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Hot ductility of Nb-V microalloyed steels

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Content

Abstract	3
Chapter 1 Introduction.....	4
1.1. Background.....	4
1.1.1. High strength low alloy (HSLA) steels.....	4
1.1.2. Microalloyed steel.....	5
1.1.3. Application	7
1.1.3.1. Microalloyed steel in offshore application and petroleum industry	7
1.1.3.2. Microalloyed steel in automobile industry	9
1.1.3.3. Constructional microalloyed steel.....	11
1.1.4. Typical microalloyed steel	12
1.1.4.1. Niobium and niobium contained microalloyed steel	12
1.1.4.2. Vanadium and Vanadium contained microalloyed steel	22
1.1.4.3. Titanium and Titanium contained microalloyed steel.	26
1.2. Hot Ductility.....	27
1.2.1. Variations influence the hot ductility.....	27
1.2.1.1. Influence of niobium and vanadium to hot ductility.....	28
1.2.1.2. Influence of Titanium on hot ductility.....	31
1.2.1.3. Influence of aluminum on hot ductility.....	32
1.2.2 Hot ductility during continuous casting	32
1.3. Research Purpose	35
Chapter 2: Experimental procedure and result analysis	36

2.1. Microalloyed Steels Rolled and as cast structure of type D and type F	36
2.2. Procedure for rolling	37
2.3. Sample preparation	38
2.4. Microstructure analysis of F and D typed before tensile test	40
2.5. Hot tensile test	44
2.6. Illustration analysis for hot tensile test result	47
2.6.1. Strengthen hardening by Nb	47
2.6.2. Hot ductility behavior analysis	50
2.7. Optical microscopy analysis	55
2.7.1. Microstructure analysis of as cast steel	56
2.7.2. Microstructure analysis of hot rolled steel	60
Chapter 3 Discussion and conclusion	64
References	66

Abstract

Microalloyed steel has become a good option in industry application for its high strength and low weight. However hot ductility problems exist during high temperature deformation which limit the commercial usage. Therefore, in this study, the hot ductility behavior of Niobium and Vanadium contained microalloyed steel with as cast structure and hot rolled structure were investigated by hot tensile test in the temperature region from 800°C to 1000°C, followed by fracturing and rapid quenching. Reduction of Area was recorded as an important parameter representing the hot ductility. High temperature yield strength, Ultimate tensile strength and elongation were obtained for study the hardening effect of microalloyed elements. Analyses of microstructure damage during tensile test were performed with optical microscope. Cracks were observed to understand the detrimental influence of Niobium and Vanadium on hot ductility. It was detected that the critical temperature zone for this microalloyed steel is between 850°C and 950°C, the rolled steel has better ductility performance than as cast steel, and rolling procedure may reduce the negative effect of Nb and V on hot ductility.

Hot ductility of Nb-V microalloyed steels

Chapter 1 Introduction

1.1 Background

1.1.1 High strength low alloy (HSLA) steels

High strength low alloy (HSLA) steels have been developed since the 1960s originally for large diameter oil and gas pipelines. Generally, it is a class of steels designed to achieve specific properties by controlled thermomechanical processing. Varying from other steels they are not made to meet a specific chemical composition, but rather to specific mechanical properties. They have a carbon content between 0.05–0.25% to retain formability and weldability. Other alloying elements include up to 2.0% manganese and small quantities of copper, nickel, niobium, nitrogen, vanadium, chromium, molybdenum, titanium, calcium, rare earth elements, or zirconium, of which copper, titanium, vanadium, and niobium are added for strengthening purposes. Most HSLA are microalloyed steels.

The elements added into are intended to alter the microstructure of carbon steels, which is usually a ferrite-pearlite aggregate, to produce a very fine dispersion of alloy carbides in an almost pure ferrite matrix. On one hand, this maintains and increases the material's strength by refining the grain size, which in the case of ferrite increases yield strength by 50%, and on the other hand, precipitation strengthening plays a minor role. Their yield strengths can be anywhere between 250–590 MPa. Due to their higher strength and toughness HSLA steels usually require 25 to 30% more energy to form, as compared to carbon steels.

According to its high strength and stiffness, HSLA is widely used in automotive industry to replace low-carbon steel parts with thinner cross-section parts for reduced

weight without sacrificing strength and dent resistance. Trucks, construction equipment, off-highway vehicles, mining equipment, and other heavy-duty vehicles use HSLA sheets or plates for chassis components, buckets, grader blades, and structural members outside the body. In equipment such as power cranes, cement mixers, farm machinery, trucks, trailers, and power-transmission towers, HSLA bar, minimum yield strength ranging from 350 to 500 MPa is used.

Accompany to the strength increasing from 250 to 550 MPa, a 30 to 40% loss in ductility occurs, which require 25 to 30% more energy when operation on HSLA steel than structural carbon steels. Another disadvantage is that HSLA steels have directionally sensitive properties, which means formability and impact strength can vary significantly when tested longitudinally and transversely to the grain. This is because bends that are parallel to the longitudinal grain are more likely to crack around the outer edge since it experiences tensile loads. By adding Copper, silicon, nickel, chromium, and phosphorus to increase corrosion resistance, adding Zirconium, calcium, and rare earth elements for sulfide-inclusion shape control which increases formability, this directional characteristic is substantially reduced.

1.1.2 Microalloyed steel

From functional point of view, HSLA can be divided into 4 types, Weathering steels, Control-rolled steels, Pearlite-reduced steels and Microalloyed steels. Most HSLA are microalloyed steels and it has the most widely applications.

As we have mentioned before, Microalloyed steel contain small amounts of vanadium, niobium, titanium, molybdenum, zirconium, boron, and rare-earth metals, Individual elements generally less than 0.1% and total microalloying elements generally less than 0.15%. They are used to refine the grain microstructure and facilitate precipitation hardening. Performance and cost of these steels are between carbon steel and low alloy steel. Yield strength is between 500 and 750 MPa without heat treatment. Weldability is good, and can even be improved by reducing carbon content while maintaining strength. Fatigue life and wear resistance are superior to

similar heat-treated steels. The disadvantages are that the ductility and toughness are not as good as the quenched and tempered (Q&T) steels. Also, they must be heated hot enough for all of the microadditions to be in solution; after formed, the material must be quickly cooled to 540 to 600 °C.

Despite all weakness of microalloyed steel, the obvious economic advance associated with using such small additions together with the significant benefits to mechanical properties are the reasons for the popularity of MA Steels in the market place. It is estimated that of the nearly 800 Mt of steel produced in the world today about 12% could be defined as MA steel. Such steels are used in all major market sectors especially line-pipe and offshore (Table 1).

	Europe	N. America	Japan	
Linepipe	95	95	95	
Shipbuilding	40	20	75	
Offshore Steels				
Plates	90	30	70	
Sections	70	20	10	
Pressure Vessels	30	25	85	
Structural				
Sections	30	20	10	Annual World Tonnage ~800Mt HSLA Steels ~12%
Section, automotive	80	80	80	
Section, ships	15—30	20	10	
Sheet piling	25	15	100	
Rebar	100	5	10	
Plates	25	20	10—30	
Sheet and Coil (inc. Galv.)				
Automotive	30	20	30	
Building (not rebar)	95	80	70	

Table 1 worldwide production of microalloyed steel [1]

1.1.3 Application

Microalloying technology, developed for the production of flat products (plate, strip and line pipe) during the 1960's and 1970's, has been applied to "long products" such as engineering bars, sections, forgings and wire rod since about 1980. In the 1980's the main rationale to use niobium bearing steel bars and wires was to eliminate the need for a hardening process, i.e. quenching and tempering, in manufacturing heat-treated steel parts without any trade off in properties. After entering 21st century, a global trend of steelmaking increasing occurs within developing countries, which leads to supply and demand unbalance of raw material. The resulting shortages in raw materials, energy and means of transportation contributed to raising prices. From sustainable point of view, on one hand satisfying the engineering needs, on the other hand costing less steel, Microalloyed steel has taken a leading role in modern manufacturing field. By contributing to a "sustainable" growth rate of steel production, microalloyed steels fulfill a new role: they create economic value and wealth. To achieve global economic goals of underdeveloped countries, substitution may be a necessity, rather than an option.

Microalloyed steels are used in automobile industries, offshore platforms and in structural applications.

1.1.3.1 Microalloyed steel in offshore application and petroleum industry

Oil and gas industry is one of the main consumer for High Strength Low Alloy and microalloyed steel. The consumption on petroleum pipeline takes 1/5 of the whole investment of petroleum industry every year.

For economic consideration, oil and gas pipeline is the most economical way in long distance petroleum transportation, which leads to the blooming of pipeline building in the past century, and 10 million tons of steel are consumed in the petroleum pipeline manufacturing every year.

During the past 20 years, the enormous rise in need for petroleum pipeline at the same time brings in much more consideration during manufacturing, installing and maintaining. Larger and larger amount transportation request large diameter pipeline and higher pressure gas transportation; complicated service environment demand the pipe can serve in cold and corrosive service environment; offshore pipelines installed in deeper water operate under higher pressure, therefore the line pipe material has to show higher strength properties with simultaneously increased toughness. In case that the gas to be transported contains certain amount of H_2S additional requirements regarding HIC (Hydrogen Induced Cracking) resistance have to be taken into consideration. Traditional pipeline steel could not fulfill all demands but microalloyed steel make all requests above possible and makes economic value.



Fig.1.1 Offshore pipeline



Fig.1.2 Petroleum industry

1.1.3.2 Microalloyed steel in automobile industry

In early 1994, a consortium of 35 sheet steel producers from 18 countries set out to demonstrate a lightweight steel auto body structure that would meet a wide range of safety and performance targets, which is the ULSAB project. As one of the research conclusion, scientist explained how to use high-strength steels to reduce mass in a vehicle structure that is Safe, Affordable, Fuel Efficient and Environmentally responsible (SAFE).

The outstanding properties of microalloyed steel is ultra-high strength compare to hot rolled, weldable carbon steel with the same weight, which means by replacing the inexpensive carbon steel with higher value microalloyed steel, it saves enormous weight. Just considering weight reduction, cost saving can be realized from not only weight but also in the entire process route from material delivery to final component application.

The weight reduction achievable through substitution depends not only on the difference in strength but also on the mode of loading. For straight loading in tension,

the weight reduction is proportional to the difference in strength. An increase in yield strength by a factor of two may reduce the weight of steel by two - a situation found in concrete reinforcing bars. The range of weight savings is shown in Figure 1.3.

For other types of loading (e.g., bending), a two fold increase in strength may contribute to a weight reduction of 34% or more. Considering a safety factor, one microalloyed steel, being twice as strong as carbon steel, may reduce the weight by at least 25% [2].

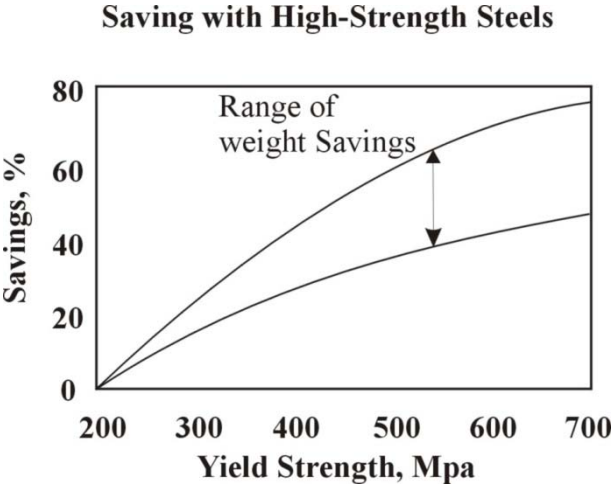


Fig. 1.3 The weight of components can be reduced substantially by substituting high-strength steel for low-strength carbon-manganese steel (SSAB Sheet Steel Handbook).



Fig. 1.4 automobile products of microalloyed steel

1.1.3.3 Constructional microalloyed steel

Microalloyed Steel products with high strength, crack resistant are used nowadays for building contemporary constructions with the high technological load and rationally matched mass, for transport means consuming low energy amounts for their propulsion, as well as for various machines and devices. Hence, container and manufacturer are interested in microalloyed steel to reduce transmission cost and improve safety. If we use 550 MPa steel as substitution of 350 Mpa plain steel, the container self-weight can reduce 20%. Corner column, up-down stringer and beam are favorable microalloyed steel products in container industry and mining industry.

Another important product is microalloyed constructional plate, which is widely used in civil engineering: heavy steel structures of welded construction, bridges with spans between 4 and 1000 m and offshore platform for oil and gas production and processing. Because of its multiple dimensions in formability, as well as the outstanding mechanical properties, the heavy plates provide for the possibility of very economical and durable constructions.

Here are an example of welded heavy microalloyed plates used in bridge with big spans (more than 150m):

The Erasmus-bridge crosses Nieuwe Maas River in Rotterdam, where a new quarter has been established on a former harbour area. The steel bridge has an overall length of 499 m with a 410 m long cable-stayed bridge composed of a 139 m-tall pylon and a 89 m-long hinged bridge.

In total, 6000 t of microalloyed heavy plates of grades S355M (thickness less than 100 mm, 4200 t), S460ML (thickness less than 80mm, 2000t) and S460QL (thickness less than 125) were used for this bridge.



Fig. 1.5 Erasmus Bridge Rotterdam

1.1.4 Typical microalloyed steel

1.1.4.1 Niobium and niobium contained microalloyed steel

Niobium was first used commercially in early 20th century. Nowadays, niobium is used mostly in alloys, the largest part in special steel such as that used in gas

pipelines and in various superconducting materials because its highest critical temperature of the elemental superconductors at atmospheric pressure. Besides, niobium plays a precipitation-hardening role in nickel-, cobalt-, and iron-base superalloys, which are widely used in Aeronautics and space technology such as jet engine components, gas turbines, rocket subassemblies, and heat resisting and combustion equipment. Furthermore, niobium alloy are lightweight and hypoallergenic (no skin reactions), suitable for jewelry production.

Niobium has threefold influence on the mechanical properties of steel, grain size refinement during thermomechanical hot forming, lowering the A_{r3} transformation temperature and precipitation hardening. To realize the grain refinement, Niobium prevent or delay recrystallization in last steps of hot forming; flattened grains as well as a high dislocation density of the austenite enhance ferrite nucleation. At the same time of lowering A_{r3} temperature, Niobium enhance the ferrite nucleation rate and reduce grain growth rate, which lead to particular fine-grained transformation structure.

Drawn from Fe-Nb phase diagram (Fig 1.6), niobium and iron are completely miscible in iron based alloy at high temperature. The equilibrium diagram for the iron-niobium system is well established. Niobium plays a role of ferrite stabilizer. However, additions of up to 0.10 to 0.20 percent lower the A_3 temperature.

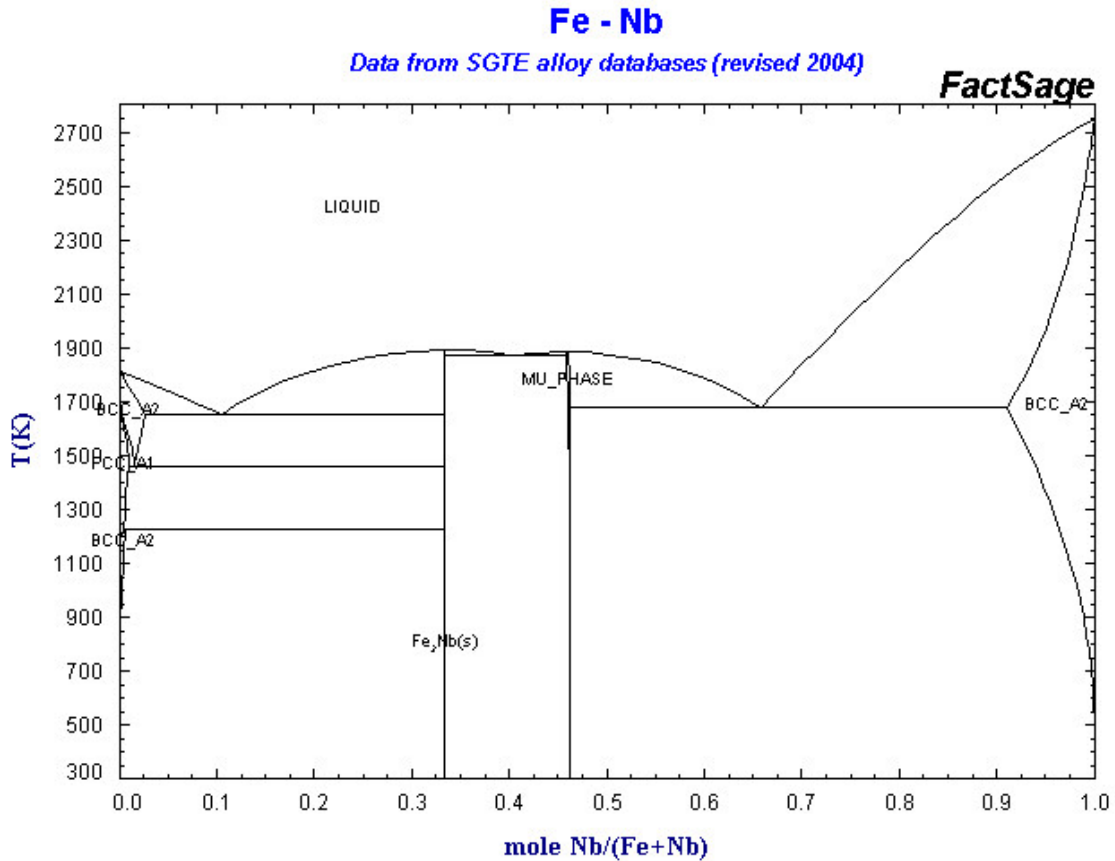


Fig. 1.6 Nb-Fe phase diagram

Here are the part to review how niobium does influence on grain size refinement and precipitation hardening. As we all know, transition metals are known to form a series of simple and solid solution compounds of oxides, sulfides, carbides and nitrides. Niobium shows a strong tendency to form carbonitrides, but relatively little tendency to form oxides, sulfides or solid solutions of these compounds, similarly like vanadium. This characteristic is quite different from Titanium, which prefers oxygen, nitrogen and sulfur better than carbon.

