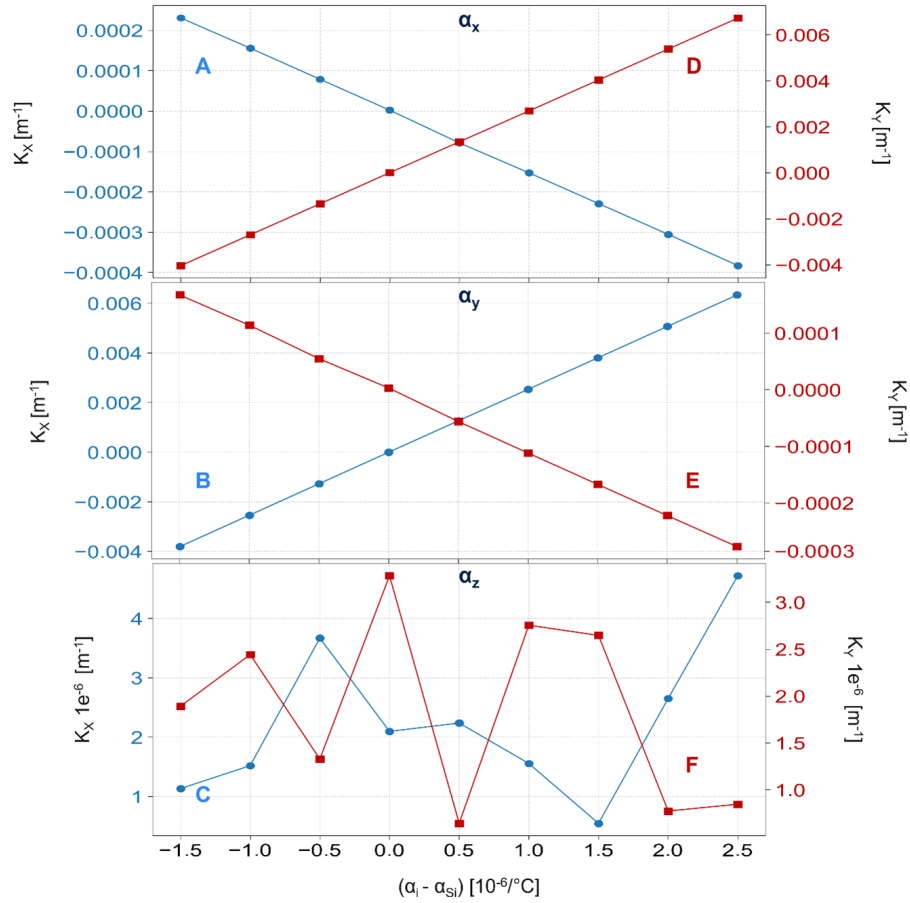


Figure 3.15: curvatures κ_x and κ_y along x and y wafer profiles reported in blue on the left and red on the right, as function of the delta between the components of homogenized layer CTE and the isotropic CTE of silicon ($\alpha_i - \alpha_{Si}$). The pedis i indicates the three components of the homogenized layer CTE, along x y and z directions. The results indicates a linear relationship between the wafer curvature on both profiles with the components x and y of the CTE (in-plane components), while the component z have no impact on the curvatures (resulting curves are related to the noise of the simulation result).

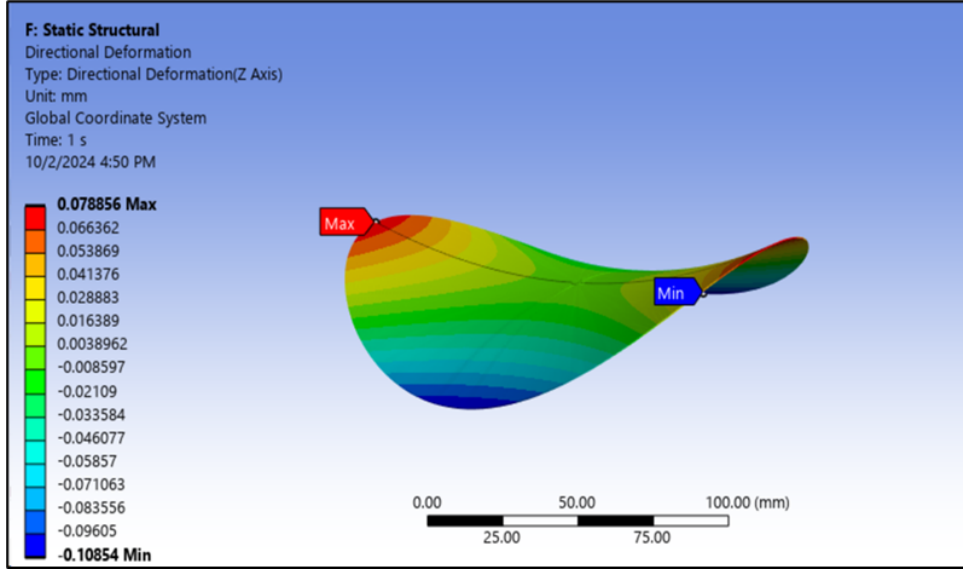


Figure 3.16: the HTM simulation made with RVE 3 gives a resulting bow along the X and Y profiles equal to $+79 \mu\text{m}$ and $-108 \mu\text{m}$, respectively, showing an error of -8% and $+4\%$ respect to the mean experimental target (reported in Table 3.1). This result confirm the effectiveness of this approach in transferring the effect of an external stress inside a homogenized model through the calculation of an effective CTE.

This approach preserves the computational advantages of homogenization while addressing the critical need to include intrinsic stress effects, ensuring that the HTM remains a viable and accurate tool for predictive warpage modeling in trench-patterned IGBT wafers. However, it lacks the predictive function of the simulation approach and should be used only to transfer the effect on an external intrinsic stress within a homogenized model.

3.5.5. Further Developments

To address the limitations observed in modeling the trench closure effect during the polysilicon C deposition step, several possibilities for further developments can be considered as potential corrective factors, such as obtaining experimentally evaluated CTEs, introducing finer meshes or considering the influence of bifurcation phenomena [154]. However, such developments are unlikely to mitigate the missing contribution of the total trench closure.

Beyond these corrective factors, more fundamental improvements to the modeling framework are necessary to capture the complex physics at play during such a production step. One promising direction is the incorporation of contact mechanics between the opposite

sidewalls of each trench. COMSOL Multiphysics provides dedicated contact nodes within its physics interfaces that allow simulation of interactions such as sliding, adhesion, and separation, which could significantly influence the stress distribution and deformation while the trenches are completely closed by the polysilicon. Moreover, it would be interesting to test the HTM also on the process steps following the Gate-Oxide Deposition, as they could reveal further information regarding the issue of trench closure.

Another important enhancement would be the transition from static, time-independent simulations to fully time-dependent analyzes that simulate the actual growth process of the polysilicon C layer. Modeling the kinetics of deposition and stress evolution dynamically could provide a more realistic representation of the intrinsic stress development and its impact on warpage, potentially resolving the discontinuities seen in static models.

Furthermore, to consolidate and generalize the developed models, it is advisable to apply the Discrete Trenches Method (DTM) to a wider range of structures featuring different pattern geometries, dimensions, and material compositions. Such parametric studies would validate the robustness of the modeling approach in various device configurations and identify key parameters influencing the behavior of warpage. This systematic exploration will enhance the predictive power of the models and support their application in optimizing manufacturing processes for next-generation trench IGBTs and related semiconductor devices.

3.6. Summary

This chapter has addressed the modeling of wafer warpage in trench-patterned IGBT devices during critical production steps, following a systematic approach grounded in experimental validation and advanced simulation techniques.

In Section 3.1 key experimental targets were defined. Warpage was quantified primarily through wafer bow measurements along two principal directions - perpendicular (X) and parallel (Y) to the trenches - at the three main process steps: Gate Oxide Deposition, Trench Poly-Filling, and Body Annealing. Auxiliary flat wafers, deposited with identical film stacks but without trench patterning, were also produced and characterized to support the evaluation of mechanical properties and residual stress decoupling.

Building upon these experimental inputs, Section 3.2 introduced a dual finite element modeling strategy designed to overcome the multiscale challenge inherent to simulating a 300 mm wafer patterned with nanoscale trenches. The Discrete Trenches Method (DTM) explicitly models a representative small portion of the wafer with detailed trench geom-

etry, capturing local stress distributions and anisotropic deformation. The Homogenized Trenches Method (HTM), on the other hand, replaces the complex trench pattern with an equivalent orthotropic homogeneous layer derived from Representative Volume Element (RVE) analyses, enabling full-wafer simulations that incorporate realistic device footprints and wafer shapes.

In Section 3.3, the crucial role of intrinsic residual stresses was highlighted. These stresses, initially neglected, were isolated through a stress decoupling procedure using the auxiliary flat wafers and incorporated into the DTM simulations as planar in-plane stresses. For HTM, the effects of intrinsic stress were introduced indirectly via an effective thermal expansion coefficient (CTE_{eff}), allowing the homogenized model to approximate the influence of intrinsic stresses on the curvature of the wafer.

The simulation results presented in Section 3.4 demonstrate that both models, once corrected for intrinsic stresses, achieve good agreement with experimental wafer bow measurements for the gate oxide deposition and subsequent process steps, with the exception of the final polysilicon C deposition step corresponding to trench closure. This step revealed significant deviations, indicating limitations in current modeling approaches and motivating further development.

It should be noted that the models developed in this chapter were specifically created for a pattern of aligned trenches characteristic of the studied IGBT devices. Naturally, different trench geometries or device architectures require the development of suitably adapted models. Nevertheless, the general methodology and modeling framework presented are sufficiently flexible to be applied to a wide variety of trench patterns and semiconductor device designs, making this approach broadly applicable within the field.

The overall modeling strategy combines the use of auxiliary flat wafers for preliminary mechanical characterization and residual stress evaluation, the DTM for detailed and accurate simulation of warpage in patterned structures, and the HTM for wafer-scale simulations that incorporate macro-scale factors such as device footprint and wafer geometry. This framework enables predictive studies of several types of variation in the design of devices. The simulation can help to verify the impact on warpage of different trench depth or width, varying deposited film thicknesses, or number of subsequent layer deposition, for example evaluating the impact with only two layers of polysilicon instead of three. By simulating these variations, the models can guide process optimization efforts, reducing experimental trial-and-error, shortening development time, and lowering costs associated with mitigating warpage effects in semiconductor manufacturing.

Key Takeaways

- The dual modeling approach effectively balances detailed local analysis with computational efficiency at the wafer scale.
- Incorporation of intrinsic stress is essential for accurate warpage prediction and is achieved directly in the DTM and indirectly in the HTM via effective CTE.
- Thanks to the stress decoupling approach the models reliably predict warpage for most process steps after the introduction of the residual stress contribution, but trench closure remains a challenge requiring further research. Quantitative results are reported in Table 3.7.
- Sensitivity analyzes highlight the dominant role of mechanical properties and trench geometry in anisotropic warpage behavior.
- The methodology is adaptable to various trench designs and supports predictive process optimization to improve manufacturing yield and device reliability.

Table 3.7: Comparison of the average bow values along X and Y profiles for the four process steps of interest between simulation and experimental values. Simulation results in green or red indicate a value between or outside acceptable targets for the simulations results (reported in Table 3.1), respectively.

Step of Production	Profile X (Across Trenches) [μm]	Profile Y (Along Trenches) [μm]
Gate Oxide Deposition	+54/+72	-118/-123
Trench Poly-Filling (Intermediate)	-16/-18	+83/+65
Trench Poly-Filling (Complete)	-108/+122	+6/+20
Body Annealing	-309/-284	-119/-141

4 | Composition-Dependent Mechanical Behavior of Silicon Nitride Thin Films

Silicon nitride (SiN_x) thin films deposited via Plasma Enhanced Chemical Vapor Deposition (PECVD) are widely used in semiconductor device fabrication as passivation and dielectric layers due to their exceptional combination of chemical, electrical, thermal, and mechanical properties [69, 155]. These films are often deposited as one of the most external layers to provide protection to the underlying device structures by serving as physical barriers against environmental contaminants and mechanical damage, thus enhancing device reliability and operational lifetime.

Despite these advantageous characteristics, a fundamental challenge associated with these films is their inherent brittleness, manifested as a high susceptibility to cracking and mechanical failure under the mechanical and thermal stresses commonly encountered during semiconductor manufacturing processes and device operation [72, 156]. Cracking in the passivation layers can lead to compromised electrical isolation, increased leakage currents, and accelerated device degradation, ultimately reducing the yield and reliability of semiconductor products. Consequently, understanding and improving the mechanical robustness of SiN_x films is of paramount importance for the semiconductor industry.

In this study, I investigated how the mechanical properties of sub-micrometric SiN_x thin films change with varying their chemical composition. In particular, the silicon-to-nitrogen (Si/N) atomic ratio has been varied in 3 different compositions, starting from a quasi-stoichiometric one and increasing the silicon content, obtaining the so-called silicon-rich SiN_x films. Such films compositions have been obtained by changing the ratio between the two precursor gasses during the PECVD procedure, namely silane (SiH_4) and ammonia (NH_3). In the literature, it is known that increasing the silicon content of SiN_x films during PECVD leads to the spontaneous formation of inhomogeneities of amorphous silicon (a-Si) dispersed through the SiN matrix $_x$ [70, 157]. Such aggregates could affect film

mechanical compliance, intrinsic stress evolution, and fracture behavior; however, research on this phenomenon has so far focused mainly on the effects related to optical properties [158–160]. Moreover, accurate characterization of fracture toughness in sub-micrometric SiN_x films requires advanced experimental techniques that account for substrate effects and the presence of residual stresses, which made these types of characterization technically complex and still not widely spread in the literature.

The present chapter builds upon these considerations by investigating the composition-dependent mechanical properties of SiN_x films through an alternative indentation-based method developed by Xia and his group [75]. In Section 4.1 an overview of classical toughness evaluation methods based on indentation is reported, with a focus on the methodology developed by Xia et al. used in this research. Sections 4.2 and 4.3 report the experimental procedures followed in this investigation and the results obtained, respectively, while Section 4.4 is dedicated to the discussion of such results. Finally, Section 4.5 reports a summary of the chapter and a condensed list of key takeaways.

4.1. Toughness Evaluation in Thin Films

This section reports a basic description of fracture toughness and proposes a short review of classical evaluation methods based on indentation. The second part of the section focuses on the methodology developed by Xia et al., which has been used in this study to evaluate the toughness of three SiN_x sub-micrometric films with varying stoichiometry.

4.1.1. Fundamentals of Fracture Toughness

Fracture toughness is a fundamental material property that quantifies the resistance of a brittle material to crack propagation under applied loading. It is a critical parameter in fracture mechanics and describes the ability of materials such as ceramics, glasses, and certain composites to resist catastrophic failure in the presence of cracks or flaws [161].

In the most general approach, the fracture process is characterized by three different modes of crack loading: mode I (opening), mode II (sliding), and mode III (tearing), i.e., the types of load that promote the propagation of a crack within a material. For each mode of loading, it is possible to define a stress intensity factor K that characterizes the stress concentration near the tip of the propagating crack. This value is expressed in units of pressure multiplied by the square root of length, generally described in $MPa\sqrt{m}$, reflecting the fact that the stress near the crack tip depends both on the magnitude of the applied stress and on the size of the crack itself. The critical stress intensity factor,

K_c , defines the threshold at which crack propagation becomes unstable and then evolves uncontrollably, reaching the material fracture in a very short time. This critical value is what is generally sought through experimental analysis, and it is associated with a material's resistance to crack propagation, also defining the general distinction between ductile and brittle materials.

For Mode I loading, this critical value is K_{Ic} , while for mode II and mode III these values are denoted as K_{IIc} and K_{IIIc} , respectively. Within the field of linear elastic fracture mechanics (LEFM) - a well-established theoretical foundation for the fracture of brittle materials, which assumes a sharp crack in an elastic body with a small-scale inelastic zone at the crack tip - Mode II and III are commonly considered non-influential, since the critical stress intensity factor of Mode I is sensibly lower and thus assumes greater practical relevance for engineering design [72]. A schematic representation of the three modes of crack loadings and a crack tip in the LEFM model is reported in Figure 4.1.

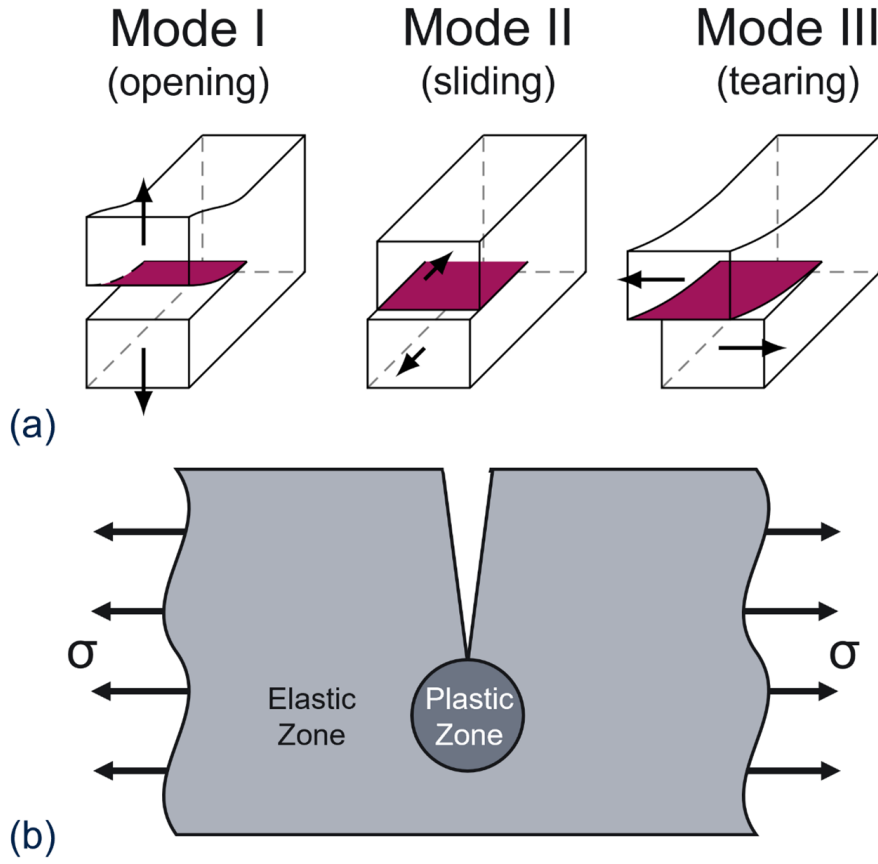


Figure 4.1: Mode I, II and III (from left to right) of crack opening (a), and a classical representation for the LEFM model featuring a crack, with a plastic zone considered on its tip front, propagating through a wide elastic plate subject to a remote tensile stress σ

Due to this classical approach, K_{Ic} is commonly considered the unique critical value when evaluating brittle failure and the same approach is used in this research, since the impact of the Mode II and Mode III crack loads is negligible for classical uniaxial indentation. For this reason, from now on, in this thesis the K_{Ic} value will be generally referred to as the fracture toughness of the material (as often happens in other studies, like also the one proposed by Xia et al.).

Brittle materials typically exhibit low K_{Ic} values, often ranging from 0.5 to 5 MPa $\sqrt{m}$, making them highly sensitive to relatively small flaws that can significantly reduce their load-bearing capacity. Moreover, fracture toughness can also be related to the amount of energy required to extend the total area of the crack, known as the critical energy release

rate G , defined as:

$$G = \frac{K_{Ic}^2}{E_r} \quad (4.1)$$

Where G is measured in J/m^2 , K_{Ic} in $\text{Pa}\sqrt{\text{m}}$, and E_r is the reduced elastic modulus of the film, measured in Pa. It is immediate to notice that, knowing the reduced modulus of a film, it is possible to evaluate the energy release rate for Mode I directly from the Stress Intensity Factor K_{Ic} . The parameter G quantifies the energy per unit area needed to propagate a crack and is closely related to the surface energy of the material and the mechanisms of dissipation of the microstructural energy.

Experimentally, fracture toughness is determined using standardized test methods and specimen geometries, including Single Edge Notch Bend (SENB), Compact Tension (CT), Double Cantilever Beam (DCB), Double Torsion (DT), and Chevron Notch specimens [73, 74, 161–163]. These tests involve measuring the critical load or displacement at which crack initiation occurs, which is then used to calculate K_{Ic} through shape factors that account for crack size and specimen dimensions. However, while these methods provide improved accuracy by isolating the film's fracture response, they often require complex sample preparation and sophisticated finite element modeling to interpret results accurately.

Plane Indentation-based methods have also been developed to estimate fracture toughness by analyzing cracks induced by hardness indenters. The most common approach in this field is the Lawn–Evans–Marshall (LEM) approach, based on the empirical correlation between indentation load, induced crack length, and material hardness, which relates fracture toughness to the size of the cracks (in the case of radial cracks) formed around the indentation site [90, 164]. The general LEM equation for a Berkovich tip is:

$$K_{Ic} = \alpha \left(\frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} \quad (4.2)$$

Where α is a correction factor related to the shape of the crack, E and H are the elastic modulus and the hardness of the film, respectively, P is the maximum load applied in the indenter and c is the measured crack length.

However, as described in Chapter 2, accurate measurement of fracture toughness in sub-micrometric films is challenging due to the strong influence of the underlying substrate, which can cause cracks to propagate into the bulk silicon wafer, thereby confounding the interpretation of the results. Moreover, another impacting factor on the effective toughness of SiN_x films is related to the residual stresses which naturally develop dur-

ing PECVD, due to factors such as thermal expansion mismatches, plasma-induced ion bombardment, and chemical gradients within the growing film [71, 165]. These residual stresses can be tensile or compressive depending on the deposition parameters and film composition, influencing crack initiation and propagation. Tensile residual stresses tend to reduce fracture toughness by facilitating crack opening, whereas compressive stresses can enhance toughness by impeding crack growth [71].

To address these challenges, we have decided in this study to employ an alternative indentation-based method proposed by Xia et al. which offers a practical and relatively straightforward approach to assess the fracture toughness of thin films by analyzing indentation-induced cracking in patterned samples [75]. This method has never been used before in STMicronics laboratories; therefore, its use has also been of procedural interest to understand its effective reliability for industrial applications.

4.1.2. The Xia Method

Zhenhai Xia et al. developed an innovative method to evaluate the fracture toughness, K_{Ic} , of thin films bonded to brittle substrates, addressing key limitations of traditional indentation-based toughness measurements on coatings. Conventional methods often suffer from substrate effects, interface delamination, and residual stresses, which complicate the interpretation of indentation cracks formed directly on coated surfaces. Xia's approach circumvents these problems by testing a film only partially coated, as represented schematically in Figure 4.2.

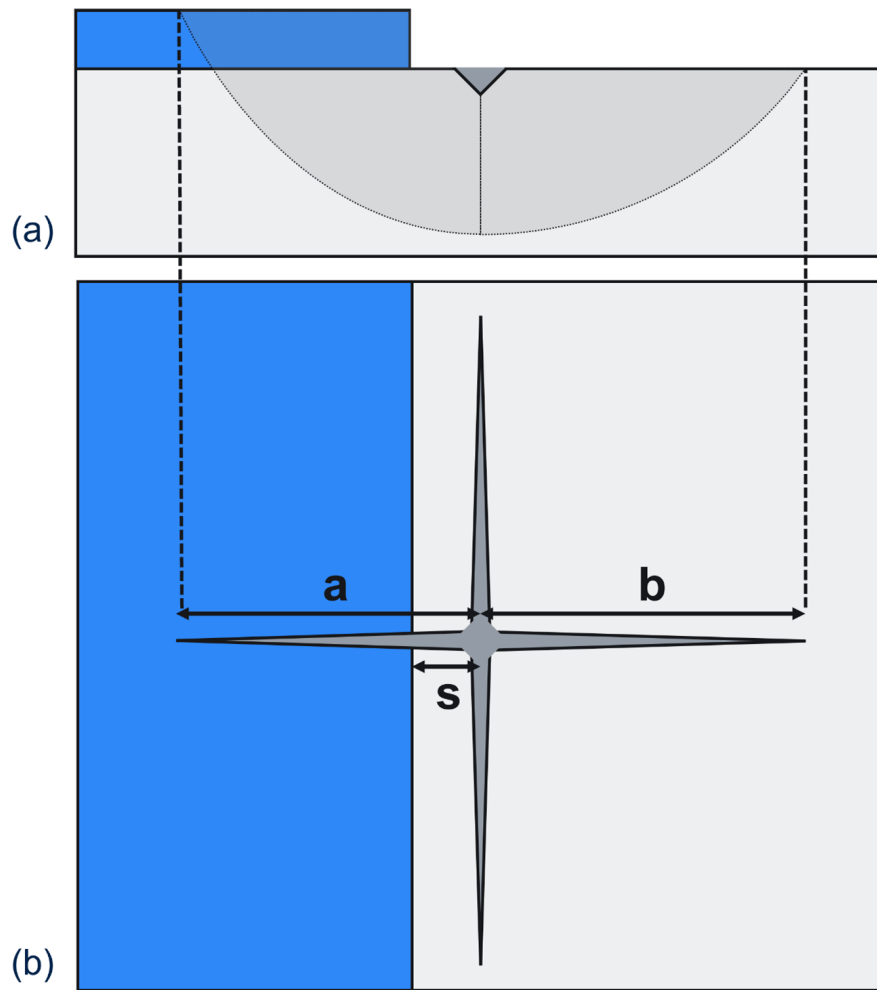


Figure 4.2: schematic representation of the pattern and indentation geometry in cross section (a) and front view (b) employed for the application of the model proposed by Xia et.al. The film coating is represented in blue and the substrate in light gray. The Vickers tip indentation, performed at a distant “ s ” from the coating edge, leads to the propagation of four cracks along a cross shape. In this study we consider the two cracks normal to the film edge, designated as “ a ” for the one across the coating and “ b ” for the one across the free substrate.

