

INCREASING THE YIELD STRENGTH OF NIOBIUM MICRO-ALLOYED REINFORCING BAR

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A research report submitted to the Faculty of Engineering and the Built Environment, University of Witwatersrand, Johannesburg, in partial fulfilment of the requirements for the degree of Master of Science in Engineering.

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DECLARATION

I declare that this research report is my own, unaided work and where sources have been used they have been credited and acknowledged by means of referencing. It is being submitted in partial fulfilment for the Degree of Master of Science in Engineering (Metallurgy and Materials Engineering) at the University of the Witwatersrand. This work has not been submitted before for any degree or examination at any other institution.

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ABSTRACT

Reinforcing bar, which it is commonly abbreviated as rebar, is used in the construction industry to impart tensile strength to concrete structures, which by nature is very brittle. At ArcelorMittal South Africa Newcastle Works, 460 MPa (minimum yield strength) rebar is traditionally produced by using Vanadium as a micro-alloying addition in order for the mild steel to attain the required strength as specified. However, the fluctuating price of Vanadium over the past years necessitated the use of alternative micro-alloying elements. Niobium is currently used successfully instead of Vanadium on the Rod mill, but not on the Bar mill, due to the difference in cooling facilities between these rolling mills.

Alternative manufacturing routes and strengthening mechanisms for the cost effective production of rebar containing Niobium on the Bar mill was investigated. It was decided to produce a trial cast containing Niobium as a micro-alloying element with a Chromium addition and subsequently roll it into 10 mm and 12 mm rebar at the Bar mill. The minimum yield strength of 460 MPa was not achieved. The average yield strength was approximately 430 MPa on both these sizes.

DEDICATION

To my Lord and Saviour Jesus Christ, who makes all things possible, to my family, who has loved and supported me through my whole life in all of my endeavors and to the love of my life, for his support, love, patience, motivation and understanding always.

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NOMENCLATURE

Aluminium	Al
Carbon	C
Carbon Equivalence	CE
Chromium	Cr
Cold Twisted Deformed rebar	CTD
Continuous Cooling Transformation diagram	CCT
Controlled Cooled Rebar	CCR
Energy Dispersive Spectrometer	EDS
Finishing Temperature	FT
Manganese	Mn
Molybdenum	Mo
Micro-alloying	MA
Nickel	Ni
Niobium	Nb
Nitrogen	N
Quench and Self Temper	QST
Reinforcing bar	Rebar
Silicon	Si
Titanium	Ti
Transmission Electron Microscope	TEM
Ultimate Tensile Strength	UTS, R_m
Vanadium	V
Yield Strength	YS, R_e

1 INTRODUCTION

1.1 Background

Reinforcing bar, abbreviated as rebar, is used in the construction industry to impart tensile strength to concrete structures, which by nature is very brittle. Rebar is therefore a vital material in modern building and engineering projects. Concrete is one of the most important building materials since its properties include good formability, resistance to weathering and fire, and it can withstand high compressive stresses, but unfortunately almost no tensile and shear stresses. A Frenchman named Monier was the first person to apply steel in combination with concrete to improve its tensile properties. Steel is considered the best material to reinforce concrete with since the expansion behaviour for both steel and concrete are similar, so under normal conditions the two materials will expand and contract almost equally. The rebar is mainly ribbed to guarantee a good joint between these two materials and to ensure the cracks remain in the tension zone of a structural component. ^(1, 2, 3)



Figure 1: Ribbed reinforcing bar (rebar)



Figure 2: Use of rebar in construction of a dam wall

Over the years, a lot of research globally has gone into the development of high strength rebar with superior properties, since the use of high strength rebar in concrete structures can greatly reduce the consumption of reinforcing steel. This is because in reinforced concrete, steel alone accounts for 30-40% of the cost. Different types of rebar are available in the market for concrete reinforcement, such as rebar produced by a cold twisted deformed process, a thermo-processed rebar and rebar produced by micro-alloying additions. ^(1, 2, 3)

This rebar must be produced according to international specifications. At ArcelorMittal South Africa Newcastle Works, the BS4449/ 1997 460B standard and SANS 920/2005 450 MPa (cert) weld is used. These standards stipulate that the yield strength of the material must be above 460 MPa or 450 MPa, respectively, and that the Ultimate Tensile Strength (UTS) to Yield Strength (YS) ratio (UTS/YS) must be at least 1.08. The minimum elongation allowable at fracture is specified at 14%. The yield strength specification is important in terms of structural stability. The minimum elongation and UTS/YS ratio provides capacity for plastic deformation and is subsequently a safety factor against fracture. Furthermore, the carbon content and carbon equivalence value, which is an indication of weldability and ductility, is capped at 0.25% and 0.51%, respectively. Weldability is important in order for economical fabrication techniques to be utilised. ^(2, 4, 5)

At ArcelorMittal South Africa Newcastle Works, 460 MPa rebar was traditionally produced using Vanadium as a micro-alloying addition in order for the mild steel to attain the required strength. However, the fluctuating price of Vanadium over the past years necessitated the use of alternative micro-alloying elements. Niobium can be used successfully instead of Vanadium in rebar rolled at the Rod mill as a cost saving initiative. The Rod mill can produce 5.5 mm to 14 mm rebar in coils or straightened lengths. The Bar mill can produce 10 mm to 40 mm rebar lengths. Customers on the local market prefer rebar lengths rolled at the Bar mill for the following reasons:

- Scale removed during straightening at the Rod mill tends to accelerate corrosion of the rebar and hence is aesthetically displeasing.
- Material appears corrugated after straightening at the Rod mill.

1.2 Problem Statement

The Niobium micro-alloyed rebar grade chemistry was designed and optimised for the Rod mill. Since the visual appearance of rebar is very important for stockists, this material was then rolled to lengths at the Bar mill as a trial. However, this was unsuccessful since the minimum yield strength of 460 MPa was not achieved. This was due to the vast differences in cooling rates experienced between the Rod and Bar mill, i.e. the Rod mill has controlled forced fan air-cooling while the Bar mill lacks this facility. Currently the Bar mill still produces rebar containing Vanadium and it requires a higher level of Vanadium alloying addition in order to achieve the mechanical properties due to the slower cooling rates prevailing in the Bar mill process. Due to the high tonnages produced as a result of customer demand for the Bar mill rolled rebar, this is very expensive for ArcelorMittal South Africa Newcastle Works.

As a result of the fluctuating steel market, it often happens that either the Bar or Rod mill has orders exceeding capacity, and since high tonnages of rebar are rolled on both mills, interchangeability can create capacity when needed if

it can be successfully accomplished. This will optimise the use of both mills. Therefore, alternate production routes or strengthening mechanisms need to be investigated in order to achieve a minimum of 460 MPa yield strength on Bar mill produced rebar. The focus will be on the 10 and 12mm sizes, since these are the highest volume demand sizes in the market.

1.3 Objectives of the Research

The primary objective is to use both mills to produce rebar complying with specifications. In order to use the mills interchangeably, it is necessary to investigate different means to achieve a minimum of 460 MPa yield strength in 10 and 12 mm rebar produced at the Bar mill. In order to achieve the primary objective, the research was sub-divided into the following objectives:

- i. Investigation of the current process routes available for the production of rebar to decide the optimum route, within the limitations imposed by the Bar mill, in a cost effective manner.
- ii. Determining the effect of different micro- and macro-alloying elements in order to design a suitable steel grade for the production of the rebar.
- iii. The effect of different microstructures on mechanical properties was investigated to design an optimum microstructure needed to achieve the required minimum strength specification.
- iv. The effect on finishing temperatures, via Gleeble simulated rolling, to obtain the optimum microstructure and hence required yield strength, was investigated.

1.4 Hypothesis

Reinforcing bar with a minimum yield strength requirement of 460 MPa can successfully be produced at the Bar mill by using suitable micro- and macro-alloying element additions.

1.5 Structure of report

The report begins with the literature survey where the various available process routes for the production of rebar and the mechanisms by which they strengthen rebar is explained. The results of various previous trials done at ArcelorMittal South Africa Newcastle Works and other plants are discussed. The reasoning behind the process route of choice for the production of rebar and the design of the steel grade for the trial is explained. The results obtained from the evaluation of the trial rebar is given and compared with rebar produced via other micro-alloying routes and /or cooling conditions. This was followed by a summary of the findings of the trial as well as recommendations for future work to be done.

2 LITERATURE REVIEW

There are three manufacturing routes shown in the flowchart in figure 3 below, which is basically employed for the production of rebar to impart strength, namely:

- Cold Mechanical Working
- In line-heat treatment
- Micro-alloying

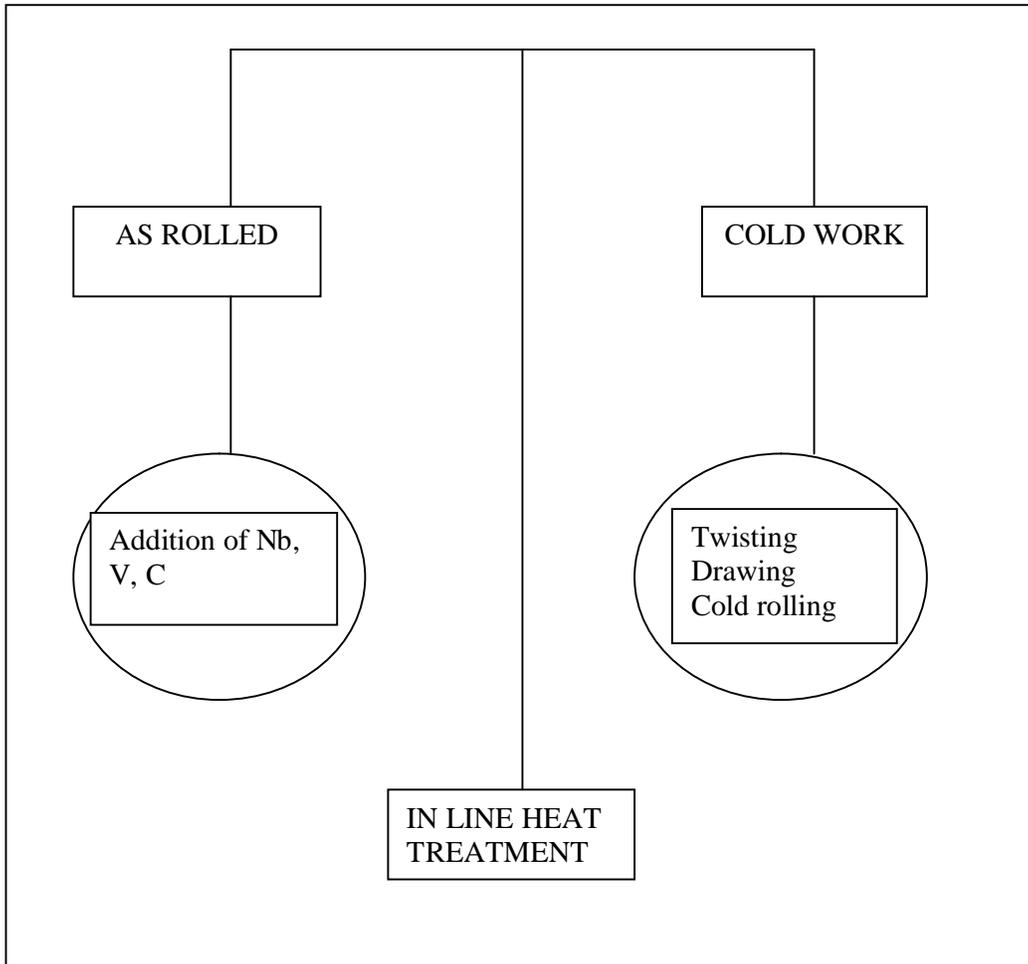


Figure 3: Methods for increasing the yield strength of rebar ⁽⁶⁾

2.1 Cold Mechanical Working

Three cold working methods can be employed to impart strength to rebar i.e. Cold Twisting, Drawing and Cold Rolling. Rebar produced by cold twisting is the most common method used and is abbreviated by CTD. ⁽⁶⁾

2.1.1 Background

The CTD process was developed in Europe in the 1970's and after only a few years of implementation, Europe and the rest of the world, except one country, India, abandoned this method. The main reason for this was that high strength was achieved at the cost of ductility (achieved elongation values were less than 12%), and therefore the high strength CTD bars did not gain global acceptance. This process remained the manufacturing process of choice in India largely due to the previous closed market conditions and significant cost savings. ^(7, 8)

It was taken for granted in India that all the CTD bars met the IS 1786-1985 Grade Fe 415 specification. The continual use of CTD bars with low ductility and the above assumption created a great risk, as most of India lies in a high seismic hazard zone. Emphasis on the UTS/YS ratio in seismic zones is important since this allows for structures to yield, but not catastrophically fail, in the event of an earthquake. To expand further, the minimum value specified ensures yielding will not be confined to a specific area, thus greater elongation of the rebar is permitted before fracture and consequently greater ductility is achieved. It is only at the turn of the century that the CTD process of manufacturing rebar slowly began to be replaced in India. ^(7, 8)

2.1.2 Principles

In cold twisting, the hot rolled mild steel is stretched and twisted beyond its yield plateau and then the load is released. (Refer to Figure 4 below)⁽⁹⁾ This operation results in a residual strain as well as in increased proof strength of the rod. A linear elastic path (modulus of elasticity = original mild steel) is followed upon reloading until the point is reached where the unloading began

- the new increased 'yield point'. Beyond the yield point, the material enters the strain hardening range. ⁽⁹⁾ In cold rolling, the hot rolled rod is passed through a series of rolls. The material is compressed and hence deforms as it is forced into the gaps between the rolls. This deformation then increases the strength of the material. In cold drawing, carbide dies are employed to reduce the cross section of the rod, thereby strengthening the material while employing cold deformation. ⁽⁹⁾

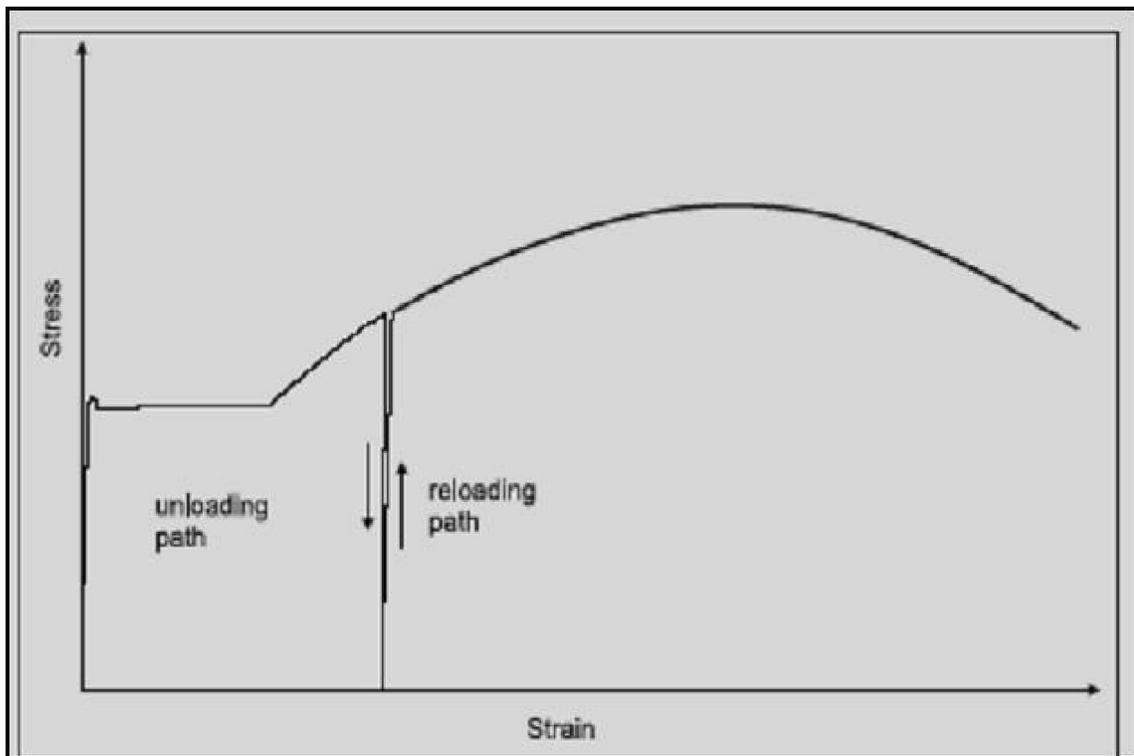


Figure 4: Effect of cold working on mild steel rebar ⁽⁹⁾

2.1.3 Properties

Although cold working increases the proof strength of the steel, it inevitably reduces the ductility of the material. The yield strength of CTD rebar is in the order of 400 MPa with an elongation of ~14%. In addition to limited ductility, this material suffers an inherent problem of poor weldability, since although the carbon content is restricted to some extent, a certain amount is however necessary to achieve the required strength of the rods using this process.