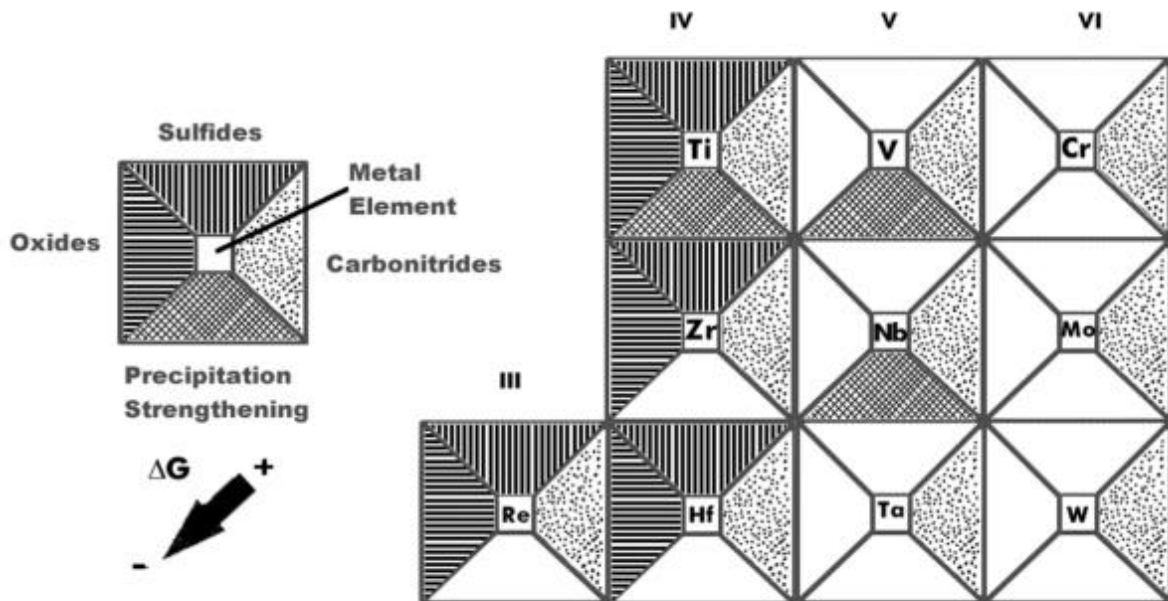
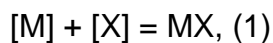


Fig.1.7. The tendency of certain metals to form oxides, sulfides, carbides, and nitrides and their precipitation-strengthening potential (arranged similar to the periodic table) [3].

As talking about the compound form between transition metal and metalloid, M (transition metal) dissolved in austenite gets restrained at a certain temperature T with metalloid X into the interstitial phase MX with the cubic lattice of the NaCl type according to the reaction:



Where $[M]$ and $[X]$ are microaddition M (Nb, Ti, V, Zr or B) and the metalloid X (N or C) portions respectively, dissolved in the solid solution γ at the temperature T , K. Solubility of the MX phases in the solid solution allowing for the activation energy of their nucleation (dissolving) is described by the relationship:

$$\log [M][X] = A - B/T, (2)$$

Where A consist of activation energy of MX phases and temperature, and B depend on the constant of phase type [4].

Making it possible to evaluate the temperatures of the beginning and end of the MX phase precipitation at a certain concentration of the microaddition M introduced into the steel. Knowledge of the temperatures of the beginning and end of precipitation of the MX phases in austenite is very important for designing the hot-working conditions for the microalloyed steel.

The effect of the dispersive particles of the MX phases on development of the fine-grained structure can be explained through steel structure changes during hot working. Taking rolling at temperature higher than austenite recrystallization temperature T_R as an example, the feedstock at the beginning of hot-work has a coarse grained structure. During rolling plastic deformation occur at the austenite grains and they are prolonged in the direction of metal flow (Fig.1.8) with their simultaneous dynamical recovery, causing the flow stress reduction. The static recovery and static recrystallization occur in deformed part after leaving the rolls, leading to the nearly complete work softening and to development of the fine-grained γ phase structure. This unstable structure has quite big grain boundaries area and its ratio to the grain volumes is unfavorable. Therefore, the destructive austenite grains growth occurs when the recrystallization is over. This process may be stepped in case of the microalloyed steels by the dispersive particles of the MX phases, developed in the austenite during plastic deformation, restraining the grain boundaries mobility. Therefore, the austenite grains size become significantly smaller with these microadditions. This effective influence is illustrated directly in Fig 1.9. The dispersive particles of the MX phases cause precipitation hardening of the steel apart from restraining the grains boundaries.

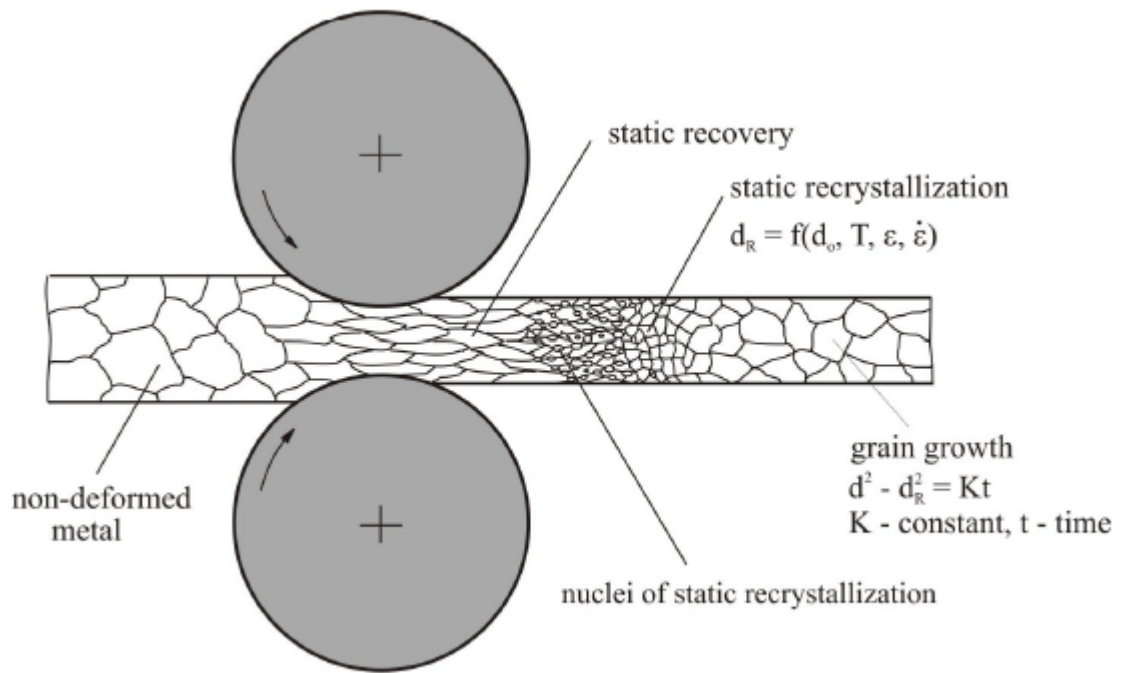


Fig.1.8. schematic diagram of structure changes of the deformed metal in the course of rolling and after its completion with a strain higher than ϵ_{cd} .

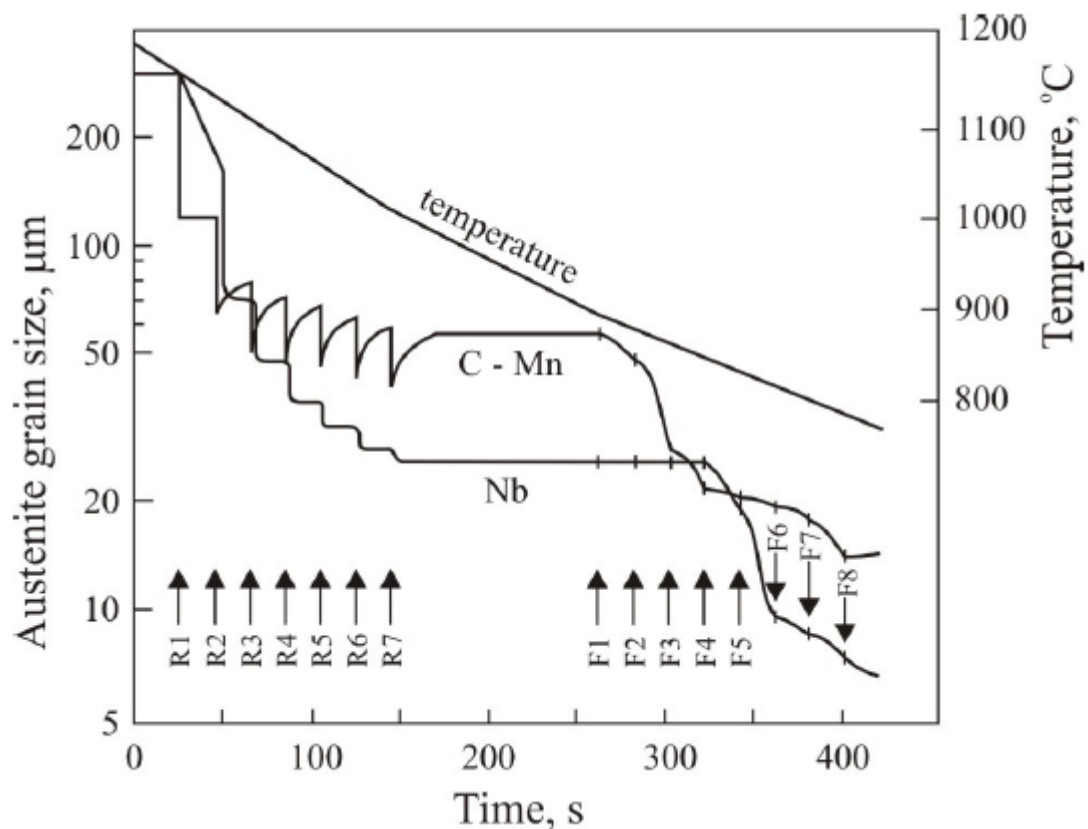


Fig.1.9. Predicted changes of austenite grain size in the course of rolling 200 mm thick slabs into 20 mm thick plates of C-Mn steel and such a steel containing 0.04% Nb [5]; R1 do R7 and F1 to F8 -subsequent passes during roughing and finishing rolling, respectively.

The MX phases of various metallic additions have different thermal stability and differentiated effect on steel properties. The optimum Niobium concentration in the low-carbon steels is 0.04% and this concentration makes it possible to increase the yield point of products by about 120 MPa due to refinement of grains and by about 160 MPa due to precipitation hardening by the dispersive NbC particles, with the simultaneous lowering of the T_{pk} temperature by about 40°C (Fig.1.10).

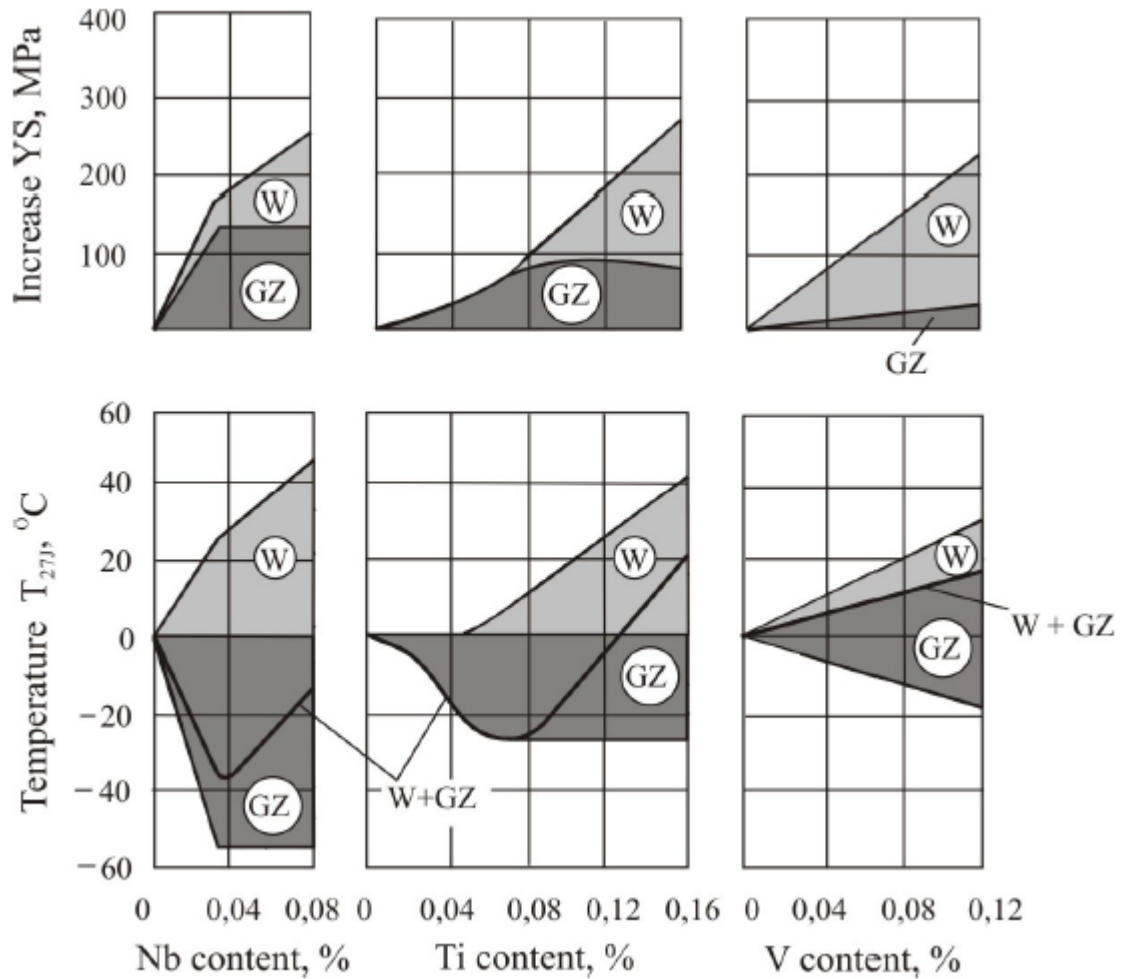


Fig.1.10 Influence of the Nb, Ti, and V content on the increase of the yield point and variation of the impact transition temperature of the low-carbon steel[4]: GZ - influence of grain refining, W - influence of precipitation hardening

The precipitation temperature range of the MX phases depends on concentrations of C and N in the steel and the type of microaddition. In order to optimum use of its metallurgical potential Niobium has to be in solid solution by an adequate reheating furnace temperature to dissolve Nb(C,N) precipitates before hot forming. Several additions are added into to make larger range of MX precipitation temperature (Fig.1.11).

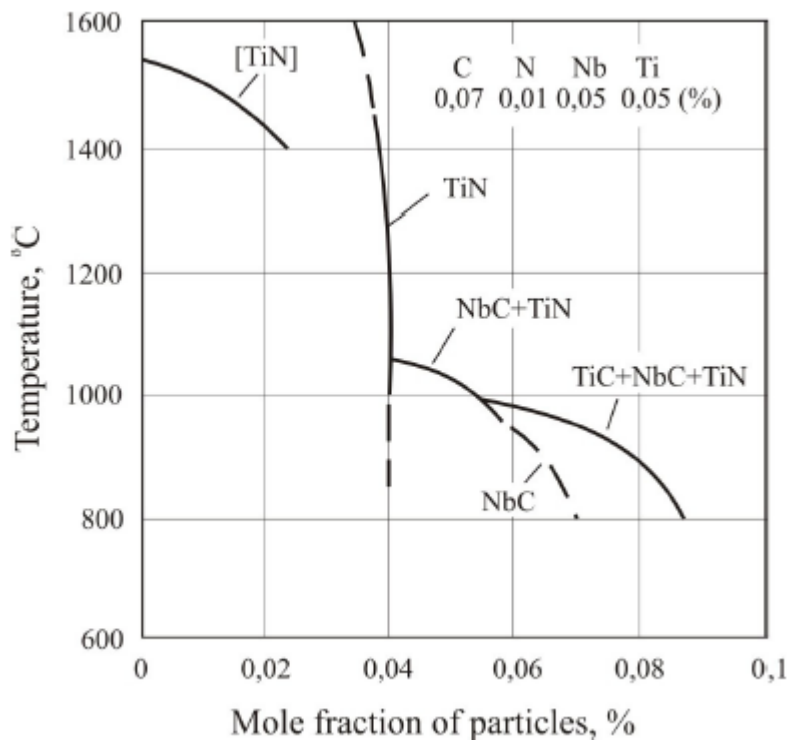


Fig.1.11. Temperature sequence of nitrides and carbides precipitation in steels with microadditions of Ti and Nb [6]

Here are some examples of niobium microalloyed steel in engineering applications:

Hot-forged Products:

For closed die forgings in the automotive industry the steel grade 49MnVS3 (0.50% C-0.25% Si- 0.70% Mn-0.04% S- 0.10% V) was established in the middle of the 1970's [7]. Though the required strength could be obtained by 0.08% Nb microalloying when high processing temperatures are applied, the limited toughness restricted its application. For improved toughness, a 0.38% C-1.00% Mn-0.07% V-0.03% Nb steel was developed and applied in the as-forged condition for automobile parts with improved safety [8].

Bucher [9] did the research on adding niobium to AISI 1141, getting the result by compare to standard AISI 1141. The structural refinement increased the hardness of

the niobium bearing steel to 97HRB vs. 92HRB, and improved toughness that was sufficient for connecting rods. The niobium-modified AISI 1141 is also used for weld yoke or universal joint couplings.

Spring Steels:

High strength quenched-tempered spring steels are required to have desirable tensile strength, proof stress, fatigue strength, sag resistance, and with increase in the design stress, corrosion fatigue strength and delayed fracture resistance. The Figure below shows the change in the design stress of coil springs in the Japanese automotive industries. In the early 1990's, light springs were necessary for reducing weight in passenger cars and high-alloy steels were developed to endure a surge in design stress accompanied by a thinner coil or plate shape. Recent developments in this field have drifted back towards reducing alloying elements in spring steel, more equivalent to 1200 MPa design stress spring.