This method was specifically developed to minimize substrate-related effects by avoiding direct indentation of the film, thereby minimizing delamination and allowing a clearer assessment of the coating’s fracture toughness through the comparison of the crack growth behavior in the two regions. Xia et al. developed their approach following four main steps: an initial experimental observation, the development of FEM Analysis to study the previous results, the development of an analytical expression, and the validation of such

expression.

Experimental Observation: The experimental samples consisted of silicon wafers partially coated with polycrystalline chemical vapor deposited (CVD) diamond films. The partial coating is achieved by masking the substrate during deposition, creating a sharp interface between the coated and uncoated areas. Indentation is conducted using a Vickers indenter on the uncoated silicon substrate in a position with a distance s from the edge of the film coating. During indentation, one of the radial cracks starts in the substrate and propagates normally toward the film and upward through its thickness, thus generating a single, clearly defined through-thickness crack with length a . In the opposite direction, another crack will propagate along the free substrate with length b . This difference arises because the coating, typically having a higher fracture toughness, resists crack growth more effectively than the substrate. Thus, the ratio and absolute values of these crack lengths could provide a measurable signature of the resistance to fracture of the coating relative to the substrate.

Development of FEM Analysis: To quantitatively interpret experimental crack length data, Xia et al. developed two different 3D finite element models that closely mimic the experimental sample geometry and loading conditions: a 45° wedge model representing a square patch of uncoated substrate surrounded by coating and a half-infinite plate model representing the experimental half-coated sample (as reported in Figure 4.2). The models incorporated the elastic properties and residual stresses of both the film and the substrate, and also the fracture toughness values of the two materials, set as a fictitious value for the polycrystalline CVD diamond film. A cohesive zone model was implemented along the crack plane to simulate crack initiation and stable propagation realistically. This model defines a traction-separation law characterized by maximum tensile strength and critical opening displacement, allowing the crack front shape and growth to evolve naturally under different applied loads, which was tested during multiple simulations. The FEM results revealed that the crack length in the coating a increases nearly linearly with the crack length on the substrate b with increasing applied load of the indenter. The models also showed the significant effect of residual tensile stresses in the coating, which increase the crack length a for a given b , effectively reducing the apparent toughness, while compressive stresses have the opposite effect.

Development of an Analytical Expression: Inspired by the FEM results, Xia et al. formulated an approximate analytical model grounded in an energy balance argument. The key premise is that the total mechanical work done by the indenter to create cracks

in the half-coated sample would be equal if the coating were made of a different material or the same material of the substrate. Since the total work performed by the indenter is definable through the energy release rate G multiplied by the opened crack areas, this premise allows us to write the following equation:

$$A_c G_c + A_s G_s = A_{sc} G_s + A_{ss} G_s \quad (4.3)$$

where G_c and G_s are the energy release rates for the coating and substrate, A_c and A_s are the cracked coating and substrate areas in the real sample, and A_{sc} and A_{ss} are the cracked areas when the coating properties are hypothesized to be identical to those of the substrate.

Considering Equation 4.1 it is possible to write this equation in function of the coating fracture toughness $K_{Ic}(c)$:

$$K_{Ic}(c) = K_{Ic}(s) \sqrt{\frac{E_r(c)}{E_r(s)} \frac{A_{ss} - A_s + A_{sc}}{A_c}} \quad (4.4)$$

Where $K_{Ic}(s)$ is the fracture toughness of the substrate, and $E_r(c)$ and $E_r(s)$ are the reduced elastic modulus of the coating and the substrate, respectively. Continuing, it is possible to estimate the areas $A_c = t(a - s)$ and $A_{sc} = t(b - s)$, where t is the thickness of the coating, but the areas A_s and A_{ss} involving subsurface cracking are unknown functions of a , b , t , and s . However, from the analysis of the results of the finite element, Xia et al. founded a relation between the quantity $[(A_{ss} - A_s + A_{sc})/A_c]^{0.5}$ and $[(\phi b - a)/t + B]$, where ϕ is a dimensionless factor and B is a constant. Introducing a proportionality constant λ , Equation 4.4 is rewritten as:

$$K_{Ic}(c) = K_{Ic}(s) \left[1 + \lambda \frac{\phi b - a}{t} \sqrt{\frac{E_r(c)}{E_r(s)}} \right] \quad (4.5)$$

Moreover, Xia et al. inserted in the key premise the possible effect of residual stresses by rewriting Equation 4.3 in:

$$(A_c G_c + A_s G_s) - (A_{sc} G_s + A_{ss} G_s) = \pm G_{rc} A_c \quad (4.6)$$

where G_{rc} is the energy stored due to residual stresses in the coating, estimated through a previous work developed by Lawn and Fuller [166], and the signs $+$ and $-$ refer to

residual tensile and compressive stresses, respectively. Following a similar approach, Xia et al. conclude with the final analytical expression defined as:

$$K_{Ic}(c) = \left\{ \left[K_{Ic}(s) \left(1 + \lambda \frac{\phi b - a}{t} \sqrt{\frac{E_r(c)}{E_r(s)}} \right) \right]^2 \pm \left[2\Psi\sigma_r\sqrt{t} \sqrt{\frac{E_r(c)}{E_r(s)}} \right]^2 \right\}^{1/2} \quad (4.7)$$

where λ and Ψ are two dimensionless material-independent factors equal to 0.45 and 0.95, respectively - obtained from the FEM calculations - and ϕ is the slope of the adjustment obtained by plotting a vs b measured with increasing loads of the indentation, and therefore can be measured experimentally without implementing any FEM. This expression effectively extends the classical indentation load–crack length relationships used for bulk materials to coating/substrate systems, incorporating the influence of residual stresses and elastic mismatch. The analytical formula provides a practical and efficient means to estimate coating toughness directly from indentation crack length measurements, without the need for any finite element simulations, and can then be applied to any system of brittle film deposited on brittle bulk substrate with the right patterning.

Validation: The equation obtained was finally applied to the polycrystalline CVD diamond films analyzed at the beginning. The lengths of the indentation cracks a and b were measured at various loads, and the residual stresses were measured independently (~ 1 GPa biaxial tensile stress in the diamond film). Using the analytical model the coating fracture toughness was determined to be approximately $8.4 \text{ MPa}\sqrt{m}$, with an uncertainty of $\pm 0.5 \text{ MPa}\sqrt{m}$ and strong consistency with previous measurements on polycrystalline CVD diamond. The analytical expression was then validated against a wide range of finite element simulations covering various coating and substrate properties (e.g., SiO_2 and PZT films on silicon), thicknesses and residual stress states. In all cases, the analytical predictions closely matched the numerical results, confirming the robustness and generality of the model.

In general, Xia’s method offers a robust and experimentally accessible technique to measure the stiffness of the fracture toughness of thin films, overcoming the challenges posed by substrate interactions and delamination by (i) avoiding the complications of direct indentation in the coatings, such as delamination and substrate influence; (ii) using a simple, easily fabricated partially coated sample geometry; (iii) incorporating the effects of residual stresses; and (iv) providing a practical approach applicable to a wide range of coating/substrate combinations.

To resume, in order to apply the method in a real experimental environment, it is necessary

to satisfy the following requirements:

- Fabricate a sample with a step-like pattern between the coating and the substrate (as reported schematically in Figure 4.1).
- Perform a sufficient number of indentations at varying loads to obtain statistically solid results.
- Measure each obtained crack length a and b .
- Plot the function a versus b to evaluate the slope ϕ or directly the intercept ($\phi b - a$).
- Evaluate the reduced modulus and intrinsic stress of the film.

After completing the steps described above, it will be possible to apply Equation 4.7 to evaluate the fracture toughness of the film under analysis.

4.2. Experimental Procedures

In this section, the procedure for obtaining the final experimental samples with adequate patterning is described for the application of the method proposed by Xia et al., followed by the procedure employed to perform all the structural and compositional analysis of this research.

4.2.1. Sample Preparation

Unlike the approach used by Xia et al., the time and tools available in the company did not allow for partial deposition of the film via pre-masking of the substrate. This process would have required an excessive effort and preparation time. It was therefore decided to opt for a planar deposition followed by a subsequent partial removal of the film.

In this study, three silicon nitride thin films with varying stoichiometry have been analyzed. All three 300 mm wafers (crystalline silicon [111]) were initially deposited with an identical 50 nm thick silicon nitride film obtained by exposing them to an ammonia atmosphere at 800°C. This preliminary “thermal deposition” is commonly adopted in STMicroelectronics to promote adhesion of following PECVD-grown SiN_x films, and is a standard practice that was impossible to bypass. In addition to these preliminary thin films, three different PECVD-grown SiN_x films with varying compositions were subsequently deposited with a thickness of 600 nm. The PECVD depositions were carried out at a substrate temperature of 380°C and a chamber pressure of 4.3 Torr, and all films were deposited with a nominal thickness of 600 nm. To study the effect of composition

on mechanical properties, the three films were deposited with varying stoichiometries by adjusting the gas flow ratios of silane (SiH_4) to ammonia (NH_3) while keeping the ammonia flux constant. The compositions included a quasi-stoichiometric film designed as Standard (St) with a $\text{SiH}_4:\text{NH}_3$ ratio of 3:4 and a nominal Si/N At% of 0.8, and two silicon-rich variants (SR4 and SR5) with ratios of 4:1 and 5:1 and nominal Si/N At% of 1.1 and 1.3, respectively. Following deposition, residual stresses in the films were evaluated by applying the Stoney Equation after wafer curvature measurement using optical profilometry with a PWG tool. Only after this measurement were all of the wafers treated to obtain the step-like patterning required to apply the method proposed by Xia et al.

Initially, various attempts were made to obtain an adequate patterning on samples of "test-size", i.e. portions of the full wafer with a dimension of 2×2 cm. This procedure was attempted to avoid the longer process required by physical etching procedures, which would have involved tools dedicated to industrial production and, thus, rarely free for engineering research. Moreover, optimizing a patterning procedure inside the STMicroelectronics laboratories, limiting the involvement of production tools, would have benefited the general toughness evaluation procedure, in the framework of validating the Xia methodology for a standardization inside the company.

For this reason, patterning was first attempted onto 2×2 cm squared samples of the St SiN_x film through two parallel strategies: the employment of a Ga-based Focused Ion Beam (FIB) milling system, or an ICP-based Plasma Etcher. The former technique was used to form $60 \times 60 \mu\text{m}$ hole patterns with a FIB HV of 30 kV and an etching time of 6 minutes. The latter was attempted using three variants of a fluorine-based recipe plasma for 1 minute on the half-covered sample with a photoresist tape. Both techniques, with all their variants, failed to produce a suitable pattern in different ways. A reduced set of images taken by SEM or optical microscopy during the characterization of the obtained patterning is reported in Figure 4.3.

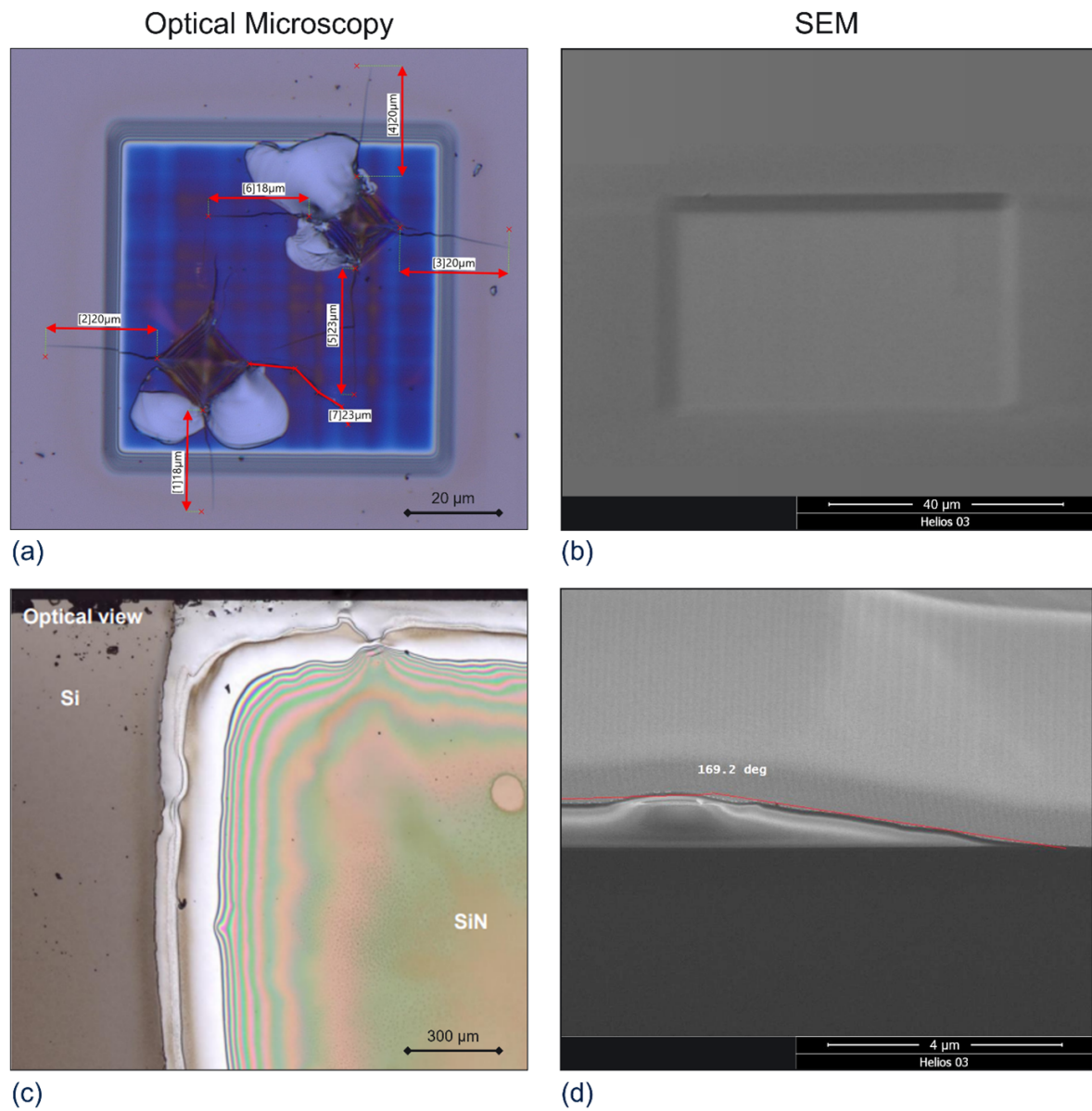


Figure 4.3: set of images taken after pattern tentative with FIB milling (a and b) and Plasma Etcher (c and d) obtained with optical microscopy (a and c) and SEM (b and d). While the formers are taken in front view, image (b) has been taken by the FIB camera with a tilted angle respect to the film plane, while Figure (d) was taken in cross section (obtained with prior FIB milling).

The most important parameters that were taken into consideration for the successful execution of the patterning are the tapering of the vertical wall - which should ideally be at 90 degrees relative to the exposed base of the substrate - and the absence of strong under or overetching, meaning an incomplete removal of the film or an undesired removal

of the underlying substrate, respectively.

The pattern obtained from the FIB milling (Figure 4.3 a-b) was not further analyzed in its cross section due to the time required to obtain it relative to its size. In fact, it was at that moment that we realized the actual number of indentations that would have been necessary to complete the analysis on each sample. Each obtained box could accommodate at most one or two indentations depending on the applied load, and this would have meant several full work days of preparation via FIB milling, a prospect that would have conflicted with the previously stated intention of creating a more streamlined approach to physical etching in the production line, especially given the high need for FIB milling preparations for a wide variety of characterizations requested from laboratories by other groups within the Company.

The pattern obtained through plasma etcher (Figure 4.3 c-d) instead shows clear signs of extremely high tapering and strong over-etching effects (the variants not shown display similar or even worse cases). These issues are likely caused by a combination of two factors: the poor selectivity of the plasma - an unavoidable factor given the strong similarity between silicon nitride and silicon - and the low efficiency of the mask produced with a photo-resistive tape. This approach is inherently less effective than a deposited photoresist mask as in classical photolithographic processes, but it is also much faster and often sufficiently adequate in cases of more selective plasmas. Unfortunately, in this case, it was not possible to employ this method, and therefore the preparation through photolithography in the production line was the only feasible option.

This option proved feasible also, thanks to the identification of a photolithographic mask suitable for the type of pattern required, that is, one that allows the formation of a sufficiently long step to enable a high number of indentations. The mask had originally been created for a particular process step of the IGBT, but it also turned out to be useful for this research work, saving the complexity of creating a custom lithographic mask. A front-view representation of the mask used is shown in Figure 4.4.

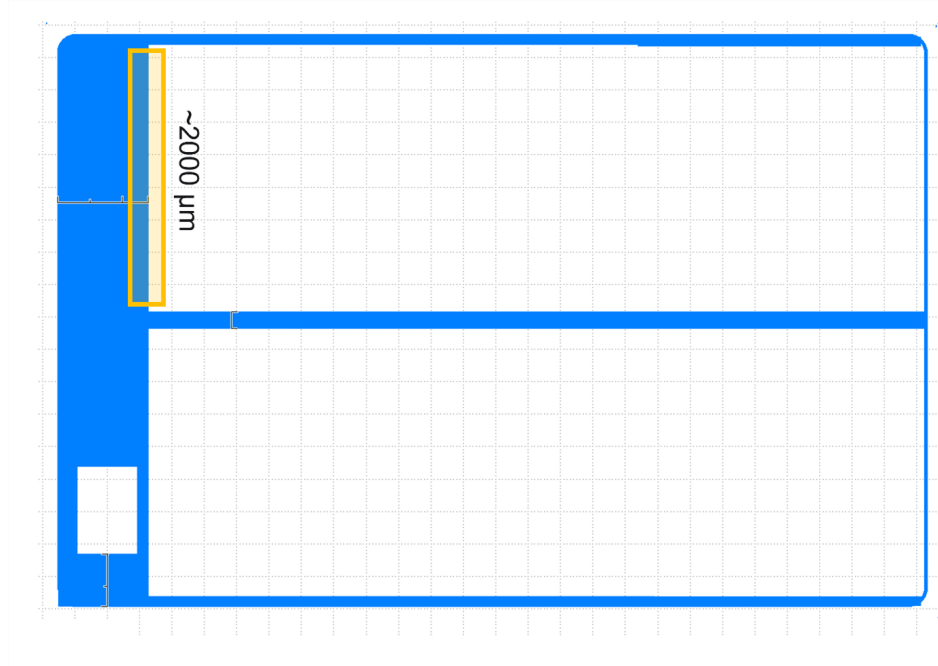


Figure 4.4: mask shape utilized for the wafer patterning (precise scales cannot be shared due to privacy reasons). The blue areas are subjected to SF_6 dry etching exposing the substrate, while the SiN_x coating remains in the white areas. The highlighted orange area is the step portion destined for indentations, featuring a length of $\sim 2000\ \mu m$.

As shown in Figure 4.4, the mask provides a feature of approximately 2 mm with a stepped morphology that lands on a sufficiently large area of exposed substrate. Furthermore, the entire wafer is patterned with the same mask across its surface, creating hundreds of these areas and thus allowing an extraordinarily high number of indentations sites. After a complex optimization procedure for the dry etch, performed with a plasma of sulfur hexafluoride (SF_6), all three types of SiN_x were patterned with acceptable results, shown in Figure 4.5.

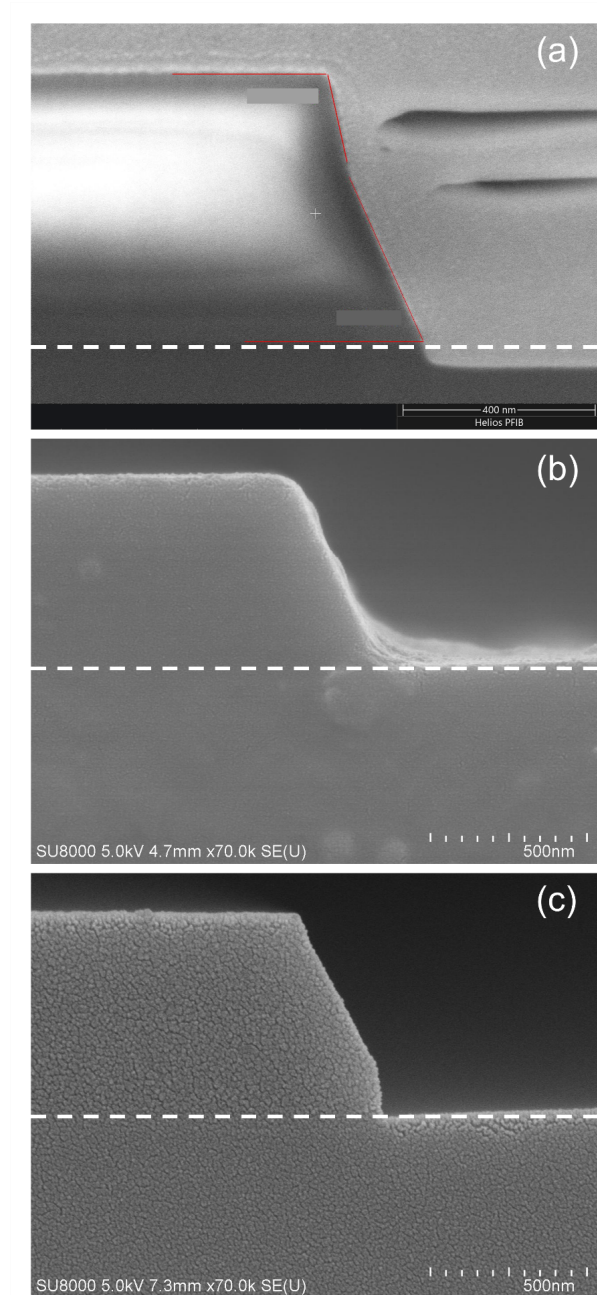


Figure 4.5: SEM cross-section after patterning through dry etching for the St (a), SR4 (b) and SR5 (c) SiN_x films. Unlike the sample synthesis method used by Xia et al. - a deposition preceded by photolithography to obtain a pattern with sharp edges - the approach used in this Work leads to the creation of less precise patterns, in favor of reduced preparation times and costs. In any case, the obtained slopes of the film sidewalls affects a portion of the film less than a micrometer, while the cracks obtained exceed several tens of micrometers. For this reason, the patterns were considered sufficiently adequate for the implementation of methodology.

The sample preparation process therefore takes place through the following steps without ever leaving the process line in a clean room:

- Deposition of SiN_x films on 300 mm crystalline silicon wafers.
- Measurement of curvature via PWG (assessment of residual stresses using the Stoney Equation).
- Patterning through dry etching using a suitable mask.

The wafers then arrive at the analysis laboratories ready to be characterized, either intact or after being sectioned into smaller samples. Despite the lower precision of the obtained patterns, which could lead to an increase in experimental error, the time and cost for sample preparation are extremely reduced compared to those of the synthesis method used by Xia et al., which involves the deposition of a negative pattern produced ad-hoc through photolithography to allow the deposition of the film of interest with a more ideal pattern. It should be noted that the samples obtained in this study still show relatively limited tapering phenomena, affecting portions of the film near the edge with dimensions smaller than a micrometer, while the cracks produced by the lower load used for film characterization will have dimensions greater than 20 μm . Similarly, the phenomenon of over-etching visible in the case of the standard sample SiN_x affects just over 20 nm, a minimal fraction of the total thickness of the film, and therefore was considered to have little impact on the final results.

In summary, the approach used for sample preparation involved a compromise between practical feasibility and accuracy of the results. Although a greater error in the toughness results is expected, the industrial environment in which this study was conducted required such compromises.

4.2.2. Structural and Compositional Characterization

In addition to the work performed for sample preparation, structural and compositional analyzes were carried out on the SiN_x films to clarify their chemical composition and microstructural characteristic. X-ray photoelectron spectroscopy (XPS) was performed using a PHI5000 Versaprobe III (ULVAC-PHI) with a photon energy of 280 eV, equipped with an argon sputtering gun to enable in-depth profiling up to 10 μm . This technique was utilized to accurately determine the film stoichiometry and confirm the intended nominal stoichiometry of the films.