These bars also have a high impact transition temperature, which is undesirable. Since the rods are subjected to torsional stresses, they become less corrosion resistant. Incorrect pitch of twisting (over - or under-twisting) can result in undesirable results; hence care needs to be taken in this regard. ^(2, 9)

Since this method involves an additional processing operation, extra investment costs are incurred. Because it is a simple process, the operating costs are at a minimum. Cold rolling results in good section control and coil presentation. However, the ductility is much lower than that achieved in hot-rolled steels. A cold drawing process also inherently reduces the ductility of the material. ^(6, 9)

2.2 In-line Heat Treatment

2.2.1 Background

The TEMPCORE® process, or Quench and Self Temper (QST) process, as it is commonly referred to, is an in-line heat treatment process applied commonly for the production of high quality rebar, especially in developed countries. ^(6, 9) The TEMPCORE® patented process for producing rebar is licensed to Centre de Recherches Metallurgiques (CRM), Belgium. The TEMPCORE® process imparts strength to rebar by using a thermo-processing control technique. THERMEX® is another patented process also employing “Quench and Temper” technology. “Quench and Temper” technology was developed in the early 1980’s in order to replace the CTD process. Rebar produced by this method gained global acceptance by civil engineers, especially since it met their requirements for seismic zones, i.e. minimum YS of 500 MPa with adequate ductility.

2.2.2 Principles

The reheated steel billet is rolled and reduced progressively through all the rolling stands in order to achieve the required final shape and size. It is only immediately after the last rolling stage that the TEMPCORE® process is applied in three successive stages. This process is illustrated in Figure 5

below and it further illustrated in relation to the continuous cooling diagram in Figure 6 below. ⁽¹⁰⁾

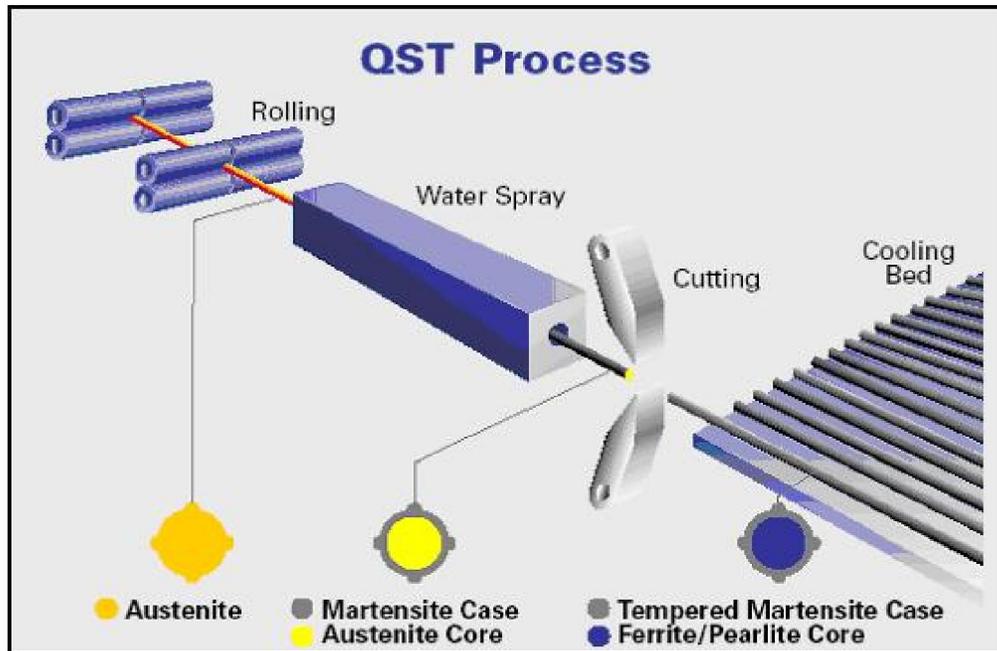


Figure 5: TEMPCORE® Process ⁽¹⁰⁾

Stage 1 - Quenching: Rapid quenching upon exit of the last rolling stand is employed by specially designed cooling water spray system. ⁽¹⁰⁾ . The cooling efficiency of the system is very high. This is due to the disruption of vapour blanket formation around the bar since the kinetic energy of the water is high and hence allows for immediate fully wetted cooling to occur. The surface layer of the bar is quenched into the hard, but brittle, martensite phase up to a certain depth below the skin, whilst the core remains austenitic. Depending upon the operating and controlling parameters of the process, a layer below the martensitic layer can be completely or partially transformed to bainite. ^(4, 6)

Stage 2 - Self-Tempering: When the bar leaves the area of drastic cooling and is exposed to air, a temperature gradient is created through the cross-section of the bars. Heat transfer then occurs from the hot core to the quenched surface layer by conduction until the temperatures are equalised. This results in the peripheral martensite, which was formed in Stage 1, being self-tempered. The core still remains austenitic to ensure that adequate

ductility is achieved and high yield strength is maintained. This temperature-equalisation stage is dependent upon bar diameter and the application of cooling conditions during Stage 1. ^(1, 6)

Stage 3 – Atmosphere cooling: This stage occurs on the cooling bed where the austenitic core transforms to ductile ferrite and pearlite due to its much slower cooling rate. The formation of this microstructure is dependent on specific alloy chemistry, bar diameter, bar entry temperature to the rapid cooling system, duration of cooling, and cooling efficiency. The final microstructure consists of a strong surface layer of tempered martensite, intermediate bainite layer and ductile core of ferrite and pearlite and it is this unique combination that ensures the strength and ductility of the rebar. ⁽¹⁰⁾

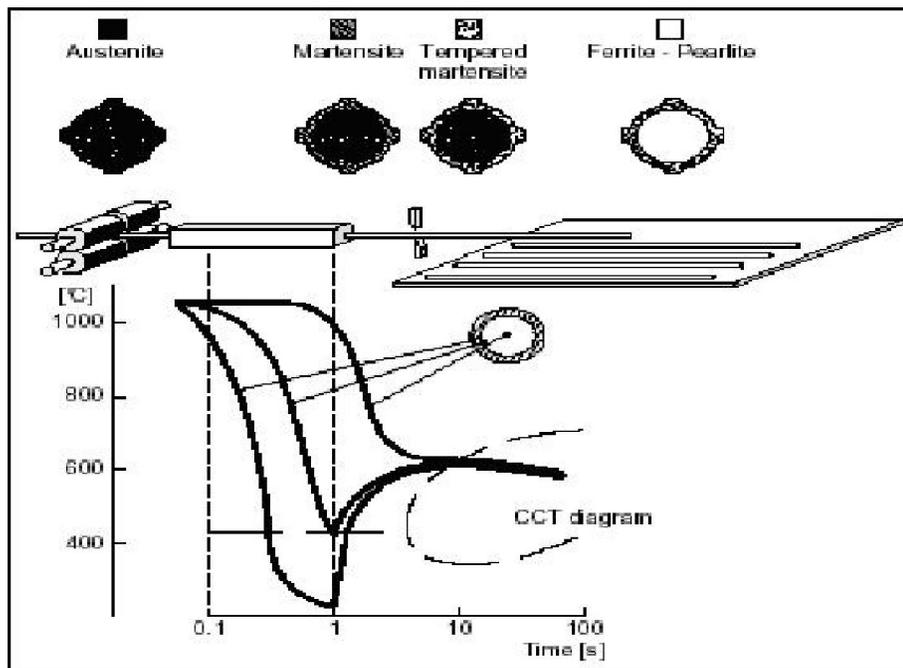


Figure 6: TEMPCORE® process in relation to the CCT diagram ⁽⁴⁾

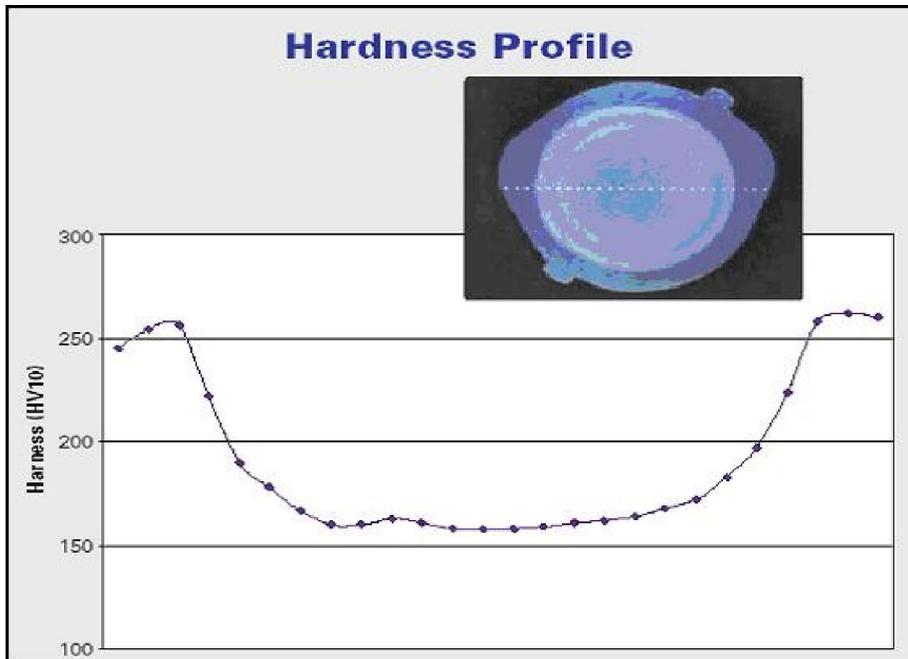


Figure 7: Hardness profile of a cross-section of a QST bar ⁽¹⁰⁾

Figure 7 above shows the typical hardness profile of a cross-section of a rebar produced by the QST process. The variations in the hardness profile are due to the different microstructures present in the rebar as shown in Figures 8, 9 and 10 below.

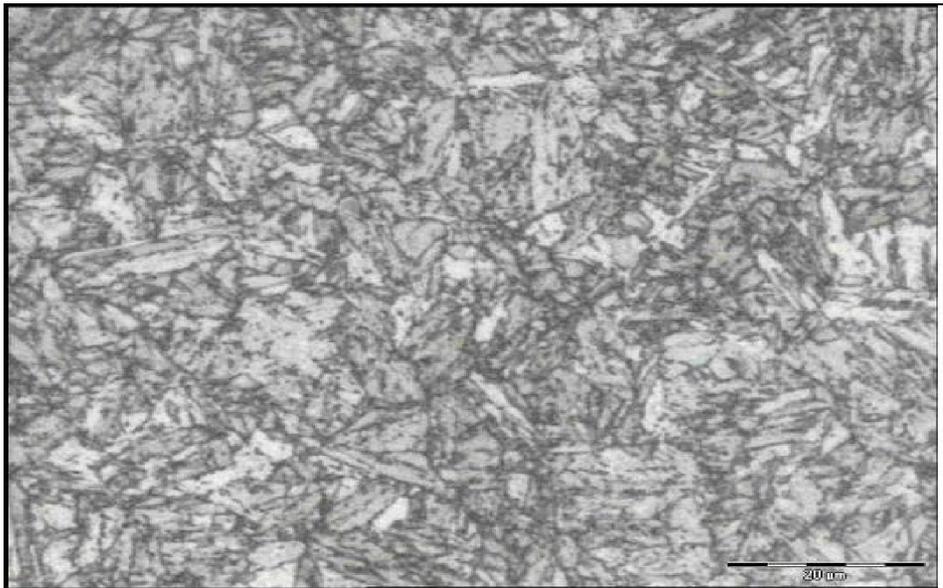


Figure 8: Photomicrograph illustrating a Martensitic microstructure ⁽¹⁰⁾
 Magnification: 500x Etchant: 2% Nital

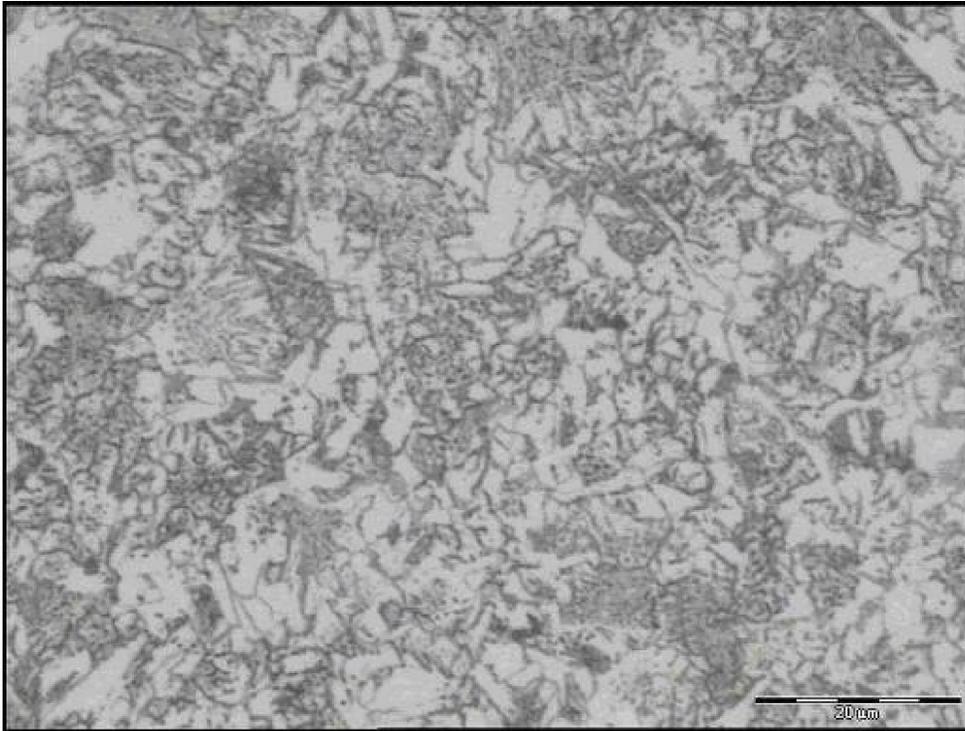


Figure 9: Photomicrograph illustrating a Bainitic microstructure ⁽¹⁰⁾
Magnification: 500x Etchant: 2% Nital

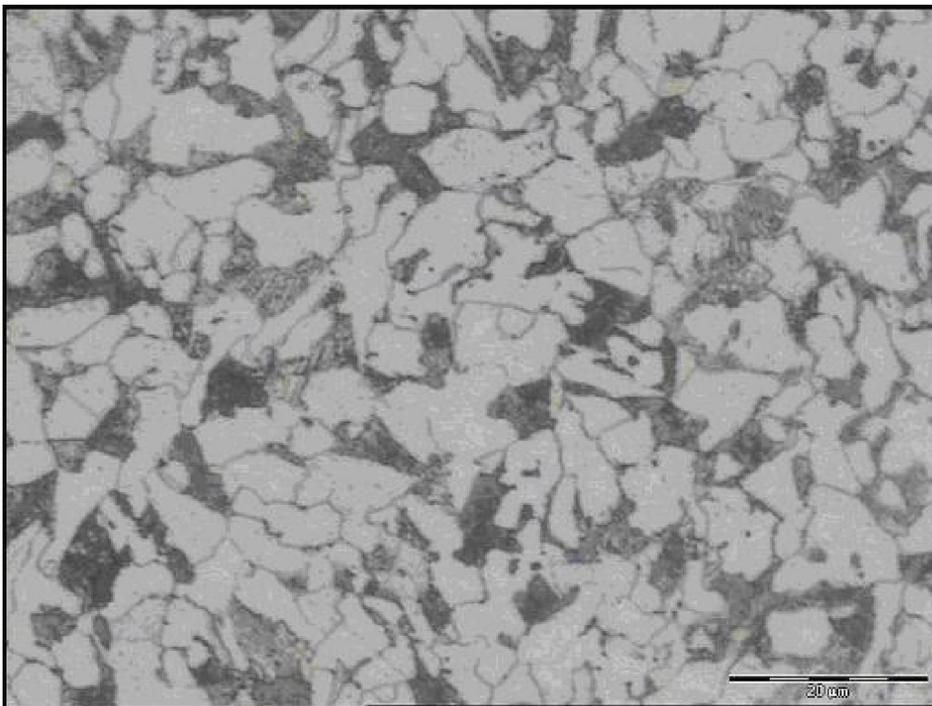


Figure 10: Photomicrograph illustrating a Ferrite and Pearlite microstructure ⁽¹⁰⁾
Magnification: 500x Etchant: 2% Nital