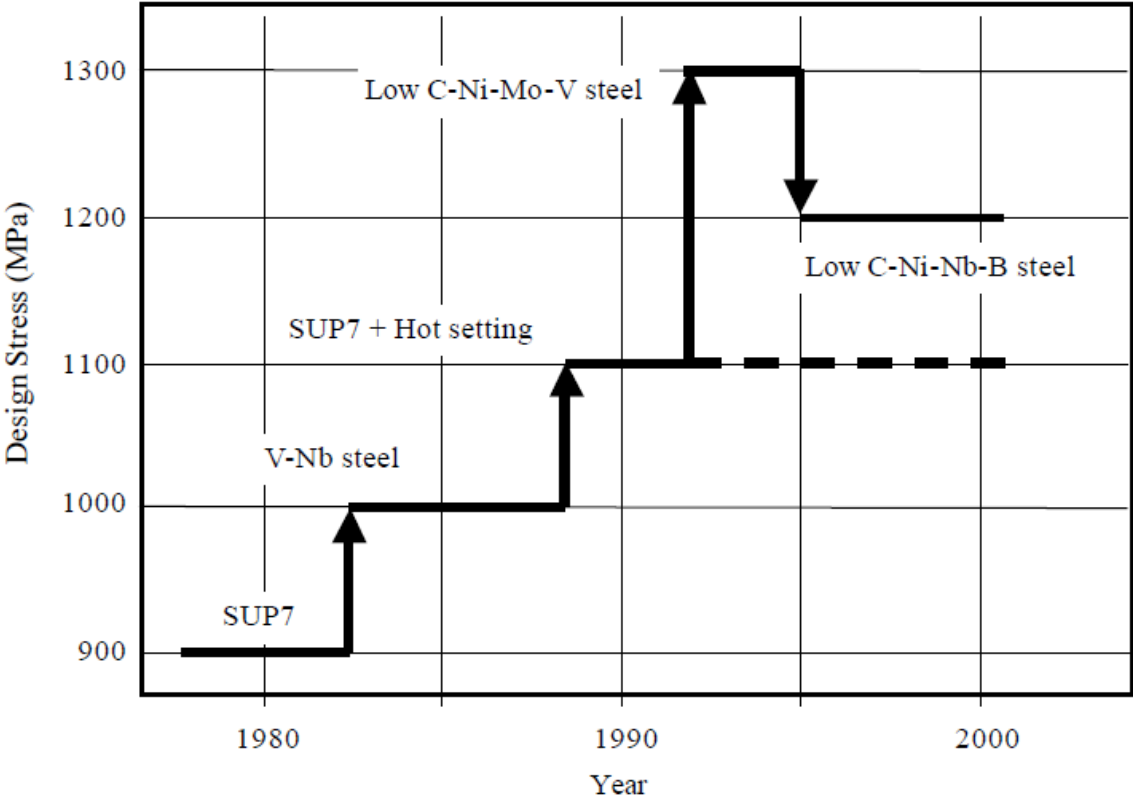


Fig.1.12: change in the design stress of commonly used hardened coil springs.

Before 1990s', with the attempt to improve sag resistance in coil springs, by adding V and Nb to JIS SUP7, superior sag resistance in vanadium and niobium-bearing steel was observed, and the design stress of coil springs was raised from 900 MPa to 1000 MPa.

For saving natural resource and reduce automobile emission, as one part of auto weight reduction program, the design stress of coil springs was successfully raised to 1330 MPa using 0.4% C-2.5% Si-0.8% Mn-2.0% Ni-0.85% Cr-0.4% Mo-0.2% V steel with a weight-saving of over 20% being achieved. However, this development can't go far on the stage because of high cost of steel as Ni, Mo and V.

A new grade for 1200 MPa design stress has been developed and is gradually increasing in commercial production. The steel composition is typically 0.4% C-1.8% Si-0.5% Ni-1.1% Cr-0.15% V-0.025% Nb-0.0015% B. Carbon is reduced for increasing corrosion fatigue life, Si remains at a relatively high level for sag resistance, Ni is added to retard pitting corrosion, and Nb and B are added for grain refinement and strengthening of the prior-austenite grain boundaries.

1.1.4.2 Vanadium and Vanadium contained microalloyed steel

Approximately 85% of vanadium produced is used as ferrovanadium or as a steel additive. After Vanadium was found to be the stabilizer of nitrides and carbides with the significant increase in the strength of the steel, vanadium steel was used for applications in axles, bicycle frames, crankshaft, gears, and other critical components. Also, vanadium plays a very limited role in biology: a vanadium-containing nitrogenase is used by some nitrogen-fixing micro-organisms.



Fig.1.13 tools made from vanadium steel

In the development of microalloyed steel theory, the most capitalized ones are grain refinement and microalloy precipitation hardening. Grain refinement adds both strength and toughness to the steel whereas precipitation increase strength but sacrifice toughness. Compared to Nb and Ti, the most significant properties of V are:

- (1) The solubility of V(C,N) is much larger; this is particularly evident at higher temperatures, for the reason that the high amount of V can be dissolved if the temperature is lower or alternatively.
- (2) Compared to other microalloying elements, V are extraordinary. In particular the solubility of its carbonitrides is much larger and the solubility of its nitride is about two orders of magnitude smaller that its carbide, similar to Ti but contrary to Nb. This suggests that N as a microalloying element has determinative role in V microalloyed steel, specially for precipitation hardening.

As to the problem of grain refinement, as we know Nb can realize this refinement during hot rolling by the pre-exist Nb carbonitirdes in austenite and its retarding

recrystallization process. The result is recrystallization stops and continued rolling the unrecrystallized austenite grains makes that flattened. The main mechanism is to increase grain boundary area of austenite.

As we mentioned before, the large solubility for V and its carbonitrides annihilate the possibility of having effective resistance for austenite recrystallization during hot rolling. Another new way called repeated austenite recrystallization, after each rolling reduction and sufficient number passes, produces a very efficient austenite grain refinement, which has a ferrite grain size as same as the former method. This method is called Recrystallization-Controlled-Rolling (RCR).

V is one of the best elements for strong precipitation strengthening (Fig.10). The mechanism is that larger solubility product of its carbonitrides leads to a lower solution temperature and a larger capacity to dissolve them when increase the temperatures. Compared to Nb carbides and nitrides, VN has much lower solubility than VC, so N plays very important role in V-steels, especially in their precipitation strengthen.

Reviewing the literature [10], by increasing N-content, the larger driving force leads to large nucleation rate for V(C,N) and then the hard particle—V(C,N) spacing is realized, which results in precipitation strengthening. With this significant effect of N-content, instead of adding strength, this effect can save alloy cost. For example, adding N from a level of 0.005% to 0.015% makes it possible to reduce V from 0.12% to 0.06%.

In Iraq, Microalloyed vanadium hot rolled steel in combination with very high strength concrete can protect high value targets from blasts—either as a new method of barrier construction or in the construction of a high value building itself. This program, initiated by military, is demonstrating the higher strength, lower cost benefits of vanadium microalloyed steel. The research show that weight of army support structures, temporary and intermediate bridge and vehicles can be improved in protection and mobility. Depending on the application, weight and cost reductions could possible be up to 40 percent.



Fig.1.14. U.S. Army maintains vanadium microalloyed steel bridge designs for rapid reconstruction during deployed operations.

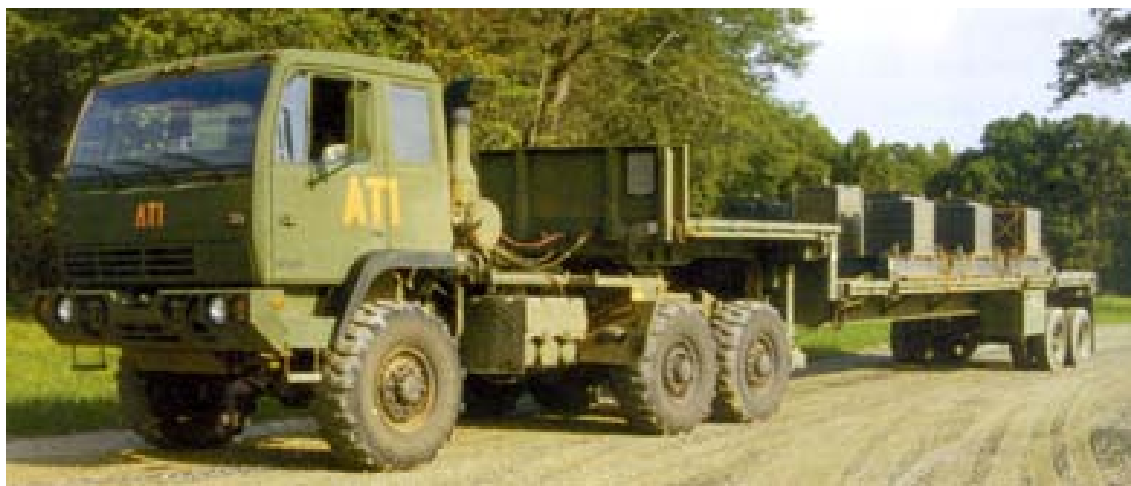


Fig.1.15. The M871A3 Trailer Weight Reduction Team will investigate the application of vanadium microalloyed steel to reduce weight and improve performance of the M871A3 trailer.

1.1.4.3 Titanium and Titanium contained microalloyed steel

Nowadays, Titanium is widely use in alloy as alloying element, to reduce grain size, to reduce carbon content with aluminum, vanadium, copper , iron and other metals. Due to its suitable properties, titanium alloy are widely use in aircraft, armor plating, naval ship, spacecraft and missile. The industrial applications of titanium in welded pipe and process equipment are mainly for its high corrosion resistance. In racing /performance automotive market, Ti is highly preferred but it is beyond the general consumer market. This metal is non-toxic and has no rejection by human body, then titanium is used in a gamut of medical applications including surgical implements and implants, such as hip balls and sockets (joint replacement) that can stay in place for up to 20 years.



Fig.1.16. The Guggenheim Museum Bilbao was the first buildings in Europe and to be sheathed in titanium panels.

Similar as Niobium and Vanadium the precipitation of Ti carbide, nitride and carbonitride can prevent austenite growing during the whole rolling process, and controlling austenite grain recrystallization. Strengthening and increasing toughness (Fig.1.10). When Ti has large content ($>0.04\text{wt}\%$), at relative low temperature, TiC will effect the properties by dispersion strengthen. By review the research of W. Saikaly

[11] on contribution of TiC to the yield strength, specimen with (wt%) 0.085C、0.96Mn、0.23Si、0.004S、0.0065N、0.045—0.13Ti, under suitable process method can increase the yield strength by 100-250 MPa.

The appearance of Ti in sulfur contained microalloyed steel can produce compound $Ti_4C_2S_2$, at the same time eliminate MnS products. This result highly reduces the properties difference within length and breadth, improving cold deformation property.

When adding other microaddition like Nb and V, small amount of Ti can produce TiN easily, occupying major free N and increasing solubility of other microaddition. Then, during cooling process, other element will precipitate small carbonitrides particle to harden the steel. Meanwhile, nitrogen content decreasing result in less Nb(C,N) precipitate on grain boundary, and this change largely improve the hot ductility of Nb alloy, reducing hot cracking during hot work.

1.2 Hot Ductility

Microalloyed steel, with high yield strength, good toughness and fine grain structure, has application preference in many fields. But during hot processing, at certain high temperature region (usually 700-1000 °C), microalloyed steel is very sensible to cracking and may fracture during deformation. There is a considerable loss in ductility which leads to fracture. To measure this loss, hot ductility tensile test is introduced. As one important result after fracture the specimen, the reduction of area values (R of A) are taken as a measure of the ductility and the value required to prevent cracking occurring is dependent on the exact test conditions. To make sure the steel do not experience a sudden failure, the reduction of area should be above 40%.

1.2.1 Variations influence the hot ductility

Reviewing the work by Mintz, the poor ductility in the trough is always related to intergranular failure at the γ grain boundaries [12]. Cracks are formed along these

boundaries, and grain boundary sliding in the austenite and transformation controlled intergranular failure are the two mechanisms leading to cracks. The first mechanism is strongly encouraged by having particles at the boundaries and the presence of a fine matrix precipitation, for instance Nb microalloyed steel, accompanied by precipitate free zones, will enhance this mechanism. For the latter mechanism thin films of ferrite growing surrounding the γ grain boundaries may lead to transformation controlled intergranular failure.

Four important variables control ductility: strain rate, grain size, precipitation and inclusion content (their size, volume fraction and distribution being important) [13]. Increasing the strain can reduce the amount of grain boundary sliding, and refining the grain size can make it more difficult for cracks to propagate along the boundaries. Both of the above methods give rise to improved ductility. But it's not generally possible to alter either of these sufficiently in conventional continuous casting to make significant improve to ductility.

As to precipitation, finer precipitation leads to worse ductility. Grain boundary precipitation is particularly detrimental. Within a particular volume fraction of precipitate or inclusions, finer particles at boundaries make them closer to each other, thus cracks may interlink. Strain induced precipitation is always finer, hence more harmful to ductility than precipitation present before strain [13].

As the important possible content of microalloyed steel, Niobium, Vanadium, Titanium and other microadditions play significant role in improve the mechanical properties of steel, but on the contrary, their exist in high temperature region is not always welcome, for there effects to the hot ductility.

1.2.1.1 Influence of niobium and vanadium to hot ductility

Niobium, the most common microaddtion in microalloying, precipitates in the form of Nb carbonitrides, which is particularly effective in reducing the hot ductility and widening the trough in hot ductility curve.

For Nb containing steels, intergranular failure in the higher temperature range (900°C) invariably occurs by grain boundary sliding in the austenite. The fine matrix precipitation of Nb(C,N) increases the stress required for deformation and therefore, increases the stress in grain boundary regions. Then precipitate free zones usually appear at the boundaries, concentrating the strain in there. Furthermore, there is always marked precipitation at the austenite grain boundaries and this will facilitate voiding and elongation of cracks formed by grain boundary sliding. If the precipitation is fine with close spacing of particle as what in Nb containing steels, cracks can readily link up.

At lower temperature (800 °C), deformation induced ferrite, together with precipitation, leads to continued poor ductility. Nevertheless, fracture is by microvoid coalescence at the MnS inclusions present in these thin films and Nb(C,N) precipitations make the situation worse.

The improvement in ductility can be realized by increasing the temperature above Ae3 to eliminate deformation induced ferrite, but this improvement is small, since grain boundary sliding in the austenite then occurs, which is deleterious to ductility. In the end, the main improvement in ductility is associated with dynamic recrystallization.

For Nb containing steels, the fine precipitation of Nb(C,N) formed on deformation delays the onset of dynamic recrystallization to higher temperatures, thus widen the trough to higher temperatures. The presence of abundant volume fraction of ferrite leads to ductility recovery at low temperature end of the trough. Also, the strength variation between the γ and ferrite is reduced [14].

Reviewing the work by Mintz [15], increasing the V or N content reduces the hot ductility. This is due to the increasing of volume fraction of VN precipitation in the temperature range of the trough. In the previous statement we know that fine precipitation make concentrate the sliding which leads to poor ductility, and the trend in fig.18 shows that during increasing the V content result in worse ductility, but it seems that the V is not as sufficient as Nb through they have similar precipitations.

this is probably because at these V and N contents, precipitation is less extensive but coarser than that shown but the Nb containing. Similar as Nb, recovery of ductility at higher temperature end of the trough is related to dynamic recrystallization, which will be encouraged if the precipitation becomes more intense.

V has higher solubility in austenite than Nb does, so precipitates would also be expected to coarsen more rapidly in V containing steel according the Lifshitz-Wanger theory of particle coarsening. This is the reasonable explanation for V having less effects than Nb in reducing hot ductility, because during solidification of continuously cast strand, it forms a coarser precipitate and therefore much more are required for ductility deteriorates. Through coarser than Nb(C,N), V rich precipitates are sufficiently fine enough for strengthening in solution during reheating for rolling.

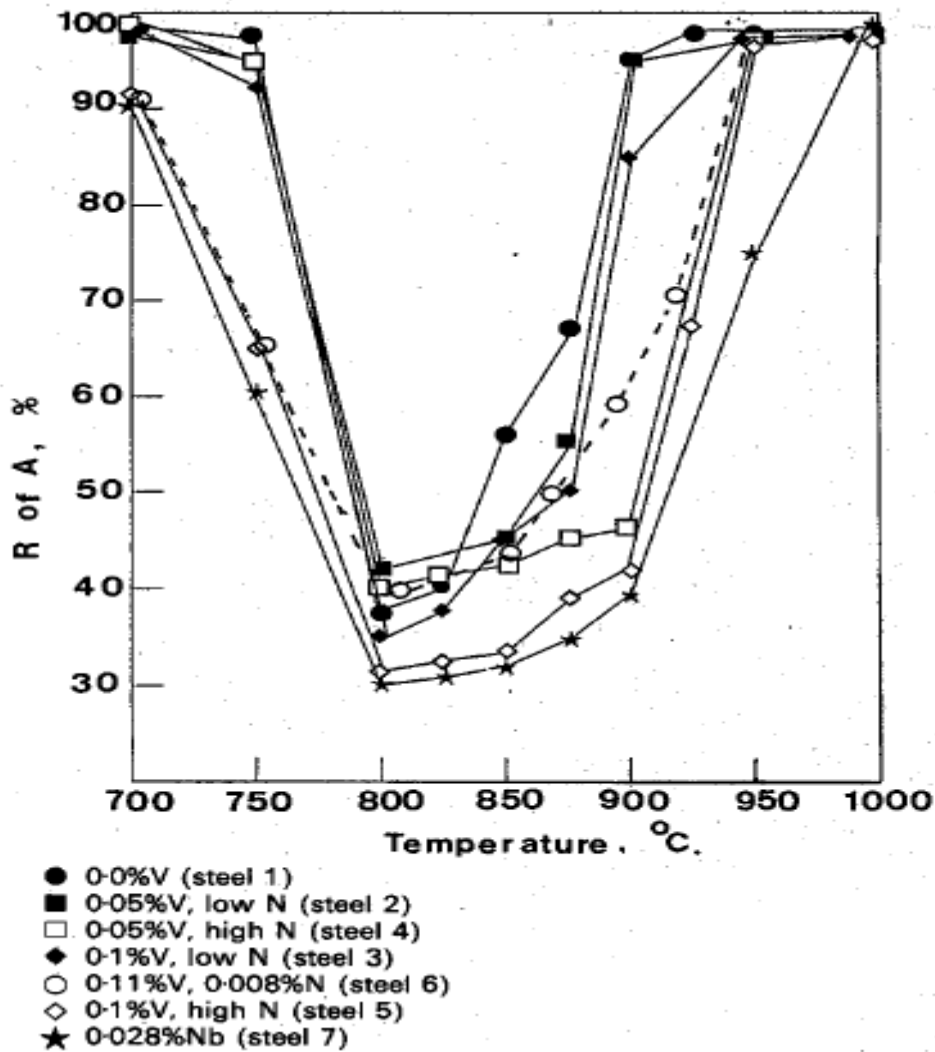


Fig .1.17 hot ductility cure of the above steels examined [15]

1.2.1.2 Influence of Titanium on hot ductility

Work by Abushosha [16] shows that the ductility of Ti containing Nb free steels is dependent on the cooling rate, particle size, and volume fraction. The increasing of cooling rate leads to refinement of sulphides at the boundaries, hence cracks can easily join up to structural failure. Similar to Nb, fine precipitation particles is detrimental to ductility by strengthening the austenite, linking up cracks as inclusions do.