Raman spectroscopy was employed to probe the structural characteristics of the silicon-rich films and verify the presence or absence of embedded silicon inclusions. Measurements

were conducted with a Renishaw InVia micro-Raman spectrometer, using an Ar^+ gas laser ($\lambda = 514 \text{ nm}$) and a 1800 grooves/mm grating. To prevent sample damage, the laser power was kept below 0.4 mW. The same instrument was used for photoluminescence (PL) spectroscopy, using a different laser wavelength ($\lambda = 457 \text{ nm}$) to investigate the optical properties of silicon-rich films, focusing on signals indicative of quantum confinement effects of possible embedded amorphous silicon nanoparticles (a-Si NPs) [158]. PL analysis was also performed after two different thermal treatments performed in the atmospheric environment at 425°C and 600°C , both for 1 hour of dwell time and a temperature ramp of $5^\circ\text{C} / \text{min}$. This analysis was performed on the two silicon rich samples to assess a potential effect on the NP distribution and average dimension through the monitoring of their PL signals.

To gain further insight into the nanoparticles that could be formed during deposition, silicon-rich films were thermally annealed at 1100°C in air for one hour. This treatment was specifically intended to crystallize the amorphous silicon nanoparticles that spontaneously formed within the silicon-rich SiN_x matrix during deposition. Crystallization was necessary to allow direct observation of the nanoparticles by transmission electron microscopy (TEM), as the amorphous nanoparticles could not be distinguished from the amorphous SiN_x matrix in their as-deposited state. TEM measurements were performed on electron-transparent lamellae using a Thermo Fisher Themis Z aberration-corrected scanning transmission electron microscope (STEM) operated at 300 kV. Dark-field TEM (DF-TEM) images were collected from the annealed samples for microstructural analysis and to determine crystal sizes, providing an independent validation and quantification of nanoparticle size and distribution consistent with prior Raman and PL findings. In addition, the images were taken in two areas, one near the substrate and the other near the surface of the SiN_x films, to observe possible gradients in the NP distributions and the average diameters along the thickness of the film. Finally, to avoid observing the oxidated thickness of the SiN_x films after annealing, estimated to be lower than 100 nm [167], the set of figures near the surface was taken under this most external thickness.

Finally, mechanical characterization was performed using nanoindentation techniques, employing a Triboscan TI300 nanoindenter. Hardness (H) and reduced elastic modulus (E_r) were determined with a Berkovich tip using the well-established Oliver & Pharr method [128] by restricting the indentation depths to a range of 8%-15% of the film thickness and exploiting a specific analysis software, called Intrinsic Thin Film [168], which implements a methodology by Li and Vlassak [169] that allows excluding the substrate effect for a thin mono-layer deposited on a bulk. The final values of hardness and elastic modulus utilized in this research are the result of averaging 12 subsequent indentation

for each tested sample. The Intrinsic Thin Film software was not suitable for toughness evaluation; thus, the method proposed by Xia et al. [75] was utilized for the accurate determination of the fracture toughness (K_c) using a Vickers tip. In this case, several trials were performed in order to obtain at least 30 clean indentations, i.e. without scaling effect (described in Section 4.3.3), for each of four different loads of indentation on each sample.

4.3. Results

4.3.1. Chemical Composition and Stoichiometry

XPS depth profiling was used to determine the chemical composition and stoichiometry of the SiN_x thin films deposited by PECVD. The analysis revealed consistent Si/N atomic ratios throughout the film thickness for all samples, except the presence of oxygen - at the surface and as small portions of inclusion inside the bulk - and a higher concentration of nitrogen near the interface with the substrate due to the initial 50 nm layer of grown silicon nitride placed for adhesion promotion, as mentioned in Section 4.2.1. Moreover, the results confirmed compositions almost the same as the nominal values intended for the two silicon-rich samples, while the St sample showed a slightly higher silicon content than the expected one. The results for the three samples are reported in Figure 4.6.

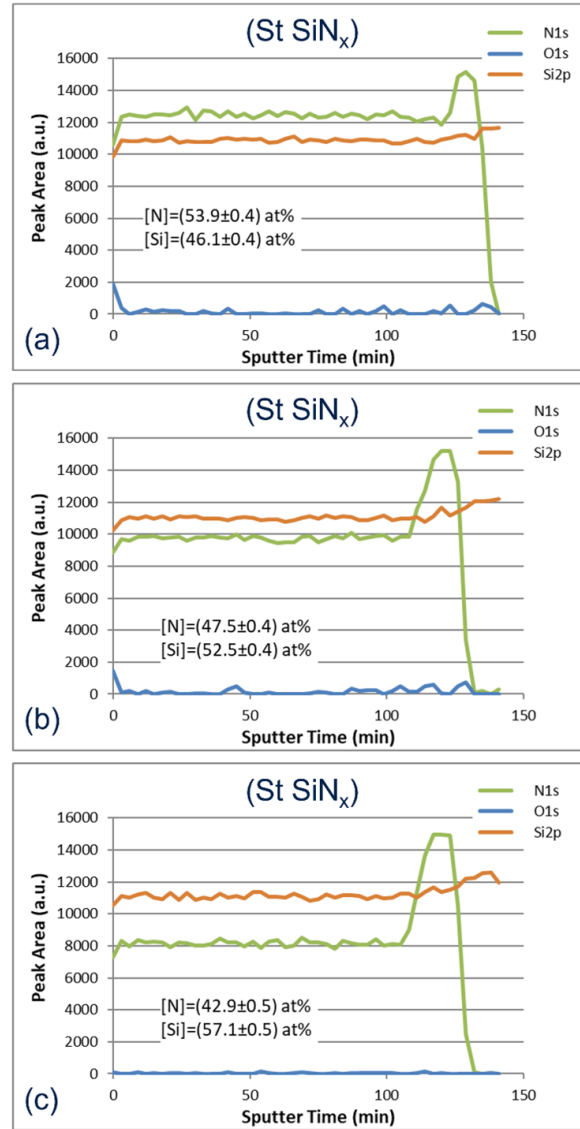


Figure 4.6: XPS depth-profiling results reporting atomic percentages of Si, N and O atomic content respect to the sputter time, i.e. the film thickness.

Specifically, the St sample exhibited a Si/N At% of 0.86 (with respect to nominal 0.80), while the two silicon-rich films (SR4 and SR5) showed increased ratios of 1.10 (equal to nominal) and 1.33 (with respect to nominal 1.30), respectively. No contamination or presence of elements other than silicon, nitrogen, or oxygen was detected, ensuring the purity of the films. A complete overview of the XPS results is reported in Figure 4.7.

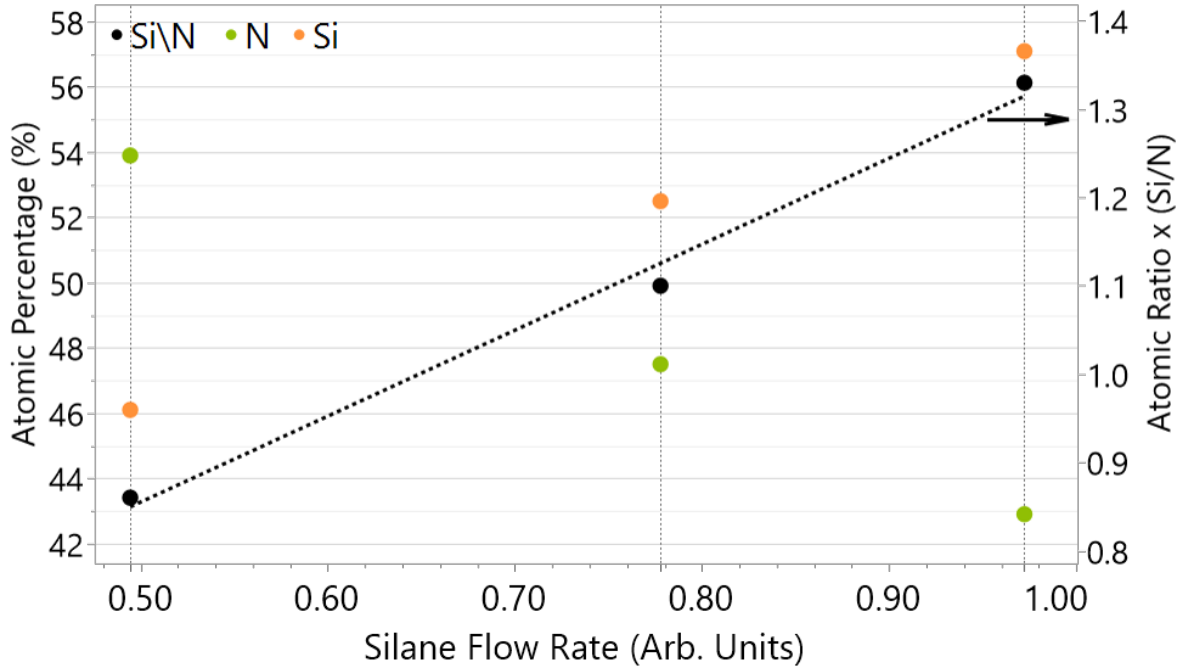


Figure 4.7: XPS results reporting atomic percentages of Si and N atomic content on the left and Si/N atomic ratio on the right. Results are reported for increasing silane flow rate during PECVD deposition considering a fixed ammonia flux. The dashed line is a guide to the eye to highlight the almost linear growth of the Si/N atomic ratio between the three samples.

These results validated the controlled tuning of the film stoichiometry, which is fundamental for investigating the influence of silicon content on structural and mechanical properties.

4.3.2. Structural Analysis

The structural characterization of the silicon nitride thin films was conducted through a combination of Raman spectroscopy, photoluminescence (PL) spectroscopy, thermal annealing, and transmission electron microscopy (TEM), providing a comprehensive understanding of the microstructural features and the presence of silicon inclusions within the films.

Raman spectroscopy revealed significant differences between the stoichiometric and silicon-rich films. The results are reported in Figure 4.8.

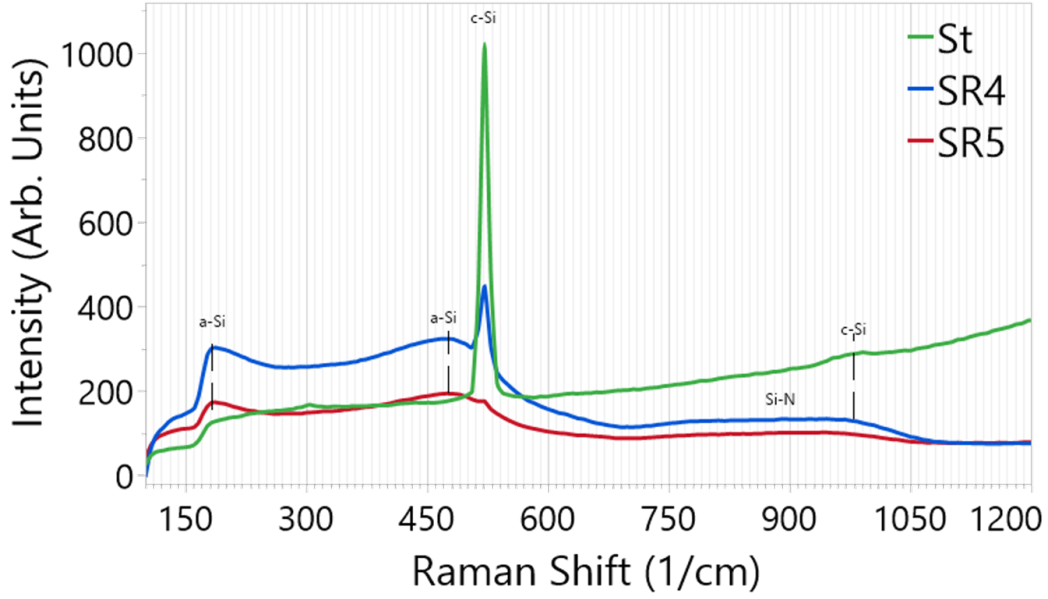


Figure 4.8: Raman spectra of St, SR4 and SR5 SiN_x samples. The two silicon rich samples shows bands related to amorphous silicon content does not present in the Standard sample. The strong peak related to crystalline silicon originating from the substrate and its decreasing intensity for the three samples indicates a lower transparency of the SiN_x films for increasing silicon content during PECVD deposition.

The St sample exhibited a strong and sharp peak at 521 cm^{-1} , corresponding to the crystalline silicon substrate beneath the film. The intensity of this peak served as an indirect indicator of the transparency of the film; the high intensity in the St sample suggested minimal absorption or scattering within the film, consistent with a homogeneous and transparent SiN_x matrix. In contrast, silicon-rich films (SR4 and SR5) showed a marked decrease in the intensity of this substrate peak with increasing silicon content, indicating growing optical absorption with the deviation from the stoichiometric composition, since all three films have the same thickness of 600 nm. The second order of crystalline silicon at 950 cm^{-1} is also visible for the St sample, whereas for the two silicon-rich samples it seems less intense and probably obscured by a band, ranging from 750 to 1000 cm^{-1} , related to the Si-N vibrational modes of silicon nitride [159, 170]. This band is not visible for the St sample which shows a rising tail of a photoluminescence broad band in that area, that for the other two samples begin for higher Raman shifts, as will be discussed later.

Further analysis of the Raman spectra of the SR4 and SR5 samples revealed broad bands centered around 470 cm^{-1} , which were absent in the St sample. These bands are attributed to the Si-Si stretching modes - centered at 465 cm^{-1} - and the transverse optical (TO)

phonon modes - centered at 480 cm^{-1} - characteristic of amorphous silicon [159, 160, 171]. An additional feature related to the presence of amorphous-silicon is the band near 180 cm^{-1} , further corroborating the presence of dispersed domains throughout the silicon nitride matrix [157, 158, 170]. The absence of such features in the stoichiometric film strongly suggests that the formation of these amorphous silicon inclusions is a direct consequence of the increased silicon content during PECVD deposition.

In addition to Raman analysis, photoluminescence spectroscopy provided further insights into the optical and electronic structure of the films, particularly concerning the embedded silicon nanoparticles (NPs). PL spectra of all the samples analyzed are shown in Figure 4.9 reported normalized to maximum intensity with black and colored solid lines for the original curves and the fitting curves, respectively.

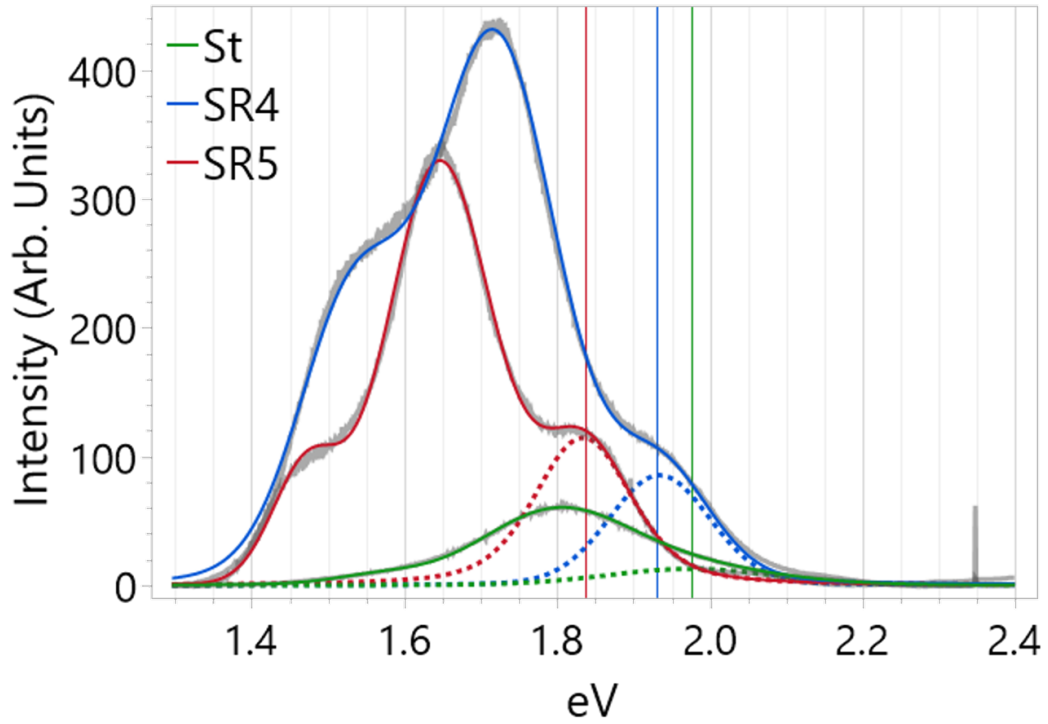


Figure 4.9: spectrum of photoluminescence obtained for the three samples reported normalized to the maximum intensity with black and colored solid lines for the original and fitting curves. The dotted lines represent the deconvoluted peaks related to quantum confinement effect. The central energy values of such peaks are linkable to the average dimension of the amorphous silicon inclusions: the lower the PL energy the bigger the inclusions. Moreover, higher relative intensities indicate higher presence of emitters, i.e. inclusions, embedded in the SiN_x matrix.

For this analysis a laser wavelength of 457 nm was used. The related exciting energy was

2.71 eV, less than the average energy gap of silicon nitride, reported to be around 4.5 eV [172]. However, different investigations - both based on experiments and simulations - report various energy gaps ranging from less than 2 eV to 5.7 eV depending on deposition parameters and various other factors [172–175]. In a similar way, unfortunately, the landscape of photoluminescence analyzes of SiN_x reported in the literature is unclear due to the strong dependence of the signals on the film composition. However, in this study, we focused on a single signal whose origin is linked to quantum confinement effects, and therefore to the presence of NPs, as discussed below.

The fittings shown in the figure for each original photoluminescence signal were obtained employing the software Fytic, which permits to evaluate the weighted sum of squared residual (WSSR) of the curves. For each sample a very good WSSR value (1 ± 0.1) has been obtained for the relative fitting, achieved by performing a deconvolution of the original curve using three peaks described by a Voigt function (Gauss-Lorentzian Function). Among these three primary components of the complete PL emission spectra, two lower-energy peaks have been associated with radiative defect states [158, 160] and band-tail emissions intrinsic to the silicon nitride matrix [172], while the higher-energy peak in the range of 1.8 to 2.1 eV has been ascribed as a signature of quantum confinement effects occurring within silicon nanoclusters or nanoparticles [158, 160]. In each of the three samples, this last deconvolution peak is reported in Figure 4.9 by a dashed line. The centers of these peaks, also identified in the figure, indicate the photoluminescence energy E_{pl} of the photoemitting NPs and are also related to the average dimension of such inclusions.

Analyzing the evolution of such signals with increasing silicon content, we can determine three pieces of information: (i) the intensity (area) of the peaks associated with quantum confinement, and thus the number of emitters - i.e., the NPs - is very similar for the Si-rich samples SR4 and SR5 (the area of the PL peak associated with quantum confinement, dashed lines in fig.4) being only 4% larger for SR4 compared to SR5; (ii) E_{pl} decreases, indicating larger average dimensions of the NPs, with increasing atomic Si content, but the NP size estimated from E_{pl} is very similar; (iii) the full width at half maximum (FWHM) of the signals related to the two silicon-rich samples is very similar, indicating a similar dimensional distribution of the NPs. Instead, we observe that the total intensity of the PL spectra is very different among the 3 samples, indicating very different radiative recombination mechanisms associated with the defect chemistry. These observations will be further elaborated in Section 4.4: Discussions.

To directly observe the presence of silicon inclusions, the silicon-rich films were subjected to thermal annealing at 1100°C for one hour in an atmospheric environment. This thermal treatment was performed to induce the crystallization of amorphous silicon nanoparticles

that spontaneously formed within the silicon-rich SiN_x matrix during deposition. Crystallization was necessary to allow direct observation of the nanoparticles by transmission electron microscopy (TEM), as the amorphous nanoparticles could not be distinguished from the amorphous SiN_x matrix in their as-deposited state. TEM measurements were performed on electron-transparent lamellae using a Thermo Fisher Themis Z aberration-corrected scanning transmission electron microscope (STEM) operated at 300 kV. Dark-field TEM (DF-TEM) images were collected from the annealed samples for microstructural analysis and to determine crystal sizes, providing an independent validation and quantification of nanoparticle size and distribution consistent with prior Raman and PL findings. In addition, the images were taken in two areas, one near the substrate and the other near the surface of the SiN_x films, to observe possible gradients in the NP distributions and the average diameters along the thickness of the film. Finally, to avoid observing the oxidated thickness of the SiN_x films after annealing, estimated to be lower than 100 nm [167], the set of figures near the surface was taken under this most external thickness.

Selected TEM images (taken from the group near the interface with the substrate) and correlated statistical analysis for the average NP dimensions are reported in Figure 4.10 for both silicon-rich samples.

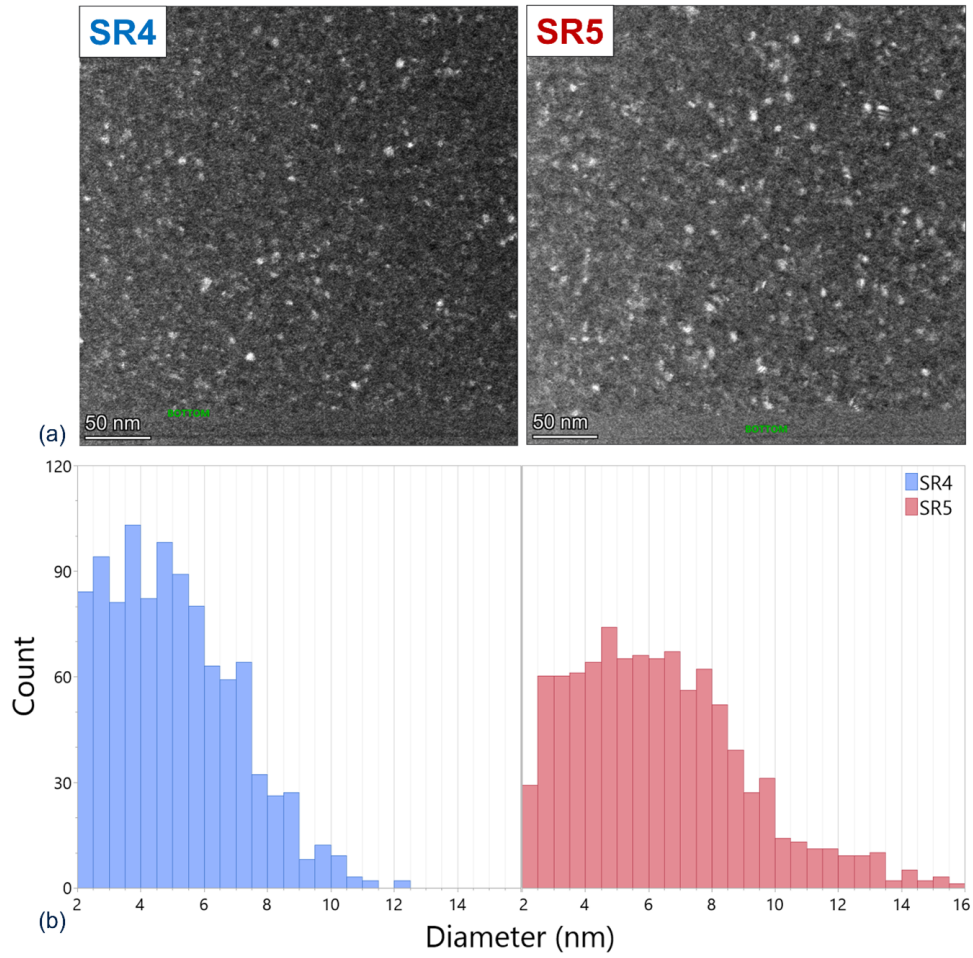


Figure 4.10: after crystallization thermal treatment at 1100°C on the two silicon-rich samples, TEM analysis allowed direct observation of embedded silicon NPs inside the SiN_x matrix (a). Further analysis on several TEM images permitted to estimate an average diameter (b) and volume fraction of the NPs for the two samples setting a lower selection limit of 2 nm of diameter.

What we observe are NPs of similar average diameter and volume fraction, with SR5 showing values of 5.4 nm and 1.1%, respectively, slightly higher than SR4 showing values of 5.1 nm and 1.0%, respectively. This crystallization process was performed mainly to have visual confirmation by TEM of the actual presence of NP inclusions in the silicon-rich films. However, since the experiment was performed identically on the different samples, it is plausible that the variation in the average diameter of the NPs after treatment is also similar. This allows to hypothesize that a similar size of crystalline NPs, both in diameter and in volume fraction, could indicate a similar relationship in the case of amorphous NPs prior to the crystallization thermal treatment.

Having confirmed the presence of the NPs only in the two silicon-rich samples, two different heat treatments were also performed at 425°C and 600°C for 1 and 4 hours of dwell time. This study was conducted to investigate the possibility of manipulating the average size of the nanoparticles through a possible interdiffusion or coalescence phenomenon without provoking crystallization. This test was performed in the hope of increasing the dimensions of NPs, enhancing a potential crack deflection phenomenon, and consequently the effective toughness of the films. However, it was not possible to attempt treatments at temperatures above 600°C due to the impracticality this approach would have in a real-world application in the semiconductor field. In fact, during the BEOL phase of device manufacturing, the metallization layers are already present, and therefore, high-temperature thermal treatments are no longer possible. Following the thermal treatments on the two silicon-rich samples, photoluminescence analysis was performed again to explore the variation of peaks related to quantum confinement, i.e. the NPs properties. The photoluminescence centers of such peaks are shown in Figure 4.11.