2.2.3 Properties

The TEMPCORE® process uses the composite microstructure in order to attain the required properties, therefore enabling the use of lower Carbon and Manganese contents to ensure better ductility and good weldability and high bendability. In rebar produced by this method, the microstructure and hence mechanical properties vary continuously from the surface to the centre of the rebar. The overall yield strength is dependent on the volume fraction of the individual phases present and is therefore in turn dependent on the process parameters, the most important being the water flow rate, quenching time (rebar finishing speed), finishing temperature and steel chemical composition. For a given chemical composition, the yield strength is dependent on the self-tempering temperature and martensitic and bainitic depth. ⁽⁶⁾

Rebar produced by this method meets a YS of minimum 500 MPa and also has relatively good corrosion resistance. Furthermore, no preheating or post heating is required during welding. The tempered martensite surface layer imparts high thermal resistance to rebars even at temperatures of up to 600°C. However, prolonged exposure at these conditions will result in a compromise of the mechanical properties. ⁽⁹⁾

2.2.4 CCR at ArcelorMittal South Africa Newcastle Works

Previously TEMPCORE® or CCR (Controlled Cooled Rebar), as it is referred to on the plant, was used as process route for the production of Rebar. It was stopped and micro-alloying with Vanadium (V) then became the production route of choice in order to achieve the required mechanical properties.

In 2005 CCR was trialed again at the Bar mill when the Vanadium price increased drastically. Reinforcing bar was successfully produced using a plain carbon steel grade without the use of expensive micro-alloying elements to comply with the specifications of the CCR method, e.g. achieving of a minimum of 460 MPa YS. However, the Bar mill production yield losses (material losses due to cobbles and time to build up the mill) were high.

Furthermore, the rolling tempo had to be decreased by two-thirds of normal production rolling tempo, which made it less advantageous. Therefore a breakeven price for vanadium was calculated on the plant, i.e., if the vanadium price exceeded a certain level, only then would the use of the CCR process be warranted.

2.3 Micro-alloy Additions

2.3.1 Background

Steels containing micro-alloying elements are considered as very important in the industry and are estimated to constitute approximately 12 % of the total world steel production. Their importance is derived from the fact that very low levels of micro-alloying elements are needed to cause major strength and toughness improvements in steels. Hence, the popularity of micro-alloyed steels in the market place is simply due to their ability to increase mechanical properties in an economically advantageous manner. The development of these micro-alloyed steels has led to the expansion of some key industries including oil and gas extraction, transportation and construction. Over the past 40 years extensive research has been performed on the addition of alloying elements such as Vanadium (V), Niobium (Nb) and Titanium (Ti) in amounts of less than 0.1 weight % to increase the strength of hot rolled structural grade material. Strengthening of rebar was found to be possible without increasing Carbon and/ or Manganese contents since the increase of these elements proved detrimental to weldability and toughness of the steel. It is interesting to note that Vanadium was reported as the first micro-alloy element to be widely used as an addition to C-Mn steels dating back as early as 1916. ^(11, 12)

2.3.2 Principles

The combinations of metallurgical factors that govern the structure and behaviour of micro-alloyed rebar include solid solution, grain refinement, dislocations and precipitation hardening. A typical breakdown of how these factors contribute to the strengthening of hot rolled steel is illustrated in Table 1 below. The mechanisms by which V, Nb and Ti influence the properties of

the steel include the solute drag effect and the formation of carbides and nitrides, which will be further explained. ^(11, 13, 14)

Table 1: Percentage contribution of strengthening mechanisms to hot-rolled steels ⁽¹⁵⁾

Strengthening Mechanism	Percentage contribution
Precipitation Hardening	32 %
Grain Size Refinement	41 %
Pearlite	4 %
Solid Solution	8 %
Base	15 %

Solute Drag Effect

In the solid solution, micro-alloying elements, as well as all other elements in steel, retard all diffusion-controlled processes. The solute drag effect is also known as diffusion retardation. It is found to be stronger with a bigger difference in atomic size of any specific element as compared to that of the iron atom. Niobium is the most effective of the three micro-alloying elements in this context, followed by Ti (See Table 2 and Figure 11 below). ⁽¹⁴⁾

During hot rolling of steel, the solute drag effect assists in grain refinement by ⁽¹⁴⁾:

- Preventing secondary grain growth during the interpass time, since grain growth is a diffusion-controlled process.
- Retarding the onset of recrystallization, by niobium carbide precipitates.

Table 2: Atomic Radii of Refractory Metals ⁽¹⁴⁾

Element	Atom radius in nm	Difference to the Fe-atom in %
Ti	0.147	+14.8
V	0.136	+6.2
Cr	0.128	~ 0
Nb	0.148	+15.6
Mo	0.140	+9.4

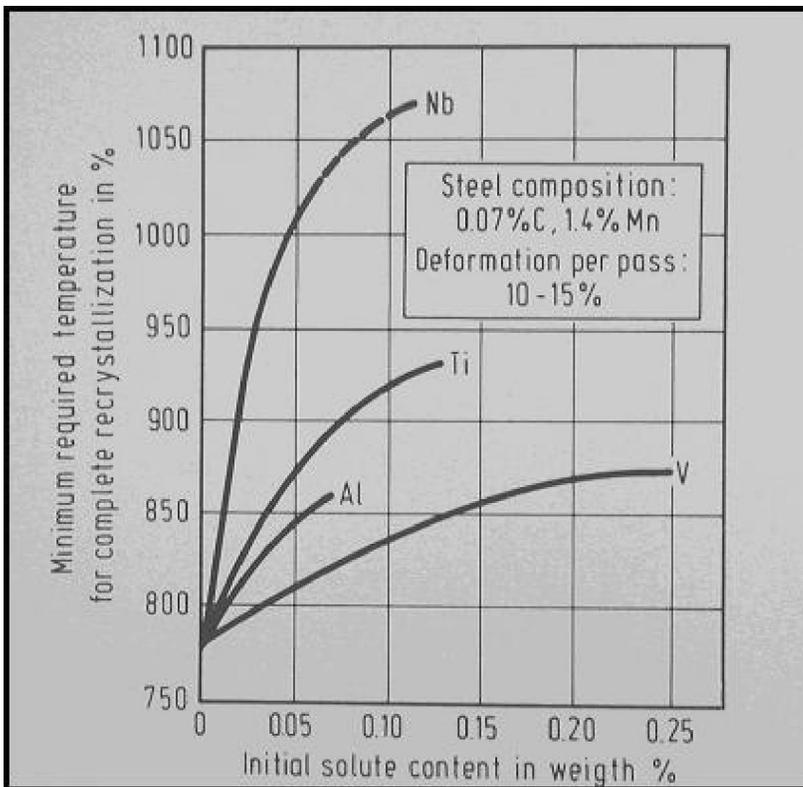


Figure 11: Retardation of recrystallization by austenite ⁽¹³⁾

The Austenite (γ) to Ferrite (α) transformation, which is diffusion controlled, is delayed due to the solute drag effect. This delay increases hardenability and not only results in higher yield strengths, but also improved toughness and ductility. ^(11, 14, 15, 16)

Formation of Carbides, Nitrides and Carbonitrides

Significant strengthening is obtained by the precipitation of these micro-alloying elements as carbides, nitrides and carbonitrides in ferrite. The main influence on the formation of carbides, nitrides and carbonitrides is their respective solubilities. The driving force for precipitation is strong supersaturation, since their solubilities in ferrite is much less than in austenite and solubility is a strong function of temperature. ⁽¹¹⁾

The solubility product describes the equilibrium conditions for the dissolution and formation of non-metallic compounds, including carbides, nitrides and carbonitrides. Figure 12 summarizes the solubility product of several carbides and nitrides in austenite. ^(12, 14)

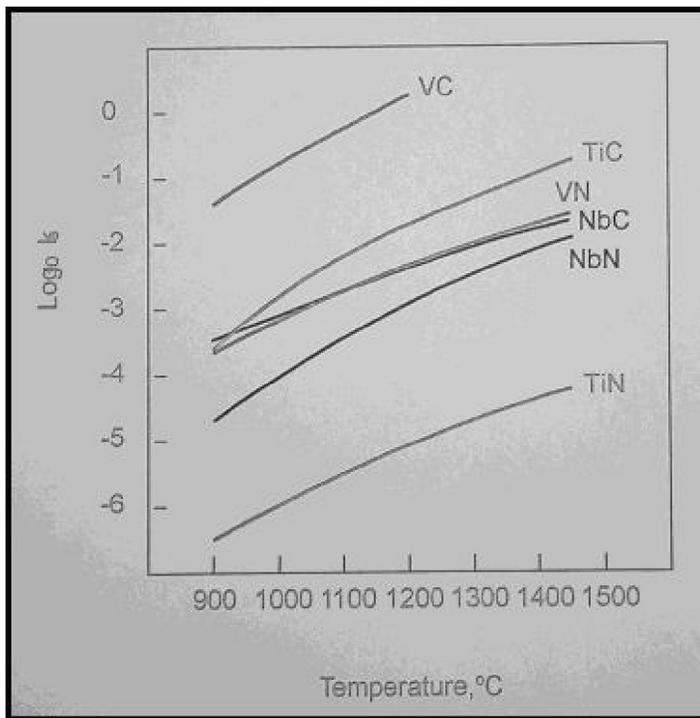


Figure 12: Solubility of micro-alloy carbides and nitrides in austenite ⁽¹²⁾

From the Figure 12, it is evident that carbides, nitrides and carbonitrides of Vanadium have much larger solubilities in austenite than those of Titanium and Niobium. The solubility of Vanadium nitride is about two orders of magnitude smaller than its carbide. Furthermore, the lower Carbon (C) content on rebar allows increased solid solubility of Nb in the austenite phase.

In order to form nitrides and carbonitrides, the presence of nitrogen in these micro-alloyed steels is vital and should be strictly monitored. The content of Nitrogen (N) present determines the density of carbonitride precipitation and hence the degree of precipitation strengthening. The effect of Nb in micro-alloyed steel depends on the N content of the steel since Nb carbonitrides are found to be present when the carbon to nitrogen ratio ranges between 1:1 and 4:1. In V micro-alloyed steel, not only is N employed in formation of nitrides, but its presence optimizes the precipitation reaction and effectively less V is needed to achieve the desired yield strength. ^(12, 15)

Characteristic Features of the effects of V, Nb, Ti in steels ^(11, 12, 14, 16)

The following are some of the characteristic features of each of the above-mentioned elements:

Ti forms titanium nitrides, which are stable at high temperatures and thus prevent austenite grain growth during reheating. Since Titanium is an effective nitrogen scavenger, by forming TiN it ensures that there is enough Niobium in solution at γ/α transformation temperatures for an effective solute drag effect.

V exhibits high solubility of its precipitates in austenite and is therefore in plentiful supply for precipitation hardening at/or after the γ/α transformation. This ensures the precipitation of a high volume fraction of fine precipitates, thus enhancing the effectiveness of precipitation hardening.

Nb which is not precipitated in austenite, will delay the γ/α transformation and the small precipitates formed during and after the transformation gives strength by precipitation hardening. The size of these precipitates is in the order of 2 nm. It also retards recrystallization during hot rolling, promoting a finer microstructure. Furthermore, it is a more effective grain refiner than V. To increase the effectiveness of Nb, the billet soaking temperature prior to rolling should be selected to ensure that almost all the Nb is in solid solution prior to rolling. The relationship between solubility of the precipitates and reheat furnace temperatures at different Nb and C contents are illustrated in

Figure 13 below. If the Nb content exceeds the equilibrium, which is usually attained during soaking, coarse Nb(C,N) precipitates of approximately 200 nm will exist which can retard austenite grain growth. It is because of the retardation action that Nb micro-alloyed grades can possess a smaller austenite grain size at the beginning of rolling and therefore a smaller recrystallized austenite grain size during rolling at temperatures above 1000°C⁽¹⁶⁾

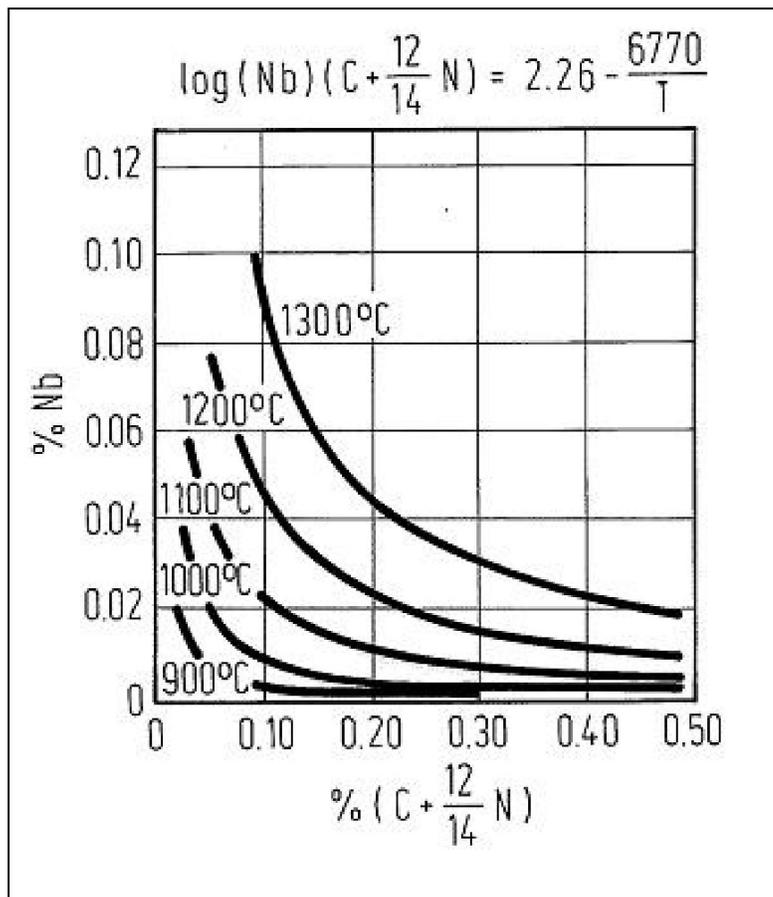


Figure 13: Solubility of Nb (C,N) in steel as a function of C content at different reheat temperatures⁽¹⁷⁾