Increasing particle size has the effect on improving hot ductility [16]. But particle size is also influenced by the Ti/N ratio, the product of $[Ti]*[N]$, cooling rate and test temperature. Coarser particle will be formed if Ti/N ratio is above 3.4:1, thus the larger

amount of Ti in solution will favor the growth of Ti containing particles. The product of $[Ti]*[N]$ is the symbol for volume fraction, where it increases, the hot ductility reduces.

For Nb containing steels, the addition of Ti generally give poor ductility [17], for Ti containing precipitation being very fine and stable at high temperatures. However, in industry application, Ti has good effect in improving the surface quality of continuously cast slabs. From the hypothesis and demonstration by Comineli [17], it appears that for limiting the volume fraction of Ti nitride, lower Ti levels is recommended to get hot ductility. Or else, the cooperation of low N level for limiting the volume fraction and high Ti level for favoring growth will give good ductility.

If N level is high, a low Ti level is inclined to limit volume fraction of Ti containing precipitate. It's possible that having a high N level at the same time a high $[Ti]*[N]$ product will encourage precipitation at higher temperatures, getting coarser precipitation.

1.2.1.3 influence of aluminum on hot ductility

Aluminium is not usually classified as a microalloying element. The precipitation of AlN can, however have strong influence on the properties of microalloyed steel, as Nb, V or Ti does. At higher temperature aluminium dissolve in austenite before rolling but AlN is stable at lower temperatures. In Titanium and niobium containing steel, Comineli [17] found that raising the soluble Al level did not influence the ductility. Nevertheless average particle size is coarser in the higher Al containing steel. Loberg et al.[18] have suggested that when Al associates with N, though no precipitate produced, the amount of Ti in solution increases, encouraging growth. Furthermore, work by Mintz [12] found that Nb suppresses the precipitation.

1.2.2 Hot ductility during continuous casting

In previous statement, microalloyed steel show their excellent properties in strengthening and grain refinement, but within some special high temperature range,

it always shows their fragile and breakage during continuous casting. Continuous cast microalloyed steel has the coarse melting structure.

Molten metal achieving the right temperature after the ladle treatment and vacuum degassing is casted with the continuous casting method in the argon temperature, protecting the steel from the secondary oxidation and nitriding. The ladle with the molten metal is transported to the continuous casting system consisting of the tundish maintaining the continuous metalstatic pressure. Copper mould allows cooling water flows inside. Pinch rollers, some of which are equipped with electromagnetic stirrers, limit development of columnar grains and development of the axial segregation in the slab, followed by bending rollers as well as the shear (Fig.1.18). The continuous slab solidifies at a certain distance from the mould, while the molten metal deficiency in the slab core, connected with metal contraction during crystallization, is replenished from the tundish under the metalstatic pressure. In newer solutions, some of the guiding system rollers fulfill also the role of the pinch rollers for the slab with liquid core, which makes a significant section change possible and limits development of the contraction cavity and shrinkage porosity in the slab. Slab section is selected according to the final product shape, making allowance for the required rolling reduction ratio. The slab is subjected to hot working after shear while hot and after additional heating up to the required temperature.

As we mentioned before, a trough in hot ductility exists over a certain range of temperature (700-1000°C) where surface cracking is likely to occur that the problem is extremely severe in microalloyed steel compared to plain C-Mn steels. The most critical factor is the upper temperature at which ductility deteriorates since bending of the slab, which is cooling continuously, must be complete before any part of it falls below this temperature [10].

A typical creep rupture behavior results in crack along austenite grain boundaries at high temperature and rather low deformation rate, which is more severe at bigger

grain sizes in cast. As the grain boundaries are softer than interiors, deformation and sliding concentrate at the grain boundaries.

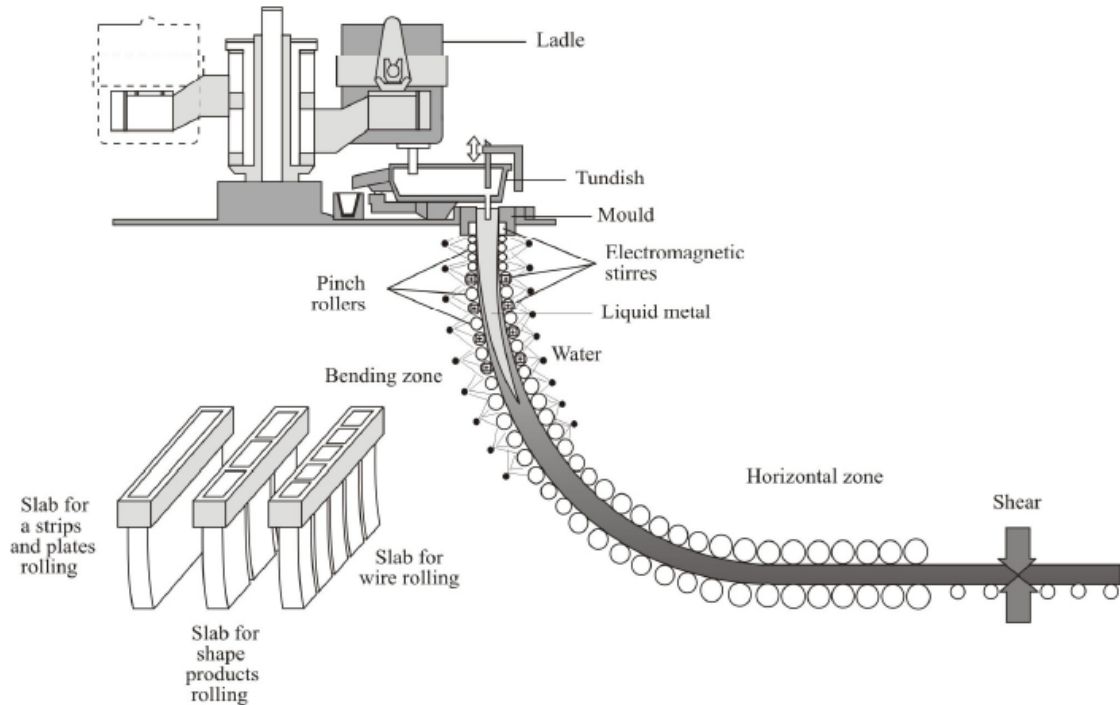


Fig.1.18 Technological equipment for continuous casting of steel

Microalloyed steel are sensitive to such cracking for the reasons below [18,19]:

1. Void initiation at coarse carbonitride particles located on austenite grain boundaries.
2. Hardening of the grain interiors by fine scale carbonitride precipitation.
3. Inhibition of dynamic recrystallization by precipitation on deformation substructures thus preventing relaxation of stresses built up at grain boundaries

When the temperature falls below A_{r3} , ferrite starts to form, commencing along the austenite grain boundaries. At a given temperature, the strength of ferrite is only about 60% that of austenite so the tendency for deformation to concentrate to the boundary regions becomes magnified with rapid localized rupture as a consequence. Only when

the structure becomes mainly ferritic at lower temperatures, the deformation becomes more uniformly and ductility is restored.

1.3 Research Purpose

In the previous section, influence of Nb, V, Ti and other microalloying element has been reviewed, similarly their effect mainly originates from the concentration of solute and deformation, particle size and morphology (fine or coarse), concentration level to affect solubility and so on, even though, full agreement concerning the mechanism of hot ductility loss still seems to be lacking. However, to any specific problem, we have a perfect frame of experimental method and sufficient relative knowledge to figure it out. A lot of literatures [13,14,15,16] have been done on the hot ductility loss of as cast steel, however, about the influence of microaddition on hot ductility of rolled steel, there are really few references to consult. Our aim is to find the hot ductility performance of rolled microalloyed steel, with respect to that of as cast steel. As to the possible great improvement or reduction in hot ductility, optical analysis is necessary to find out the influence aspect for fracture and which factors are the key leading to these properties changes.

Chapter 2 Experimental procedure and result analysis

2.1 Microalloyed Steels Rolled and as cast structure of type D and type F

The Microalloyed steels investigated and used for hot tensile tests can be divided into two types by the chemical composition, in particular by the percentages of Nb and V (Table 2). The first type of steel, represent by letter D, is characterized by a percentage of Nb and V of 0.022% and 0.052% respectively. The second steel, which is marked with the letter F, is characterized by the complete absence of Nb and V, or at least with the negligible content (0.002% and 0.003 respectively). The starting material were obtained from a continuously cast billet (Fig.2.1). Steels of both typed were produced through continuous casting without using Electromagnetic stirrers. Some of D and F steels were then hot rolled and the remaining kept unchanged, retaining as cast structure.

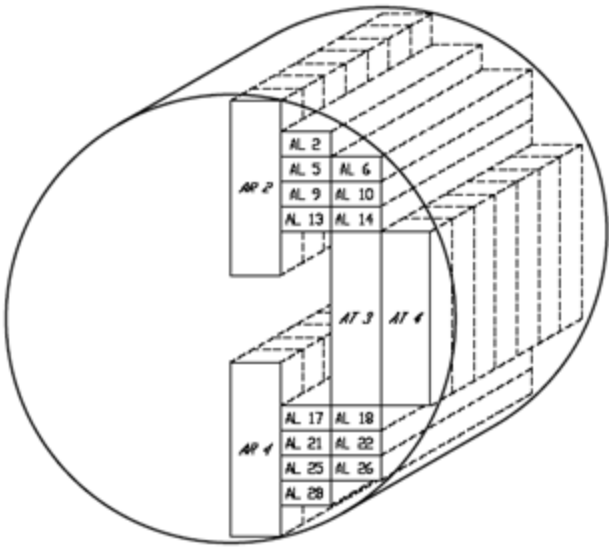


Fig 2.1 continuous casting billet

Steel	C	N	Mn	Al	Si	Nb	V	Ti	S	P
D	0,14	0,0061	1,27	0,027	0,23	0,022	0,052	0,002	0,003	0,014
F	0,15	0,0080	0,86	0,019	0,24	0,002	0,003	0,003	0,007	0,014

Table 2, composition of the steel (wt%)

2.2 Procedure for rolling

The specimens (D-type and F-type) were heated by furnace to 1200 ° C before rolling. Then the hot rolling procedure reduced their thickness from 20 mm to 5 mm (75% reduction).

The rolling was carried out in two steps: an initial one in which the thickness was reduced from 20 mm to 10 mm in steps of 2 mm with 150 rpm(1m/min) rolling speed; the second stage in which the thickness was reduced from 10 mm to 5 mm in steps of 1 mm, mill speed at 300 rpm. The interval time between each rolling is 5 minutes, keeping the specimens in oven.

One third of the samples, both D type and F type, were rolled at 1200 ° C, through the two staged mentioned before. These specimens will be marked with F₁₂ and D₁₂. Another third of the samples, were rolled at 1200 from 20mm thick to 10mm and then at 800 from 10mm to 5mm. they were marked with D₈ and F₈. The samples left, with the as cast structure, were named by D_{AC} and F_{AC}.

The mill featured the following data:

--Cylinders diameter: 135 mm

--Cylinder-surface hardness: 70 HRC

--Power: 3 KW



(a)



(b)

Fig 2.2 (a) rolling mill and (b) resistance furnace for heating the sample

2.3 Sample preparation

Expose the specimens in air for cooling, and take some small pieces of the material (1 cm³) for making samples.

The pieces were taken by cutting along the rolling direction (RD), and the extraction corner is shown in Figure 2.4. Later this small sample was put into the mounting machine, squeezed with thermosetting resin under a thermal cycle consisting a heating (up to 175 ° C), and then, it was maintained isothermal (6 minutes), cooled subsequently.



Fig.2.3 Mounting equipment

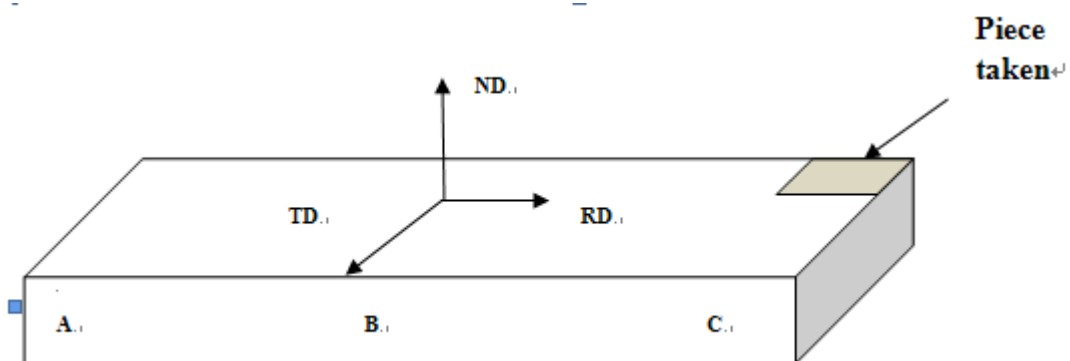


Fig.2.4 Specimen for making samples (RD rolling direction, ND normal direction, TD transversal direction)

The next step was to prepare the sample for optical microscopy. Emery papers were used with the Grit designation increasing from P120 to P1200. After getting this smooth surface, subsequent procedure was to polish them with finer diamond suspension, size 6 μ m 3 μ m and 1 μ m, to eliminate the scratches, hence phases and grain boundary were clearly identified without the disturbance of scratches.

After polishing procedure, in case of surface oxidation, for emphasize the vision of microstructure, immediate etching was necessary. The samples were etched in a chemical solution named Nital 2% for 15s, with the composition of 98% ethanol (CH₃CH₂OH) and the remaining 2% nitric acid (HNO₃). Longer etching may cause over etch, that is corrode the surface to make the grain indistinguishable. The optical microstructure was then visual in microscope, which would be in comparison with the final structure of sample cut from the steel after hot tensile fracture.

2.4 Microstructure analysis of F and D typed before tensile test

Microstructure of the as cast steel, both F and D type obtained under 500X magnitude, are shown in Fig. 2.5 and 2.6

Fig 2.5 represents F type steel with negligible content of Nb and V, and Fig 2.6 represents D type steel with effective content of Nb (0.022%) and V (0.052%).

In both the two kinds as cast steels, allotriomorphic polygonal ferrites (white part) occupy half area in microstructure, and surround the dark pearlite. Ferrite nucleates at the austenite grain boundaries, growing and forming the shape following the grain boundary contours. Grain boundaries are clearly visible in the latter picture.

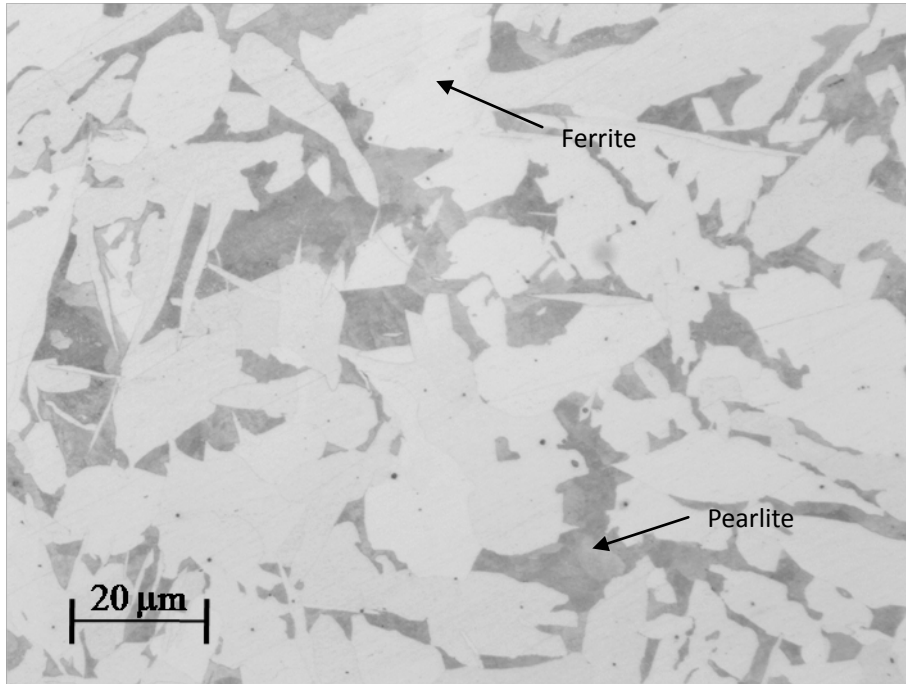


Fig 2.5 microstructure of F type steel as cast structure 500X

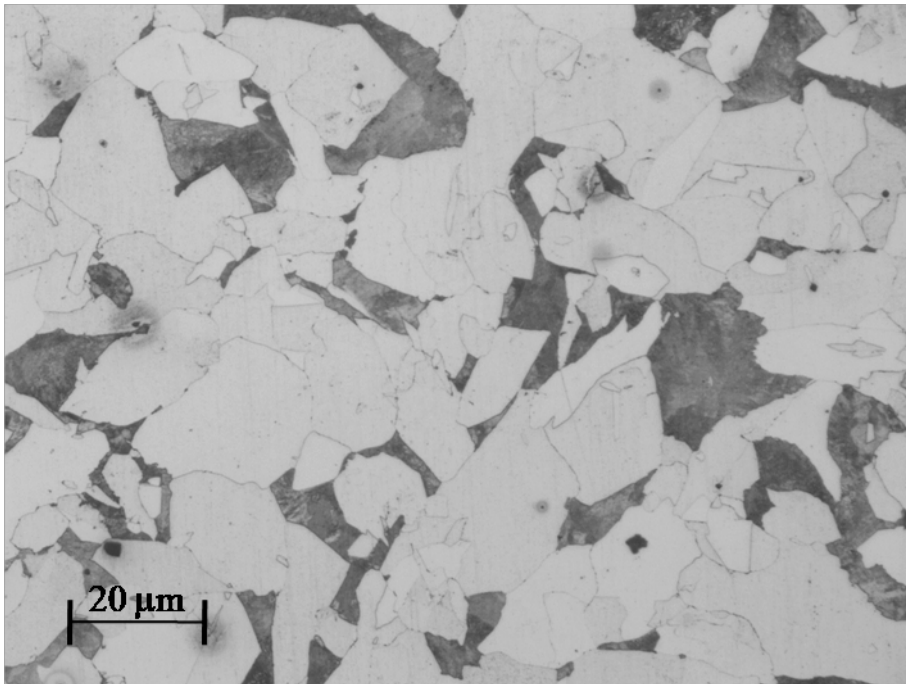


Fig 2.6 microstructure of D type steel as cast structure 500X

Analysis of microstructure is also done on rolled steel type D₈, D₁₂, F₈ and F₁₂. Fig 2.7 represent the microstructure of F type after rolling from 20mm thickness to 10mm

at 1200°C followed by 800°C from 10mm to 5mm, and Fig 2.8 shows the D type with the same condition.