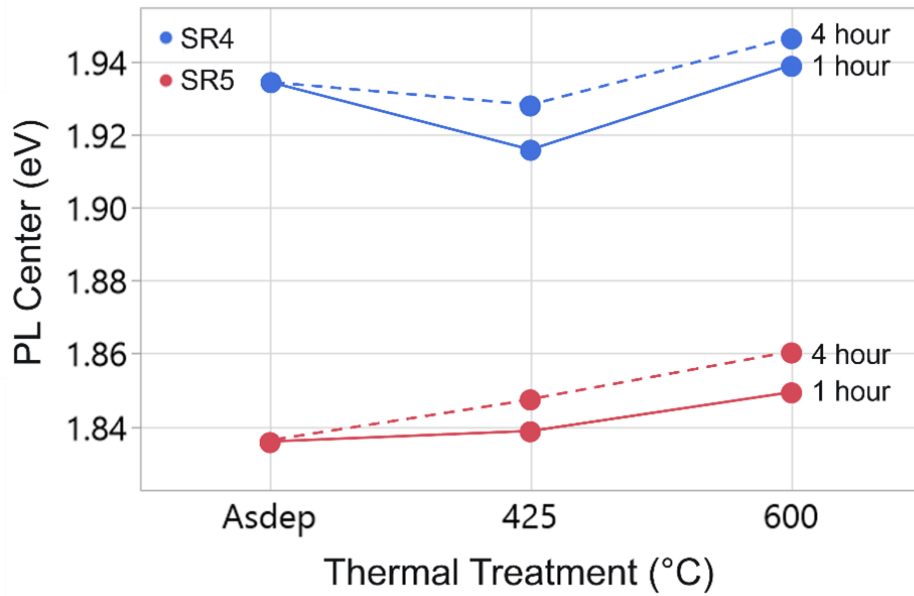


Figure 4.11: variation of the photoluminescence energy at the center of the peak related to quantum confinement for varying post-deposition thermal treatments and dwell time on the two silicon-rich samples. The PL center energy is correlated with the average dimension of the emitting nanoparticle. The results shows a slight variation of the energy following thermal treatments, highlighting the limited manipulability of NP sizes following post-deposition thermal treatments.

From the results, a distinction can be made between the two silicon-rich samples, with

SR5 consistently showing lower E_{pl} - and therefore larger average NP sizes - compared to sample SR4. Looking at individual trends, it can be observed that sample SR5 shows an increase in E_{pl} with increasing treatment temperature and exposure time. The treatments therefore seem to decrease the size of the NPs. Sample SR4 shows a slightly different behavior: for both thermal treatments, the dwell time has the same effect as observed for sample SR5, but the treatment at 425°C instead seems to lower E_{pl} , leading to an increase in size, which then decreases again at a higher temperature of 600°C. This particular trend could be connected to an energy balance system between the bulk phase of silicon nitride and the heterogeneities of amorphous silicon, in which the silicon atoms tend to migrate from one phase to another to minimize the system's energy. However, it should be noted that the most important information is related to the scale of these changes, which is almost negligible; in fact, the variations in E_{pl} for all observed cases are very limited.

To conclude the study on the impact of thermal treatments, the average size of NPs in the deposited SR4 and SR5 samples were evaluated using a phenomenological relationship that correlates E_{pl} and the average diameter of amorphous silicon nanoparticles derived by Kim et al. [176] and Park et al. [120] in their studies:

$$d_a = \sqrt{2.40/E_{pl}^{-1.56}} \quad (4.8)$$

where d_a is the average diameter of an amorphous silicon particle and the two constants are phenomenologically obtained. The values of E_{pl} and the average diameter of NPs for all the cases analyzed are reported in Table 4.1.

Table 4.1: Collection of all measured E_{pl} through photoluminescence analysis on SR4 and SR5 samples as-deposited and after thermal treatments (TT).

	SR4		SR5	
	E_{pl} (eV)	d_a (nm)	E_{pl} (eV)	d_a (nm)
As-deposited	1.933	2.5	1.836	3.0
TT 425°C 1 hr	1.917	2.6	1.841	2.9
TT 425°C 4 hr	1.925	2.6	1.848	2.9
TT 600°C 1 hr	1.939	2.5	1.846	2.9
TT 600°C 4 hr	1.948	2.5	1.861	2.8

Moreover, recalling the results of the analysis after crystallization - showing values of the average diameter and volume fraction of 5.4 nm and 1.1%, respectively, for SR5, and of 5.1 nm and 1.0%, respectively, for SR4 - this analysis brings four main results:

- Thermal treatments below 600°C and 4 hours of dwell time (i.e., the reasonable limits for these procedures in the semiconductor field) do not produce any useful variation in the average diameter of the embedded NPs in both SR4 and SR5 samples.
- The average size and volume fraction (V_{NPs}/V_{Tot}) of the crystalline NPs appears to weakly vary with increasing silicon content.
- The difference in the average size of the NPs between SR4 and SR5 is higher for the amorphous NPs in the samples deposited (+16%) compared to the crystalline one (+6%) in the samples that underwent thermal crystallization treatment.
- The average sizes of the NPs are larger after the crystallization thermal treatment. This last phenomenon could be induced by clustering effects promoted above 750°C, which could lead to atom migration or smaller NPs merging during crystallization to form larger ones [165].

We can then observe that, as sizes and distributions of the crystalline NPs are similar between the two silicon-rich samples, a similar size distribution can be expected also for the amorphous NPs, confirming the indications obtained by PL measurements. Moreover, even considering the possible difference in the size and distribution of amorphous NPs between samples SR4 and SR5, the NP sizes are too small to indicate a potential toughening effect due to crack deflection, as it can occur in certain composite materials. However, the XPS results confirmed the presence of significantly higher amounts of silicon in the SR5 sample compared to that present in the SR4. Thus, it would be plausible that part of the silicon remained within the film in other forms, such as inclusions, interstitial atoms, or particles too small to be detected.

In general, structural analysis confirms that increasing the silicon content in PECVD silicon nitride films leads to the formation of amorphous silicon nanoparticles embedded within the SiN_x matrix. These inclusions evolve in size and concentration with composition and very slightly with thermal treatment; however, the dimensions of such NPs are always no more than a few nanometers.

In summary, the main results of this section are as follows:

- Silicon-rich PECVD SiN_x films contain amorphous silicon inclusions that vary modestly in size and concentration but do not dictate the differences in toughness alone.
- Post-deposition thermal treatments seem to have no impact on the dimensions of the embedded inclusion, leaving the deposition parameters as the only effective way to manipulate their characteristics.

- The residual stress state varies linearly with the silicon content, moving from tensile to compressive and influencing fracture toughness.
- The hardness remains initially stable but starts to decrease with a sufficiently strong silicon over-stoichiometry (57.3% in this study) that influences the fracture toughness.
- The Xia et al. indentation method provides a practical approach to fracture toughness measurement in sub-micrometric films overcoming the substrate effect challenge.

4.3.3. Mechanical Properties

The mechanical characterization of PECVD silicon nitride films focused on elucidating the influence of the chemical composition and microstructural features on intrinsic residual stress, hardness, reduced elastic modulus, and fracture toughness. These parameters are critical to understanding the reliability and performance of films as passivation layers in semiconductor devices.

Intrinsic residual stress was measured by wafer curvature analysis combined with the Stoney equation prior to patterning for the application of the Xia et al. method. The St sample exhibited a tensile residual stress of approximately 120 MPa, while the silicon-rich films showed a progressive reduction in tensile stress, with SR4 at 15.2 MPa tensile and SR5 transitioning to a compressive stress state of -85.3 MPa. This trend could indicate compositional control over the intrinsic stress state, likely related to variations in film densification, atomic packing, and microstructural evolution induced by excess silicon. The absolute values of residual stress are relatively low compared to typical PECVD SiN_x films, which can reach values of the order of GPa strongly depending on the gas flow ratio, RF power, and chamber pressure during deposition [165]. However, the shift from tensile to compressive stress with increasing silicon content is significant, as residual stresses are known to influence crack initiation and propagation behavior in brittle thin films.

In the patterned samples, nanoindentation experiments were initially performed using a Berkovich tip, and the classical Oliver & Pharr method was applied to extract hardness (H) and reduced elastic modulus (E_r), with corrections implemented to exclude substrate effects thanks to the Intrinsic Thin Film methodology offered by Bruker with their tools. The results showed a clear compositional dependence: the reduced elastic modulus decreased linearly from approximately 150 GPa in the stoichiometric sample to 125 GPa in the SR5 film, reflecting a softening of the film with increased silicon content. Hardness values remained nearly constant between St and SR4 samples (15.5 GPa) but decreased to approximately 13.5 GPa in SR5. This reduction in hardness suggests an increase

in mechanical compliance and a potentially enhanced energy dissipation capacity in the silicon-rich film with the highest silicon content.

All results from mechanical characterization preliminary to toughness analysis - and values considered for crystalline silicon taken from literature - are reported in Table 4.2.

Table 4.2: Collection of all mechanical characterization prior to the application of the methodology developed by Xia et al. for toughness evaluation. Values for crystalline silicon (c-Si) [100] have been taken from literature [75, 146].

	E_r (GPa)	H (GPa)	σ (MPa)
St	150.32 ± 2.26	15.47 ± 1.03	120.11 ± 2.61
SR4	138.68 ± 2.06	15.77 ± 0.13	15.25 ± 1.84
SR5	125.12 ± 3.53	13.47 ± 0.23	-85.31 ± 4.50
c-Si [100]	180 ± 1	11.8 ± 0.2	/

The main experimental effort for mechanical characterization referred to the analysis of fracture toughness (K_{Ic}), which was evaluated using the method developed by Xia et al. The practical application of the methodology revealed some experimental challenges that were initially not considered. Apart from the proper optimization of the necessary pattern - an initial challenge by itself - Xia's method required carrying out a larger number of indentations and a longer post-processing work than expected.

The loads used to perform the tests on all three SiN_x samples were: 1300 mN, 1900 mN, 4500 mN, and 6000 mN. A fifth load of 9000 mN was attempted, but in this case, the scaling phenomena - the partial detachment of portions of the substrate following indentation - were too significant. The scaling phenomena were indeed a substantial source of error not mentioned in the reference article of Xia et al.: these brittle fracture events of the substrate consume a significant portion of the work done by the tip, thus undermining the assumption underlying the methodology reported in Equation 4.3. For this reason, scaling events that were considered too intense led to the discarding of the related indentation, and therefore the total number of indentation required for each load increased significantly. Specifically, a total of 30 "correct" indentations were chosen for each load, i.e. in the absence of substantial scaling phenomena, as the minimum value to obtain solid statistical results and consequently nearly double the number of indentations was carried out compared to this value due to the many discarded tests. Figure 4.12 shows some of these results.

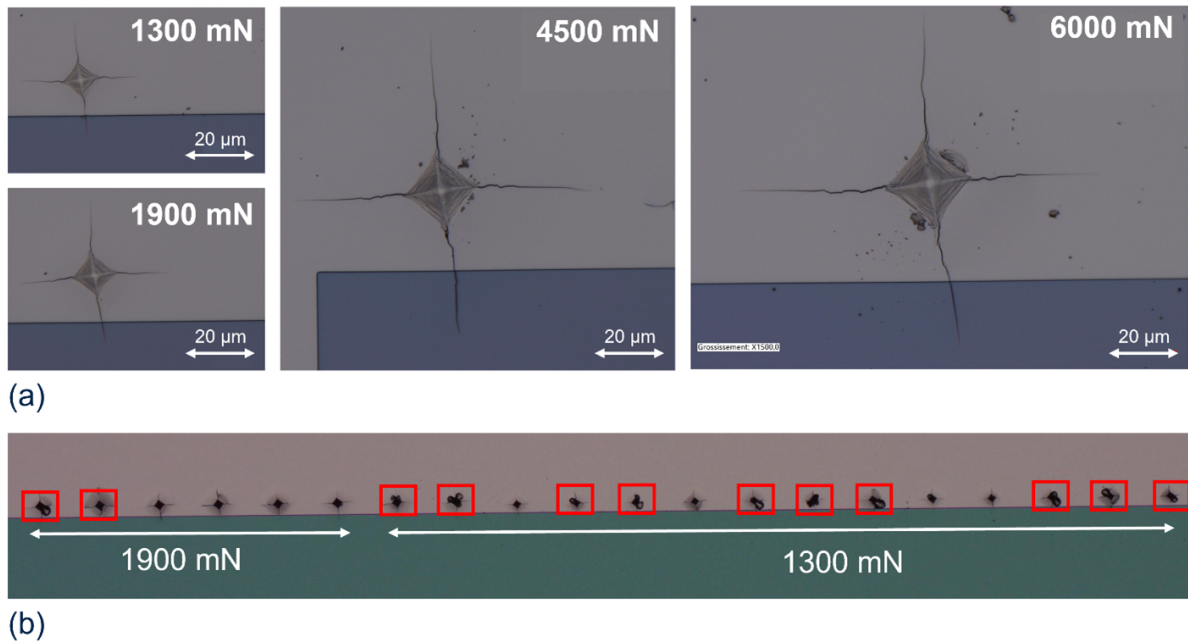


Figure 4.12: front view optical images of indentations at varying loads for the Standard SiN_x sample at higher magnification (a) and lower magnification (b). In the latter image it is possible to observe several indentations showing scaling effect highlighted in red which have been discarded for the following post-processing required to evaluate the film toughness.

A second experimental challenge involved performing indentations with the correct orientation of the samples relative to that of the Vickers tip. In fact, it was necessary to align the two so that the impression made by the tip on the samples, and thus the propagation of cracks, was as perpendicular as possible to the direction of the coating step. This was also necessary to take advantage of the automation processes made available by the tool in use: thanks to the considerable length of the step obtained with dry etching patterning, it was possible to carry out a good number of indentations (variable depending on the load used) by automating a regular movement of the tip along the axis of the step itself. However, to do this, it was necessary to correctly align the two components to prevent each indentation from being a smaller or larger distance than the previous.

Once at least 30 “correct” indentations were obtained for each load and sample analyzed, it was necessary to measure the lengths a and b of each crack. Due to the large number of cracks, it was not possible to carry out these measurements using SEM, which would have required too long operating time; thus, an optical microscope was used. After performing all measurements, it was possible to plot the lengths a against b for each sample. The results obtained are shown in Figure 4.13.

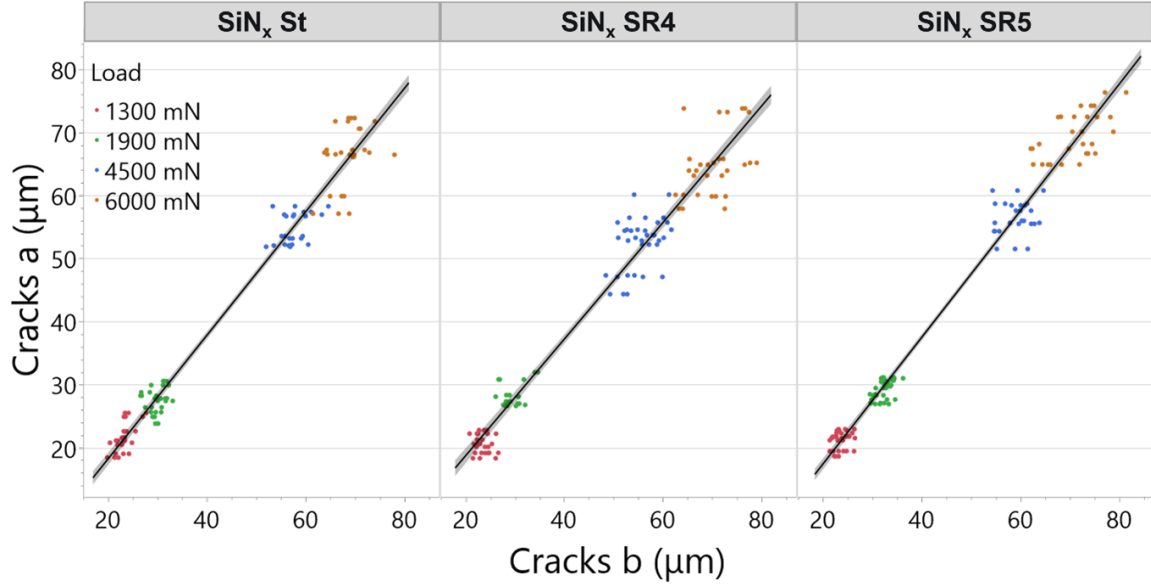


Figure 4.13: coating crack lengths a vs. substrate crack lengths b at various indentation loading from 1300 mN to 6000 mN; at least 30 points have been taken for every load and each sample. It is possible to notice that as the load increases, the scatter of the results grows, as expected from the classic brittle fracture response; however, the trend remains clearly visible, and the fitting maintains an acceptable error.

Since ϕ is the slope of the fittings obtained, the quantity dependent on the geometric factors in the final equation to calculate the toughness obtained by Xia et al. (Equation 4.7), that is, the portion “ $(\phi b - a)$ ”, can be calculated directly from the intercept of the fittings themselves. Having all the other required parameters available - also considering reduced modulus and toughness of the substrate as 180 GPa and $0.83 \text{ MPa}\sqrt{\text{m}}$, respectively, for [100] crystalline bulk silicon [75, 146, 177] -, it is therefore possible to evaluate the toughness of the analyzed films SiN_x . The experimental error of the values obtained K_{Ic} was assessed by developing the standard error from Equation 4.7, considering as variables (with their experimentally measured errors) the quantities “ $(\phi b - a)$ ”, E_r and σ . The final results are reported in Figure 4.14.

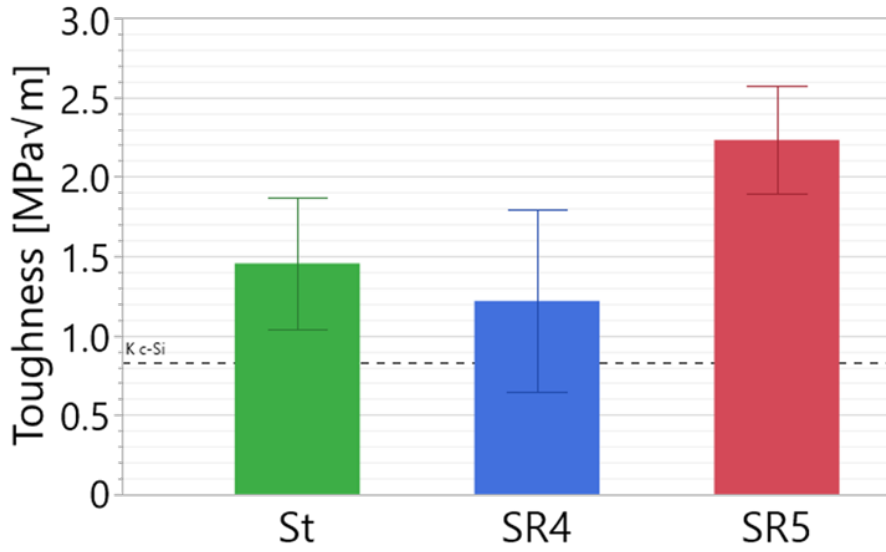


Figure 4.14: toughness results from characterization with the methodology developed by Xia et.al. for the three SiN_x samples. The horizontal dashed line indicates the toughness value of the crystalline silicon [100] substrate. The results shows high standard errors, expected within the study of brittle failures, but it is possible to state that the toughness values for the standard and SR4 samples are statistically undistinguishable between themselves, while SR5 samples clearly shows a higher value.

The measured fracture toughness values revealed that the stoichiometric films (St) and intermediate silicon-rich films (SR4) exhibited similar toughness levels, equal to $1.45 \text{ MPa}\sqrt{m}$ and $1.22 \text{ MPa}\sqrt{m}$, respectively, which are consistent with the average toughness values encountered in the literature for similar PECVD silicon nitride films [162, 176]. In contrast, the SR5 film demonstrated a significantly improved fracture toughness of $2.23 \text{ MPa}\sqrt{m}$, indicating a significant toughening effect occurring beyond a certain silicon content.

Finally, a general analysis of the mechanical data obtained also revealed an inverse correlation between hardness and fracture toughness, consistent with other phenomenological observations in brittle coatings where increased toughness is often accompanied by reduced hardness [13, 70]. The result is reported in Figure 4.15.

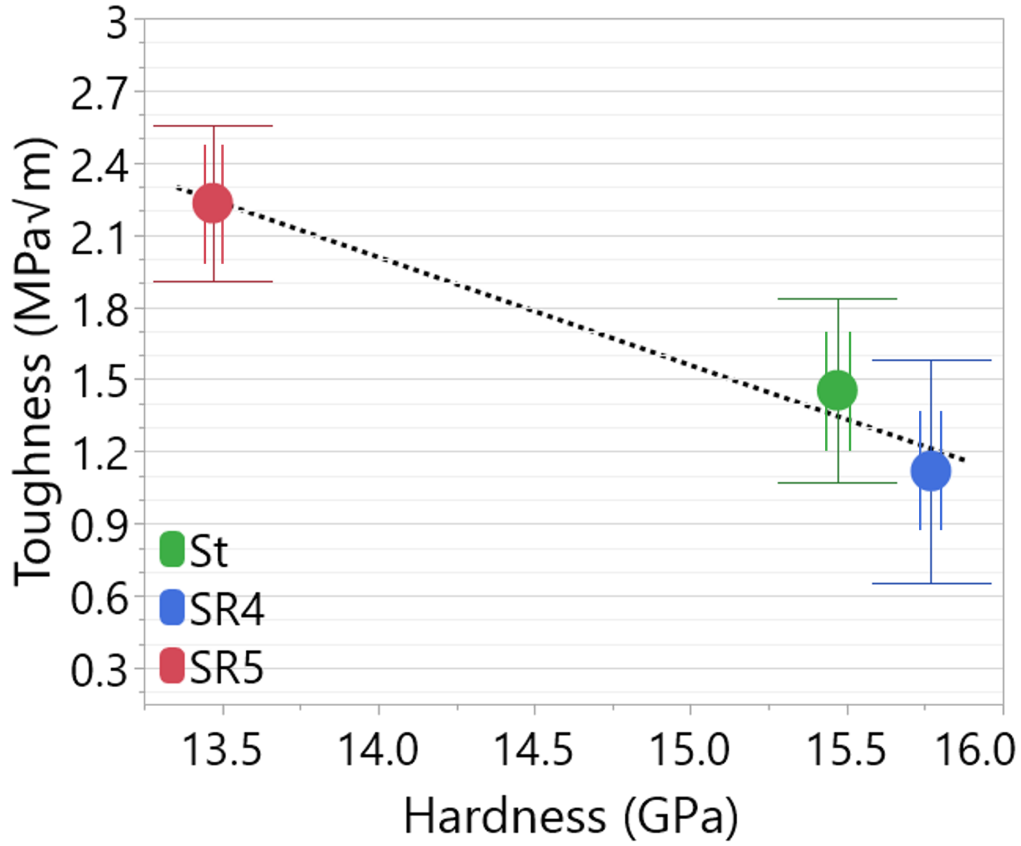


Figure 4.15: fracture toughness vs. hardness for the three analyzed SiN_x films. A linear relation between the two values has been observed, as already indicated in similar studies.

The lower hardness of the SR5 film combined with higher toughness suggests a shift in mechanical behavior towards greater compliance and improved resistance to crack propagation, possibly due to mechanisms related to the presence of inclusions of amorphous silicon or different intrinsic stress.

In summary, mechanical characterization demonstrates that careful compositional tuning of PECVD silicon nitride films, particularly increasing silicon content to form silicon-rich matrices with embedded amorphous silicon inclusions, can effectively modulate residual stress, reduce hardness, and improve fracture toughness in a significant way. These findings highlight the critical role of microstructural engineering in optimizing the mechanical performance of thin-film passivation layers for semiconductor applications.

In summary, the main results of this section are as follows:

- Residual stress and Elastic modulus decrease monotonically as the silicon content increases (from tensile to compressive for stress).
- The hardness drops significantly and the fracture toughness increases significantly only

for the film with the highest silicon-content, while St and SR4 show very similar results.

- Despite some encountered experimental challenges, the Xia et al. method effectively measures toughness proving compositional tuning of SiNx films mechanical properties.

4.4. Discussion

4.4.1. Assessment of the Applicability of the Xia et al. Method

The implementation of the Xia et al. method for evaluating fracture toughness in silicon nitride thin films within a practical laboratory and industrial context has illuminated both the strengths and limitations of this approach.

One of the most underestimated issues of this methodology is its time-consuming experimental procedure. Each sample requires extensive indentation campaigns across multiple loads, with a large number of indentations necessary to achieve statistically meaningful results. This demand arises in part from the brittle nature of both the films and the silicon substrate, which leads to frequent scaling phenomena. Consequently, the total number of indentations performed often doubles the minimum required, extending the duration of experiments to several days per sample and hindering the possibility of measuring the crack lengths through more sophisticated analysis like SEM in favor of faster ones, like optical microscopy.