2.3.3 Properties

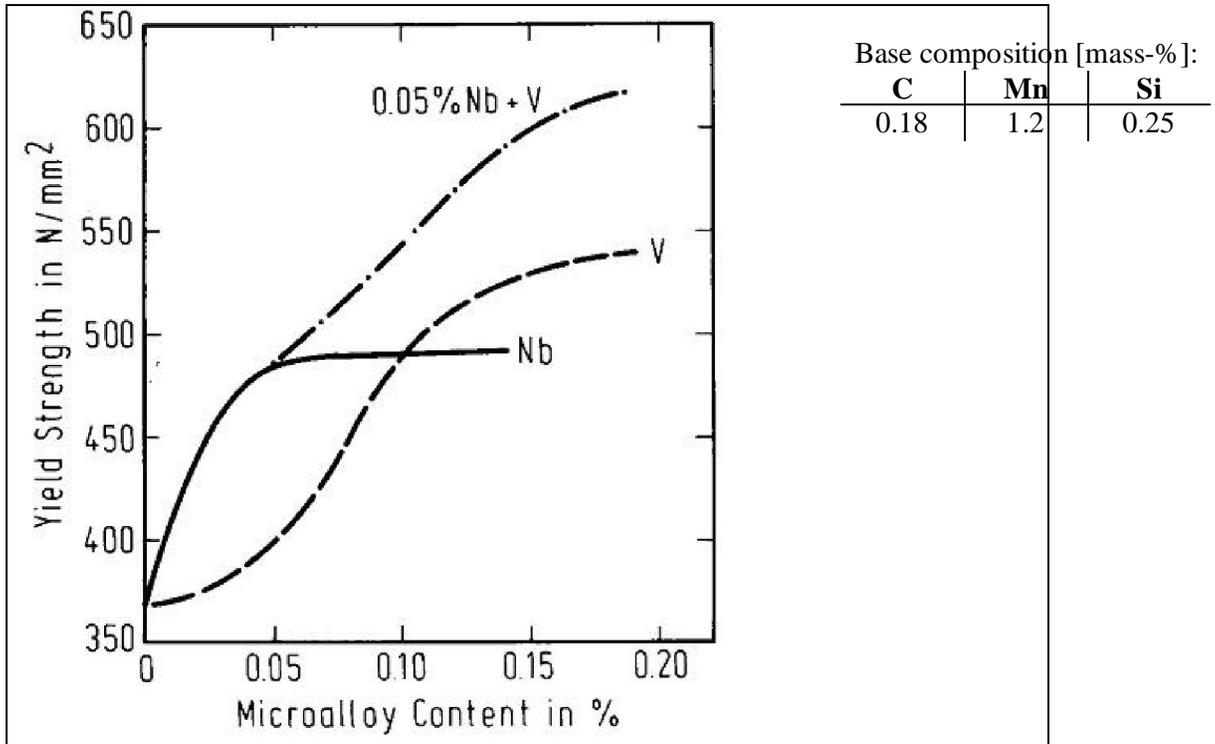


Figure 14: Yield strength of micro-alloyed ferrite pearlite steel⁽¹⁶⁾

Figure 14 above shows the yield strength for various Nb and V contents in air cooled 0.18 % carbon steel. It is evident that Nb is the most effective micro-alloy element in small additions, since only 0.03% Nb produce a yield strength of about 480 MPa while twice as large a V addition would be required to achieve the same properties. Nb enhances the effect of V when used in combination.

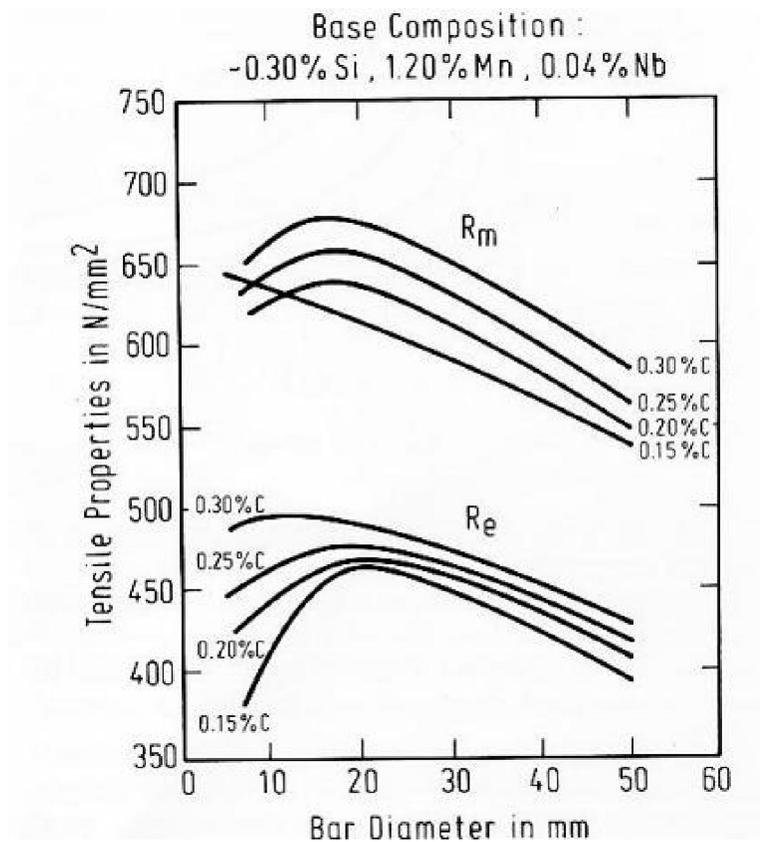


Figure 15: Tensile properties of Nb micro-alloyed rebar for various C contents and diameters ⁽¹⁶⁾

An inspection of Figure 15 above, which illustrates tensile properties of Nb micro-alloyed rebar of various C contents and diameters, indicates that in bar diameters below about 20 mm, the yield strength (R_e) and tensile strength (R_m) drops significantly. This is due to the formation of bainite and sometimes martensite together with ferrite and pearlite. With such a microstructure, the yield point is suppressed and it is attributed to continuous yielding caused by internal stresses. ^(16, 18)

2.3.4 Overview of the Development of Micro-alloyed rebar in the Steel Industry

Chemical Analysis and Substitution

Although Nb and V can both be used to strengthen rebar, the properties of the product that result will be different since these elements are not interchangeable and cannot be substituted for each other without considering not only commercial, but also technical aspects. Nb and V respond differently in terms of steel chemistry, continuous casting, hot rolling practice, and preferred strengthening mechanism, which was previously explained in detail. Furthermore, the solubility of Nb depends on the carbon content of steel, i.e. when carbon is less than 0.1%, its solubility is high whereas the solubility of V is independent on the carbon content of the steel. This dependence of Nb on carbon content can be seen in Figure 13. In some literature, the presence of nitrogen in Nb steels is said to be negative since it makes casting more difficult and promotes precipitation at high temperature. It can be scavenged by a titanium addition if necessary. By contrast, in V steels nitrogen acts as a useful alloying addition. The phenomenon of ageing is not eliminated in Nb steels. ^(16,19) Literature studied states that nitrogen is required and hence is beneficial, since it is needed for the formation of niobium carbonitride precipitates that contributes to strengthening. ^(12, 15)

In 2005, experimental work was conducted by the Central Iron and Steel Research Institute, jointly with Nanchang Iron & Steel Co. Ltd., in China in order to develop rebar with yield strength of 400 MPa minimum. Their current successful production of rebar at that stage was via a Vanadium micro-alloying (20MnSiV) route. Since the rising high cost of Vanadium becomes a problem it necessitated development work on Nb micro-alloyed rebar. Table 3 below indicates the chemical analysis of the first two trials casts that were produced. Only rebar rolled from Trial Heat 2 met the specification, which is explained below. ⁽²⁰⁾

Table 3: Chemical Analysis of the Trial 20MnSiNb rebar material

Trial Heat	%C	%Mn	%P	%S	%Nb	%Si
1	0.20	1.47	0.032	0.026	0.030	0.65
2	0.18	1.38	0.033	0.036	0.026	0.53

This material was then rolled down to 12 mm and air-cooled. Heat 1 exhibited continuous stress – strain curves lacking a marked yield point. Therefore 0.2% proof stress was used for Heat’s 1 evaluation of yield strength, which resulted in average values of maximum 380 MPa, that was below the required specification value. Heat 2 met the requirement by achieving a 410 MPa yield point. In Heat 1, the microstructure consisted mainly of bainite, with fragmented martensite present within the bainite, as compared to the ferrite and pearlite structure with a little bainite found in the samples of Heat 2. The higher Mn content of Heat 1 promoted the mixed bainitic + martensite structure and was subsequently responsible for the lack of a marked yield point on the stress-strain curves. The grain size number for Heat 1 rebar was 9.7 and 9.5 for Heat 2, which are both relatively fine. ⁽²⁰⁾

Continuous Casting

Steels containing micro-alloying elements are sensitive to the formation of transverse cracks during continuous casting, due to the formation of micro-alloy precipitates at the grain boundaries that lowers the ductility. Nb steels are more sensitive to transverse crack formation than V steels since the former’s compounds precipitate at a higher temperature than those in V steels. This sensitivity increases in the presence of nitrogen. ^(16, 19)

Reheating, Rolling and Cooling Effects

Very few of the literature reports studied with regards to the strengthening effect of Nb addition in rebar, gives any detail about the effects of the processing parameters, such as reheat temperature and cooling rate after rolling, on mechanical properties. In order for maximum grain refinement to be achieved in Nb steels, the finishing temperature should be low which is not

easy to achieve when rolling long products. This is not the case for V steels, where a high finishing temperature is adequate. Nb steels require heavier rolling loads since the rolling needs to be controlled at a lower temperature. Subsequently roll wear has to be considered.⁽²¹⁾

As mentioned previously, reheat temperature is important since it affects the solid solute Nb content and austenite grain size, which influence the mechanical properties of the steel. In a joint trial conducted by CBMM Asia (now Kobeko Research Institute) and Kobelco Research Institute in Japan on 0.25% C – 0.5% Si – 1.2% Mn – (0.05% Nb, 0.05% Nb+ 0.05% V) the above-mentioned parameters were investigated.⁽²²⁾ These steels were referred to as 0NB for the base steel of 0.25% C – 0.5% Si – 1.2%Mn, 5NB where 0.05% Nb was added and 5NV, where 0.05% Nb+ 0.05% V are added to the base steel. In the laboratory experiments, the reheat temperature was varied from 1050 to 1250 °C and the reheat time was 30 min at each temperature while the finishing temperature was controlled at 900 °C before air-cooling. It was found that the yield strength in 5NB increased from 440 MPa as the reheat temperatures increased at 1050 and 1100 °C to 485 MPa at 1250 °C. The tensile strength also showed an increase of about 40 MPa with an increase in reheating temperature in 5NB. Total elongation was higher than 28% but was found to decrease with increasing reheating temperature.⁽²²⁾

The influence of cooling rate on mechanical properties was investigated from 900 – 700 °C by varying the water density while keeping the reheat temperature constant. The yield strength in 5NB increased 20-30 MPa as the cooling rate was increased from 0.8 °C/s (only air cooling) to 40 °C/s (water cooling). Similarly an increase in tensile strength was noted, but to a lesser extent. However, the total elongation was not drastically affected. In addition to these trials, the microstructures observed in 5NB reheated at 1150 °C consisted of ferrite and pearlite. No bainite or martensite was present even after the cooling rates were increased. The ferrite grain size was found to have decreased with an increase in cooling rate. The prior austenite grain size was observed since it contributes to final grain refinement in 5NB after

reheating at 1150 °C for 30 minutes followed by a quench and was found to be ASTM grain size number 3.7. ⁽²²⁾ It was concluded from the trial that micro-alloying the base steel with Nb or Nb + V was effective in increasing strength, especially at high reheating temperature, and that it could be ascribed to precipitation and grain refinement hardening. Furthermore, an accelerated cooling up to 700 °C was effective to increase strength and yield point elongation after hot rolling.

Similar trial results were achieved by Caribbean Ispat Limited (CIL) in Trinidad where the effect of reheat temperature on Nb micro-alloyed rebar was evaluated. ^(22, 23) In Trial 1 the billets were reheated at 1080 – 1100 °C and Trial 2 the reheat temperature was increased to 1100 – 1160 °C. These billets were then rolled to 10 mm rebar. It was found that yield strengths in excess of 460 MPa were achieved with the higher reheat temperatures. The higher temperatures allowed an increased amount of Niobium Carbides and Niobium Nitrides {Nb (C, N)} to be taken up in solution. Subsequently, an increased, finer dispersion of Nb (C, N) was precipitated in the ferrite, and a decrease of the austenite to ferrite transformation temperatures occurred. Furthermore, the Nb (C, N) particles that were not taken into solution, contributed to grain refinement which also increased the strength. ^(22, 23)

In the trials by CIL, cooling conditions of the rebar was also investigated and once again similar results to that obtained by the Japanese workers as discussed above was achieved. ^(22, 23) The reheat temperatures for these trials were kept constant at 1130 °C and the material was rolled to 12 mm. In Trial A, the billets were rolled with water cooling before the finishing block and forced air cooling conditions with all the fans set at maximum after rolling on the conveyer. For Trial B, no water-cooling was applied to the steel before the finishing block and all the fans were off. The rebar, which experienced the aggressive cooling, exhibited desirable mechanical properties, i.e. yield strength up to 500 MPa was achieved. The water-cooling applied to the material prior to finishing affected the precipitation behaviour of Nb (C, N) by enhancing it and thereby increasing the strength. The forced air-cooling after

rolling enhances the fast transformation from austenite to ferrite which favoured nucleation over growth and caused a further increase in the material's strength. ^(22, 23)

From examining the plant trials conducted in Japan as well as Trinidad, it is observed that both these plants are in agreement that high billet reheating temperature prior to rolling and accelerated cooling of the rebar after rolling are beneficial in increasing the mechanical properties of rebar. The reheat temperature at the Bar mill is between 80-100 °C lower than the actual furnace temperature, therefore for the Bar mill, the actual reheating temperature is approximately 1180 °C. Although it is possible to increase this temperature at the Bar mill to 1250 °C, it is not feasible, because if the billets are surface ground, they tend to adhere to each other in the furnace, and hence it becomes a production problem. Furthermore, the Bar mill is not equipped with controlled cooling facilities and this poses a major limitation in increasing the mechanical properties of rebar.

2.3.5 Micro-alloying rebar at ArcelorMittal South Africa Newcastle Works

At ArcelorMittal South Africa Newcastle Works all rebar is currently being produced via the micro-alloying process route. Traditionally 460 MPa rebar is produced using V as a micro-alloy addition. However, the fluctuating price of V over the past two years necessitated the use of alternative micro-alloying elements. Nb is used successfully instead of V in rebar rolled to sizes less than 14 mm at the controlled cooled Rod mill as a cost saving procedure. For sizes greater than 14 mm, which is rolled at the Bar mill that lacks forced air cooling facilities, V is still used. It requires a higher level of V alloy addition in order to achieve the required mechanical properties due to the slower cooling rates prevailing in the Bar mill process. Previous trials on the Bar mill with the successful Nb Rod mill chemistry, was unsuccessful due to the difference in cooling facilities (the Stelmor cooling conveyer on the Rod mill provides fast and effective cooling).

In 2003 experimental work was conducted on the plant in order to develop rebar with high yield strength. This was attempted by the production of trial

heats with additions of Nb, Ti, V, Silicon (Si) and N. The idea was to influence the mechanical properties through mechanisms of grain refinement, solid solution strengthening and precipitation hardening. However, the emphasis was placed on producing the rebar with the traditional V addition since only one of the five different casts produced contained Nb. Table 4 gives the chemical analysis for the two types rebar that were of particular interest.