In both of the two microstructures, great changes in grain size and shape have taken place. Ferrites not long exist in polygonal form. By comparing the following two, we found that the morphology is quite different and grain sizes in F₈ are larger than that in D₈. From our previous statement, Nb and V can form fine precipitation during reycrystallization at the austenite grain boundary, effectively restrain the grain boundary, preventing them growing. During our experiment, hot rolling at 1200°C, the dynamic recrystallization may occur, resulting in the microstructure we observed. .

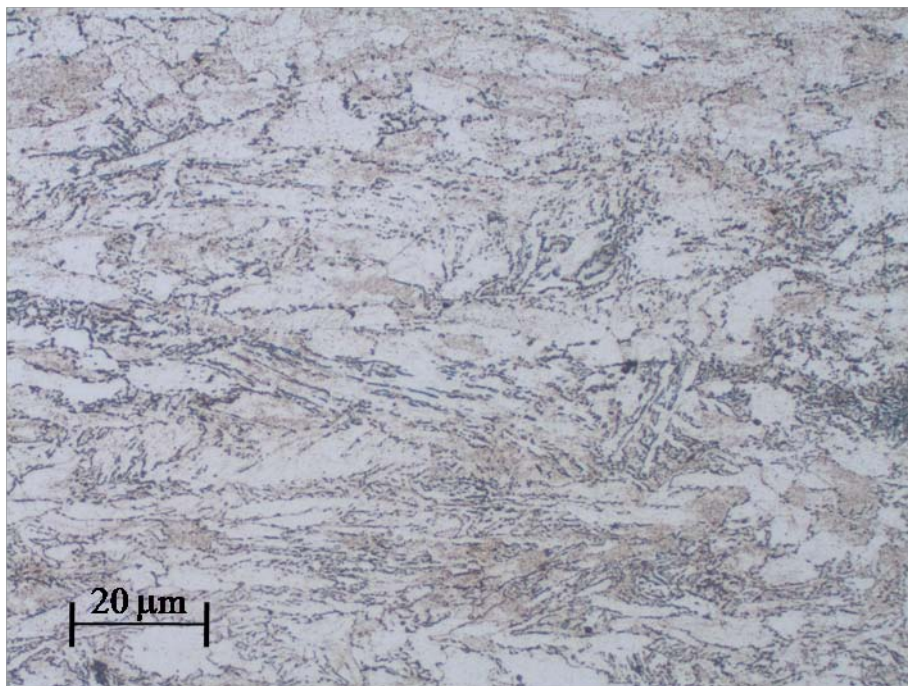


Fig 2.7 microstructure of F₈ rolled at 1200°C followed by 800°C (500X)

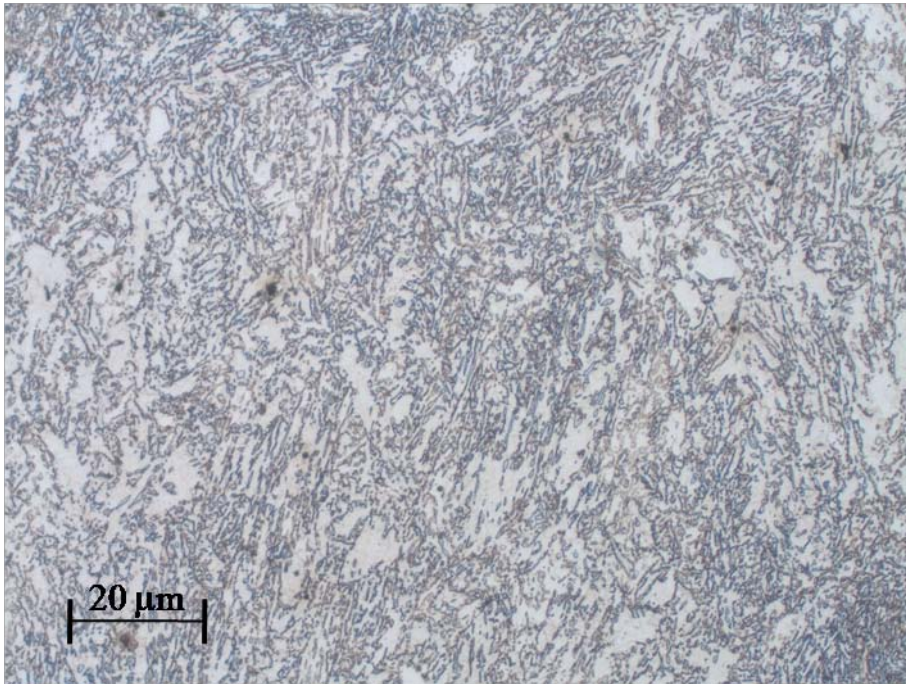


Fig 2.8 microstructure of D₈ rolled at 1200°C followed by 800°C (500X)

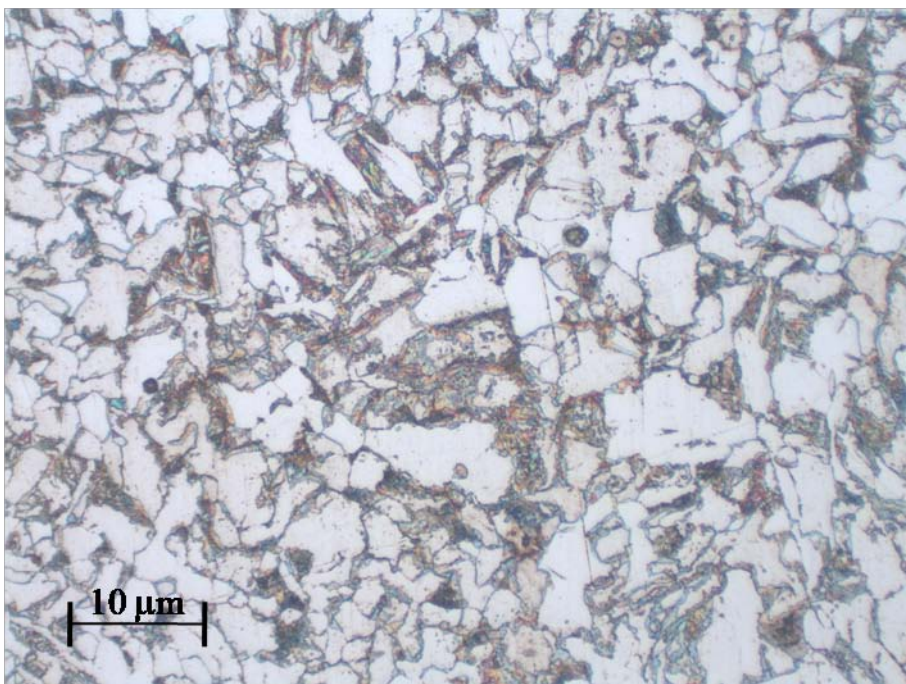


Fig 2.9 microstructure of F₁₂ rolled at 1200°C (1000X)

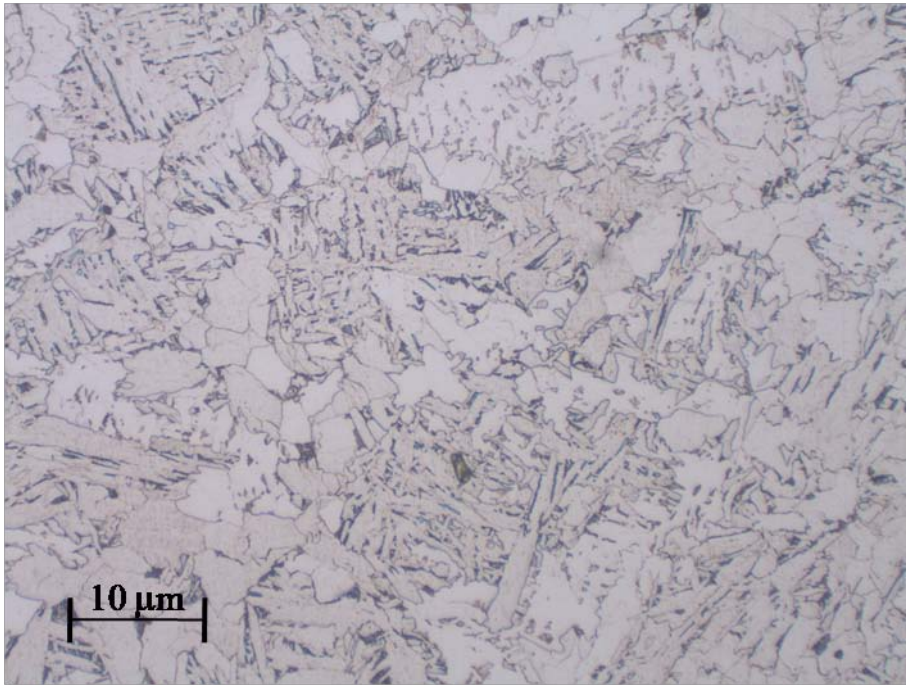


Fig 2.10 microstructure of D₁₂ rolled at 1200°C (1000X)

Fig 2.9 and 2.10 are the microstructure from sample F₁₂ and D₁₂, with hot rolling at 1200°C by 75% reduction. We can easily define recrystallization by the appearance of equiaxed ferrite grain and the black pearlites around in sample F₁₂. The effect became weaker in the microstructure of D₁₂ under the influence of Nb and V carbonitrides, thus grain growth was restrained by the precipitations located on the boundaries.

2.5 Hot tensile test

Before hot tensile, all the specimens were cut into the shape shows in the following picture, to handle them in the clamps for tensile test.

The hot tensile tests were carried out using a tensile machine (INSTRON 1195, USA) equipped with a tubular furnace (Figure 2.13).

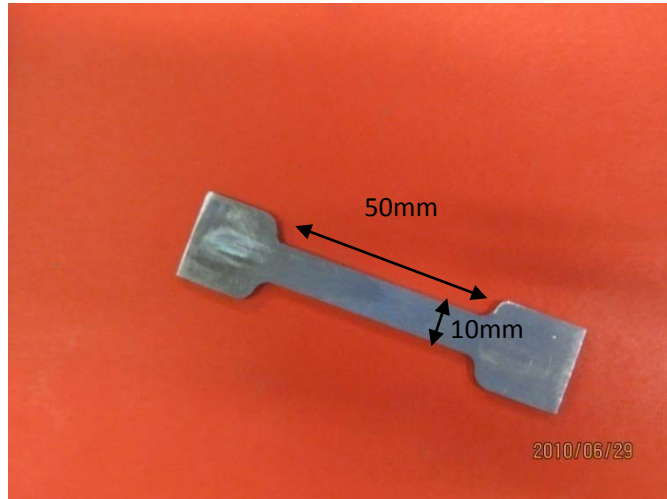


Fig 2.11 Specimen shape

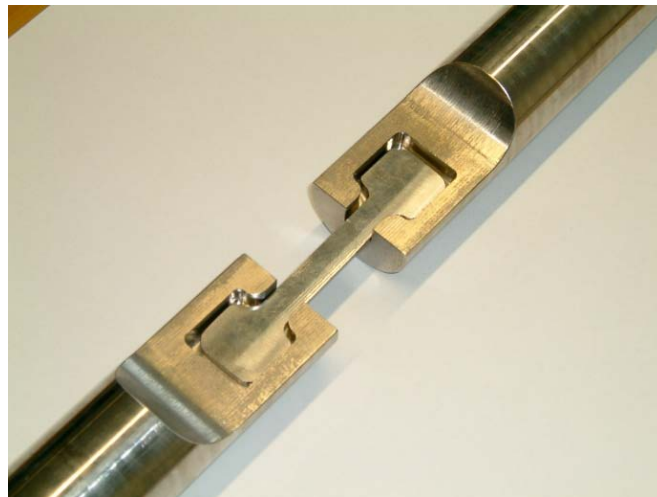


Fig 2.12 Clamps for holding specimen



Fig 2.13 Furnace

All of the specimens were pulled in tension respectively at 800°C, 850°C, 900°C, 950°C and 1000°C, up to 40% deformation (to make complete fracture) in length of the specimen, and the test speed was kept at 5 mm / min. A temperature controller was also used for keeping the furnace fulfill experimental request, with a thermocouple bounded to the clamps touching the test specimen and reading the central temperature during test.



Fig 2.14 Temperature controller

Each specimen was hold for 7-10 min at test temperature before it was strained, to ensure uniform temperature throughout the gauge length.

Once the specimen was fractured, cooling in water was necessary to immediately "freeze" the resulting microstructure so we can analyze it in an optical microscope.

2.6 Illustration analysis for hot tensile test result

2.6.1 Strengthen hardening from Nb

The following two graphs consist of stress-strain curves for both F and D as cast steel tested at all the five temperatures. Comparing each curve of them at the same temperature, it can be noticed that Dac steel has higher yield strength and ultimate tensile strength than Fac steel (Table 3,4), which is the effect Nb precipitation

Temperature (°C)	800	850	900	950	1000
Yield Strength Fac (MPa)	56	49	44	46	38
Yield strength Dac (MPa)	75	52	57	45	39
Yield strength F12 (MPa)	52	37	47	39	29
Yield strength D12 (MPa)	49	53	50	45	36
Yield strength F8 (MPa)	61	44	50	40	32

Table 3 Yield strength of all samples

strengthening and grain refinement.

Temperature (°C)	800	850	900	950	1000
UTS Fac (MPa)	66	69	57	55	45
UTS Dac (MPa)	84	77	74	57	45
UTS F12 (MPa)	71	52	61	50	38
UTS D12 (MPa)	65	73	65	50	40
UTS F8 (MPa)	74	69	62	49	40