In addition to this, a second concern regards the repeatability of the experiments. The method yields relatively high standard deviations in fracture toughness values, reflecting the intrinsic variability of crack initiation and propagation in brittle thin films and the sensitivity of the measurement to subtle experimental factors such as surface roughness and local film heterogeneity. The necessity of aligning the Vickers indenter precisely perpendicular to the coating edge to ensure consistent crack propagation adds further complexity to the procedure, and there is no single criterion to decide whether a scaling phenomenon can be considered negligible or whether it results in rejection of the indentation test.

Lastly, the analytical model, while robust and validated against finite element simulations, is still an approximation. It assumes linear elastic fracture mechanics, uniform residual stresses, and idealized crack geometries. Real films may exhibit more complex stress distributions, anisotropic mechanical properties, or microstructural heterogeneities that are not fully captured by the model. Additionally, the parameters of the model, such as dimensionless constants λ and Ψ , are derived from simulations on specific material systems, and even if the test was run for a large set of materials, they may not universally

apply without recalibration.

On the other hand, one of the most significant advantages of this method lies in its ability to circumvent the substrate effect without requiring complex sample preparation such as microfabrication of freestanding films or micro-cantilever structures, such as the one necessary to apply a single edge notch bend (SENB), Double Cantilever Beam (DCB), or other similar approaches [73, 74, 161–163]. Although in the case of this study the preparation of the samples involved some challenges, it is clear that, unlike the previously mentioned techniques, Xia’s approach can be implemented using standard indentation tools combined with photolithographic patterning, which is readily available in semiconductor fabrication environments. It is important to note that the synthesis method used in the original research by Xia et al. requires some additional steps, since lithography must be used prior to film deposition to obtain an almost perfect pattern. However, the process used in this research - a flat deposition followed by dry etching - allows for a more approximate geometry but with shorter times, especially if the methodology becomes routine and it is therefore not necessary to perform an etching optimization procedure. Moreover, in many cases this approach could be implemented on real devices at any step of the process that shows an appropriate pattern, without, therefore, needing to create a custom wafer. It is sufficient to consider that in the work presented here, we used a mask initially created for the processing of the IGBT device; therefore, it is plausible that, among all the process steps of a device, there could be some featuring the appropriate pattern for performing such analyzes. This accessibility makes it particularly attractive for industrial research and quality control, where throughput and simplicity are critical.

Moreover, there are no thickness-related limits to the applicability of the method; this is one of its main strengths, since the classical LEM method is considered unapplicable under thicknesses of 1 μm . By indenting near the edge of the film on the bare substrate and comparing the lengths of the cracks propagating into the coated versus uncoated regions, it effectively isolates the fracture behavior of the coating from the influences of the substrate regardless of its thickness. Moreover, the method can also account for residual stresses by incorporating this contribution into an analytical model validated by finite element simulations. This comprehensive consideration improves the reliability of the toughness values obtained in comparison to classical approaches based on the indentation directly of the film of interest.

Overall, the Xia et al. method represents a valid approach for the measurement of the fracture toughness of thin films, providing a relatively practical and straightforward solution to a historically challenging problem. Its application in this study has demonstrated its feasibility in an industrial research setting, offering valuable comparative insights into

the mechanical behavior of silicon nitride films with varying composition. A summary of the pros and cons identified for the methodology proposed by Xia et al. is reported below:

• **Pros**

- Allows us to avoid challenges related to substrate-effect phenomena.
- Requires relatively accessible instrumentation and sample preparation.
- Applicable regardless of the film thickness (not limited on micrometric films).

• **Cons**

- Time consuming (around 2-3 days per sample considering indentations and crack length measurements).
- Low repeatability and high standard deviation (tendency towards a qualitative approach).
- Based on a poorly grounded analytical model.

4.4.2. Results on Toughness Variation with Si/N Ratio

The main objective of this research is to understand if and how the variation of SiN_x stoichiometry during PECVD synthesis affects its mechanical properties. The results have shown that a toughening effect is possible when depositing SiN_x coatings with a sufficiently high ratio between the flux of silane and ammonia, i.e., a sufficiently strong over-stoichiometry of silicon in the film composition. However, it is not straightforward to understand the exact mechanisms that reside at the origin of such variation in the mechanical behavior of silicon nitride.

A clear information, already widely reported in the literature, concerns the spontaneous formation of amorphous silicon clusters within the silicon nitride matrix when deposited with a sufficiently high over-stoichiometry of silicon precursors during PECVD. Indeed, silicon-rich films have been shown to emit Raman and PL signals related to the presence of amorphous silicon, which was not present in the standard sample. TEM images obtained after a thermal crystallization treatment at 1100°C confirmed the absence of NPs in the St sample and provided further information on the similarity between NPs in the two silicon-rich samples: although the crystallized particles will differ from the amorphous ones, the heat treatment, applied identically to both samples, should modify them in a similar way.

The subsequent investigation focused on the possibility of modifying the size and distribu-

tion of these amorphous silicon clusters through post-deposition annealing at 425°C and 600°C, but even in this case the results were discouraging. Indeed, photoluminescence spectroscopy conducted after such annealing revealed negligible changes in the average size of amorphous silicon nanoparticles, as inferred from shifts in quantum confinement-related emission peaks. These subtle variations suggest that thermal treatments within the allowable BEOL thermal budget do not promote nanoparticle coarsening or clustering enough to promote any further toughening phenomenon. Therefore, these initial investigations have made it possible to determine that although silicon-rich SiN_x films do contain clusters of amorphous silicon - absent in nearly stoichiometric films - their average sizes (~ 3 nm) cannot be modified effectively after the synthesis of the films and are too small to activate any known toughening phenomena, such as crack deflection or shielding. Moreover, they are too similar between the two silicon-rich samples to indicate a possible toughness difference between them.

The preliminary results of the mechanical characterization indicated a compositional dependence on the silicon-to-nitrogen ratio in the films of both intrinsic residual stress, elastic modulus, hardness, and fracture toughness. Residual stress measurements showed a monotone trend from tensile in the stoichiometric film (St) to compressive in the film with the highest silicon content (SR5). This transition is significant because the residual stress state directly influences crack initiation and propagation: tensile stresses facilitate crack opening and reduce toughness, whereas compressive stresses impede crack growth, effectively enhancing toughness. Similarly, the reduced elastic modulus decreased linearly with increasing silicon content, reflecting a softening of the film matrix, while the hardness remained relatively constant between the stoichiometric and moderately silicon-rich film (SR4) but decreases notably in the film with the highest silicon content (SR5). This reduction in hardness suggests an increase in mechanical compliance and energy dissipation capacity, which are consistent with the observed increase in fracture toughness. Once preliminary analyzes were completed, it was possible to begin toughness analyzes using the method proposed by Xia et al., which gave results consistent with average toughness values encountered in the literature for similar PECVD silicon nitride films [162, 176] in the case of the St and SR4 samples, but a significantly higher result for SR5. As expected for brittle failure analyzes [72], the standard deviations obtained for the three toughness values are large, and it is therefore difficult to evaluate the increase of the result related to the SR5 sample compared to the SR4 or St samples. However, the average increase with respect to the St sample is 54%, with a minimum 20% considering the standard deviation. Moreover, regardless of the specific results, allowed a better understanding of the strengths and weaknesses of the methodology itself, as discussed in section 4.4.1.

On top of these considerations, the XPS results clearly show that the SR5 sample has a significantly higher atomic percentage of silicon (Si/N ratio equal to 1.33) compared to the SR4 sample (which is only slightly over-stoichiometric) and the St sample (slightly substoichiometric). Both silicon-rich films contain amorphous Si nanoparticles, but increasing significantly the silicon content during the PECVD growth of a SiN_x film does not lead to an increase in the size or concentration of segregated Si nanoparticles, but rather to an excess of silicon which remains dispersed as interstitial atoms, Si atoms bonded to N (e.g. filling dangling bonds), or as very small clusters (not contributing to PL), impacting in a stronger way the mechanical properties of the SiN_x matrix. Interstitial atoms can modify the evolution of intrinsic stress during growth and amorphous clusters are known to possess a lower hardness compared to stoichiometric silicon nitride [13], therefore their incorporation into the matrix may be expected to facilitate localized plasticity, thus improving fracture resistance. Although exact mechanisms relating to local atomic structure and mechanical properties certainly require further investigation, this can explain why an excessive amount of dispersed silicon would lead to a reduced hardness of the SiN_x matrix and a shift toward compressive stress formation simultaneously, thus leading to an increase in the effective toughness of the material.

4.5. Summary

This chapter presented a comprehensive investigation into the composition-dependent mechanical behavior of silicon nitride (SiN_x) thin films deposited by plasma-enhanced chemical vapor deposition (PECVD). The primary focus was to understand how variations in the silicon-to-nitrogen atomic ratio influence critical mechanical properties such as intrinsic residual stress, hardness, elastic modulus, and mainly fracture toughness, which are essential for the reliability of semiconductor passivation layers.

The study confirmed that increasing the silicon content during PECVD leads to the spontaneous formation of amorphous silicon nanoparticles embedded within the SiN_x matrix. These inclusions were detected only for the two silicon-rich films through Raman and photoluminescence spectroscopy and were observed directly after crystallization through thermal annealing via transmission electron microscopy (TEM). However, despite their presence, the nanoparticles were too similar between the two films, and their slight variations could not fully explain the significant differences observed in fracture toughness between the SR4 and SR5 samples. Instead, the mechanical property variations were closely correlated with stoichiometry-driven changes in residual stress and elastic modulus. Specifically, an increase in silicon content shifted the residual stress from tensile

to compressive and reduced the elastic modulus and hardness, resulting in increased mechanical compliance and fracture toughness.

The fracture toughness was evaluated using the indentation-based method developed by Xia et al., which effectively mitigates substrate effects and incorporates residual stress contributions. Although not without flaws, this method provided reliable comparative toughness values, showing a pronounced toughening effect beyond a critical silicon content threshold. The inverse relationship between hardness and toughness observed is aligned with phenomenological models in brittle coatings, suggesting a trade-off between stiffness and fracture resistance mediated by microstructural and compositional factors.

In general, the findings highlight the critical role of compositional tuning and microstructural engineering in optimizing the mechanical performance of PECVD silicon nitride films. The presence of dispersed amorphous silicon, likely in forms beyond detectable nanoparticles, appears to modulate the properties of the matrix and the evolution of residual stress, thus enhancing fracture toughness without compromising the protective functions of the film.

These insights have important implications for the semiconductor industry, suggesting that careful control of silicon content during PECVD deposition can be a practical strategy to improve passivation layer durability, especially in the context of device scaling and wafer size transitions. Future research should focus on elucidating the detailed mechanisms by which amorphous silicon inclusions influence other properties such as electrical performance and moisture resistance, in order to have a broader overview of the properties that can be achieved for the silicon nitride passivation layers by simply manipulating the film growth parameters during PECVD.

Key Takeaways

- Silicon-rich PECVD films SiN_x contain amorphous silicon inclusions that vary modestly in size and concentration but do not dictate the differences in toughness alone.
- Post-deposition thermal treatments seem to have no impact on the embedded inclusion's dimensions, leaving deposition parameters as the only effective way to manipulate their characteristics.
- Residual stress state varies linearly with silicon content, going from tensile to compressive and influencing fracture toughness.
- Hardness remains initially stable but starts decreasing over a sufficiently strong silicon over-stoichiometry (57.3% in this study) influencing fracture toughness.

- The Xia et al. indentation method provides a practical approach to fracture toughness measurement in sub-micrometric films overcoming the substrate effect challenge. A resume of the quantitative results obtained for each material studied is reported in Table 4.3.

Table 4.3: Resume of the results obtained for the mechanical characterization of the material involved in this line of research. Specifically, the table report the values of Elastic Modulus (E_r), Hardness (H), Residual Stress (σ) and Toughness (K).

	E_r (GPa)	H (GPa)	σ (MPa)	K (MPa $\sqrt{m}$)
St	150.32 ± 2.26	15.47 ± 1.03	120.11 ± 2.61	1.45 ± 0.63
SR4	138.68 ± 2.06	15.77 ± 0.13	15.25 ± 1.84	1.22 ± 0.77
SR5	125.12 ± 3.53	13.47 ± 0.23	-85.31 ± 4.50	2.23 ± 0.53
c-Si [100]	180 ± 1	11.8 ± 0.2	/	0.83 ± 0.14

5 | In-Situ vs Ex-Situ Indentation for Adhesion Evaluation of Nitride/Organic interfaces

Polymeric materials play a pivotal role as protective layers in advanced microelectronic devices, providing essential mechanical protection and electrical insulation to sensitive underlying structures. Among these polymers, polybenzoxazole (PBO) and polyimides (PI) are widely utilized due to their excellent thermal stability, mechanical robustness, and chemical resistance, which are critical to ensuring device reliability and operational longevity in demanding environments. In particular, PBO has gained importance in the semiconductor industry due to its additional photosensitive properties, which make it suitable as a masking polymer for photolithography steps, in addition to its more traditional protective role [76]. This aspect is of interest for a company which will prefer the employment of materials with multiple characteristics and is one of the reasons why PBO was included as a protective polymer after the switch to 300 mm - and consequently the move to a different factory - for the production of the IGBT device.

Despite these advantageous properties, the long-term reliability of polymer-coated devices strongly depends on the quality of adhesion between the polymeric film and the inorganic substrate, which in the semiconductor field is typically a passivation layer such as silicon nitride (SiN_x) [155, 178]. Silicon nitride is extensively employed in semiconductor manufacturing, however the interface between SiN_x and polymeric coatings remains a critical locus for potential failure, as poor adhesion can lead to delamination, cracking, and ultimately device malfunction or reduced yield. A question about the possible impact of the film stoichiometry on its adhesiveness with organic coating is still open. Moreover, substrate pre-treatments are a known strategy that can be followed to enhance adhesion, counterbalancing the possible distance with the coating affinity [179–183].

Another aspect strongly impacting adhesion is the environmental factors, particularly temperature and relative humidity, that significantly influence the integrity of an or-

ganic/inorganic interface [80, 81, 184, 185]. Moisture absorption by the polymer induces hygroscopic swelling, generating internal stresses that promote interfacial degradation and delamination [82, 186–189]. Thermal cycling further exacerbates these stresses through mismatched expansion coefficients, accelerating mechanical failure mechanisms [39, 190]. Consequently, understanding how environmental conditions affect adhesion is essential for designing robust interfaces capable of withstanding operational stresses.

By comparing adhesion behavior across a matrix of substrate compositions, surface plasma treatments, and ambient conditions, this chapter presents a systematic study of adhesion between PBO and SiN_x films with varying stoichiometry, evaluated through complementary in-situ and ex-situ nanoindentation techniques under controlled environmental conditions. Section 5.1 reports a description of the procedures followed to obtain the analyzed samples, the characterization performed to analyze the surface of the substrates before the PBO deposition, and the characterization methods used to analyze the adhesion in controlled and ambient environments. In the following, Section 5.2 reports the main results of this study, focusing first on the analysis of the substrates and then on the adhesion results in ambient and controlled environments, the latter obtained with in-situ and ex-situ tests after aging treatments. Section 5.3 contains a discussion on the results obtained, also focusing on their industrial implications, and Section 5.4 closes this chapter with a final summary.

5.1. Materials and Experimental Methods

In this section, the procedure for obtaining the final experimental samples is described, followed by the procedure used to perform the substrate characterization before PBO deposition, and the setup used to perform the indentation-based adhesion analysis while considering the influence of the environment.

5.1.1. Sample Preparation

The experimental investigation focused on a set of six samples designed to systematically evaluate the adhesion between PBO polymer films and SiN_x substrates with varying stoichiometry and surface treatments. The SiN_x films were deposited by Plasma Enhanced Chemical Vapor Deposition (PECVD) with a nominal thickness of 1 μm onto 300 mm planar silicon wafers (crystalline silicon [111]) with a thickness of 775 μm . Two primary stoichiometric variants of SiN_x were prepared: a standard near-stoichiometric composition with a nominal Si/N atomic ratio equal to 0.80 and a silicon-rich composition with a nominal Si/N atomic ratio equal to 1.10. Each stoichiometric group included three

surface conditions: as-deposited (no treatment), oxygen plasma treated (O_2), and carbon tetrafluoride plasma treated (CF_4), resulting in a total of six distinct substrate types, summarized in Table 5.1.

Table 5.1: List of SiN substrates analyzed in this study. All samples are composed of a bulk silicon support, a SiN_x substrate of $1\ \mu m$ thickness with varying stoichiometry and surface treatments, and an upper-lying $9\ \mu m$ PBO film.

Tag	Si/N	Treatment
St_Asd	0.8	As-deposited
St_ CF_4	0.8	Plasma CF_4
St_ O_2	0.8	Plasma O_2
SR_Asd	1.05	As-deposited
SR_ CF_4	1.05	Plasma CF_4
SR_ O_2	1.05	Plasma O_2

The surface preparation of the inorganic substrate is a proven strategy to enhance adhesion through the modification of the surface chemistry and roughness, thus improving chemical affinity and mechanical interlocking with the polymer [180, 191]. In this work, the oxygen and fluorine plasma treatments were performed using an inductively coupled plasma reactor (ICP) with an exposure duration of 10 minutes for each sample. These treatments were selected to modify the surface chemistry and roughness of the SiN films, thereby influencing the subsequent adhesion characteristics with the PBO layer. Moreover, the choice of these specific plasmas was also dictated by the availability provided by the company STMicroelectronics, which often uses them to treat the surfaces of passivating films. This allowed the realization of the treated wafers without leaving the production line, thus minimizing experimental error.

Following substrate preparation, a uniform PBO film with a nominal thickness of $9\ \mu m$ was deposited on all samples via spin coating. The PBO precursor solution was carefully formulated and processed to achieve consistent film thickness and curing behavior, but cannot be shared in this work for privacy reasons. The curing step was performed at $350^\circ C$ under controlled thermal conditions to initiate the oxazolation reaction, which converts the PBO precursor into a thermally stable cross-linked polymer network essential for device protection [76, 192]. Finally, to enable adhesion testing, the samples were sectioned into smaller pieces ($\sim 2 \times 2\ mm$) suitable for nanoindentation analysis.

5.1.2. Surface Characterization of the Substrates

Before polymer deposition, the substrates were characterized on the surface to assess roughness, chemical composition, and other potentially influential morphological features.

Atomic Force Microscopy (AFM) was employed to quantify the surface roughness parameters. A Bruker Dimension IconXR equipped with a Nanoscope V controller was used, operating in Peak Force Tapping Mode in ambient air. A silicon cantilever with an Adama AD 2.8-AS tip (spring constant 2 N/m) was used to scan a $10\ \mu\text{m} \times 10\ \mu\text{m}$ area with a resolution of 512×512 points at a scan rate of 0.5 Hz and a peak force setpoint of 5 nN.

X-ray Photoelectron Spectroscopy (XPS) coupled with argon sputtering for depth profiling and Transmission Electron Microscopy (TEM) with Energy Dispersive X-ray Spectroscopy (EDX) provided detailed information on surface chemistry and the presence of any treatment-induced oxide layers or contaminants. XPS was performed using a PHI5000 Versaprobe III (ULVAC-PHI) with a photon energy of 280 eV, equipped with an argon sputtering gun to enable in-depth profiling up to $10\ \mu\text{m}$. TEM was performed using a Thermo Fisher Themis Z equipped with an EDX PHI5000 Versaprobe III based on an Al mono source.

5.1.3. Nanoindentation for Adhesion Measurement

The adhesion between PBO and the SiN_x substrates was initially evaluated in all six samples through macro-mechanical testing methods such as four-point bending [77, 193] and pull-off tests [194]. However, in both cases, the delamination occurred in the glue used to fix the sample to the test dummy between the PBO and the substrates. Therefore, these techniques resulted in the evaluation of a lower bound of the minimum delamination energy values for the PBO, equal to $30\ \text{J/m}^2$, but no critical values were obtained, since the methods were unable to initiate the delamination of the polymer. Although the lower bound found was sufficiently high by the STMicroelectronics standards, the company proposed to continue the research with the use of the nanoindentation technique, which has been often used as a methodology to evaluate adhesiveness used in front-view [78, 185, 195] - or even in cross-section indenting near the interface between the coating and the substrate [79] - and in this research allowed to achieve the delamination of the PBO for the full set of samples. For this reason, the research was therefore focused on a comparative study between the analyzed samples rather than on obtaining an absolute value of the delamination energy.

In this work, indentations were conducted on the surface of the PBO film in a front-view

configuration, with the indenter applying a normal load perpendicular to the film surface. All tests were performed using a Bruker TI980 tribo-indenter equipped with a high-load transducer and a sphero-conical indenter tip with a curvature radius of $20\ \mu$. The relatively large tip radius was selected to generate a stress field that extended sufficiently through the polymer thickness to reach the PBO/ SiN_x interface, thus promoting delamination under controlled loading conditions. Such delaminations are detectable as abrupt changes - referred to as “decay events” - in the load-displacement curve. The critical load at which these delamination events occur serves as a direct indicator of the adhesion strength between the polymer and the substrate. The primary metric extracted from the nanoindentation testing is the critical load of delamination, which reflects the mechanical integrity of the polymer/SiN interface. Figure 5.1 reports an example of an indentation test performed on the SR_Asd sample where the decay events and the correlated critical load are highlighted.

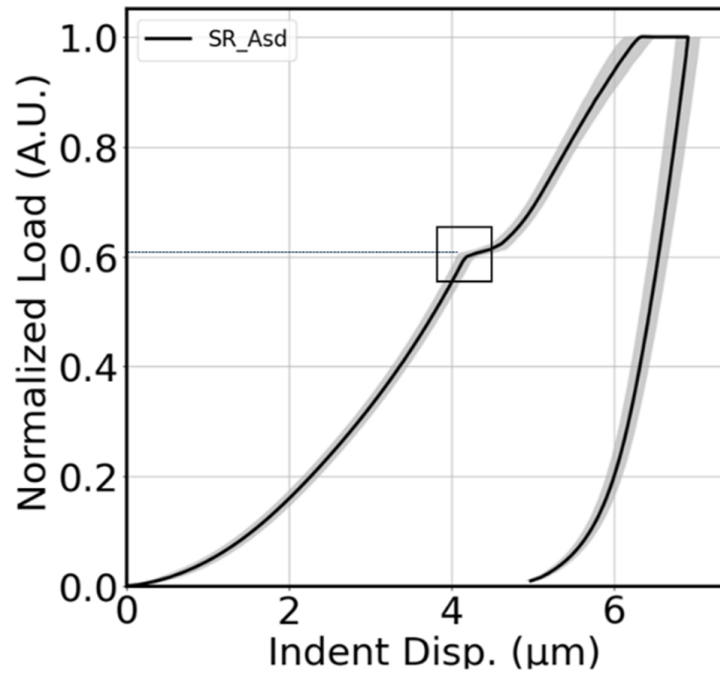


Figure 5.1: load-displacement curve obtained by averaging six separated indentations on the SR_Asd sample. The average curve is represented by the marked black line, while the gray band represents the standard deviation. The abrupt change in the slope, i.e. the decay event, permits to evaluate the critical load of delamination.

Experimentally, each sample was subjected to a series of indentations, with six consecutive tests performed per sample for each environment tested - both in-situ or ex-situ - to ensure statistical robustness. The average curve shown in Figure 5.1, for demonstration purposes, was obtained using the same method applied to all load-deformation curves presented in

this study. Specifically, the data were processed through a Python analysis, calculating the mean value and the associated standard error for all the sets of points collected along the Y-axis. These points were grouped into intervals of arbitrarily wide width equal to 10^{-5} units. Reconstructing the mean values for each interval, we obtain a continuous curve, while the bars corresponding to the standard error define the transparent band of uncertainty around the curve.

The critical delamination load of each sample in a specific environment was determined by these plots individuating the average loads across the six indentations corresponding to the decay events. The values were then normalized to the maximum applied load of 600 mN. This step has been carried out on all measurements since, in the early stages of this research, different maximum loads had been tested, making it necessary to perform normalization for a better comparison of the results. Subsequently, normalization remained a convenient practice, especially to mask the absolute values of the loads obtained when sharing the data during the participation of conferences outside of the company.

To conclude, it is important to state again that this approach allows for sensitive detection of interfacial delamination at a certain critical load but not for the extrapolation of the energy of delamination of the film, a parameter that is most often used to evaluate the adhesiveness of a coating. However, the critical load of delamination is a clear indicator, which allows us to compare samples analyzed in similar environments to assess the relative results between them and create a ranking of the most adhesive films with respect to the others.

5.1.4. Environmental Control for Adhesion Testing

Adhesion characterization methods in controlled environments often rely on ex-situ tests following ambient aging treatments [39], i.e. the maintenance of a sample in a climate chamber under controlled temperature (T) and relative humidity (RH) for a certain period of time, which often exceeds several hours or days. In contrast, in-situ adhesion testing under controlled temperature and humidity offers the potential to capture transient effects and reversible degradation phenomena, providing a more realistic assessment of the interface performance.