Table 4: Chemical Analysis of the 2003 Trial rebar material

	%C	%Mn	%P	%S	%Nb	%Si	%V	%Ti	% N	%Cr
Nb + Ti + N	0.22	1.45	0.027	0.007	0.020	0.49	0.060	0.023	0.014	0.030
V + Cr + Si	0.23	1.41	0.021	0.009	0.005	0.69	0.110	0.004	0.013	0.260

These casts were then rolled to 10 mm at the Rod mill and 25 mm at the Bar mill in order to investigate and compare the effect of cooling rates and different levels of deformation. It was found that the 10 mm rebar containing V + Chromium (Cr) + Silicon (Si) achieved the highest YS and UTS, namely 734 MPa and 1029 MPa, respectively. The microstructure of this material consisted of bainite with traces of pearlite. The 10 mm Nb-Ti-N cast also achieved a yield strength above 600 MPa, (average 650 MPa), while its microstructure consisted mostly fine pearlite with traces of bainite. ⁽²⁴⁾ On the 25 mm rebar rolled at the Bar mill that contained both V and Cr, the highest yield strength and ultimate tensile strength achieved were 607 MPa and 777 MPa respectively. The microstructure of this material consisted of 45% ferrite with the remainder being pearlite. The 25 mm Nb-Ti-N cast achieved a yield strength above 500 MPa on this trial, (average 523 MPa), while its microstructure consisted of 60% ferrite and the rest was pearlite. Large titanium nitrides were also visible in the microstructure. However, these results could not be repeated on a subsequent trial. ⁽²⁴⁾

On the Nb-Ti-N rebar, the intention was to form Niobium carbonitrides Nb(C, N) and / or Niobium carbides (NbC) for precipitation strengthening. At that stage it was thought that the formation of titanium nitrides (TiN) had a negative influence on the precipitation strengthening mechanism since nitrogen was depleted in the form of titanium nitrides, leaving less nitrogen available to combine with Nb for precipitation formation. Furthermore, when the titanium nitrides were analysed, niobium was also found to be present. ⁽²⁴⁾

For rebar containing V-Cr-Si, it was envisaged that the improved strength was achieved through the precipitation of vanadium carbides and/or carbonitrides and Cr-carbides. The Cr addition increased the hardenability of the material significantly and yielded positive results. On the 25 mm material, this cast yielded the most superior mechanical properties. The fine grain structure when compared to the others (grain size = 8.5) contributed to the high strength of the material. ⁽²⁴⁾ From this set of trials it was concluded that Nb retarded the austenite growth and recrystallisation during rolling, but not the ferrite growth during cooling after rolling. This is evident when comparing mechanical results of the 10 mm rebar which experienced forced cooling after rolling on the Rod mill and the 25 mm results which had slow / non-controlled cooling on the Bar mill. ⁽²⁴⁾

In 2004 further development work on 460 MPa minimum yield strength rebar continued and focused on the use of alternate micro-alloying elements instead of V after its price began rocketing and nearly doubled. At that stage the Nb price decreased and the Ti price also doubled. Rebar containing Nb and a combination of Nb and Ti was investigated and compared against the normal vanadium grades rolled at the Rod and Bar mill. The Nb-Ti rebar also contained higher Nb and Manganese (Mn) additions than the Nb-only containing grade. These casts were then rolled into 10, 12 and 14 mm coils at the Rod mill and 20, 25 and 32 mm lengths at the Bar mill in order to investigate and compare the effect of cooling rates and different levels of deformation in addition to effectiveness of alloy additions on mechanical properties. ⁽²⁵⁾ Table 5 summarises the chemical compositions of these two Nb containing trial rebars as well as the standard V grade.

Table 5: Chemical Analysis of the 2004 Trial rebar material

	%C	%Mn	%P	%S	%Nb	%Si	%V	%Ti	% N
Nb grade	0.230	1.400	0.018	0.010	0.025	0.480	0.006	0.002	0.005
Nb + Ti grade	0.235	1.565	0.015	0.009	0.033	0.510	0.004	0.013	0.004
V grade	0.210	1.330	0.028	0.006	0.003	0.430	0.056	0.002	0.011

On the 10 and 14 mm rebar rolled at the Rod mill both the V-only and Nb-only achieved mechanical results exceeding the minimum mechanical specifications, averaging a YS above 600 MPa for the 10 mm sizes and greater than 530 MPa for the 14 mm sizes for both these grades. No Nb-Ti rebar material was rolled to this size. The microstructure of the Nb-only material consisted of bainite with islands of fine pearlite and a ferrite-pearlite structure on the V-only rebar. On the 12 mm size, the Nb-Ti (with an average YS = 680 MPa) exhibited superior mechanical properties when compared with the standard vanadium grade (average YS = 631 MPa). Both these grades exhibited mechanical properties within the specification values. No Nb-only material was rolled to this size. The microstructure of the Nb-Ti material consisted completely of acicular ferrite and bainite. ⁽²⁵⁾

On the 20 mm rebar rolled at the Bar mill, the V-only steel grade achieved mechanical properties complying with the minimum mechanical specifications, with an average YS of 546 MPa. The Nb-only rebar achieved an average borderline yield strength of 460 MPa. No Nb-Ti rebar material was rolled to this size. The microstructure of the Nb-only material consisted of coarse

ferrite-pearlite with large pearlite grains, while the V-only rebar revealed a fine ferrite-pearlite structure with well-distributed pearlite grains.

Similar results were achieved on the 25 mm rebar rolled at the Bar mill. However, the V-only steel grade averaged a YS of 520 MPa while the Nb-only rebar achieved 452 MPa average, which did not meet the specification value. No Nb-Ti rebar material was rolled to this size. The microstructures achieved were similar to that of the 20 mm rebar for both these grades. ⁽²⁵⁾ For rebar rolled to 32 mm, the V-only steel grade once again yielded superior results, averaging yield strength 526 MPa. The Nb-only rebar, once again, did not meet the yield strength specification. However, the Nb-Ti rebar achieved an average borderline yield strength of 460 MPa. The microstructures observed in the V – only and Nb-only rebar were similar to that of the 25 mm rebar. The microstructure consisted of a slightly coarse ferrite-pearlite structure with well-distributed pearlite grains on the Nb-Ti material. ⁽²⁵⁾

On the Rod mill trials it was concluded that rebar which meets the BS4449 specification could be produced containing Nb or a combination of Nb and Ti in addition to the standard V grade. The mechanisms as to how the required mechanical properties were achieved were previously explained in Section 2.3.2. ⁽²⁵⁾ The finishing temperatures immediately after the last rolling stand on the Bar mill measured 1000 ° C on average. With respect to the Bar mill trials, borderline results were obtained on the Nb - only and the combination Nb-Ti material only. The reason is that the solute drag effect works in collaboration with the cooling rate of the rebar and on the Bar mill there is not controlled cooling facilities and hence the lack of fast and effective cooling.

In the 2004 trials only two of the three different steelgrades were rolled per size at the mills, except for the 32 mm rebar which made it difficult for a thorough evaluation and comparison to occur. Furthermore reheat furnace temperatures were not monitored in these trials. It should be noted that no Transmission Electron Microscopy (TEM) work was done to evaluate the precipitates formed in any of the previous trials performed on rebar.

To summarise the plant trials conducted at ArcelorMittal South Africa Newcastle Works, it can be said that industrial experimentation with Nb micro-alloying began in 2003 and continued vigorously in 2004. The success of the Nb alloyed rebar's dependence on cooling conditions became more and more evident, mainly due to the solute drag effect. Hence, the rebar produced on the Rod mill was successful while those on the Bar mill was not. Summaries of previous trials conducted at both the Rod and Bar Mill at ArcelorMittal South Africa Newcastle Works are shown in Table 6 and 7 below.

Table 6: Summary of previous trials conducted on rebar rolled on the Rod Mill at ArcelorMittal South Africa Newcastle Works ^(24, 25)

Rebar Type	Size (mm)	YS (MPa)	UTS (MPa)	UTS/YS (1.08 min)	% Elongation (14 min)	Microstructure	Compliant with specifications (Yes / No)
V only	10	645	744	1.15	28	ferrite + pearlite	Yes
V-Cr-Si	10	734	1029	1.40	25	bainite + pearlite	Yes
Nb only	10	607	770	1.27	30	bainite + islands of fine pearlite	Yes
Nb-Ti-N	10	650	1020	1.57	24	fine pearlite + traces of Bainite	Yes
V only	12	631	723	1.15	27	ferrite + pearlite	Yes
Nb -Ti	12	680	759	1.12	30	acicular ferrite + pearlite	Yes
Nb only	14	558	728	1.30	27	bainite + islands of fine pearlite	Yes
V only	14	531	741	1.40	21	ferrite + pearlite	Yes

Table 7: Summary of previous trials conducted on rebar rolled on the Bar Mill at ArcelorMittal South Africa Newcastle Works ^(24, 25)

Rebar Type	Size (mm)	YS (MPa)	UTS (MPa)	UTS/YS (1.08 min)	% Elongation (14 min)	Microstructure	Compliant with specifications (Yes / No)
V only	20	546	727	1.33	26	Fine ferrite + pearlite	Yes
Nb only	20	460	656	1.31	27	coarse ferrite + pearlite	Borderline
V only	25	520	704	1.35	26	Fine ferrite + pearlite	Yes
V-Cr-Si	25	607	777	1.28	24	ferrite + pearlite	Yes
Nb only	25	452	670	1.48	25	coarse ferrite + pearlite	No
Nb-Ti-N	25	523	686	1.31	24	ferrite + pearlite, large TiN	Lack of repeatability
V only	32	526	693	1.32	26	Fine ferrite + pearlite	Yes
Nb only	32	447	626	1.40	25	coarse ferrite + pearlite	No
Nb - Ti	32	460	679	1.48	26	Slightly coarse ferrite + pearlite	Borderline

2.4 Comparison of Manufacturing Methods

2.4.1 Technical considerations

Since not all rebar is made via the same manufacturing route, these differences have important implications for maintaining strength and ductility through the construction process. ⁽²⁶⁾ The advantages of both Micro-alloying (MA) and Quench and Self Temper (QST) manufacturing routes over Cold Twisted Deformed (CTD) is enormous in terms of material properties, such as strength, ductility, weldability, bendability and corrosion resistance characteristics. It is therefore understandable why this process route is

obsolete globally for the production of high grade rebar. Over the past 30 years Quench and Self Tempered (QST) has become the most common method of manufacturing rebar, mainly due to the cost of alloying elements used in the Micro-Alloy (MA) route.

Properties of MA rebar are relatively homogenous in terms of chemistry, crystal structure, strength and ductility through a section as compared with QST bar and this should always be considered in subsequent processing operations. If QST bar is heated above the tempering temperature (as low as 450 °C) for a certain period of time, the outside case will revert back to the internal core properties and the bar will lose its strength. Therefore hot bending and welding a QST rebar will not be possible without losing some strength if the cooling is not controlled. ⁽²⁶⁾ Furthermore, cutting a thread into a MA rebar will be quite different compared with QST rebar, as MA rebar is homogenous throughout a section and QST bar is not. For a MA rebar, loss of strength will be proportional to material loss but for QST bar loss of strength will be disproportional since the outer hardened case will be removed. ⁽²⁶⁾ The technical consideration of substituting the micro-alloy Vanadium with Niobium was discussed previously in Section 2.3.4. The factors considered included the actual steel chemistry, continuous casting, hot-rolling practice, preferred strengthening mechanism as well as the effect of Nitrogen.

2.4.2 Economic considerations

Cold Processed Bar

Since CTD technology is outdated, prices of the required equipment could not be obtained. Furthermore, due to the strict mechanical requirements of rebar, this process will not be considered further as an option for the manufacturing route.

Micro-alloying with Niobium versus Vanadium

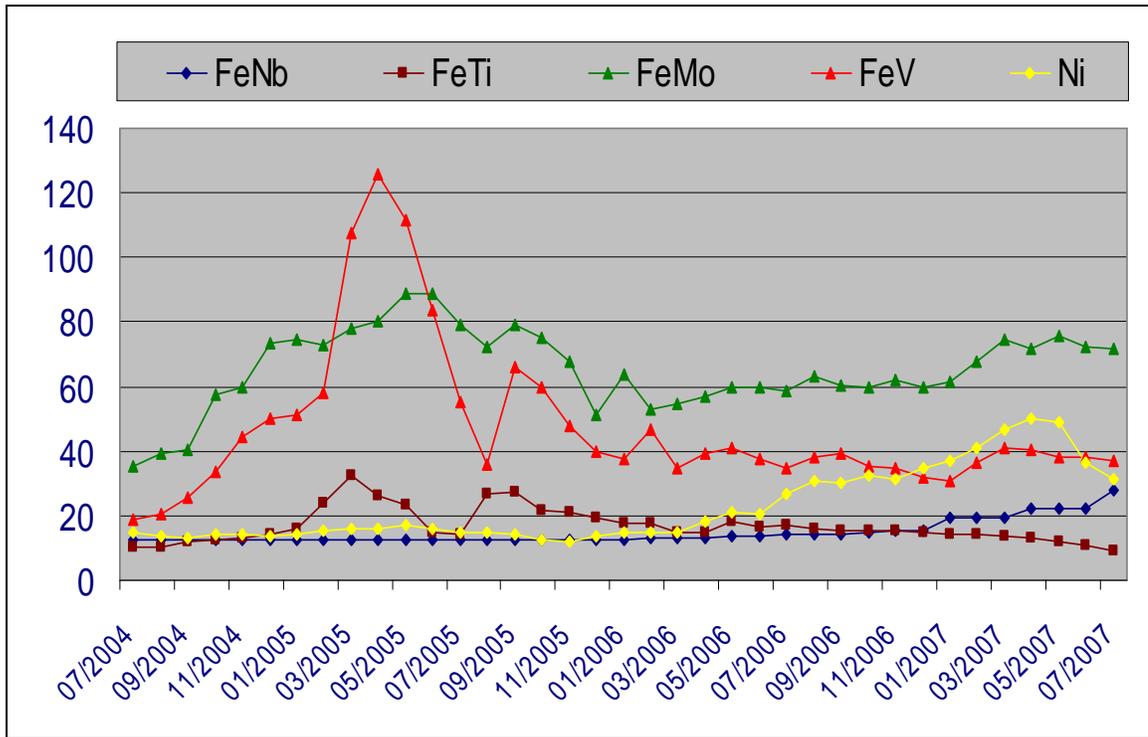


Figure 16: Prices of selected Ferroalloys (US\$ / Kg) ⁽²⁷⁾

It is a well known fact that in a free market economy, the law of supply and demand rules the prices of raw materials. Price increases may therefore be attributed to a temporary imbalance in supply and demand and / or speculative trading. Figure 16 above illustrates how the price of Vanadium reached record highs in 2005 (130 US\$ / Kg) which necessitated the investigation into alternative micro-alloying elements. Since there are a limited number of producers of Niobium, its price is usually stable. However, the price of Niobium remained relatively stable until about this year when the price began to increase while still remaining lower than that of Vanadium. This was mainly due to a Niobium shortage. ⁽¹⁹⁾

It should further be noted that Vanadium is added in much larger quantities (up to double the amount in the current Rod mill rebar) as compared to Niobium in order to achieve the required mechanical properties in rebar. Thus, even if the micro-alloy prices are the same, it would still be economically advantageous to add Niobium to the steel instead of Vanadium for strength.

The calculated micro-alloy savings of replacing Vanadium with Niobium on the 10 and 12 mm rebar rolled at the Bar Mill based on 2005 and 2006 despatches was approximately R 10 million.

Thermoprocessing unit

In order to calculate the viability of installing a thermoprocessing unit for QST a suitable non micro-alloyed steelgrade to be used for QST had to be created. From communication with another plant in the group, the following plain Carbon, Manganese with a Silicon addition for deoxidation was designed.

Table 8: Proposed steel chemistry for QST rebar

	%C	%Mn	%P	%S	%Nb	%Si	%V	%Ti
QST grade	0.11	0.50	0.030 max	0.030 max	-	0.20	-	-

If the above steelgrade in Table 8 is to be used, it would replace the current 10 and 12 mm Niobium and Vanadium rebar rolled at the Rod Mill and Bar Mill respectively. Therefore the micro-alloy cost saving on 10 and 12 mm rebar using the plain Carbon grade based on 2005 and 2006 despatches was calculated to be in the region of R 12 million.

A quote received from SMS MEER only for the supply of a new thermoprocessing unit for the Bar Mill amounted up to 1.7 million euros.⁽²⁸⁾ Even though the savings in the micro-alloy costs would warrant this large capital expenditure, installing and commissioning the new unit would incur huge production losses. The plant would not be able to supply its current orders and hence, this would have enormous monetary implications and great customer dissatisfaction due to not receiving material.

3 EXPERIMENTAL PROCEDURE

3.1 Manufacturing Route

After investigating the three existing manufacturing methods for the production of rebar, it was decided to adhere to the current production route on the plant, i.e. via micro-alloying. As previously explained, the dramatic price increases and fluctuations of the Vanadium price made Niobium the micro-alloying element of choice. It should be noted, that in addition to the commercial analysis, a technical analysis was also taken into account to avoid unforeseen cost penalties. This decision to remain with micro-alloying was made since the CTD technology was out-dated and purchasing a new thermo-processing unit would require a large capital expenditure and the loss of production. More importantly, the cost saving for using Niobium instead of Vanadium is not excessively less when compared to the cost savings of using QST for the rebar.