Table 4 Ultimate Tensile Strength

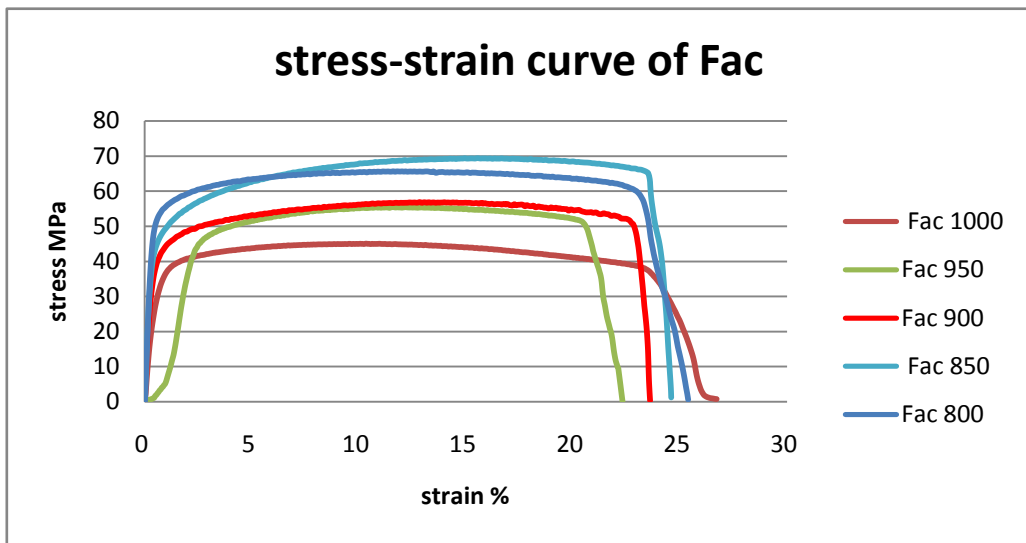


Fig 2.15 stress-strain curve of Fac steel

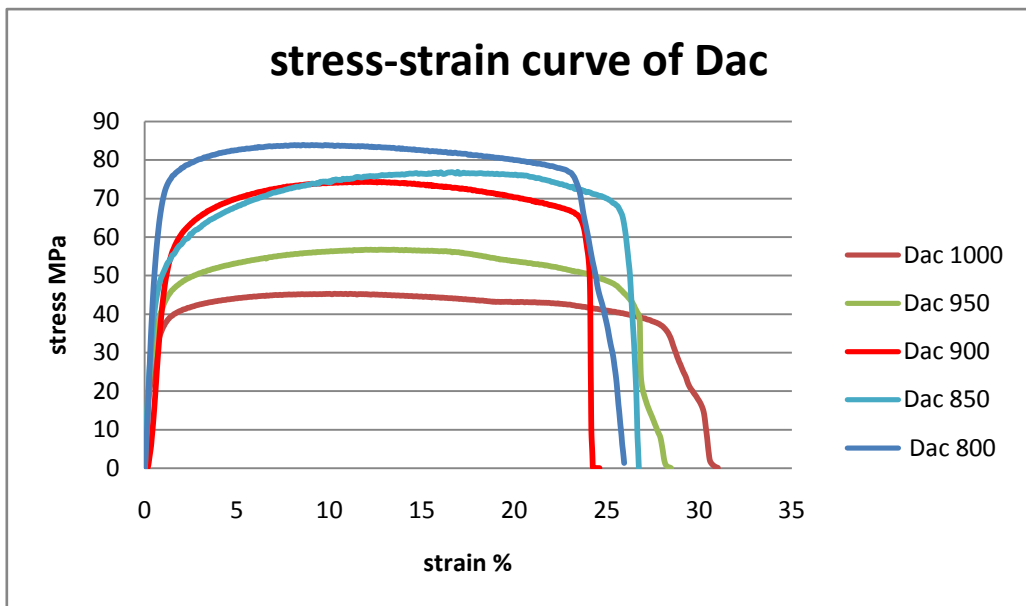


Fig 2.16 stress-strain curve of Dac

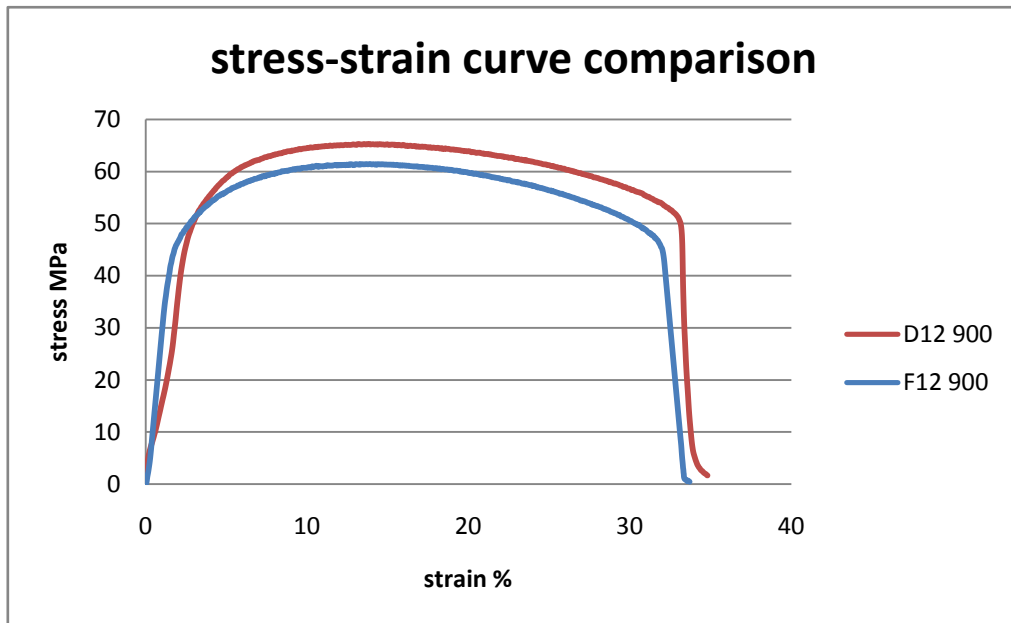


Fig 2.17 comparison between D12 and F12 at 900

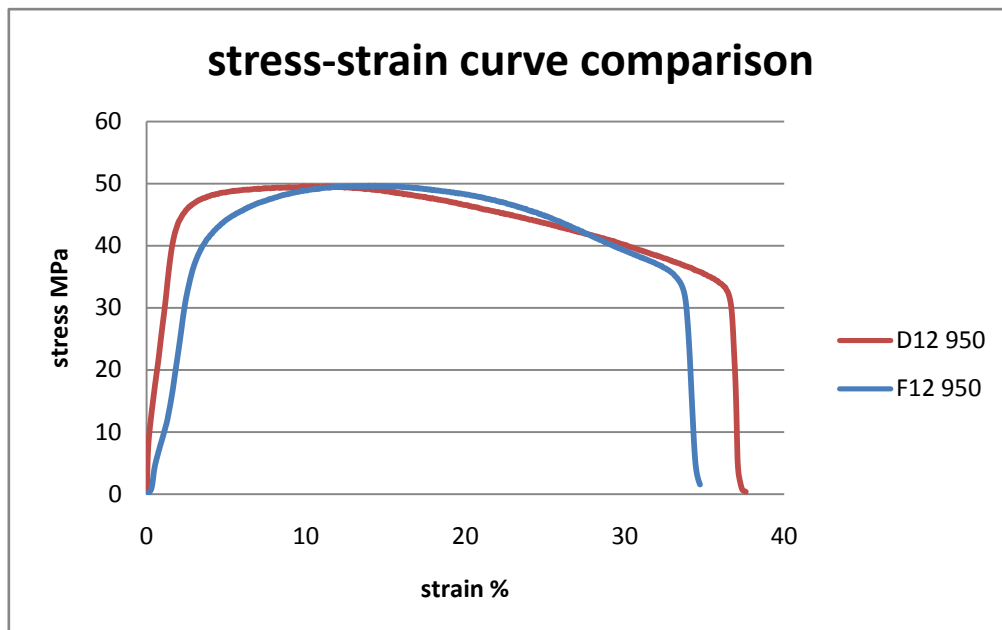


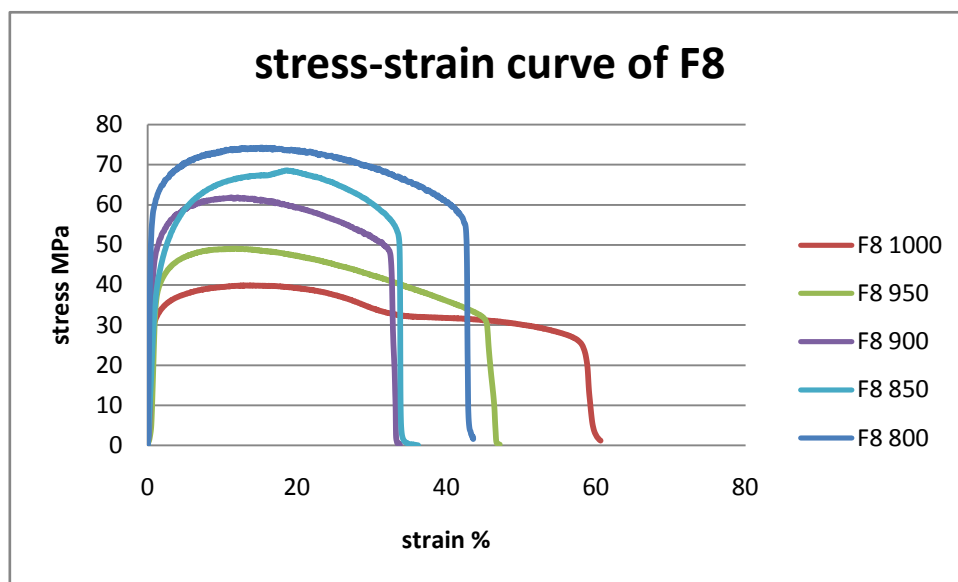
Fig 2.18 comparison between D12 and F12 at 950

Fig 2.17 and 2.18 also show the effect of Nb which strengthen the material a lot compared to the low Nb content steel. Besides yield strength, Table 4 shows us the

data of Ultimate Tensile Strength of different samples at the all test temperature. Most of D typed steels have great enhance in this aspect, proving the excellent hardening effects of Nb and V in steel.

2.6.2 Hot ductility behavior analysis

From Fig 2.19, not only can we define the yield strength (Table 3) of this material at any experimental temperatures, but also find each fracture elongation (Table 5) at different temperature. The largest fracture elongation comes from 1000°C tensile test but at 850°C and 900°C, this number become relatively small, only half of that at 1000°C. This phenomenon is known as the loss of hot ductility.



F 2.19 stress-strain curve of F8

There are two ways to quantify the hot ductility, one is using elongation distance measured from the internal distance between the two shoulders of the clamps, the other is to measure the area of fracture section, calculating the reduction fraction of area compared to initial state, the latter way is always applied in literatures on hot ductility research.

Temperature (°C)	800.00	850.00	900.00	950.00	1000.00
Elongation Fac (mm)	11.68	11.54	11.31	9.95	11.67
Elongation Dac (mm)	11.52	12.88	11.67	13.38	13.63
Elongation F12 (mm)	18.27	13.01	16.31	16.43	12.67
Elongation D12 (mm)	18.83	17.49	16.47	18.16	25.92
Elongation F8 (mm)	21.34	16.96	16.12	22.08	28.38

Table 5 elongation of all tested samples

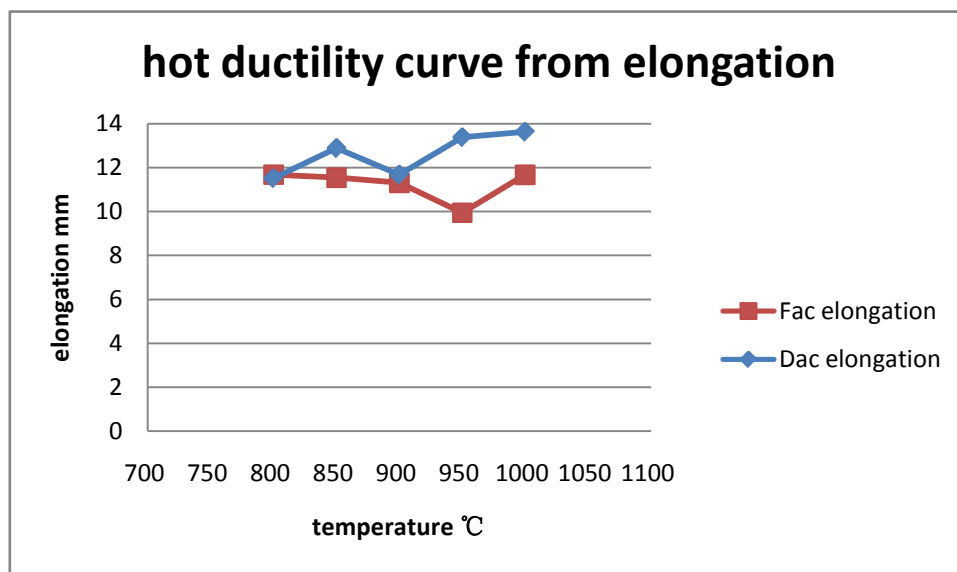


Fig 2.20 comparison of elongation hot ductility curve of as cast steel

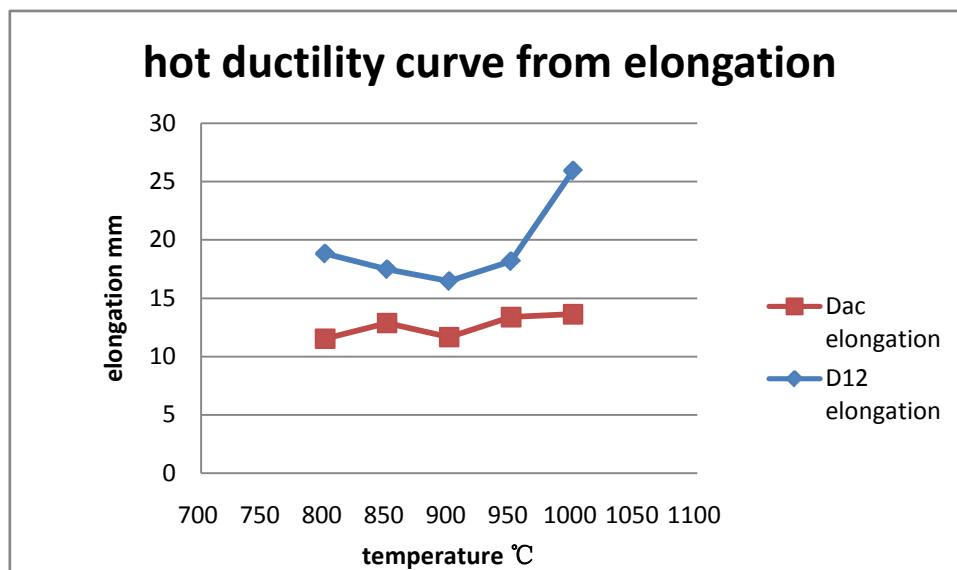


Fig 2.21 hot ductility curve from elongation data

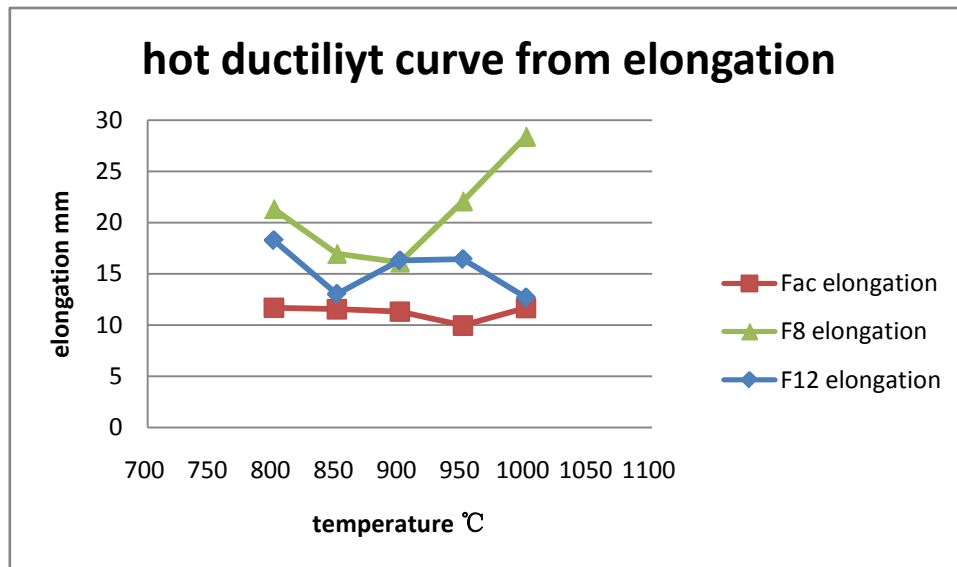


Fig 2.22 hot ductility curves of F steel from elongation data

Data of elongation are present in table 5. From previous experience mentioned in literatures, Nb is detrimental to hot ductility, thus D typed steel should be more brittle than F steel, and elongation of D steel should be generally shorter than F. However, Fig 2.20 shows us a completely different result that D steel outweighs F steel in hot ductility. Also, Fig 2.21 and 2.22 exhibit the comparison of different D and F steels respectively, only D12 and F8 perform determinate trend while others having irregular curves. Considering all the aspect, the data from elongation seems not reliable, that we need to take reduction of area as the criteria in our investigation.

Curves in Fig 2.23 were plotted base on the data of reduction of area from all Dac and Fac samples fractured at all temperatures. Clearly drawn from the figure, Fac has better hot ductility in region 800 to 930°C, but both of them follow a similar trend, ductility loss. As what we have mentioned before, Nb and V play a deleterious role in decreasing the ductility. They form fine precipitation on the grain boundaries, encouraging the grain boundaries sliding into austenite, leading to cracks on the boundaries, which we will discuss in the following section from microstructure image of the fractured samples. Also from Fig 2.19 and 2.23, we conclude that the critical temperature for the material we use is in the region between 850°C and 950°C, where

this microalloyed steel has the worst ductility, indicating to avoid this area in industry application.

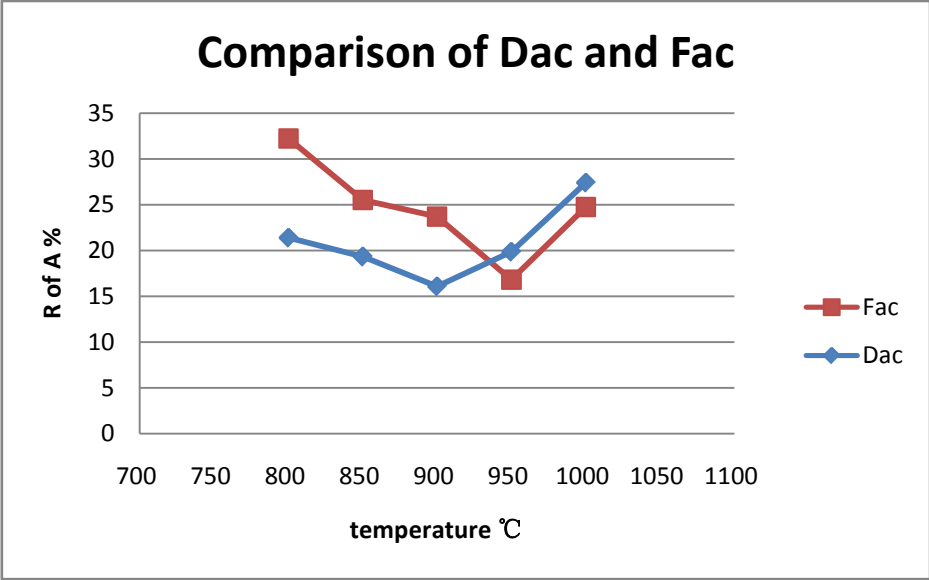
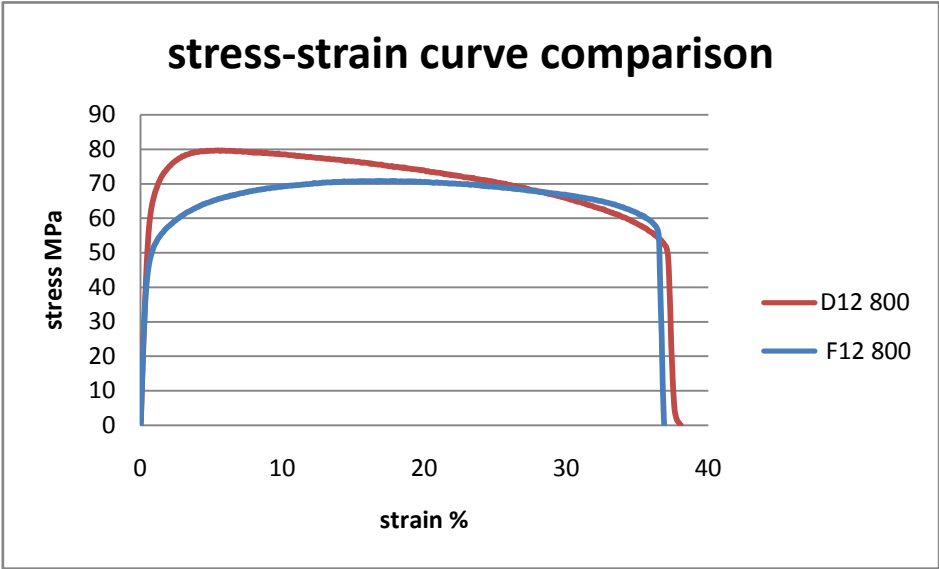