One objective of this work was also to assess and study the scale of the different output following adhesion characterizations when performed in-situ or ex-situ (i.e. immediately after aging). In the former case, adhesion measurements were carried out within a controlled environment chamber integrated with the nanoindenter, specifically the “xSol-Humidity” module produced by Bruker [137]. This setup allows for real-time control and monitoring

of temperature and relative humidity directly on the sample surface during indentation testing, eliminating the need to transfer samples between the climate chamber and the environment of the mechanical analysis. The in-situ approach captures the transient and reversible effects of environmental exposure on adhesion, providing a realistic assessment of interface behavior under operational conditions. The tests were carried out on a range of temperatures up to 75°C and relative humidity levels up to 75%, corresponding to dew points representative of severe ambient conditions. On the other hand, samples for ex-situ testing were subjected to controlled aging treatments in a climatic chamber LIB DHPV-04_PV 04 under varying temperature and humidity conditions (up to 85°C and 85% RH) for 2 and 7 days. Following aging, samples were removed from the chamber and immediately tested via nanoindentation under ambient laboratory conditions. This method simulates long-term environmental exposure and allows the evaluation of permanent or time-dependent effects of adhesion degradation.

Figure 5.2 shows all the environmental conditions evaluated in this study for both in-situ and ex-situ analyzes, designated by a diamond and a circle, respectively. Three of the points related to the in-situ tests are represented as empty to indicate that such points have been tested only for the Standard samples and not for the silicon-rich ones. Indeed, these tests were performed last in chronological order, and due to time constraints and equipment availability - provided by the STMicroelectronics laboratories - it was not possible to perform them on all of the samples in the study. The purpose of these additional tests was to evaluate the behavior of PBO adhesion as T varies while RH is kept constant, and vice versa. Moreover, they allowed for the evaluation in-situ of adhesion at a constant dew point, as explained below.

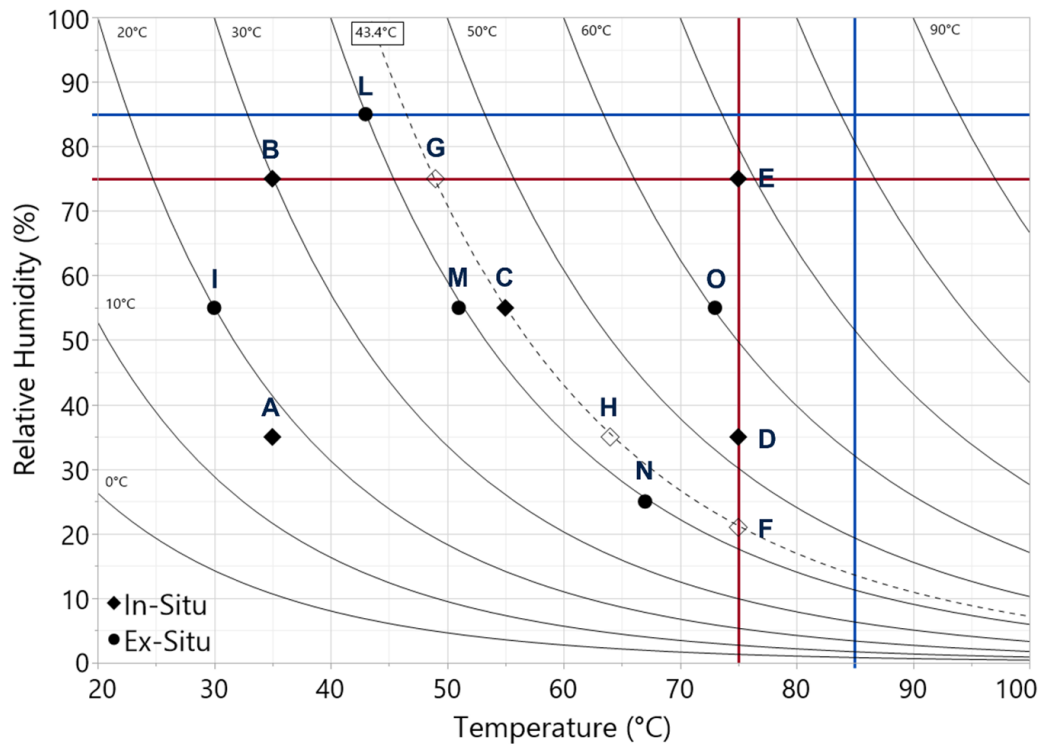


Figure 5.2: T-RH plot showing the lines of constant dew point (values reported along the plot edge) and the points characterized in this study in-situ by xSol-Humidity module or ex-situ by climatic chamber. The in-situ points not fully colored (only outlines), are referred to extra tests made only with the Standard samples. The red and blue lines indicate the operational limit of the xSol-Humidity module and climatic chamber, respectively. The letters near every points refers to Table 5.2.

In addition to temperature and relative humidity values, the figure also includes the constant dew point curves, which allow for a univocal identification of the environmental severity of each condition. Indeed, due to technical limitations of the climatic chamber tool - and its availability within the framework of the various research programs currently underway at STMicroelectronics -, it was not possible to set the same T and RH tested in-situ for the series of tests made ex-situ after aging treatments. For this reason, in this work the ambient is also classified through the dew point temperature (T_{DP}) instead than only by T and RH.

The dew point is the temperature at which the air becomes saturated with moisture, causing water vapor to condense into liquid water. It depends directly on the relative humidity and temperature of the environment and represents the value to which such a temperature should drop to bring the relative humidity to 100% and therefore lead to

the formation of liquid water (dew) in the sample [196]. By univocally associating the environment - defined by T and RH - with the dew point, in this work, it was possible to compare the PBO adhesion on the various substrates even between environments defined by different T and RH values.

The dew point can be defined by knowing T and RH using the Magnus-Tetens formula [196]:

$$T_{DP} = \frac{b \cdot \gamma(T, RH)}{a - \gamma(T, RH)} \quad (5.1)$$

with:

$$\gamma(T, RH) = \frac{a \cdot T}{b + T} + \ln \frac{RH}{100} \quad (5.2)$$

for $T > 0$, where:

$$a = 17.7, \quad b = 243^{\circ}C \quad (5.3)$$

Where T_{DP} and T are the dew point and the ambient temperature, respectively (both in $^{\circ}C$), RH is the relative humidity and a and b are the phenomenological values of the Magnus-Tetens formula. To conclude, all tested points are reported in Table 5.2. The environments marked with one or two asterisks were specifically chosen along the same dew point line, at $\sim 43^{\circ}C$ for the in-situ tests and $\sim 40^{\circ}C$ for the ex-situ tests, respectively. These tests allowed to observe the behavior of PBO adhesion in environments with different T and RH but the same T_{DP} , to experimentally identify any issues with the approach of considering environmental severity through the dew point. The additional points tested in-situ on the Standard samples were also carried out for the purposes of this investigation.

Table 5.2: List of all the ambient tested points, indicated by the couple T-RH or univocally by the corresponding T_{DP} , evaluated by Magnus-Tetens formula (Eq. 5.1). Each point is tagged with a letter for quicker reference in the following text.

* environments tested in-situ on the same dew point (only on standard samples).

** environments tested ex-situ on the same dew point.

Tag	T (°C)	RH (%)	T_{DP} (°C)	Tool	Analysis
A	35	35	17.4	xSol Humid	In-Situ
B	35	75	29.9	xSol Humid	In-Situ
C*	55	55	43.4	xSol Humid	In-Situ
D	75	35	52.9	xSol Humid	In-Situ
E	75	75	68.7	xSol Humid	In-Situ
F*	75	21	43.2	xSol Humid (Only St Samples)	In-Situ
G*	49	75	43.5	xSol Humid (Only St Samples)	In-Situ
H*	64	35	43.1	xSol Humid (Only St Samples)	In-Situ
I	30	55	20.0	Climatic Chamber	Ex-Situ
L**	43	85	40.0	Climatic Chamber	Ex-Situ
M**	51	55	39.6	Climatic Chamber	Ex-Situ
N**	67	25	39.6	Climatic Chamber	Ex-Situ
O	73	55	60.2	Climatic Chamber	Ex-Situ

5.2. Results

In this section, all the results of this work are reported, starting from the preliminary characterization of the substrates, followed by the adhesion results in ambient, in-situ, and ex-situ subsequently.

5.2.1. Analysis of the Substrates

Before polymer deposition, the SiN_x substrates with varying stoichiometry and surface treatments were characterized using complementary analytical techniques to assess their morphological and chemical status.

Atomic Force Microscopy (AFM) was employed to quantify surface roughness, providing insight into the topographical features that could influence mechanical interlocking and effective contact area at the polymer/inorganic interface. Figure 5.3 reports reference images for the two as-deposited samples.

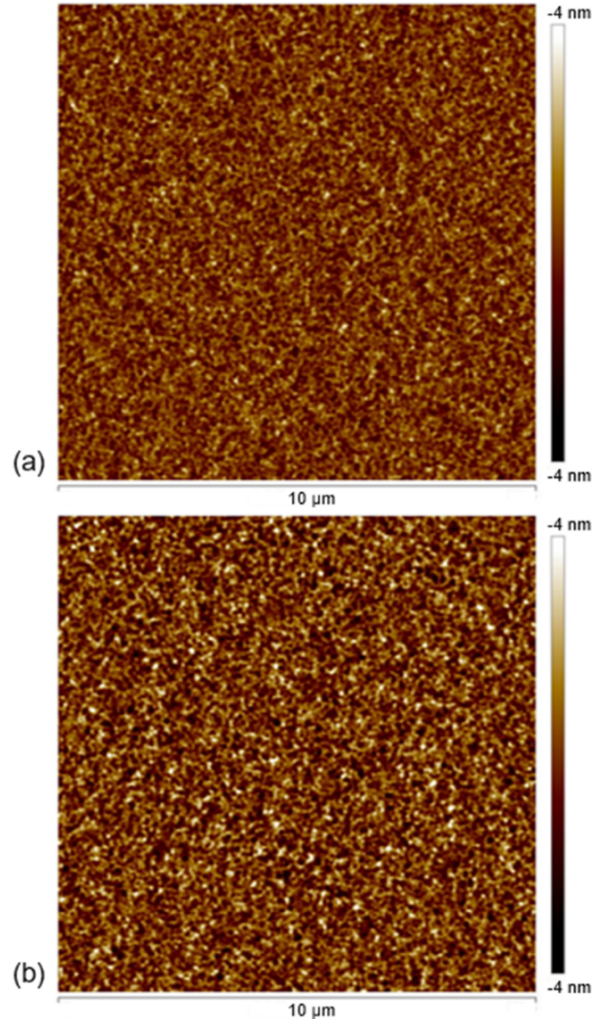


Figure 5.3: front view of the standard and silicon rich SiN_x as-deposited substrates analyzed by AFM.

The values of the mean square roughness (R_q), reported for all samples in Table 5.3, revealed a clear distinction between the standard stoichiometric variants SiN_x and the silicon-rich variants, with the latter exhibiting significantly higher roughness, with an increase exceeding 40%. This increase in surface roughness is a direct consequence of the altered film microstructure associated with excess silicon content during PECVD deposition.

Another distinct difference is noted in the impact of plasma treatments: while CF_4 plasma does not affect the roughness of the substrates, O_2 plasma effectively reduces this value by approximately 14% across both standard and SiRich substrates, suggesting a smoothing effect likely related to surface oxidation and reorganization. Indeed, in the literature, SiO_2 is known to have a rougher surface than SiN films [197], further demonstrating the

Table 5.3: AFM analysis results of each substrate by the values of root mean square roughness (R_q) and maximum peak-valley depth (R_{max}).

Sample	R_q (nm)	R_{max} (nm)
St_Asd	0.77	7.18
SR_Asd	1.12	10.1
St_ CF_4	0.78	7.88
SR_ CF_4	1.12	11.0
St_ O_2	0.66	6.84
SR_ O_2	0.97	8.63

probable oxidation of the substrate surface after plasma treatment with oxygen.

The chemical composition and surface chemistry were investigated through XPS coupled with argon sputtering for depth profiling, complemented by TEM/EDX analysis. The results of the XPS characterization are reported in Figure 5.4.

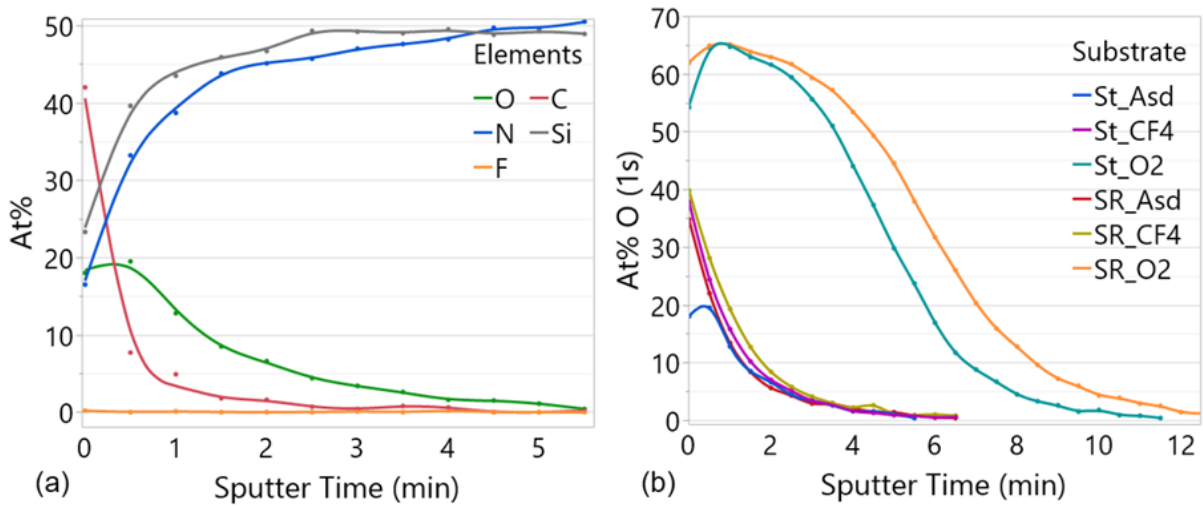


Figure 5.4: XPS analysis reported for the St_Asd substrate used as reference (a), and for oxygen content in all substrates (b).

The results of the analysis for the St_Asd substrate are shown in Figure 5.4 (a) as a reference for all other substrates investigated. Results confirmed the nominal stoichiometry of the substrates, with the standard SiN exhibiting a silicon-to-nitrogen atomic ratio close to 0.80 and the silicon-rich substrates near 1.05. Notably, the observed oxygen content for the two samples treated with O_2 plasma results to be strongly higher near their surface, while, even in this case, CF_4 plasma treated samples shows no clear differences with the two as-deposited ones. The presence of fluorine has also been searched in all samples, but never found higher than 2 At%, even for samples treated with plasma CF_4 . Lastly,

both plasma treatments were observed to lead to a reduction in carbon content near the interface, probably as a result of a “cleaning” effect of the treatments.

TEM/EDX analyzes corroborated these findings, revealing the formation of a distinct silicon oxide layer on O_2 plasma-treated substrates. This oxide layer is known to be rich in reactive silanol (Si–OH) groups, which enhance chemical affinity and bonding potential with the PBO polymer during curing [198–201]. In contrast, plasma treatment CF_4 resulted in only minor increases in surface oxygen and no evidence of a continuous oxide film, suggesting that its enhancement in adhesion arises primarily from surface cleaning effects rather than oxidation. The result related to the sample of St_ O_2 is reported in Figure 5.5.

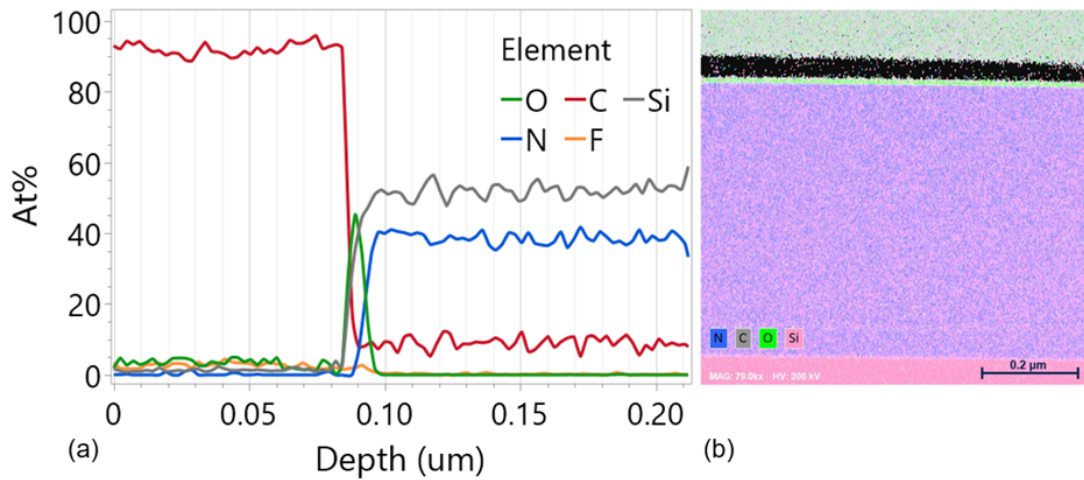


Figure 5.5: surface EDX signals of the St_ O_2 substrate (a) and related cross-section TEM image (b). Formation of an oxidized layer on the substrate interface is deductible by the oxide peak at the interface (revealed by rapid decrease of C content and increase of Si and N content) and directly visible on the TEM image as a green line.

Moreover, evaluating the full width half maximum (FWHM) of the oxygen-related peak in EDX profiling a qualitative thickness of the oxidized film has been evaluated, equal to 10~nm for the sample St_ O_2 and 14 nm for the SR_ O_2 , indicating a stronger plasma oxidation process on the silicon-rich samples than on the standard one.

In summary, the surface characterization established that the treatment of oxygen plasma creates a chemically active, oxidized surface layer with reduced roughness that should also promote chemical bonding with PBO because of the increased presence of silanol groups. In contrast, plasma treatment CF_4 maintains surface roughness, but enhances adhesion primarily through surface cleaning and modification of chemical groups. These distinct

surface states set the stage for subsequent adhesion analyzes and provide a mechanistic basis for interpreting differences in polymer interface performance.

In summary, the main results of this section are as follows.

- Surface roughness increases by 40% from standard to silicon-rich SiN_x , while between the two plasmas only O_2 has an impact reducing it by 14%.
- O_2 plasma modifies the surface chemistry of both SiN_x samples by inducing the creation of a thin oxidized film (most probably SiO_2).
- CF_4 plasma does not lead to clear variations in roughness or surface chemistry, but only operates a cleaning effect removing the previous C presence on the surface.

5.2.2. Adhesion under Standard Ambient Conditions

Initial adhesion measurements were conducted under standard laboratory ambient conditions to establish baseline values for the critical delamination load of the PBO films on the various silicon nitride substrates. These tests provided a reference framework for evaluating the effects of substrate stoichiometry, surface treatments, and subsequent environmental exposures. All results are reported in Figure 5.6.

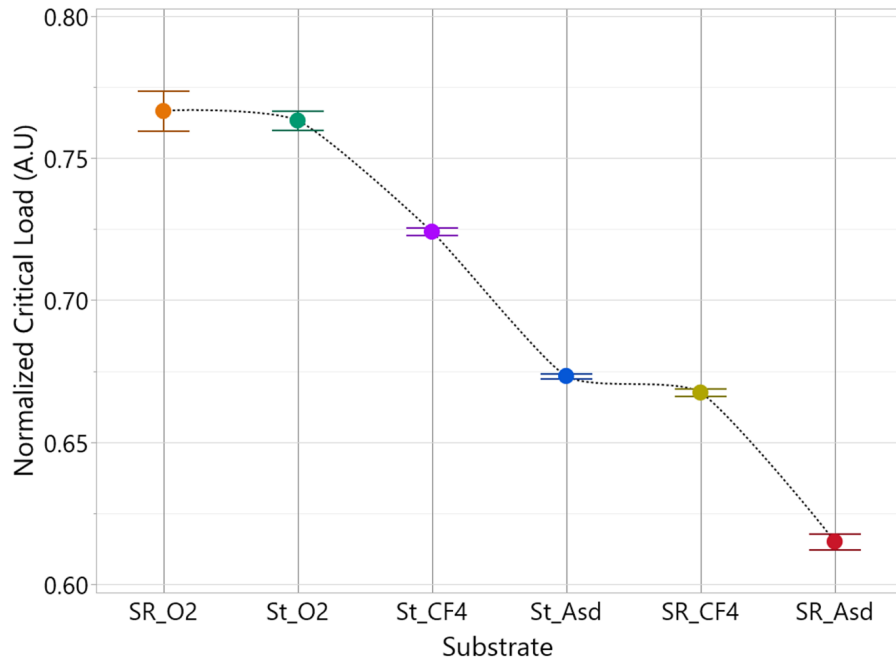


Figure 5.6: critical load of the six samples obtained by nanoindentation testing in ambient conditions. The dotted line connecting the results is placed to help visualize the differences between the various samples.

The results revealed a clear ranking in adhesion performance across the samples. In particular, the as-deposited standard SiN substrate (St_Asd sample) exhibited approximately 10% higher critical delamination load compared to the as-deposited silicon-rich SiN substrate (SR_Asd sample), despite the significantly higher surface roughness of the latter. This suggests that factors beyond surface topography, such as chemical composition and surface energy, play a dominant role in governing initial adhesion strength. An almost identical variation is observed between the two samples treated with CF_4 plasma - St_ CF_4 and SR_ CF_4 - but in this case shifted toward a higher critical load of approximately 8%. This result further indicates the low impact on the chemical and morphological properties of the samples, while apportioning an identical increase in adhesion to PBO most probably due to its surface cleaning effect from the presence of organic contaminants and other weakly bound species that could interfere with the PBO curing process and interfacial bonding. The effect of O_2 plasma is, instead, different, as reported in Table 5.4.

Table 5.4: Percentage increment in critical delamination load of as-deposited substrates after plasma treatments.

Initial Substrate	CF_4 Plasma	O_2 Plasma
Standard	8%	13%
Si-Rich	8%	24%

Indeed, oxygen plasma treatment produced the most pronounced enhancement, increasing the critical delamination load by approximately 13% on the stoichiometric substrate and by 24% on the silicon-rich substrate. This substantial improvement is attributed to the formation of the thin layer of silicon oxide, which promotes stronger chemical bonding and improved wettability with the PBO film [199–201]. As a result, the adhesion strength with PBO of the two substrates after O_2 plasma treatment converged, effectively equalizing their interfacial strength. This result also indicates that the thickness of the oxidized film does not affect the adhesion with the upper-lying polymer.

These findings underscore the interplay between substrate chemistry and surface morphology in determining adhesion quality. Although increased surface roughness in silicon-rich films might be expected to enhance mechanical interlocking, the lower initial adhesion observed suggests that chemical affinity and surface energy are more critical factors in this system.

In summary, the main results of this section are as follows.

- Stoichiometric SiN substrates inherently provide stronger adhesion to PBO than silicon-rich variants.

- CF_4 plasma treatment offers moderate adhesion enhancement through surface cleaning effects.
- Oxygen plasma treatment significantly improves adhesion by chemically modifying the substrate surface, promoting stronger interfacial bonding, and equalizing adhesion performance across different substrate stoichiometries.

5.2.3. Dew Point as a Univocal Parameter for Environment Severity

In the characterization of polymer/inorganic interface adhesion under environmental stress, it is common practice to evaluate the effects of temperature (T) and relative humidity (RH) independently or in a fixed combination. However, this approach often requires testing multiple points with either fixed temperature and varying humidity or fixed humidity and varying temperature, which complicates direct comparison of results and may obscure the true environmental impact on adhesion.

To overcome this limitation, the temperature of the dew point (T_{DP}) can be used as a univocal parameter that uniquely characterizes the severity of the ambient environment by combining the effects of temperature and humidity into a single physical meaningful metric. To ensure that this approach did not lead to incorrect conclusions, all standard samples (St_Asd, St_ CF_4 , and St_ O_2) were tested at points in the environment with the same T_{DP} but different T and RH; specifically, points G, C, H and F, which present a dew point of approximately 42°C. It should be noted that between the two most external points, i.e., point F (T=75°C and RH=21%) and point G (T=49°C and RH=75%), the difference in T and RH is 27°C and 53%, respectively.

As in the rest of the indentation analysis, also for this study, each sample was tested with six indentation for each environment. Then each result was analyzed with a statistical approach using the JMP software to evaluate the impact of the parameters T and RH on the variability of the obtained load of delamination. The analysis is reported in Figure 5.7.