3.2 Hardenability and Microstructure

The current Nb micro-alloyed rebar grade chemistry, which meets the specifications, was designed and optimised for the Rod mill. As stated previously, it was unsuccessful on the Bar mill due to the difference in cooling facilities. On the Rod mill, the microstructure of the successful Nb-alloyed rebar contains acicular ferrite and bainite. The microstructure of the material from previous trials on the Bar mill, which failed to meet the minimum YS requirement of 460 MPa, contained polygonal ferrite and pearlite. Therefore methods to influence the microstructure were concentrated upon. Cooling faster can influence the microstructure. A schematic representation of a continuous cooling transformation (CCT) diagram for steel, where fast cooling results in crossing the “noses” of the hard phase regions (bainite, martensite), as opposed to cooling slower where the slower cooling rate crosses the softer ferrite, pearlite regions during transformation from austenite, is shown in Figures 17 and 18, respectively.

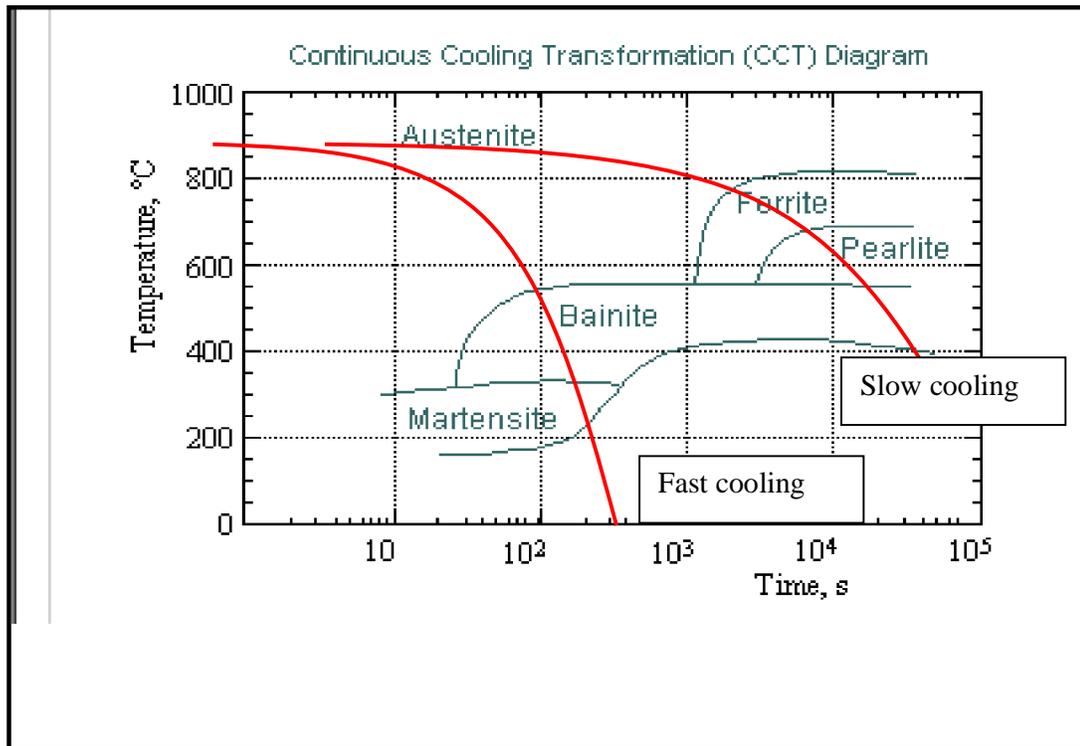


Figure 17: A schematic Continuous Cooling Transformation Diagram for steel illustrating the effect of cooling rate

The Bar mill is, however, limited by its cooling facilities; therefore cooling faster in order to achieve a bainitic microstructure is not an option. The following method, which involves shifting the nose of the CCT, as indicated in Figure 18, to influence the microstructure, will be considered.

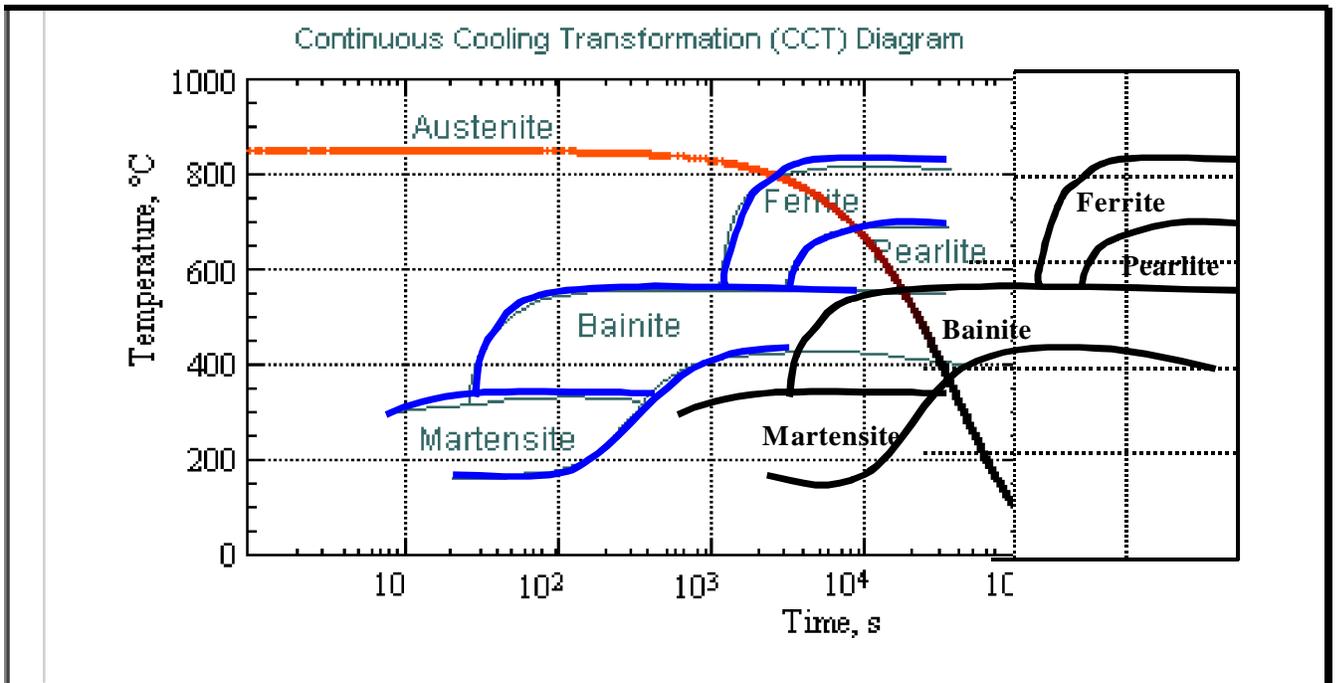


Figure 18: A schematic Continuous Cooling Transformation Diagram for the same steel as in Figure 15 illustrating the effect of the shifting noses of CCT while at a constant cooling rate.

A method employed to shift the nose, is to increase the hardenability of steel by the addition of suitable alloying elements. Hardenability is also influenced by austenite grain size. The larger it is, the greater the hardenability. The method of coarsening the austenite grain size to increase the hardenability will not be used and is not generally practised, since it had adverse effects on other properties of the material, such as increasing the brittleness of the material and causing a loss of ductility.⁽²⁹⁾

3.3 Steel grade design

After much consideration, it was decided to use the successful Rod mill Nb steel grade chemistry as the base composition and make suitable adjustments to it for the production of the trial material. The alloying elements were considered in conjunction with the rebar chemical specification, and its

influence on other properties of the steel, as well as the impact of production on the plant. Relative alloy costs were another consideration.

3.3.1 Grain Size Refinement

As previously explained, the strengthening mechanism of grain refinement contributes a large percentage to increasing the yield strength of steel. The Nb content of the trial cast was chosen to remain at the current aim of 0.020 – 0.030%, since it is found to be effective in these amounts as a result of its solubility.⁽¹⁶⁾ In addition to Niobium, which strengthens steels by this mechanism, an Aluminium (Al) addition was also initially considered as a grain refiner. Sufficient amounts of Al would have to be added for it to be an effective grain refiner since it is a major de-oxidiser in steel.⁽³⁰⁾ Furthermore, after discussions held with plant personnel, this could have a negative influence on the castability at the Continuous Caster due to potential clogging. Subsequently, the sequence lengths of rebar heats that are cast, which are currently long, would decrease dramatically and hence negatively influence production at the steel plant.

3.3.2 Hardenability and Weldability

The alloying elements, which increase hardenability, include Carbon (C), Manganese (Mn), Molybdenum (Mo), Chromium (Cr), Silicon (Si) and Nickel (Ni). However Mo and Ni will not be considered as possible additions due to their relatively high costs.⁽²⁶⁾ The BS4449 international specification limits the Carbon Equivalence (CE) and Carbon content to 0.51% and 0.25%, respectively. The formula used to calculate CE is indicated below:⁽³¹⁾

$$CE = \% C + Mn/6 + (Cr + Mo + V)/5 + (Ni + Cu)/15$$

Since C is cheaper than Mn and its effect on increasing hardenability is greater than that of Mn, it was decided to adjust the C aim specification from its current 0.15-0.18% to 0.21-0.25 % to increase the strength of the material. Furthermore, the Mn content remained unadjusted to compensate for the increase in C, so that the CE limit is still adhered to. The aim for Mn is already towards the upper limit of the 1.60% max specification.

The Si aim specification was also adjusted from its current 0.22-0.28% to 0.45-0.55%. This was done since Si has a strengthening effect and a mild hardenability effect, i.e. between that of Cr and Ni. Furthermore, it does not affect the CE calculation. Although Cr is a weaker hardenability agent than Mn or Mo, it is highly cost effective (degree of hardenability increase/relative alloy cost) and was therefore selected as a macro-alloying addition. Cr is also a strong carbide former.⁽³⁰⁾ After inserting the new C aims into the CE formula, the limit on the maximum Cr addition was calculated to be 0.30%. It was decided to aim for 0.15-0.20% Cr in the trial cast since the specification limits it to 0.30% maximum. The chemistry for the trial material as compared with the Rod Mill Nb micro-alloyed rebar steel grade is given in Table 9.

Table 9: Chemistry comparison of current Nb Rod Mill rebar with Trial Bar Mill

		%C	%Mn	%P	%S	%Nb	%Si	%Cr
Specification for NH33201 (Rod Mill)		0.25 Max.	1.60 Max.	0.050 Max.	0.050 Max.	0.050 Max.	-	0.30 Max.
Mittal Aim Specification for NH33201 (Rod Mill)	Min Max	0.15 0.18	1.35 1.50	0.040 Max.	0.040 Max.	0.020 0.030	0.22 0.28	0.25 Max.
Mittal Aim Specification for NH33201 + Cr (Bar Mill trial)	Min Max	0.21 0.25	1.35 1.50	0.040 Max.	0.040 Max.	0.020 0.030	0.45 0.55	0.15 0.20

3.4 Production of the trial cast

A development trial (DT) was drawn up for the production and rolling of the experimental trial material and distributed throughout the plant. (See APPENDIX A). A Basic Oxygen Furnace of 160 tons in capacity was used for steel-making. After tapping, alloy trimming and heating was done at the ladle furnace. The heat was continuously cast on a six-strand bloom caster. Square blooms of 260 mm x 260 mm were cast and cut to required length. The trial cast was produced successfully under normal standard practice instructions for rebar at the steel plant with the adjusted chemistry.

3.5 Rolling of the trial material

Twenty tons of the trial material in 260 mm square blooms was scheduled for rolling into 101 mm square billets at the Billet mill for intermediate size rolling. These 101 mm billets were to be used as input material into the Bar mill. At the Bar mill, ten tons of the 101 mm billets were scheduled for rolling into 10 mm rebar and ten tons of 101 mm billets were scheduled for rolling into 12 mm rebar. The trial material was rolled under normal rebar rolling practice to lengths and then normally air-cooled. (See figure 3 and APPENDIX A)

NEWCASTLE WORKS REINFORCING STEEL PRODUCTION FLOWCHART AT THE BAR MILL

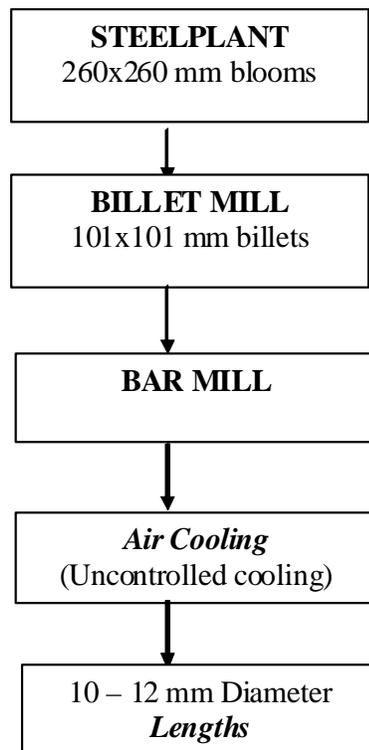


Figure 19: Flowchart illustrating rebar production using the trial material at the Bar mill

3.6 Evaluation of the trial material

3.6.1 Overview of test procedures

In order to produce rebar of acceptable quality it is important that an adequate quality control system be established and constantly maintained. This system includes mechanical and metallurgical testing together with inspection of surface and internal defects. If defects pass through the systems undetected, they are later reflected in the poor mechanical properties of the rebar in terms of low strength and ductility, inconsistent rebar geometry and inferior surface quality. Therefore for this trial, critical inspection and testing of the trial material was performed in order to remove defective material thereby ensuring a true representation of the effects on the alloy additions to the rebar. ⁽⁹⁾

Mechanical testing as specified in the BS4449/ 1997 460B standard and metallographic evaluation was conducted on samples of rebar produced from the trial material. Only if the material meets the specification, would it be despatched to a customer for evaluation on processability and customer satisfaction would be assessed. The surface quality of the cast blooms was inspected to determine the sensitivity of transverse crack formation in the trial cast. The macro-structure of the blooms was evaluated to reveal internal soundness. Tensile, hardness and metallographic evaluations of samples of rebar produced from the trial cast were compared to vanadium alloyed rebar produced on the Bar mill and niobium alloyed rebar produced at the Rod mill.

Furthermore, in an attempt to establish Bar mill rolling conditions for the 10 and 12 mm rebar, Gleeble simulations were performed on the samples at the Industrial Metals & Minerals Research Institute (IMMRI), University of Pretoria. The Zener-Holloman Parameter, Z, which combines the relationships between the resulting grain size, temperature and applied strain rate for a hot rolling process on the Gleeble machine was used (see APPENDIX B). The first objective was to attempt to reproduce the current as-rolled microstructure during the simulation in order to establish a base line. This was followed by an

attempt to establish the finishing temperatures required on the Bar mill in order to produce the desired microstructure and hardness. The effectiveness of Nb as a precipitation strengthener as compared to V was also investigated. The effect of age-ing was also investigated on the trial material to study precipitation behaviour further. This age-ing was done artificially.