Fig 2.23 hot ductility curve by RA



F 2.24 comparison between D12 and F12

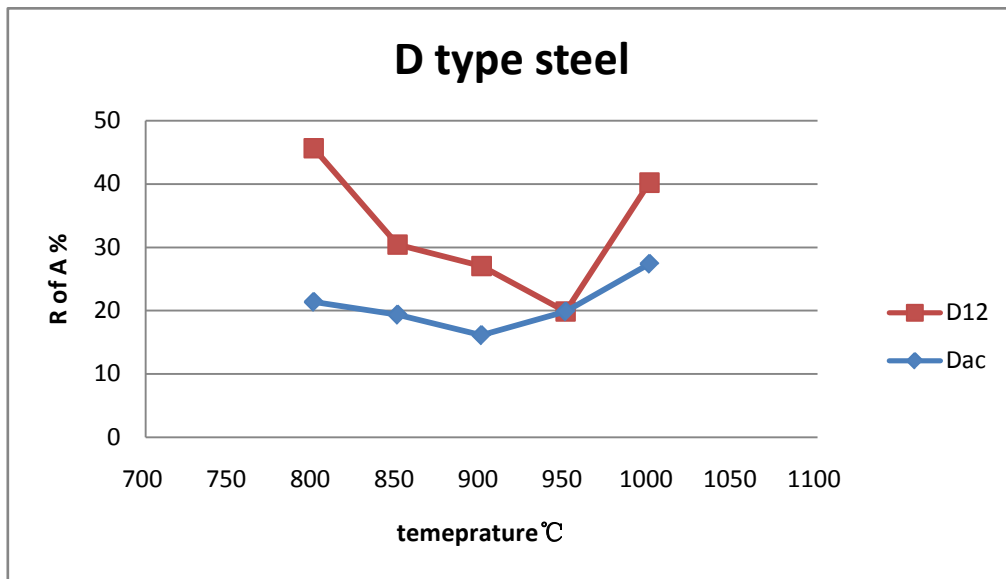


Fig 2.25 hot ductility comparison within D type steel

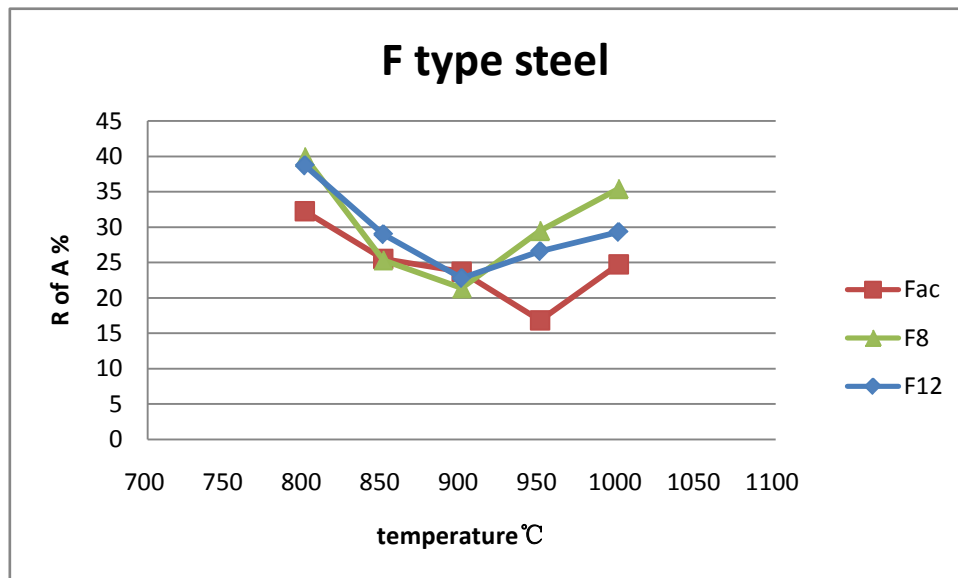


Fig 2.26 hot ductility comparison within F type steel

Obviously seen from the above three plots, samples subjected to hot rolling has better ductility than as cast steels. This is particularly evident in the higher temperature region, which can be explained from the phenomena of dynamic recrystallization in Fig 2.27. Besides that, during deformation, movement of grain boundaries effectively avoid microvoids linking up, decreasing the possibility of cracking, thus the material has good performance in ductility.

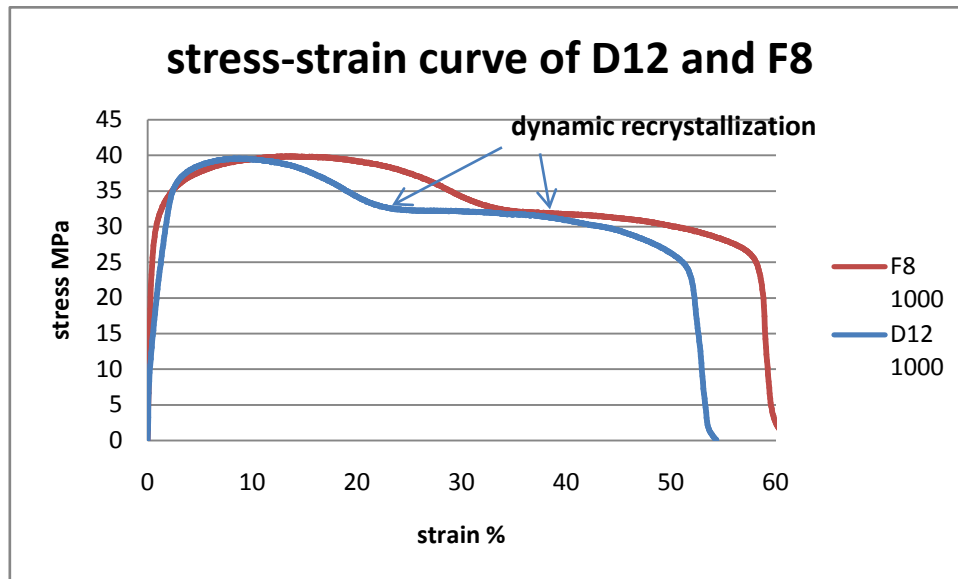


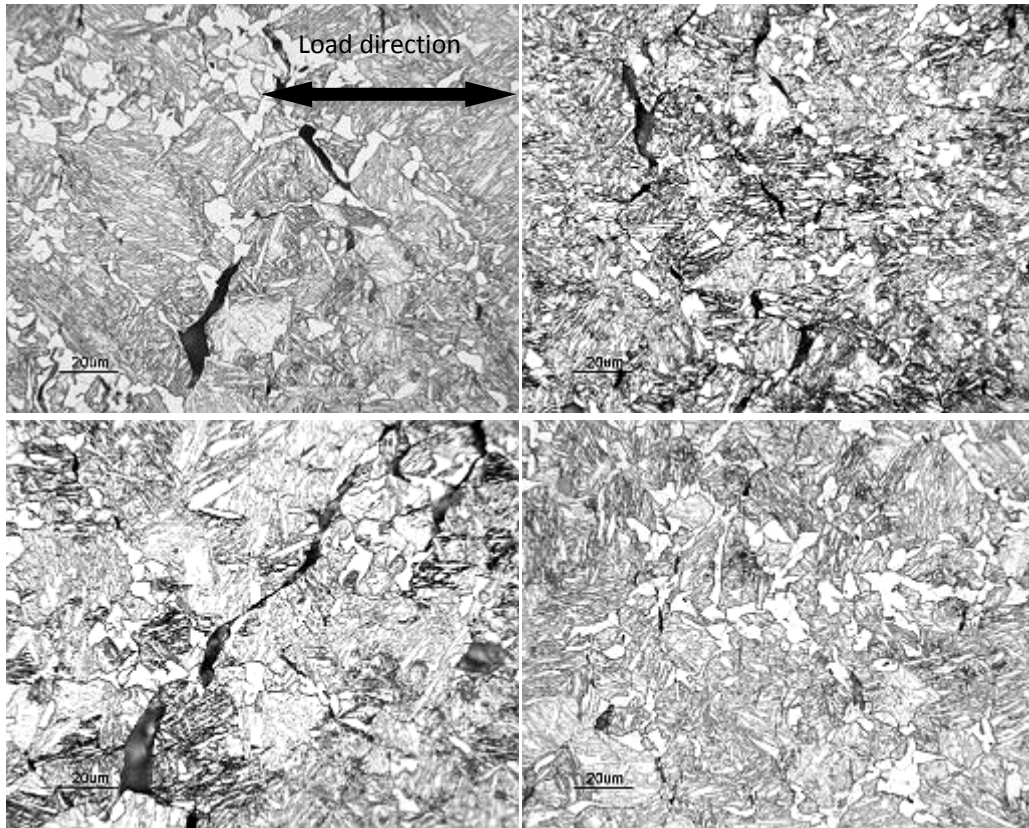
Fig 2.27 evidence of Dynamic recrystallization

2.7 Optical microscopy analysis

Further steps for sample analysis after hot tensile test have been performed. Cut along the transverse direction, the pieces were made into samples. The sample in the vision of microscope must be horizontal, which means tension applied must be horizontal in the picture of microstructure (Fig 2.28). This uniform load direction may help us in finding crack propagation mechanism.

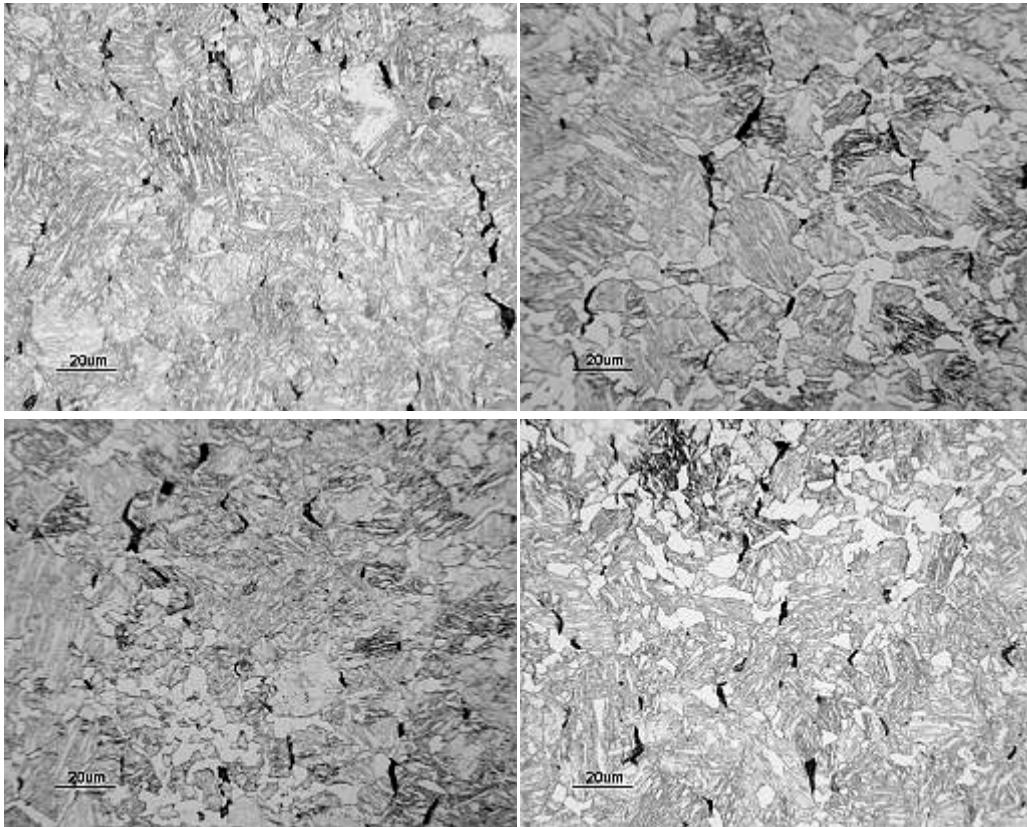
In this case, etching procedure was necessary to highlight the metallographic microstructure and micro-cracks present in the steel. Etching can be realized by reagents Marshall (5ml H_2SO_4 , 8g $COOH_2$, 100ml H_2O) and hydrogen peroxide (30%). Before etching, polishing was done in common ways, and then silicon suspensions were added for eliminating oxidized surface. Mix the two reagents right after polish finishing, immediately put the samples into and shake the sample frequently in 60 seconds. The etched surface must be clear by water to prevent from residual etching solution and over etched.

2.7.1 Microstructure analysis of as cast steel



F2.28 microstructure of Fac strained at 950°C 500X

In Fig 2.28, we can easily define the polygonal ferrite surrounded by martensite, which was formed in quenching after hot tensile test. The black area, thin and long, normally orthogonal to horizontal direction, present at between the grain boundaries, are cracks, the origin for intergranular failure. With the same hot working condition, microstructure of Dac steel shows us similar ferrite and martensite structure, but much more cracks appear in the figures, covering more area than in Fac. Cracks in microalloyed steel are formed from the microvoids concentration, and this is greatly enhanced by the appearance of Nb, V and other microadditions. Their fine matrix precipitations, pinning on grain boundaries, also block and link up the voids to form cracks.



F2.29 microstructure of Dac strained at 950°C 500X

The following two images describe the microstructure after hot working respectively at 900°C and 800°C. From the covering area of cracks, considering the previous condition at 950°C, this trend directly prove our estimation on hot ductility behavior of F as cast steel.