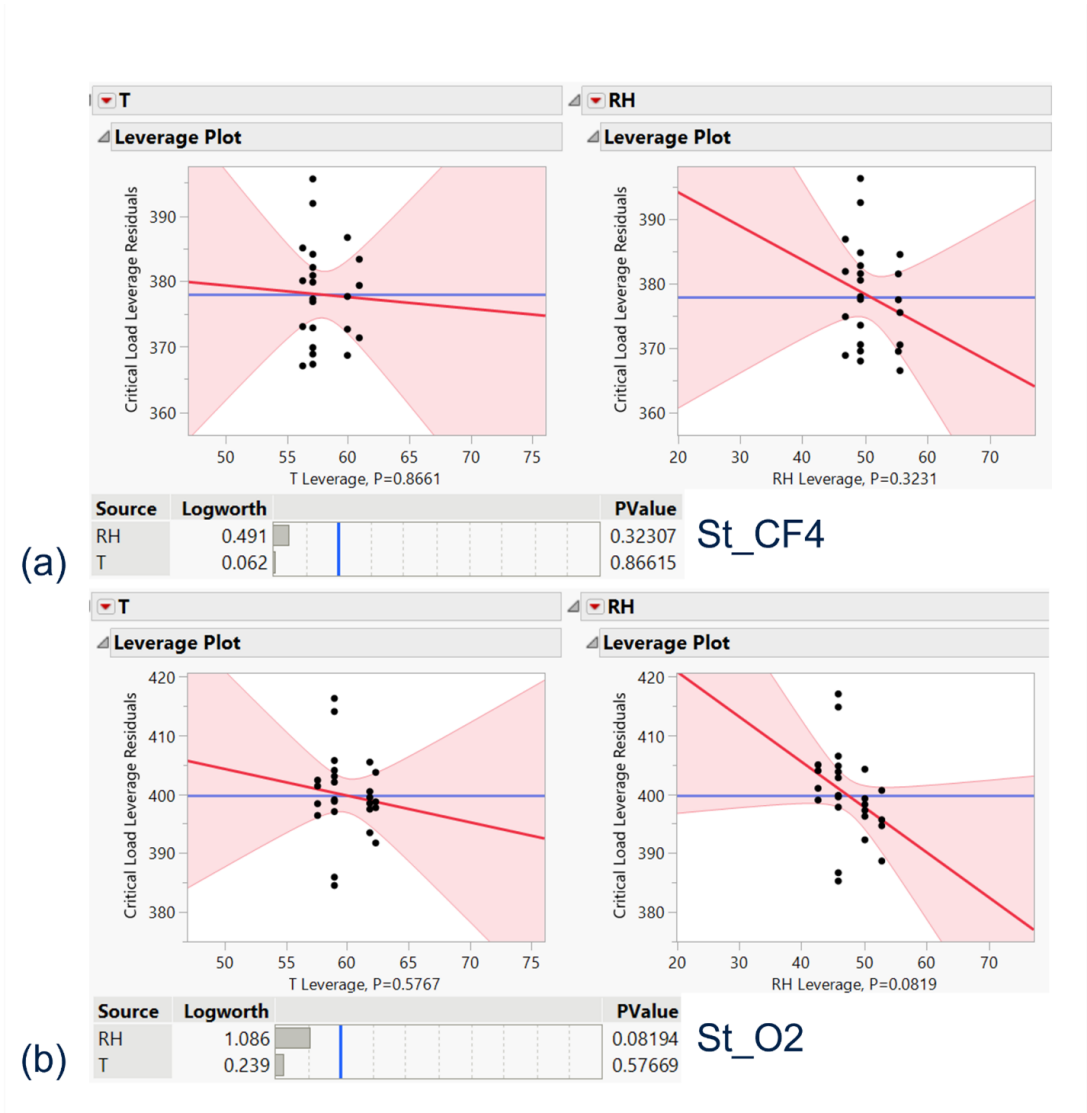


Figure 5.7: results of the statistical analysis of indentations for points G, C, H, and F ($T_{DP} \sim 42^{\circ}\text{C}$). The analysis shows that, at a fixed T_{DP} , the parameters T and RH, with a delta of 27°C and 53% respectively at the four points analyzed, do not affect the delamination loads resulting from the indentations. In other words, maintaining a constant T_{DP} , the critical load remains unvaried even for different T and RH values of the environment.

The statistical analysis presented in the figure evaluates the individual contributions of temperature and relative humidity to the critical delamination load of PBO/SiN interfaces at a constant dew point of approximately 42°C . Using leverage plots and regression analysis performed with JMP software [202], the study examined whether T or RH significantly influence adhesion independently.

A steep slope of the red line and a P value below 0.05 indicate a significant effect of the variable on the critical residual load.

Key statistical indicators include:

- **Leverage Plot:** Visualizes the influence of each variable on the residuals of the regression model, helping to identify trends and outliers. Each point represents an experimental data point, while the red line indicates the trend of the relationship between the variable considered and the residual load, considering the effect of the other variables. The pink area highlights the regression confidence interval, while the blue line represents the mean of the residuals.
- **P-Value:** A measure of the probability that the observed effect has random bases. A P-value below a chosen significance threshold (commonly 0.05) indicates a statistically significant effect.

A pronounced slope of the red line and a P value below 0.05 indicate a significant effect of the variable on the residual critical load. The results show that neither temperature nor relative humidity alone achieved statistical significance (no P-value < 0.05), indicating that within the range tested at the constant dew point, these variables do not independently explain the adhesion variation. Thus, these results show that the dew point can be used as a discriminant to evaluate the effect of the environment on adhesion.

However, relative humidity showed a stronger trend than temperature, particularly for the stoichiometric stoichiometric treated with oxygen plasma SiN_x , likely due to a broader experimental range in RH compared to T, specifically a range of 53% for ΔRH compared to a range of 27°C for ΔT . This outcome suggests that evaluating adhesion effects solely by varying T or RH could be necessary when testing points with strongly different values, while for restricted ranges - like those used in this work - using the dew point as the unique variable for discriminating environments is an effective strategy.

Adopting the dew point as the principal parameter for environmental severity leads to the following advantages.

- **Unified Environmental Descriptor:** T_{DP} inherently accounts for the combined influence of temperature and humidity, reflecting the actual moisture content that affects the polymer film and the interface.
- **Simplified Experimental Design:** Enables testing across a continuous range of environmental severities without the need to fix one variable while varying the other, reducing experimental complexity.

- **Improved Data Comparability:** Facilitates direct comparison of adhesion results obtained under different temperature and humidity combinations that yield the same dew point, enhancing the robustness of conclusions.

Based on these considerations and supported by statistical analysis, the dew point will be employed as the univocal parameter to compare the adhesion performance of PBO/SiN interfaces across all tested environments. This approach ensures that variations in adhesion are interpreted in the context of absolute environmental moisture severity, providing a more accurate and physically relevant understanding of interface behavior under operational conditions.

5.2.4. Adhesion under Controlled In-Situ Environmental Conditions

To investigate the influence of environmental factors on the adhesion between PBO and SiN_x substrates, nanoindentation tests were performed under carefully controlled in-situ conditions using the xSol-Humidity environmental control module integrated with the nanoindenter instrumentation. This setup enabled the real-time modulation of temperature and relative humidity, allowing the assessment of adhesion behavior at various dew points (T_{DP}) that simulate a range of operational environments. The set of all load-displacement curves of the six samples is reported in Figure 5.8. As stated in Section 5.1.3, all samples were tested with six subsequent indentations for every selected environment and the results were averaged to obtain a single load-indentation curve. Thus, the solid lines shown in the figure are the mean curves of the six indentations, with each blurred band indicating the relative standard error. Moreover, for a clearer view of the trends, only the test results from three environments were shown, specifically environments A ($T_{DP} \sim 17^\circ\text{C}$), C ($T_{DP} \sim 43^\circ\text{C}$) and E ($T_{DP} \sim 69^\circ\text{C}$). Finally, the indentation load on the ordinate axes have been reported to be normalized to the maximum load of indentation, equal to 600 mN.

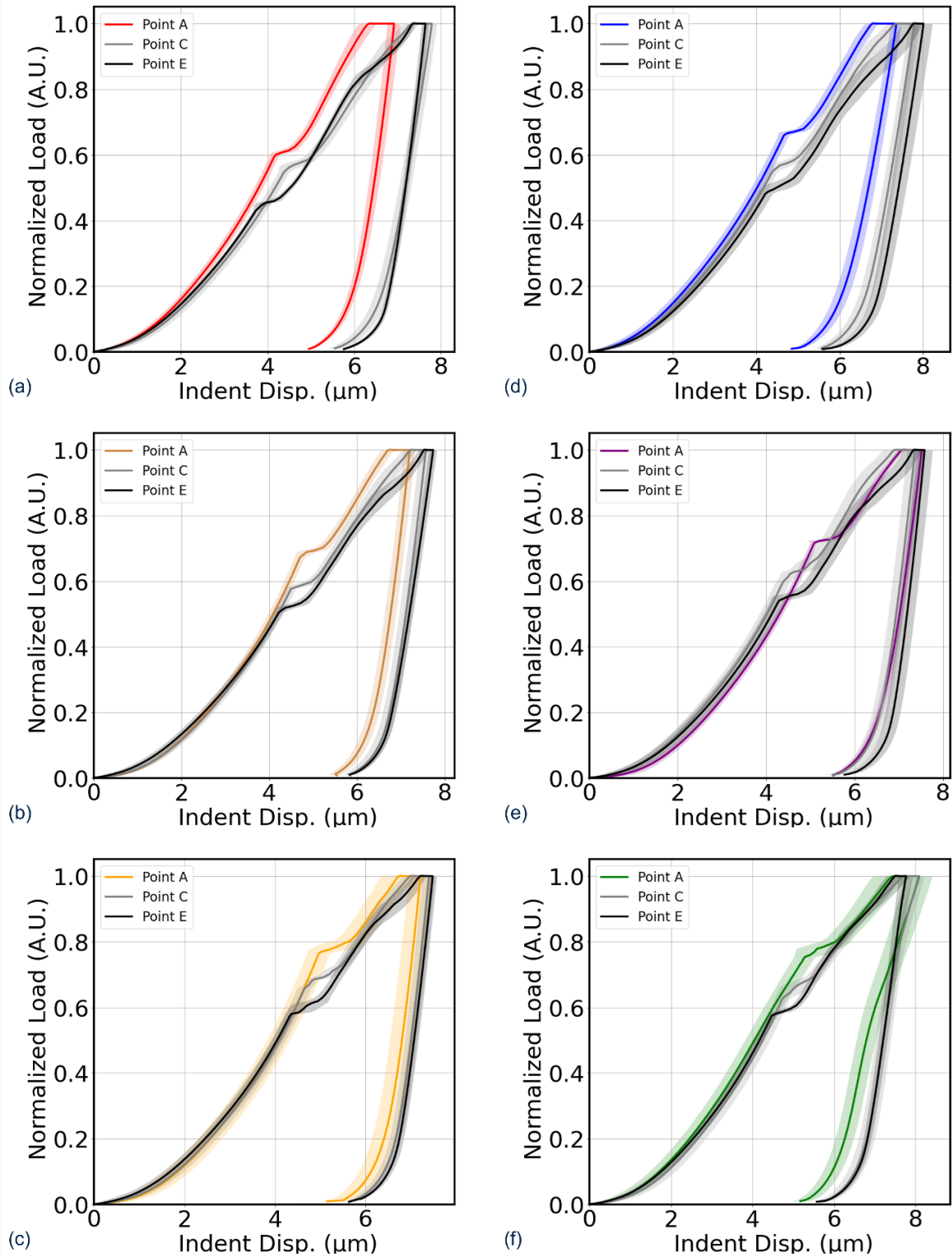


Figure 5.8: load-displacement curves for the samples SR_Asd (a), SR_CF4 (b), SR_O2 (c), St_Asd (d), St_CF4 (e) and St_O2 (f) for a limited group of all tested environments, specifically the points A ($T_{DP} \sim 17^\circ\text{C}$), C ($T_{DP} \sim 43^\circ\text{C}$) and E ($T_{DP} \sim 69^\circ\text{C}$). Each curve is obtained by averaging the results of 6 indentations; the solid lines represent the average curves, while the transparent bands show the corresponding standard deviations.

The load–penetration curves obtained during these in-situ tests exhibited consistent loading slopes in all tested conditions, indicating that the mechanical compliance of the PBO film remained essentially unchanged despite variations in ambient temperature and humidity. This observation validates the direct comparison of critical delamination loads - observed by the decay events of each curve - measured under different environmental conditions, as changes in adhesion can be attributed primarily to interfacial phenomena rather than bulk polymer compliance. Figure 5.9 shows the load-displacement curves of all six samples in ambient E ($T=75^{\circ}\text{C}$ and $\text{RH}=75\%$, $T_{DP} \sim 69^{\circ}\text{C}$), also demonstrating the same observation between different samples.

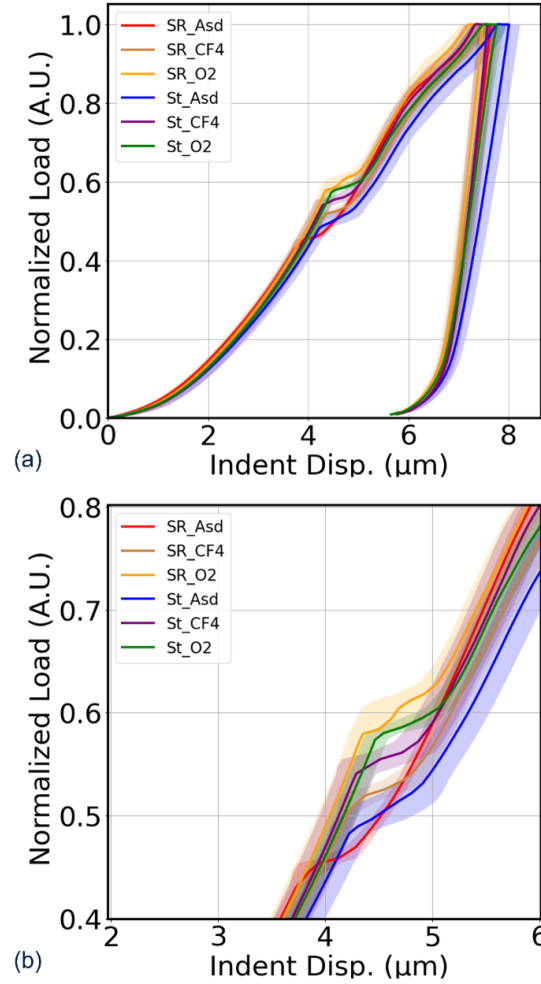


Figure 5.9: load-displacement curves for all six samples in point E ($T_{DP}\sim 69^{\circ}\text{C}$) (a) and a zoom of the results in the area showing the decay events (b) from which the critical load of delamination is extracted.

Finally, Figure 5.10 reports the critical loads evaluated for all six samples in ambient conditions and in environments C and E. A zoom-in in the zone of the decay events is also reported to allow for better data visualization.

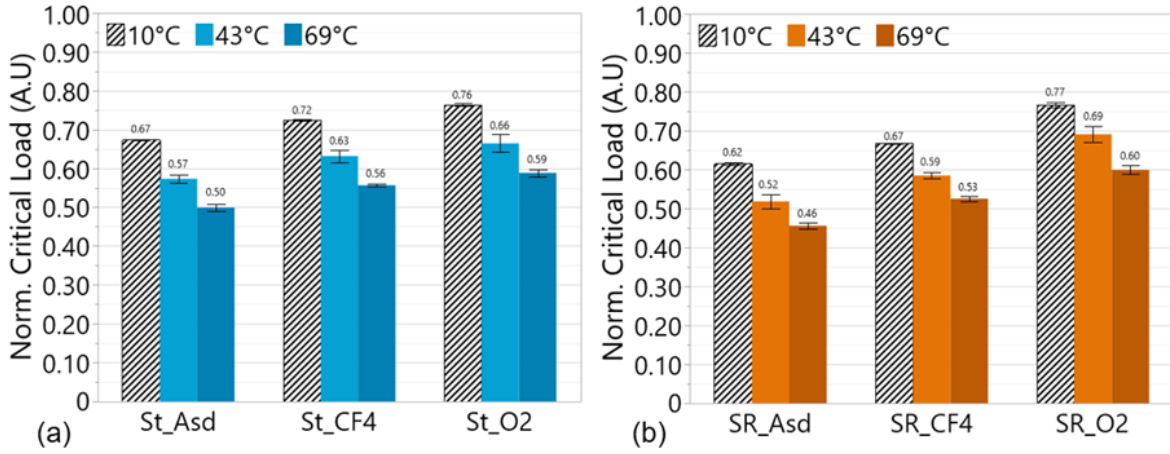


Figure 5.10: critical load results for standard SiN samples (a) and silicon rich SiN samples (b). While the dashed bar indicates the value obtained in ambient condition (prior to any environmental control), the colored bar reports the results in ambient C ($T_{DP} \sim 43^\circ\text{C}$) and ambient E ($T_{DP} \sim 69^\circ\text{C}$) in the tested range. The mean value of the critical delamination load is reported above each bar.

Analyzing all obtained results, adhesion measurements revealed three key trends with increasing environmental severity (higher dew points):

1. **Preservation of Adhesion Ranking:** the relative adhesion performance observed under ambient conditions persisted in the range of tested dew points, maintaining the same ranking observed under ambient conditions, as reported in Figure 5.6. Samples treated with oxygen plasma on both stoichiometric and silicon-rich SiN substrates consistently exhibited the highest critical delamination loads, while as-deposited silicon-rich substrates showed the lowest adhesion. This indicates that substrate stoichiometry and surface treatment effects remain significant even under harsh environmental conditions.
2. **Systematic Adhesion Degradation:** all samples experienced a reduction in critical delamination load of approximately 25% when exposed to the most severe in-situ environment tested, i.e., ambient E ($T_{DP} \sim 69^\circ\text{C}$), with respect to their initial values under ambient conditions. This uniform decrease across different substrates and treatments highlights the dominant role of moisture and temperature in weakening

the polymer/inorganic interface.

3. **Independence from Substrate Modification:** Despite the absolute adhesion improvements conferred by plasma treatments, the rate of adhesion loss with increasing dew point was similar for all samples. This suggests that while surface treatments improve baseline adhesion, they do not significantly mitigate the detrimental effects of moisture absorption and thermal stress on interface integrity.

Further analysis of the evolution of critical load as a function of the dew point revealed a nonlinear behavior characterized by an initial rapid decline at low dew points, followed by a plateau at intermediate values and a renewed decrease at higher dew points. This complex trend suggests competing mechanisms influencing adhesion, such as moisture-induced swelling, polymer plasticization, and possible interfacial chemical changes. The results of such an analysis are reported in Figure 5.11 for all samples.

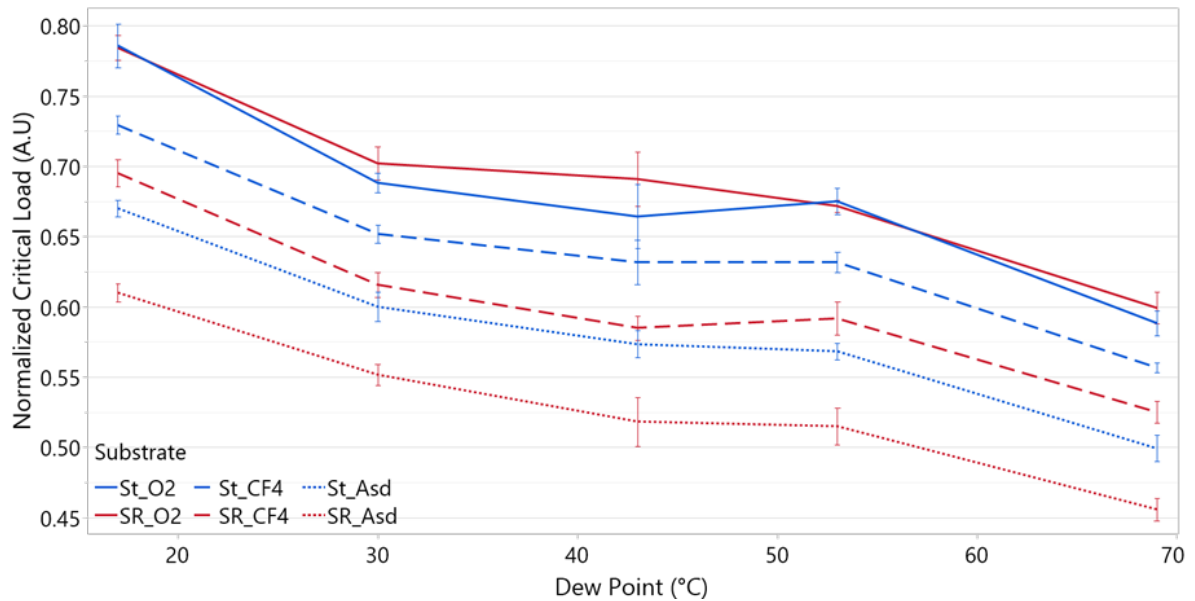


Figure 5.11: the evolution of the critical load with increasing dew point tested in-situ is reported for all the six samples with plasma treatment. In all samples a similar trend is observed, with rapid critical load decline for low and high dew point and a plateau for medium dew point with respect to the investigated range.

Finally, the extra points F and G - tested only for the Standard samples - provided a third critical load value, which allowed one to observe the trend of PBO adhesiveness with varying T and constant RH (points B, G, E) and varying RH and constant T (points F, D, E). Such results are reported in Figure 5.12.

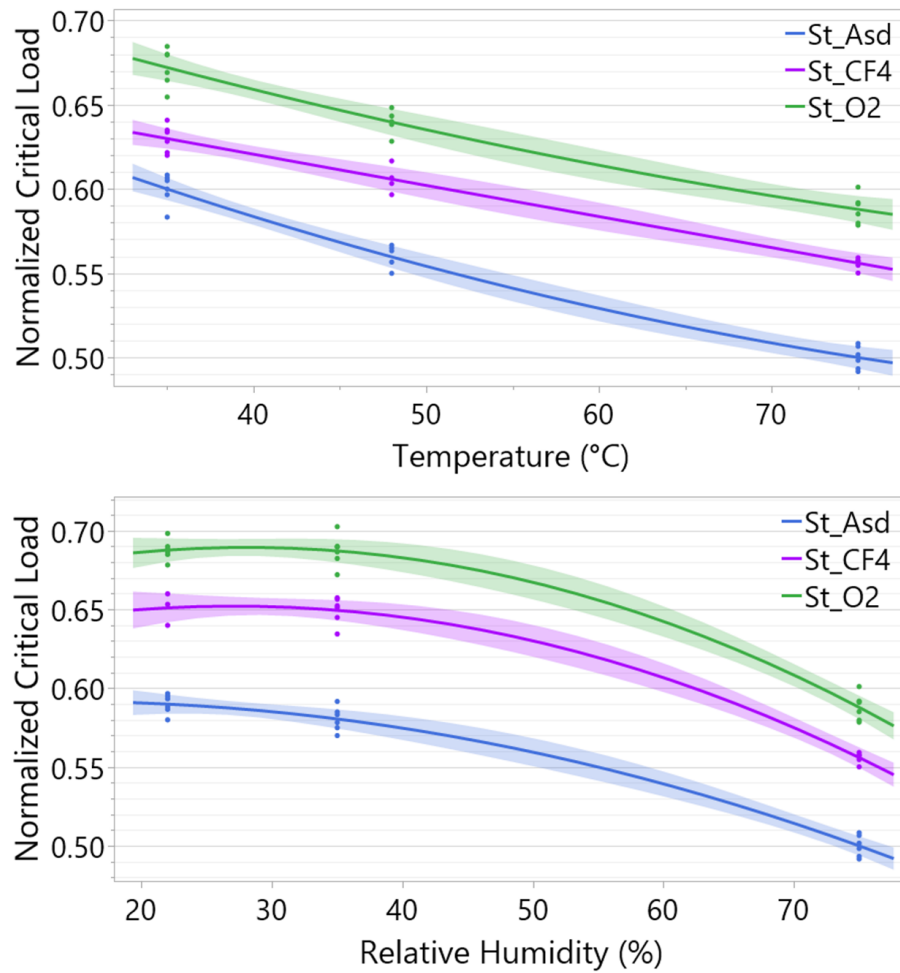


Figure 5.12: by analyzing the results at environmental points with constant RH (B, G, E) - at the top - and constant T (F, D, E) - at the bottom - it was possible to evaluate the trend of PBO adhesiveness in the Standard samples as one of the two environmental parameters, T and RH, varied. In the first case (constant RH), a linear trend of adhesiveness with increasing temperature is observed, while in the second case (constant T), a non-linear trend is observed with increasing relative humidity. Interestingly, in the case of variable relative humidity, the critical delamination loads seem to remain almost unchanged as the RH varies from 22% to 35%.

Given the availability of only three experimental points for each sample, in this case the results of each individual indentation were reported directly. First, the results obtained show behavior almost independent of the type of substrate, which affects the baseline adhesion with PBO, but not its variability with changes in the environment, as previously observed with varying dew point. Furthermore, the results show an almost linear trend with respect to temperature dependence, whereas in the case of variable RH a nonlinear

trend is observed. Similar results have also been reported in the literature [81]. Gupta et al. for example show a very similar trend in adhesion interface strength depending on the temperature, while in the case of RH the reported behavior has a different concavity. However, in the study by Gupta et al., the RH varies from 50% to 70%, while the first two points evaluated in this study are lower relative humidity. It is therefore possible that, similar to what has been observed for the behavior of the critical load with the variation of the dew point, in the case of RH the adhesiveness remains more or less constant up to a critical range, beyond which a decline is observed. The work of Gupta et al., carried out on a polyimide - which therefore has rheological properties not very different from those of PBO - could indicate that in our case this critical range could also be around 50% to 60% RH.