3.6.2 Metallographic testing

For light optical microscopy, specimens were cut and prepared using standard laboratory techniques for grinding and polishing and subsequently etching in 2% Nital to reveal the microstructure. In addition to light microscopy, transmission electron microscopy (TEM) was performed to determine if the as-cast precipitates of the trial cast were dissolved and also to see how the Nb(C,N) size and distribution compares to that of the V micro-alloyed rebar. Although solubility calculations can provide a good indication of the amount of dissolved Nb, TEM analysis was used to verify the state of these precipitates. The TEM used in the investigation was equipped with Energy Dispersive Spectrometer (EDS). Specimens were prepared in the same way as for the optical microscopy. Carbon extraction replicas were prepared for precipitate characterisation. After etching, the samples were coated with ~30 nm carbon, which was followed by another etch in order to float off the carbon extraction replicas. The carbon film (with the adherent precipitates) was subsequently rinsed with distilled water, mounted on a copper (Cu) grid and dried and was then investigated with the TEM. ⁽³²⁾

3.6.3 Mechanical testing

Tensile tests were carried out on the samples according to the EN 10 002 Part 1 specification. ⁽³³⁾ This test method covers tensile testing of metallic materials in any form at room temperature, specifically the methods of determination of yield strength, yield point, tensile strength, elongation and reduction in area. The tensile machine used was a Messphysik model TTM600 of 600 kN maximum capacity. Hardness of the various as-rolled rebar was performed on the transverse samples on a Vickers micro-hardness tester using a load set at 25 according to ASTM E92-82 standard. ⁽³⁴⁾ After the

Gleeble simulation at IMMRI, the Vickers hardness was performed on the specimens at a load of 1 kg. ⁽³²⁾

3.6.4 Macro-structural evaluation

Macro-etching, which is the etching of specimens for macro-structural examination at low magnifications, usually a visual inspection, is a frequently used technique for evaluating steel products such as billets and blooms. Macro-etching was performed on ground, transverse bloom samples according to the ASTM E381-01 standard. A procedure for rating the steel samples by a graded series of photographs showing the incidence of certain conditions is also included in this test method. ⁽³⁵⁾

Sulphur prints, also referred to as Baumann prints, are used at the Works, not only to reveal the distribution of sulphide inclusions or the solidification pattern in steels, which is its intended purpose, but also to provide an indication of internal soundness of the steel. Sulphur prints of the ground, transverse bloom samples were performed according to the ASTM E1180 standard. ⁽³⁶⁾

3.6.5 Grain size measurement

The grain size of the steel was measured in accordance with ASTM E112 – 96. ⁽³⁷⁾ The carburising procedure usually referred to as the McQuaid-Ehn test was followed which is suitable for carbon and alloy steels with carbon generally below 0.25 %. The process steps involved is described below:

- The specimens were placed in a carburising box and carburised at 927 ± 14 °C for 8 hours
- The specimens were then furnace cooled to a temperature below the lower critical (600°C was used in this case) at a rate slow enough to precipitate cementite in the austenite grain boundaries of the hypereutectoid zone of the case.
- Once cooled, the specimens were sectioned to provide a fresh-cut surface, and then etched with 2% Nital to reveal the grain size.

3.6.6 Rolling simulation

Gleeble simulations were performed on samples in order to establish the relationship between finishing temperature and grain refinement and further to determine optimum microstructures of the rebar. In the Gleeble simulation, the bar specimens were cut to 14 mm lengths, reheated, soaked and subjected to three passes of 0.33 strains with an interpass time of 15 seconds. The final deformation temperature was measured between 829 and 950 °C and corresponded to a Bar mill finishing temperature ranging between 980-1110 °C. The samples were subsequently cooled. After cooling to room temperature, the samples were prepared for light optical microscopy and further hardness and TEM evaluation.⁽³²⁾ The ageing treatment to induce precipitation was done at a temperature of 700 °C for 30 minutes.

4 RESULTS

4.1 Chemical Analysis

The final chemical composition of a heat is given as the average of two samples taken after 15 minutes and 30 minutes respectively, in the tundish during casting of the heat at the Steel Plant. There is an allowable deviation on chemical analyses between the cast and actual rolled product due to segregation of elements in the steel and it is for this reason that chemical analyses was also done on samples of rebar. Five samples were randomly selected and submitted for chemical analysis. The chemical compositions of the samples were determined using an Optical Emission Spectrometer and a Leco Combustion analyser. The results of these analyses are tabulated in table 10:

Table 10: Chemistry comparison of actual chemistry of trial cast versus aim specification

		%C	%Mn	%P	%S	%Nb	%Si	%Cr	% Ti	% N
Specification for NH33201 (Rod Mill)		0.25 Max.	1.60 Max.	0.050 Max.	0.050 Max.	0.050 Max.	-	0.30 Max.	-	-
Mittal Aim Specification for NH33201 + Cr (Bar Mill trial)	Min Max	0.21 0.25	1.35 1.50	0.040 Max.	0.040 Max.	0.020 0.030	0.45 0.55	0.15 0.20	0.020 Max.	0.010 Max.
Trial Cast (Actual)		0.23	1.41	0.010	0.009	0.024	0.53	0.19	0.0023	0.0048
Product analyses	Ave	0.23	1.42	0.009	0.007	0.024	0.52	0.19	0.0024	0.0046

The chemistry of the trial cast was within the requirements stated in BS4449 specification.⁽⁵⁾ Furthermore, the chemistry was within the aim specification as per the development trial (See APPENDIX A). The Carbon Equivalence of the cast and product samples was calculated to be 0.50, which is just below the maximum allowable value by the BS4449 specification⁽⁵⁾ of 0.51. Although Ti and N additions were not made to this cast, residual contents of 0.0023 and 0.0048 resulted in a Ti:N ratio of 0.479. The C:N ratio was calculated to be 47.9. Although it is stated in some literature that a hypostoichiometric Ti:N ratio (less than 3.42) is beneficial to Nb steels in terms of grain size control, 0.479 is too low to have an effect when compared to the ideal value of 2.⁽¹⁷⁾

4.2 Surface Quality

The surface quality of the cast blooms and rolled billets was inspected to determine the sensitivity of transverse crack formation in the trial cast as well as to determine the presence of other surface defects. This was a concern for this trial steel grade since the hardenability was increased, combined with the fact that it contains Nb, which previously explained is susceptible to transverse crack formation. The surface quality was evaluated by visual inspection on 100% of the material on 3 faces of the blooms and billets. The blooms and rolled billets had a good surface finish and no detrimental surface defects were visible.

4.3 Macro-etching Results

The macro-structure of the blooms was evaluated to reveal internal soundness. Transverse bloom slices from five random blooms were cut and prepared for macro etching. The results revealed good, sound structures on all blooms tested (see figure 20). The ratings of the blooms according to the ASTM E381-01 are tabulated in table 11 together with typical ratings of Nb-only bloom slices for the purpose of comparison.⁽³⁵⁾

Table 11: Typical Transverse bloom ratings according to ASTM E381 Plate 1: Graded series

Conditions	Ratings of Nb-Cr	Ratings of Nb-only
Subsurface conditions	S2	S2
Random conditions	R2	R2
Centre segregation	C2	C2



Figure 20: Digital photograph showing the transverse Macro-etch section of a bloom sample

4.4 Sulphur Prints

In addition to the macro-etches, sulphur prints were also used to reveal the internal soundness of the trial blooms. Sulphur prints of five ground transverse bloom slices were prepared and the results revealed no centre line cracks or subsurface blowholes. Due to low levels of sulphur, the sulphur prints were unclear and thus excluded from this report.

4.5 Mechanical Results

4.5.1 Tensile Tests

In order to comply with the BS4449 specification for reinforcing steel, the yield strength needs to exceed 460 MPa. In addition to this, the tensile strength to yield strength ratio must be greater than 1.08 and the % elongation larger than 14. Due to the absence of marked yield point on the tensile graphs, 0.2 % offset method was used to determine the yield strength as illustrated in

Figure 21 below. The mechanical results achieved on the 10 mm and 12 mm rolled material from the trial cast are tabulated in table 12 and are graphically plotted in Figures 22 - 25. Typical results achieved from micro-alloying with Vanadium at the Bar Mill and results from Nb micro-alloyed grade when rolled at the Rod mill are included for comparison purposes.

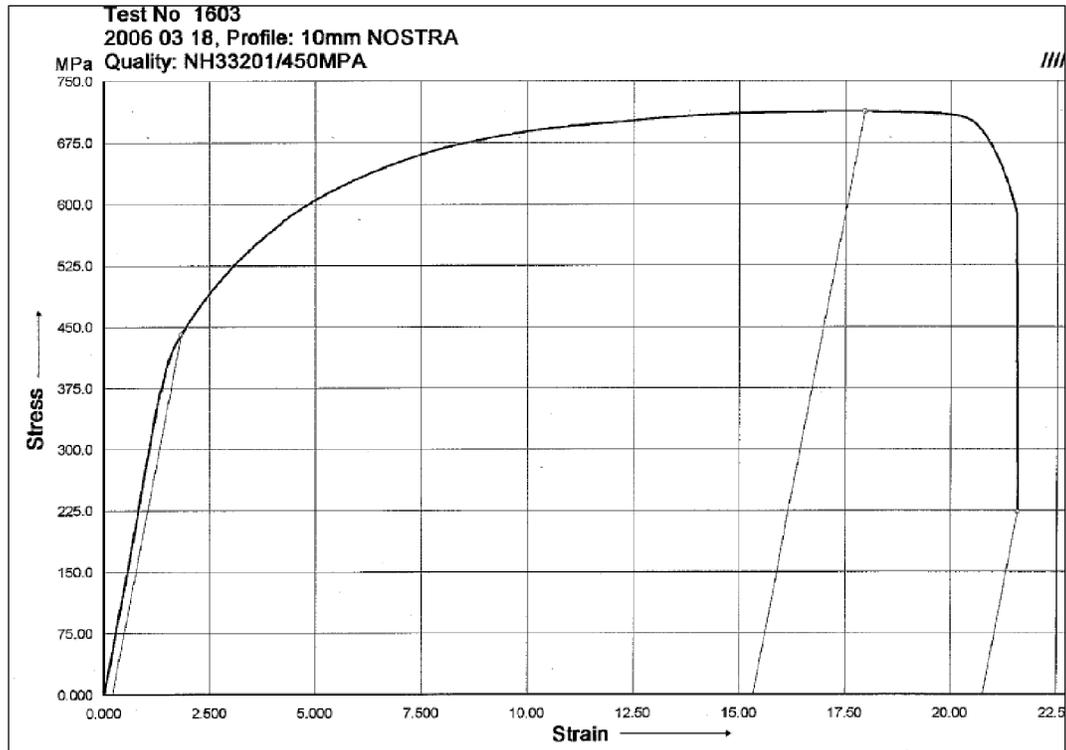


Figure 21: Tensile test graph for 10 mm Nb-Cr rebar showing 0.2 % offset method for determination of yield strength

Table 12: Average mechanical properties of as-rolled rebar lengths and comparisons

Plant	Alloys	Steelgrade	Size (mm)	YS (MPa)	UTS (MPa)	UTS/YS	% Elongation
BS4449 Specification	-	-	-	460 min.	-	1.08 min	14 min
Bar Mill	Nb + Cr	NH33201 (Nb+Cr)	10	429	727	1.69	19
Bar Mill	Nb + Cr	NH33201 (Nb+Cr)	12	436	762	1.74	16
Bar Mill	V	VH33215 (V)	12	540	675	1.25	25
Rod Mill	Nb	NH33201 (Nb)	10	500	665	1.33	22
Rod Mill	Nb	NH33201 (Nb)	12	509	640	1.26	25