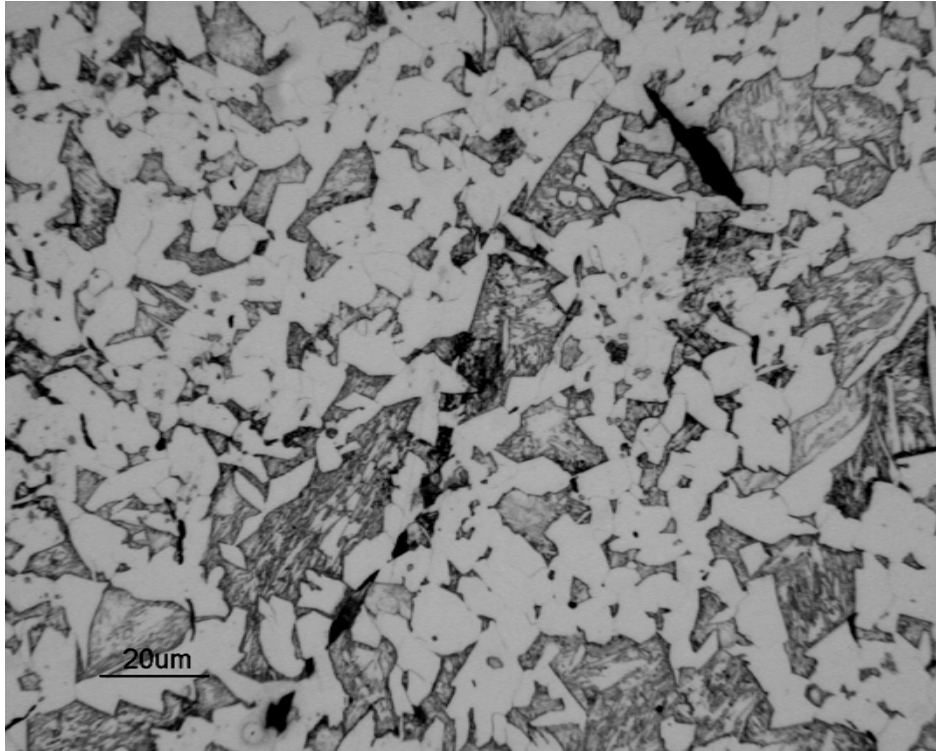


Fig 2.30 microstructure of Fac strained at 900°C 500X

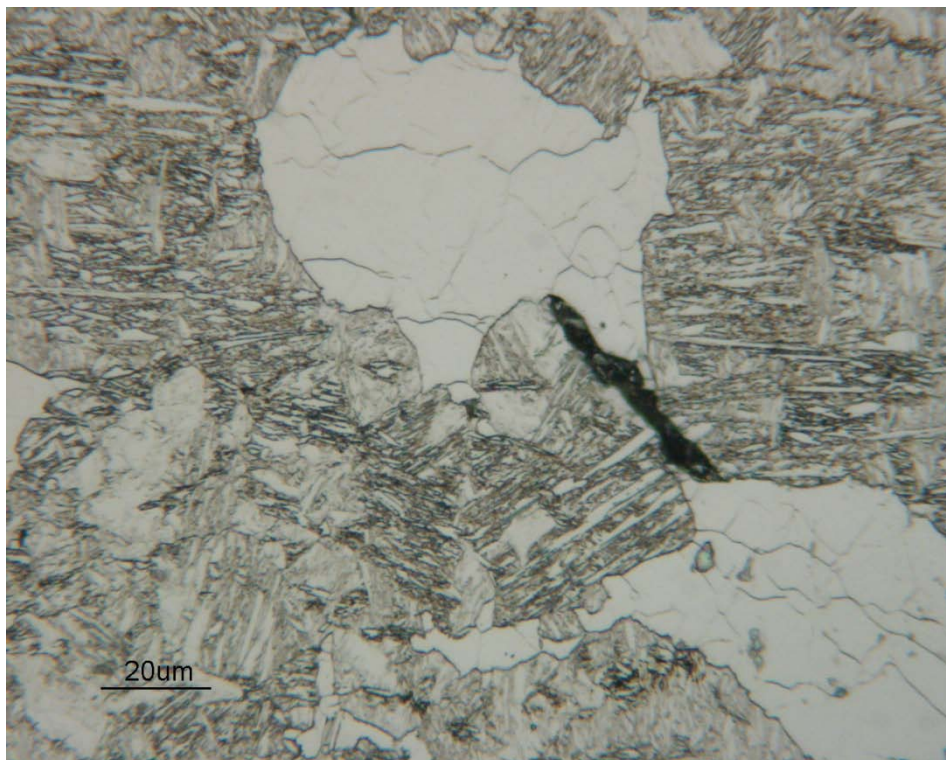


Fig 2.31 microstructure of Fac strained at 800°C 500X

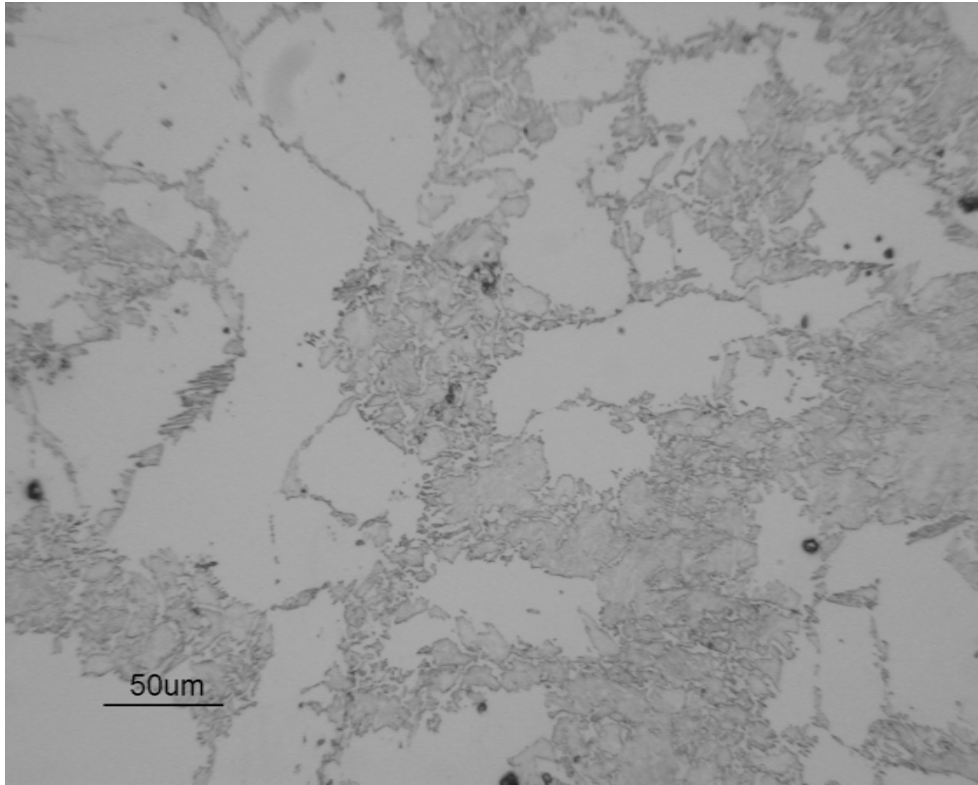


Fig 2.32 microstructure of Dac strained at 800°C 200X

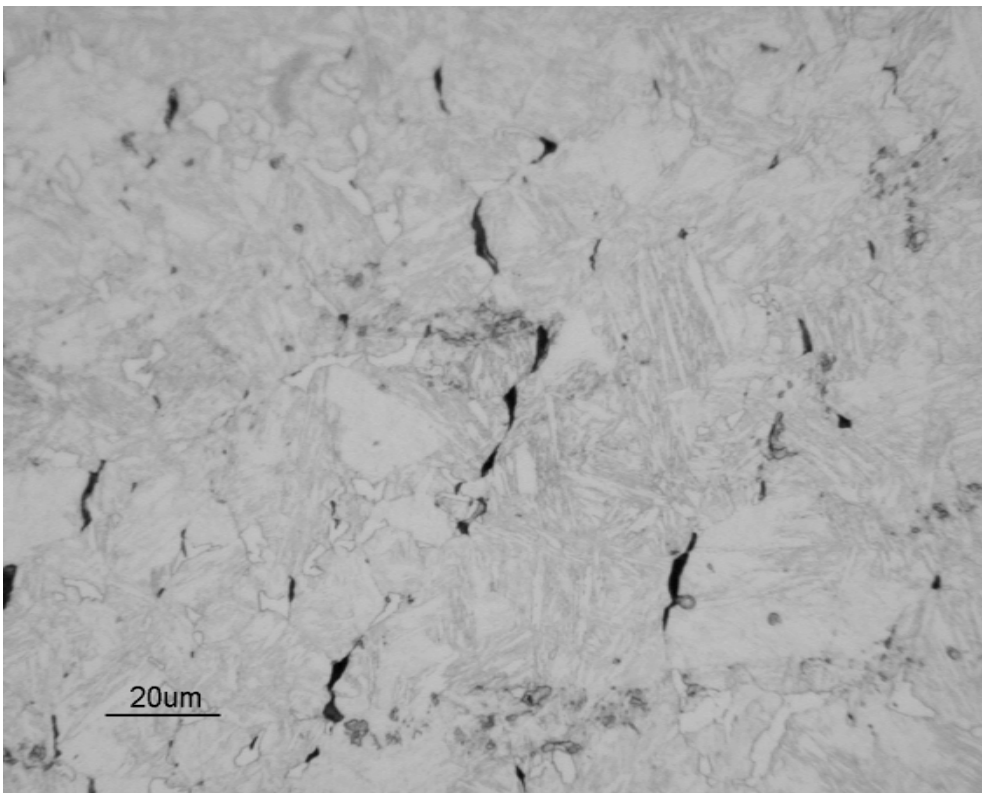


Fig 2.33 microstructure of Dac strained at 850°C 500X

In the pictures of Dac rolled at 850°C, clear intergranular cracks are formed between ferrite and martensite, located right on the boundary. However a significant ductility recovery occur at 800°C that we barely see any cracks in the picture, only some dark spot can be observed inside the ferrite grains. We estimate them to be the inclusions that affect the hot ductility, though not as effectively as Nb carbonitrides do. Strained at 850°C and 900°C, Dac steel has the largest number of cracks, that prove our conclusion on Dac hot ductility from the reduction of area.

2.7.2 Microstructure analysis of hot rolled steel

The structure in hot rolled steel changes into only martensite structure, ferrite disappears. A comparison between steel F8 in Fig 2.34 with steel D8 in 2.36 does not reveals significant changes with the structure, only cracks disperse in the F8 steel with longer size but fewer numbers. The effect of Niobium in encouraging the cracks by precipitations has been well acknowledged, so that it is easy to explain why D8 has more cracks than F8 does. However, from ductility curve data, F8 steel has worse ductility than D8 steel, which disobeys the normal understanding. Through deeper investigation, we found the martensite grain size in F8 steel is larger than that in D8 steel and very few ferrite still exist in the latter. When they were hot rolled at 1200°C, dynamic recrystallization occur in both of them, but D8 steel gets much smaller grain size for the effect of Nb grain refinement while grain growth to a considerable size in F8 steel. Even in hot tensile test, dynamic recrystallization and Nb precipitation lead to smaller grain size in D8 steel. In small grain size structure, propagation of cracks in different direction becomes even harder, which give it higher toughness and better ductility. Although, this explanation is not sufficient, further research need to be done.

Similar in F12 and D12, there are a few ferrite grains on the boundaries of martensite, but much more intergranular cracks. D12 steel senses the critical ductility at 950°C (Fig 2.38), which is smaller than F12 steel. It seems that the negative effects by Nb overweigh positive work by dynamic recrystallization at this temperature.

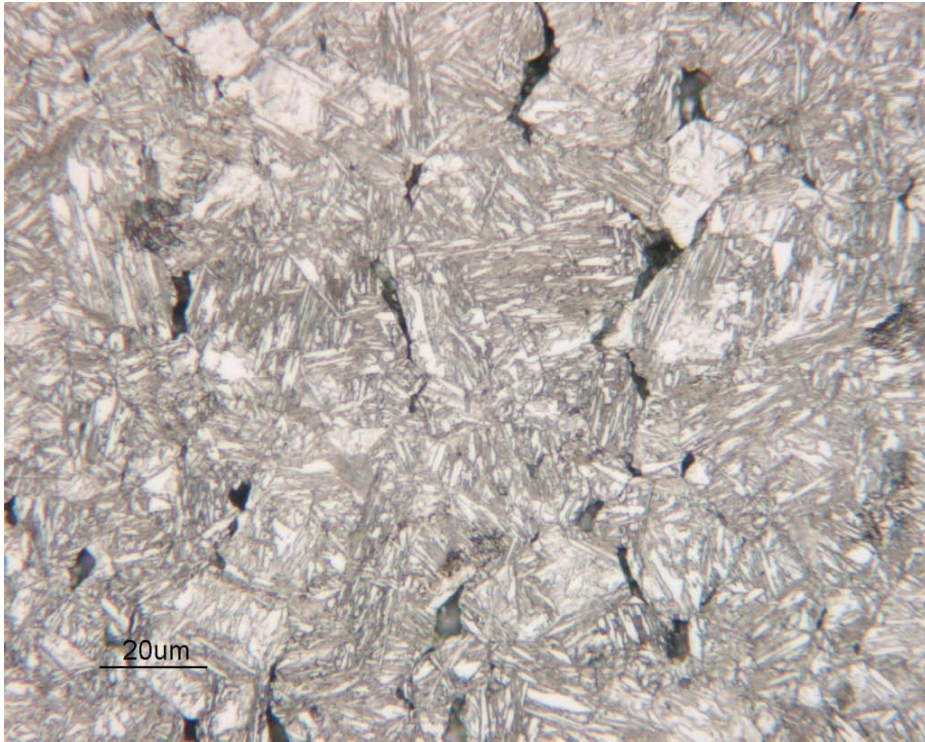


Fig 2.34 microstructure of F8 strained at 950°C 500X

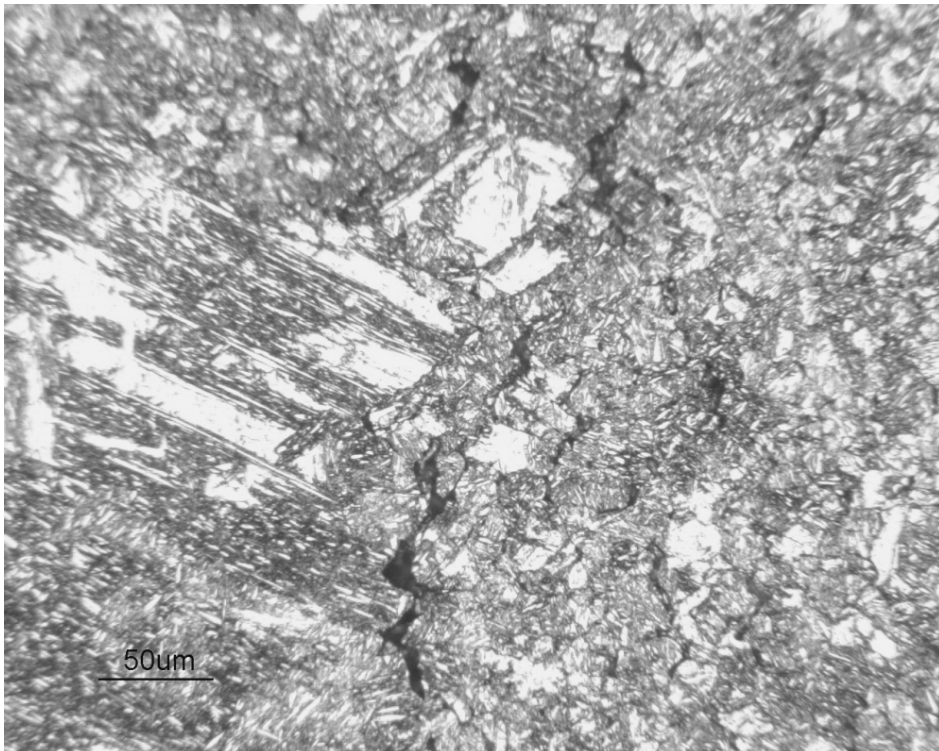


Fig 2.35 microstructure of F12 strained at 950°C 200X

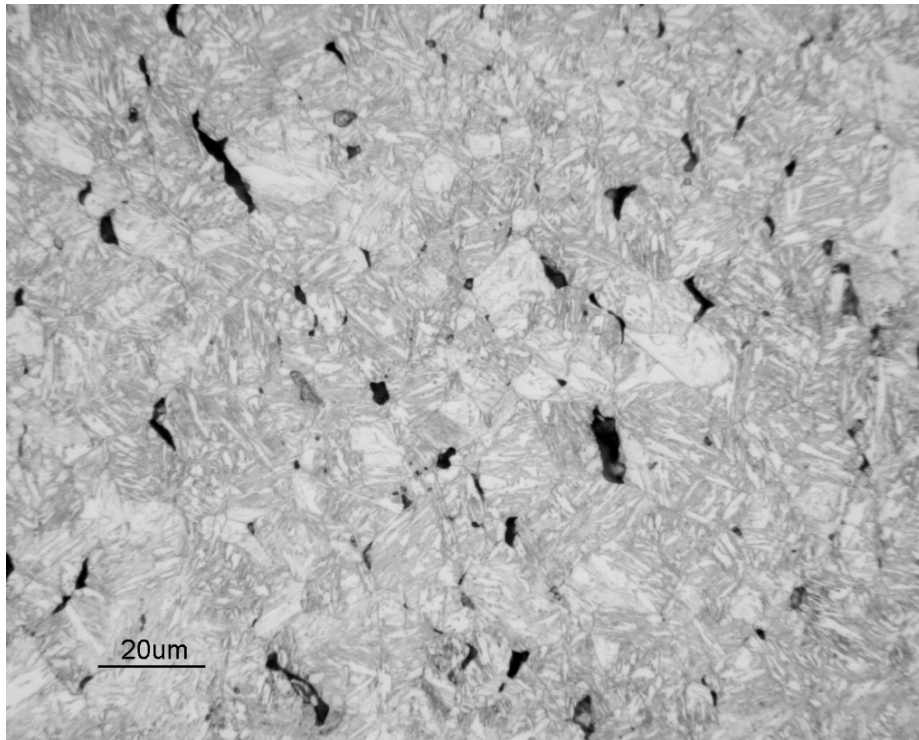


Fig 2.36 microstructure of D8 strained at 950°C 500X

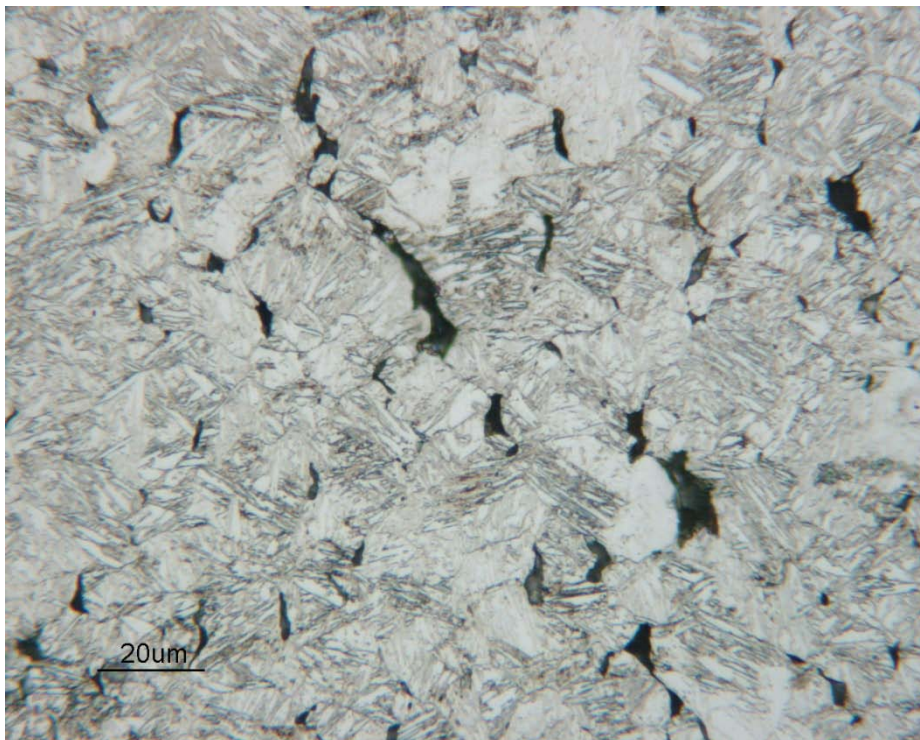


Fig 2.37 microstructure of D12 strained at 950°C 500X

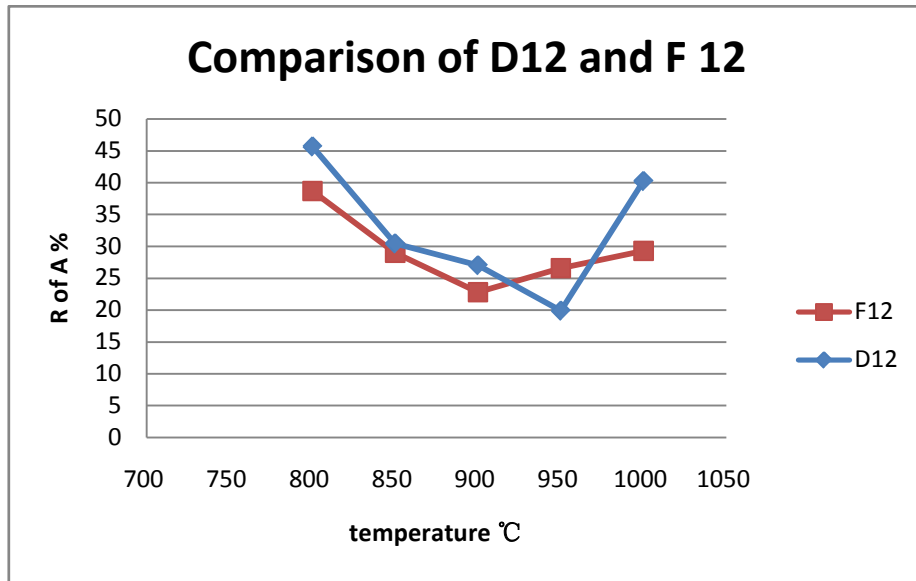


Fig 2.38 Critical ductility of D12 at 950°C

Chapter 3 Discussion and conclusion

The aim of this thesis was to study the hot ductility properties of two kinds microalloyed steels, both in as cast state and hot rolled state. The difference of these two steel lies in composition content of Niobium and Vanadium, and the steel labeled as D containing Nb and V and steel F, without such element.

The aim of microstructural analysis was to collect a reference from the structure of the steels. Polygonal ferrite consist most of as cast structure, while in hot rolled structure, small grains and allotriomorphic ferrites dominated.

Hot tensile tests were carried out at temperatures from 800°C to 1000°C, in order to get the data of stress strain relation and area reduction, both necessary for drawing stress-strain curves and hot ductility curves. Subsequently, observation of microstructure after hot tensile test were an important evidence of the fracture mechanisms compared to the previous microstructure.

By analyzing the stress-strain curves, we find that the D type steel has generally higher yield strength but lower fracture deformation than the F type. This demonstrates that Nb and V are effective elements for strengthening and hardening. They precipitate on grain boundaries and inside of the grains, pinning the boundaries and the dislocations, thus increasing the yield strength.

The hot ductility was determined by fracture elongation data. In a certain temperature region, from 700°C to 1000°C, microalloyed steel always sense a loss in ductility, even worse in the presence of Nb. Nb affects the hot ductility by precipitating on austenite grain boundaries, stimulating void nucleation at ferrite films or during grain boundary sliding [20], which are the main mechanisms of intergranular fracture.

For both of F and D steels, ductility recovers at 1000°C, and in the stress strain curves at this temperature, we find evidence of dynamic recrystallization, which is the reason of hot ductility recovery.

The analysis of microstructure after hot tensile tests was aimed at defining the microstructure and counting the crack number and area. Martensite, formed during quenching from the high temperature of the tests, is the main structure. Microalloying precipitation induced voids, at the grain boundaries leading to cracks. We found that the cracks in D steel were normally greater than those in F steel and their trend within the same steel followed the trend of the hot ductility curves. According to our research, a more cracked structure usually has lower ductility. Nevertheless, in comparison with the as cast steel and hot rolled steel, D12 steel has much more cracks than Dac, but it has much better hot ductility simultaneously.

Drawn from both the two aspects, hot ductility curves and cracks numbers, the critical temperature for hot ductility is between 850°C and 950°C.

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