These in-situ results provide valuable information on the dynamic response of the PBO / SiN_x interfaces under realistic environmental stresses. The observed adhesion degradation underscores the critical importance of controlling moisture exposure in device packaging and highlights the limitations of surface treatments in preventing the effects of environmental aging.

In summary, the in-situ adhesion testing demonstrated that:

- The mechanical compliance of PBO remains stable under varying temperature and humidity conditions.
- The adhesion strength decreases significantly - by up to 25% - at high dew points regardless of substrate stoichiometry or surface treatment.
- Plasma treatments improve initial adhesion but do not alter the susceptibility of the interface to moisture-induced degradation.
- The loss of adhesion with increasing dew point follows a nonlinear trend, indicating complex interfacial phenomena.

5.2.5. Adhesion after Ex-Situ Aging Treatments

To complement in-situ adhesion measurements and evaluate the effects of prolonged environmental exposure, ex-situ aging tests were conducted on the PBO/ SiN_x samples. These samples were subjected to controlled temperature and humidity conditions in a climatic chamber for a duration of 2 and 7 days, simulating extended operational aging.

Following aging, the samples were removed from the chamber and immediately tested via nanoindentation under ambient laboratory conditions to assess the residual adhesion

strength. A comparison between in-situ and ex-situ results (for both dwell times) is reported in Figure 5.13 for the samples treated with oxygen plasma, i.e., St_O₂ and SR_O₂ samples.

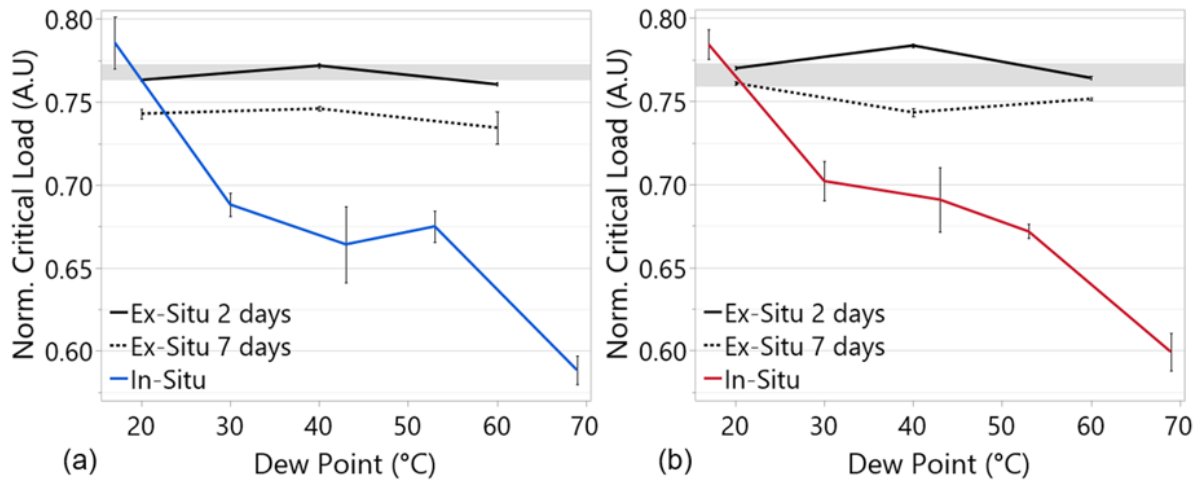


Figure 5.13: comparison between the critical load evolution with increasing dew point between in-situ and ex-situ results for the standard (a) and silicon rich (b) SiN_x samples treated with oxygen plasma. Initial values taken in ambient condition are reported in gray bands which represent the standard deviation of the measurements.

The ex-situ results revealed a markedly different adhesion behavior in comparison to the in-situ tests. After 2 days of aging, the critical delamination loads measured in points I, M, and O of the environment (see Table 5.2) were largely restored to values comparable to those obtained prior to environmental exposure - reported in figure with a gray band indicating the standard error of the measurement - indicating a significant recovery of adhesion properties after removal from the humid environment. This suggests the plausible presence of short-term moisture-induced degradation of the interface, which would be predominantly reversible, likely due to desorption of absorbed water and relaxation of hygroscopic swelling stresses within the polymer layer.

However, samples aged 7 days exhibited a small but measurable reduction in adhesion strength, with an average decrease of approximately 4% relative to the values obtained prior to aging treatments. Importantly, this reduction appeared to be largely independent of specific environmental conditions (temperature and humidity) during aging, implying the onset of irreversible degradation mechanisms at the interface over longer timescales. Potential contributors to this permanent adhesion loss could include chemical hydrolysis, oxidation reactions, or the formation of weak boundary layers catalyzed by moisture and

environmental contaminants already present in the climatic chamber, used for a variegated group of samples in the context of the company R&D laboratory.

Comparing ex-situ and in-situ data highlights a critical distinction: while in-situ testing captures real-time adhesion degradation under environmental stress, ex-situ measurements may overestimate adhesion recovery by missing transient interface weakening present during exposure. The rapid recovery observed after short aging periods underscores the dynamic nature of moisture-related adhesion phenomena and suggests that ex-situ tests alone may not fully represent interface performance under actual operating conditions.

These findings have significant implications for the qualification and reliability assessment of polymer/inorganic interfaces in semiconductor packaging. They emphasize the necessity of employing in-situ testing methodologies to capture the full spectrum of adhesion behavior, or to necessarily introduce ambient cycling during the aging treatments to simulate the transient swelling effect of the polymeric coatings under harsh environmental conditions.

In summary, the ex-situ aging study demonstrated that:

- Adhesion recovers substantially after short-term (2-day) aging, indicating reversible effects of moisture.
- Prolonged aging (7 days) leads to a modest but permanent reduction in adhesion, regardless of the severity of the environment.
- Ex-situ testing may underestimate the real-time adhesion loss that occurs under humid and thermal stress.
- Irreversible degradation mechanisms likely involve chemical and structural changes at the interface.

5.2.6. Discussion

5.2.6.1. Effect of Surface Pre-Treatment on Adhesion

The surface preparation of SiN_x substrates prior to PBO deposition plays a critical role in determining the quality of the polymer/inorganic interface. In this study, two distinct plasma treatments - oxygen (O_2) and carbon tetrafluoride (CF_4) - were applied to stoichiometric and silicon-rich SiN films to investigate their effects on surface chemistry, morphology, and ultimately adhesion performance. To discuss such aspects, it is helpful to observe the monomeric structure of PBO as-deposited and after the curing process, as

shown in Figure 5.14.

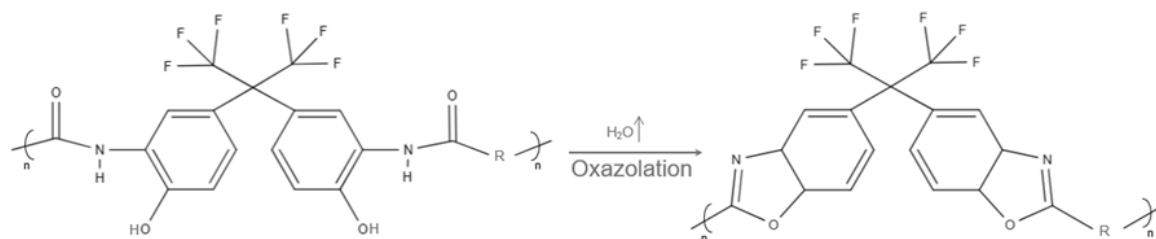


Figure 5.14: schematic representation of the analyzed PBO monomer before and after the curing step. As de-hydrolysis occurs the open carbon rings close up forming oxazole rings through the so-called oxazolation process.

After being deposited through spin-coating, each monomer component of the PBO will show two closed aromatic cycles, each with an amide group that forms a bond with the next monomer through an additive radical group and is linked to each other by phosphate groups that give the polymer its hydrophobic properties [76]. The specific additive radicals are not known due to privacy in the supplier's recipe, but it is known that they are intermittently aromatic and aliphatic groups and therefore likely serve to stiffen and increase the packing density of the chains and lower the glass transition temperature (T_g) and the conductivity of the polymer, respectively [203]. The as-deposited polymer is photosensitive and can be used in photolithography processes. After curing for one hour at 350°C, the polymer loses its photosensitivity due to the evaporation of solvents and the oxazolation process, in which the amide group and the alcohol attached to each aromatic ring react with each other to create a new oxazole ring. Before this structural modification, it is plausible that the OH radicals present in the polymer, known to be highly active [192], interact with the substrate.

O₂ plasma treatment was found to significantly enhance the adhesion of PBO in both substrate types, most likely due to the formation of a thin oxidized layer on the SiN_x surface identified by XPS and TEM/EDX analyzes. This chemically active layer would present several silanols (Si-OH), which are highly reactive and hydrophilic, facilitating strong chemical interactions with the PBO precursor prior to the curing process, specifically with the aforementioned OH radicals. These two groups, i.e., silanols and alcohols, could undergo de-hydrolyzation processes to form a covalent bonding through an oxygen atom that produces a water molecule as a byproduct [192]. In addition, the oxide layer increases surface energy and improves wettability, promoting better polymer spread and

intimate contact at the interface [198].

CF_4 plasma treatment also improved adhesion, but to a lesser extent ($\sim 8\%$ increase) and with a different underlying mechanism. Unlike O_2 plasma, the beneficial effect of CF_4 treatment is hypothesized to stem from surface cleaning, effectively removing organic contaminants and weakly bound species that could interfere with the curing and bonding of PBO. This cleaning effect likely reduces the presence of parasitic bonds or interfacial layers that compromise mechanical integrity [181]. By improving the efficiency of the PBO curing process and promoting a cleaner interface, CF_4 plasma treatment contributes to improvement of adhesion regardless of substrate stoichiometry. The uniform increase in adhesion observed across both stoichiometric and silicon-rich substrates supports this interpretation.

Finally, AFM measurements revealed that silicon-rich SiN substrates exhibit a significantly higher surface roughness compared to stoichiometric ones. However, contrary to some reports in the literature suggesting that roughness can enhance adhesion through mechanical interlocking [191], the as-deposited silicon-rich substrates showed lower adhesion than stoichiometric substrates. This indicates that surface chemistry and chemical affinity dominate over topographical effects in this system. Furthermore, O_2 plasma treatment reduced surface roughness in both substrate types, likely due to the formation of a smoother silicon oxide layer. This reduction in roughness coincides with improved adhesion, reinforcing the notion that chemical surface modifications rather than increased roughness are key drivers of enhanced interfacial bonding.

In summary, the distinct effects of O_2 and CF_4 plasma treatments underscore the importance of tailoring surface chemistry to optimize polymer/inorganic adhesion. However, it is important to note that, while these treatments affect the initial adhesion of the PBO, none of them led to a change in the way the adhesion of the coating deteriorates as the severity of the environment increases. Therefore, substrate surface treatments influence the baseline adhesion, but not its durability, under varying environmental conditions.

5.2.6.2. Influence of the Environment

Moisture absorption emerges as a critical factor affecting the adhesion performance of PBO films on silicon nitride substrates, profoundly influencing the integrity and durability of the polymer/inorganic interface. The experiments conducted in this study, which combine in-situ and ex-situ adhesion testing under controlled environmental conditions, provide valuable insights into the mechanisms by which temperature and humidity degrade interfacial bonding.

In-situ nanoindentation tests performed under varying temperature and relative humidity conditions revealed a consistent and significant decrease in the critical delamination load with increasing ambient dew point, reaching reductions of up to approximately 25% in the most severe environments tested. This uniform loss of adhesion across all samples, regardless of substrate stoichiometry or surface treatment, underscores the dominant role of moisture in weakening the interface. The observed adhesion degradation is mainly attributed to hygroscopic swelling of the polymeric PBO film. Moisture uptake causes volumetric expansion within the polymer matrix, inducing internal stresses that promote interfacial delamination. This swelling effect is well-documented in polymeric materials and is known to generate reversible mechanical stresses that compromise adhesion under humid conditions [80, 82, 204].

In contrast to the in-situ findings, ex-situ adhesion measurements after environmental aging exhibited a markedly different behavior. After short-term aging (2 days), adhesion largely recovered to near-initial levels upon removal from the humid environment, indicating that moisture-induced swelling and associated stress are reversible if exposure is limited. However, prolonged aging (7 days) led to a modest but persistent reduction in adhesion ($\sim 4\%$), suggesting the onset of irreversible degradation mechanisms. This partial recovery phenomenon implies that while short-term exposure to moisture primarily causes reversible physical effects - such as swelling and plasticization - extended exposure can initiate chemical degradation processes at the interface. Possible mechanisms include the hydrolysis of interfacial bonds, oxidation reactions, or the formation of weak boundary layers catalyzed by moisture and environmental contaminants, which permanently reduce the adhesion strength [184].

The observed reversible and irreversible adhesion changes align well with the existing literature on moisture-induced degradation in polymer/inorganic systems [204]. Hygroscopic swelling is recognized as a primary driver of temporary adhesion loss, with diffusion coefficients for water in thin PBO films suggesting that moisture penetration and desorption occur on timescales consistent with the recovery observed after 2 days of aging. Chemical degradation mechanisms, such as the hydrolysis of hydrogen bonds or covalent linkages at the interface, typically require longer exposure times and may be exacerbated by impurities or pollutants present during aging [82]. These processes weaken interfacial bonding and contribute to the permanent adhesion loss detected after extended aging periods, which would be justified by the fact that the climatic chamber used for this experiment - provided by the laboratories of STMicroelectronics - actually operates on a continuous cycle, taking large amounts of different samples entering and exiting the controlled environment, potentially leaving various impurities inside.

5.2.6.3. Implications for Industrial Interface Qualification

From the initial analyzes performed using four-point bending and pull-off tests, it was possible to observe that all six samples analyzed in the environment showed a lower bound of adhesion energy of $30 \sim \text{J/m}^2$, a fairly good value according to STMicroelectronics standards (although there is no precise reference value). However, the study was of interest to the company for the possibility of comparing the results among the six samples analyzed, providing interesting information on the dependence of adhesion on substrate surface treatments and the environment. Moreover, comparative analysis of in-situ and ex-situ adhesion testing under controlled environmental conditions highlights critical considerations for industrial process control and quality assurance.

In-situ adhesion testing, performed under real-time environmental control, offers a direct and dynamic assessment of interface behavior as devices experience fluctuating temperature and humidity. This approach captures transient and reversible degradation phenomena, such as moisture-induced swelling and plasticization, which are often overlooked or underestimated by conventional ex-situ aging tests. The observed 25% adhesion loss at high dew points during in-situ tests underscores the sensitivity of the interface to operational conditions and the necessity of evaluating adhesion within relevant environmental contexts.

In contrast, ex-situ testing after prolonged aging provides valuable insight into the long-term stability and irreversible degradation mechanisms affecting interface durability. However, the partial recovery of adhesion observed after short-term aging and the modest permanent loss after extended exposure suggest that ex-situ tests may overestimate interface robustness if not carefully interpreted. The discrepancy between in-situ and ex-situ results emphasizes the need for a balanced testing strategy that incorporates both methodologies to obtain a comprehensive understanding of interface performance. Alternatively, ex-situ tests would be necessary after dynamic aging treatments, with environmental cycles intensifying the detrimental effects of swelling and plasticization of the polymeric coatings. Hutchinson et al. [205] demonstrate the difference between adhesion analyzes of an organic film subjected to static aging and dynamic aging (thermal cycling), highlighting the greater effectiveness of dynamic aging in assessing the true resistance of the interface. The possibility of performing in-situ tests allows for obtaining adhesion results in a shorter time, but does not consider the time it takes the sample to maintain within the environment. Consequently, in any case, the two analysis techniques are complementary and cannot replace each other.

This study also demonstrates that surface pre-treatments, particularly oxygen plasma,

can substantially enhance baseline adhesion and standardize interface quality across different substrate stoichiometries, as already stated in the literature [198–200]. However, the limited effect of plasma treatments on mitigating moisture-induced adhesion degradation indicates that surface modification alone is insufficient to ensure long-term interface stability under humid conditions. Therefore, process engineers must consider additional protective strategies, such as encapsulation or barrier layers, to reduce moisture ingress and preserve adhesion integrity.

Based on the insights gained, several recommendations can be made for integrating adhesion qualification into semiconductor manufacturing workflows:

- **Incorporate In-Situ Environmental Testing:** where feasible, implement in-situ adhesion measurements under controlled temperature and humidity to monitor interface behavior under realistic operating conditions. This approach enables the early detection of moisture sensitivity and guides material and processes.
- **Inefficiency of Static Ex-Situ Aging:** static ex-situ aging tests failed to evaluate ambient-related degradation, demonstrating limitations in capturing transient effects. The implementation of dynamic ex-situ aging tests is most probably more effective, even if in this work it was not possible to test this kind of analysis.
- **Optimize Surface Treatments:** standardize plasma treatment protocols, particularly oxygen plasma, to enhance initial adhesion, removing surface pollutant, and reducing variability due to substrate stoichiometry.
- **Consider Moisture Barrier Solutions:** given the pronounced impact of moisture on adhesion, integrate effective encapsulation or barrier layers in device packaging to minimize environmental exposure and extend device lifetime.

5.3. Summary

This chapter has presented a comprehensive study on the adhesion behavior of polybenzoxazole (PBO) films deposited on silicon nitride substrates (SiN_x) with varying stoichiometry and surface treatments, evaluated under both in-situ and ex-situ environmental conditions. Although the lower bound of adhesion energy, identified using macro-mechanical analysis techniques (equal to $30 \sim J/m^2$), has been determined as a value sufficiently high for company standards, this research highlights the critical interplay between substrate chemistry, surface modification, and ambient factors such as temperature and humidity in determining interface durability, which is paramount for the reliability of semiconductor devices.

Key findings demonstrate that oxygen plasma treatment significantly enhances adhesion by forming a reactive silicon oxide layer rich in silanol groups, effectively equalizing adhesion across stoichiometric and silicon-rich substrates. Carbon tetrafluoride plasma also improves adhesion, primarily through surface cleaning effects, though to a lesser extent than oxygen plasma. These surface treatments modify the chemical affinity and wettability of the substrates, thereby strengthening interfacial bonding with the PBO layer.

Environmental control experiments reveal that moisture absorption critically degrades adhesion in a reversible manner during short-term exposure, as evidenced by substantial ($\sim 25\%$) adhesion loss under high dew point conditions during in-situ testing. Ex-situ aging tests indicate that adhesion recovers after short aging periods but experiences a small irreversible decline after extended exposure, suggesting the onset of chemical degradation mechanisms at the interface.

The comparative analysis of in-situ and ex-situ methodologies underscores the importance of incorporating real-time environmental testing in adhesion qualification to capture transient degradation phenomena that traditional aging tests may overlook. This dual approach provides a more accurate and comprehensive understanding of interface performance under operational stresses.

From an industrial perspective, these insights emphasize the need for optimizing surface treatments and environmental protections to ensure robust polymer/inorganic adhesion. The findings support the integration of in-situ adhesion testing into process control frameworks and highlight the limitations of relying solely on ex-situ aging for reliability assessment.

In conclusion, this work advances the understanding of the adhesion of the PBO/ SiN_x interface in semiconductor packaging, offering practical guidance for material selection, surface engineering, and environmental testing strategies aimed at enhancing the longevity and yield of the device.

Key Takeaways

- O_2 plasma treatment forms a reactive oxide layer that significantly improves and standardizes SiN substrates adhesiveness with PBO, while CF_4 plasma treatment enhances adhesion mainly by surface cleaning, with moderate improvements.
- Surface chemistry is the main parameter that affects adhesion, overcoming the effects of surface roughness.
- The dew point can be used as a univocal parameter to assess ambient severity, allowing

comparisons between environmental points with different temperatures and relative humidity at the same time as long as the delta between the ambient descriptors is limited.

- Ex-situ tests fail to capture the transient effect, as moisture absorption causes reversible adhesion loss under short-term exposure. The observed irreversible degradation with prolonged aging is related to contaminants in the climatic chamber.
- In-situ adhesion testing under controlled environments captures transient effects missed by ex-situ aging, showing strong losses of adhesiveness in the most severe environments tested.

6 | Conclusions

This doctoral research has tackled critical materials-related challenges that arise from the transition to 300 mm wafer-scale manufacturing of Insulated Gate Bipolar Transistors (IGBTs), focusing on three interconnected scales: wafer-level warpage, thin-film mechanical behavior, and polymer/inorganic interface adhesion. Combining advanced modeling techniques, rigorous experimental characterization, and controlled environmental testing, this work has provided valuable insight and practical tools aimed at improving device reliability and manufacturing yield.

At the wafer scale, the development of two complementary finite element modeling approaches - the Discrete Trenches Method (DTM) and the Homogenized Trenches Method (HTM) - has enabled accurate simulation of warpage induced by the complex trench patterns characteristic of IGBT devices. A key finding is that incorporating intrinsic residual stresses, experimentally separated from thermal stresses, is essential to achieve close agreement between the simulated and measured wafer bow. The DTM offers detailed local stress analysis, but is computationally intensive and limited to small wafer portions, whereas the HTM reduces computational demand by homogenizing patterned regions, allowing full-wafer simulations with realistic device footprints. Despite their strengths, both methods currently fail to capture warpage behavior during the final trench closure step, indicating a need for further refinement, possibly through dynamic process modeling and contact mechanics.

At the thin-film scale, the study of PECVD silicon nitride films (SiN_x) with varying silicon-to-nitrogen ratios has revealed that increasing silicon content leads to the spontaneous formation of amorphous silicon nanoparticles embedded within the matrix. These inclusions, confirmed by Raman, photoluminescence, and transmission electron microscopy, are similar in size and volume fraction across silicon-rich films, suggesting that they alone do not explain the observed mechanical property variations. Mechanical testing demonstrated a clear compositional dependence of residual stress, elastic modulus, hardness, and fracture toughness. In particular, a transition from tensile to compressive residual stress and a significant increase in fracture toughness is observed with increasing silicon

content between the two silicon-rich samples studied. The indentation-based fracture toughness measurement method developed by Xia et al. was effectively applied, overcoming substrate effects and providing reliable comparative data. These results underscore the importance of compositional and microstructural tuning for optimizing the mechanical robustness of SiN_x passivation layers.

At the interface scale, the adhesion between polybenzoxazole (PBO) polymer films and SiN_x substrates was systematically investigated under varying substrate stoichiometries, surface plasma treatments, and environmental conditions using both in-situ and ex-situ nanoindentation techniques. In particular, the dew point has been proven to be an effective parameter to describe univocally the characteristics of the environment in place of considering both the temperature and the relative humidity. Oxygen plasma treatment significantly improved adhesion by forming a reactive silica oxide surface layer rich in silanol groups, equalizing adhesion strength across substrate types, while carbon tetrafluoride plasma improved adhesion primarily through surface cleaning. Environmental testing revealed that moisture absorption critically degrades adhesion in a reversible manner during short-term exposure, with substantial adhesion loss at high dew points observed in in-situ tests. Ex-situ aging showed partial adhesion recovery after short exposures, but indicated irreversible degradation after prolonged aging, likely due to chemical and structural changes at the interface induced by external pollutant inside the climatic chamber used for the aging treatments. The comparative analysis highlights the need for in-situ testing to capture transient degradation phenomena and inform surface treatment and environmental protection strategies to ensure long-term interface reliability.

Looking forward, several promising directions emerge from this research. For warpage modeling, incorporating dynamic process simulations that capture the time-dependent evolution of intrinsic stresses and trench closure mechanics will improve predictive accuracy. Integration of contact mechanics and multi-physics coupling models will further improve relevance to device performance.

For silicon nitride films, the atomic-scale formation and effects of amorphous silicon inclusions have been investigated. Exploring the influence of stoichiometry and microstructure on thermal and most importantly electrical properties will provide a more comprehensive understanding that is necessary for optimized synthesis processes of films customized on their functions.

For polymer/inorganic interfaces, extending adhesion testing to dynamic environmental aging protocols that better simulate real-world device conditions is critical to having an effective comparison between in-situ and ex-situ experiments. Further study of chemi-

cal degradation mechanisms at the interface will guide the design of more robust surface treatments and polymer formulations. Incorporating advanced encapsulation and barrier materials will mitigate moisture ingress and improve durability. Finally, standardizing combined in-situ and ex-situ adhesion testing methodologies will improve industrial quality control.

In conclusion, this thesis has developed a comprehensive framework that addresses the mechanical and interfacial challenges associated with scaling semiconductor device manufacturing to 300 mm wafers. The integration of modeling, experimental, and environmental approaches has yielded actionable insights and validated methodologies that contribute to improving device reliability and manufacturing efficiency. The collaboration with STMicroelectronics has ensured industrial relevance and access to state-of-the-art materials and tools. Continued research along the outlined directions will further bridge fundamental science and industrial application, supporting the advancement of more reliable, efficient, and scalable semiconductor technologies.

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