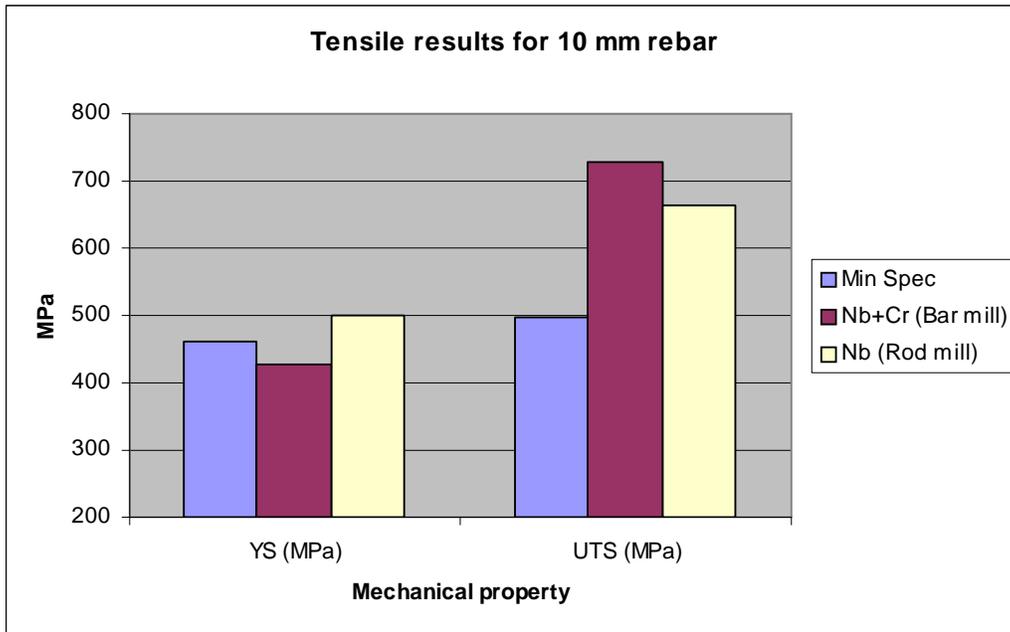


Figure 22: Tensile results for 10 mm rebar

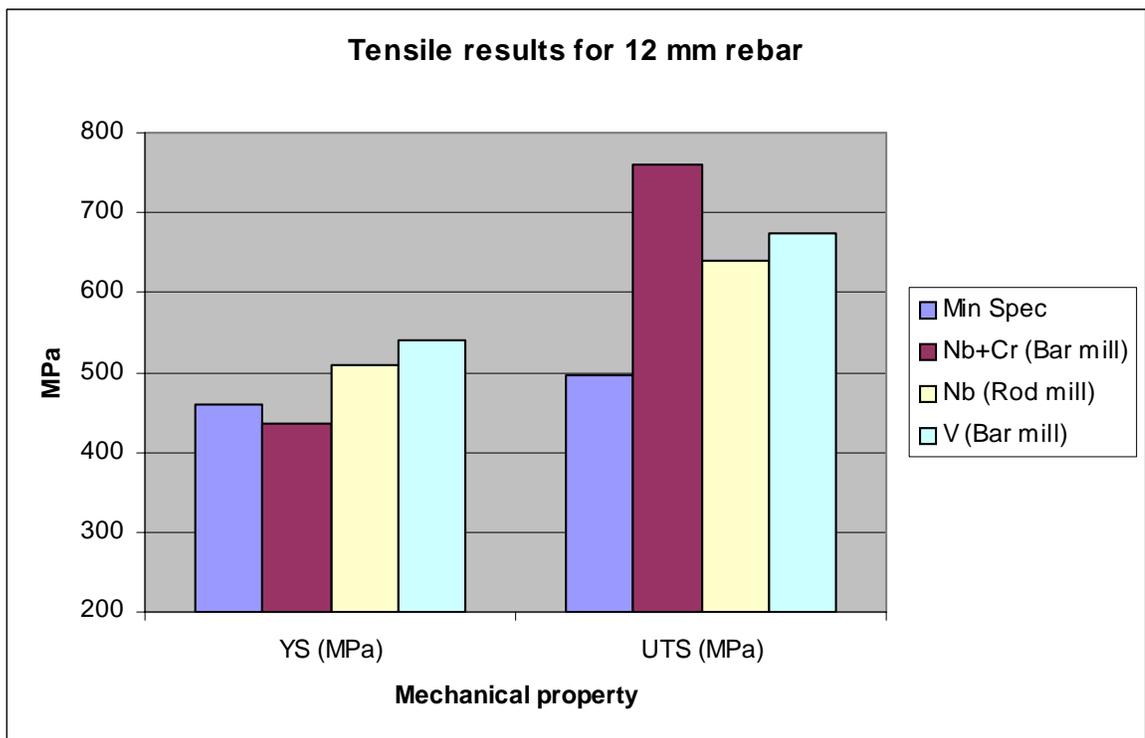


Figure 23: Tensile results for 12 mm rebar

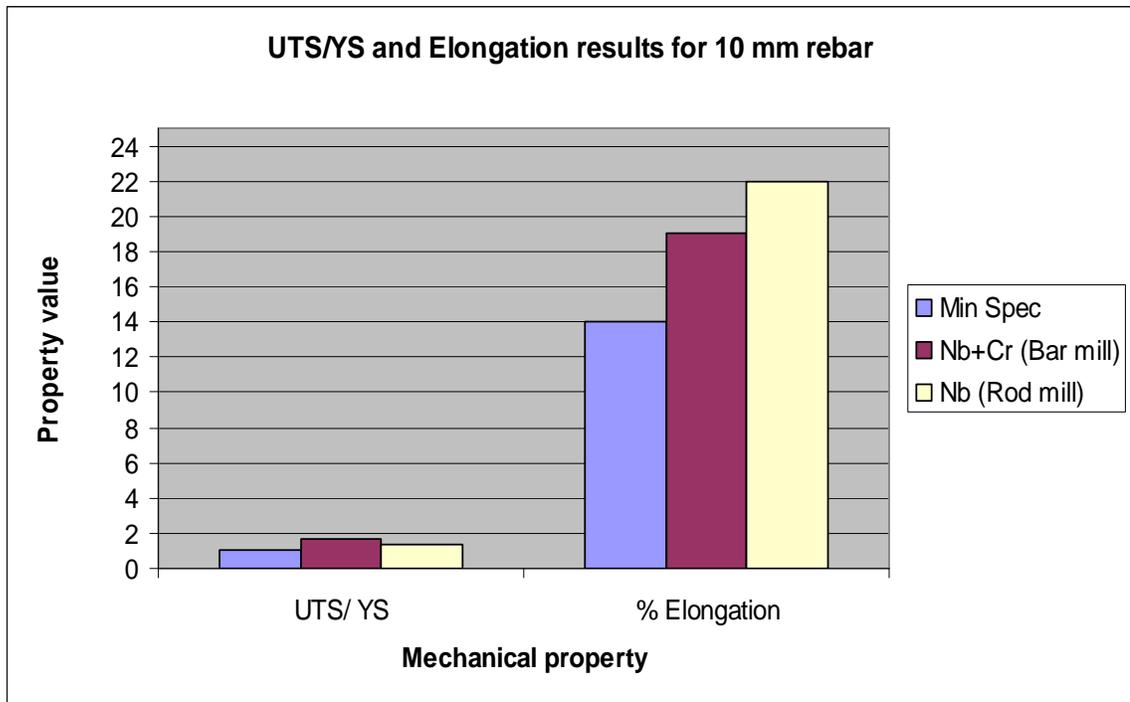


Figure 24: UTS/YS ratio and % Elongation results for 10 mm rebar

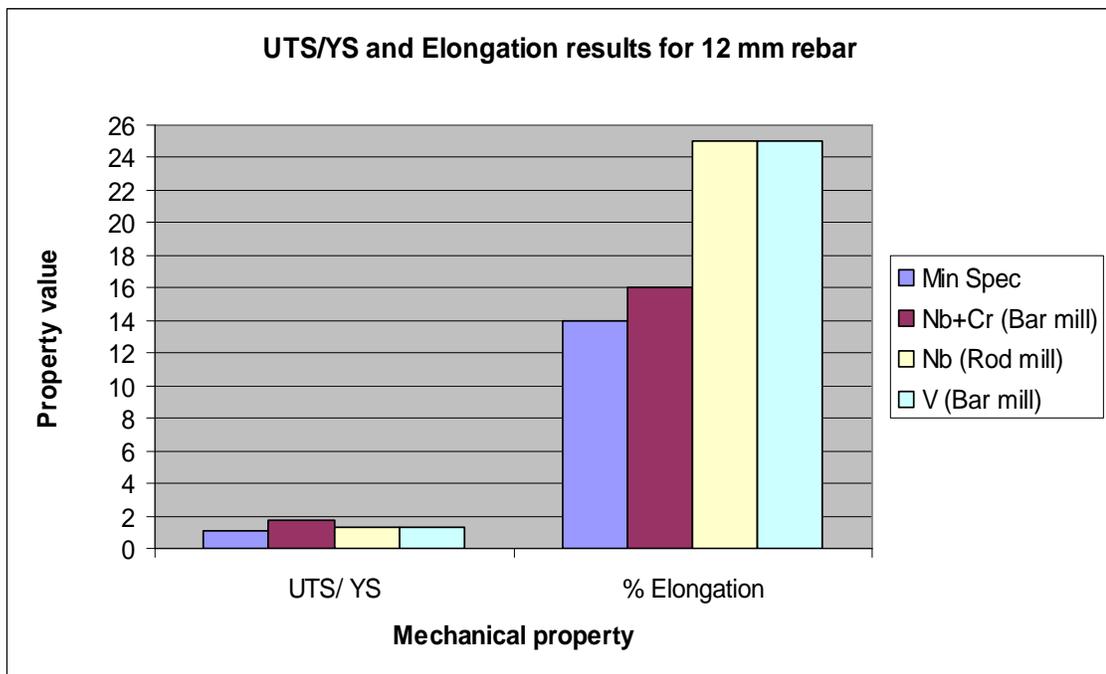


Figure 25: UTS/YS ratio and % Elongation results for 12 mm rebar

The rebar produced from the trial chemistry did not meet the minimum yield strength requirement of the BS4449 460B specification for both the 10 and 12 mm. For the 10 mm rebar, the yield strength achieved was on average 31 MPa below specification and for the 12 mm, 24 MPa below specification. It can be noted that the UTS/YS ratio and minimum % elongation specification was met for both the 10 and 12 mm rebar. When compared with the mechanical properties achieved from the V micro-alloyed rebar on the Bar mill and the Nb micro-alloyed rebar on the Rod mill, it can be seen that the trial rebar is inferior to the currently produced material.

4.5.2 Hardness Tests

The hardness results achieved on the trial rebar are tabulated in table 13 below, together with typical results achieved from micro-alloying with V at the Bar mill and results from Nb micro-alloyed grade when rolled at the Rod mill are included for comparison purposes. The hardness results of Gleeble simulated trial rebar in order to investigate the influence of finishing temperatures are shown in table 14 below. The higher hardness's exhibited on the 10 mm and 12 mm Nb – Cr trial rebar correlate to the high UTS values previously found on these samples.

Table 13: Average hardness values of as-rolled rebar and comparisons

Sample Number	Average (HV)	Steel grade	Mill
Cast 702510 - 10mm	203.3	VH33216	Bar
Cast 602134 - 10mm	230.3	NH33201 + Cr	Bar
Cast 602134 - 12mm	223.7	NH33201 + Cr	Bar
Cast 703723- 12mm	219.1	VH33216	Bar
Cast 703695 -12mm	225.9	NH33201	Rod
Cast 700251 -10mm	262.1	NH33201	Rod

Table 14: Average hardness values for Gleeble simulated 10mm Nb +Cr rebar

Average (HV)	Gleeble FT (°C)	Equivalent Bar Mill FT (°C)
237	875	1017
240	912	1063
237	950	1112

4.6 Metallographic Results

4.6.1 Microstructural analysis

Transverse samples of the as – rolled trial rebar as well as the lab simulated rolled rebar were cut and prepared for metallographic investigation using standard techniques and also compared with samples from the V micro-alloyed rebar rolled on the Bar mill and the Nb micro-alloyed rebar rolled on the Rod mill. These results revealed the following:

- A polygonal ferrite-pearlite-bainite microstructure on the 10 and 12 mm trial rebar (see Figures 26, 27 and 29)
- A microstructure containing similar amounts of polygonal ferrite-pearlite-bainite after simulated bar rolling on 10 mm Nb – Cr trial rebar. The finishing temperature (FT) of 875 °C on the Gleeble simulation, which was equivalent to FT of 1017 °C on the Bar mill (see Figure 28)
- The desired acicular ferrite-bainite microstructure was achieved during simulated bar rolling after increasing the finishing temperatures from Gleeble FT of 912 °C = 1063 °C (Bar mill) to Gleeble FT of 950 °C = 1112 °C (Bar mill) on the 10 mm Nb-Cr rebar (see Figures 30 and 31)
- An acicular ferrite-bainite microstructure on the 12 mm Nb micro-alloyed rebar rolled on the Rod mill (See Figure C1, APPENDIX C)
- A relatively coarse polygonal ferrite and pearlite microstructure on the 12 mm V micro-alloyed rebar on the Bar mill (See Figure C2, APPENDIX C)



Figure 26: Photomicrograph showing a microstructure containing polygonal ferrite–pearlite-bainite on 10 mm NH33201 (Nb + Cr) as-rolled rebar sample etched in 2% Nital.

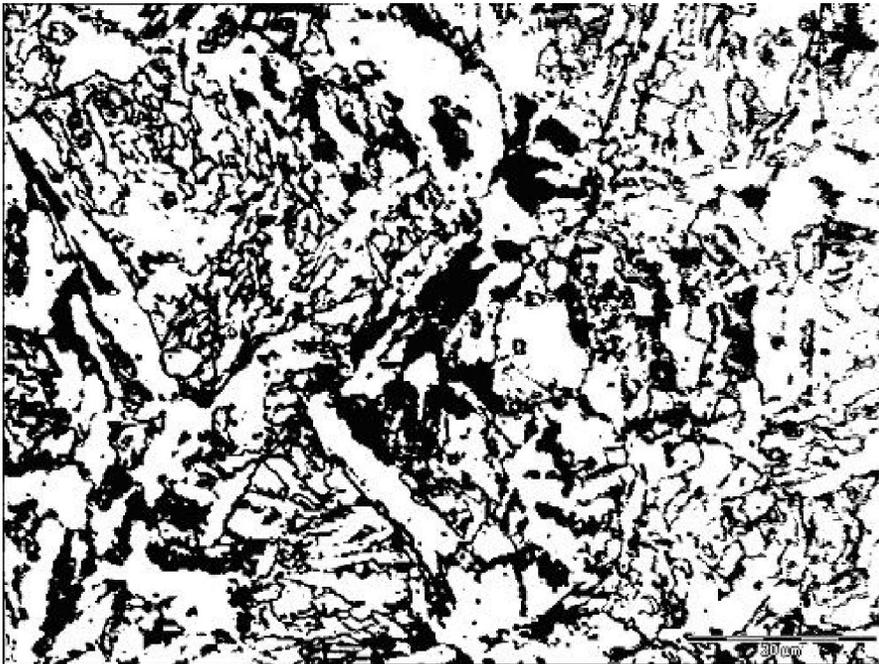


Figure 27: Photomicrograph showing a microstructure containing polygonal ferrite–pearlite-bainite on 12 mm NH33201 (Nb + Cr) as-rolled rebar sample etched in 2% Nital

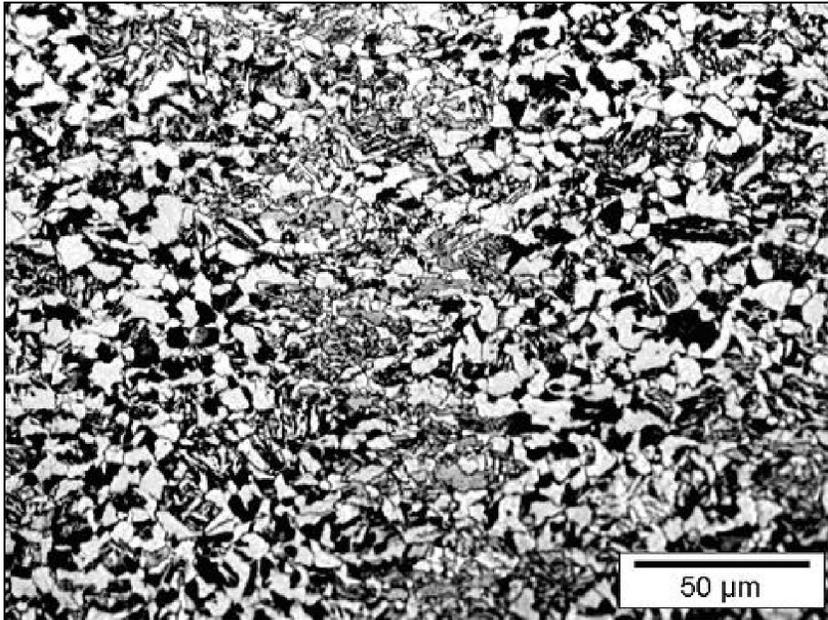


Figure 28: Photomicrograph showing a similar microstructure as Figure 23, achieved under simulated rolling (finishing temperature = 875 ° C) of 10 mm NH33201 (Nb + Cr) rebar etched in 2% Nital. $Z = 1.59 \times 10^{18}$

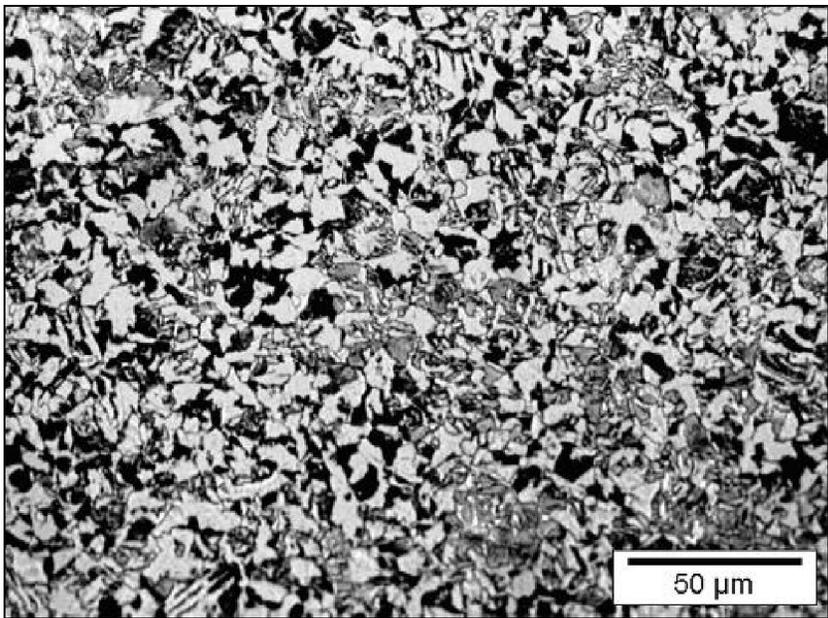


Figure 29: Same figure as in Figure 20 but photomicrographed at a lower magnification for comparison purposes.

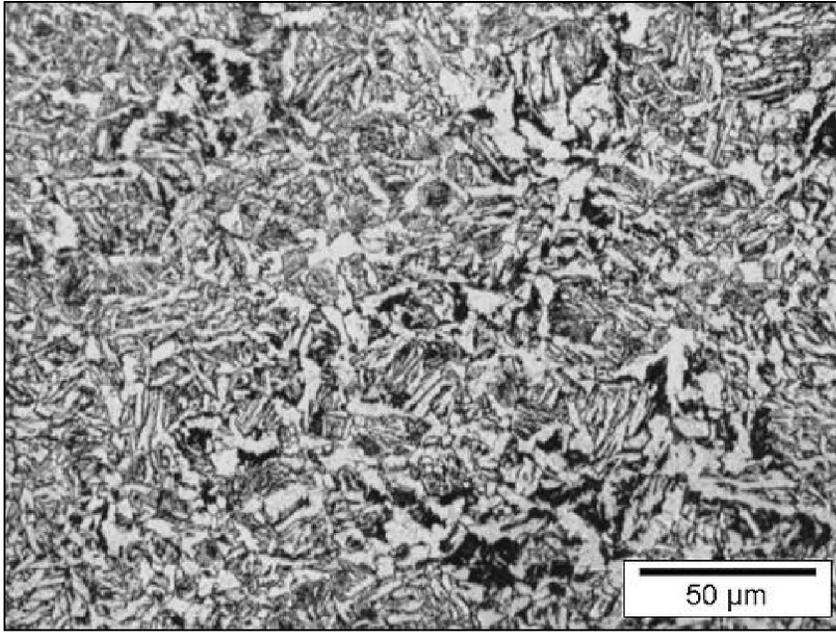


Figure 30: Photomicrograph after simulated rolling at Gleeble FT of 912 °C-1063 °C (Bar mill) of 10 mm NH33201 (Nb + Cr) rebar etched in 2% Nital. $Z = 4.29 \times 10^{17}$

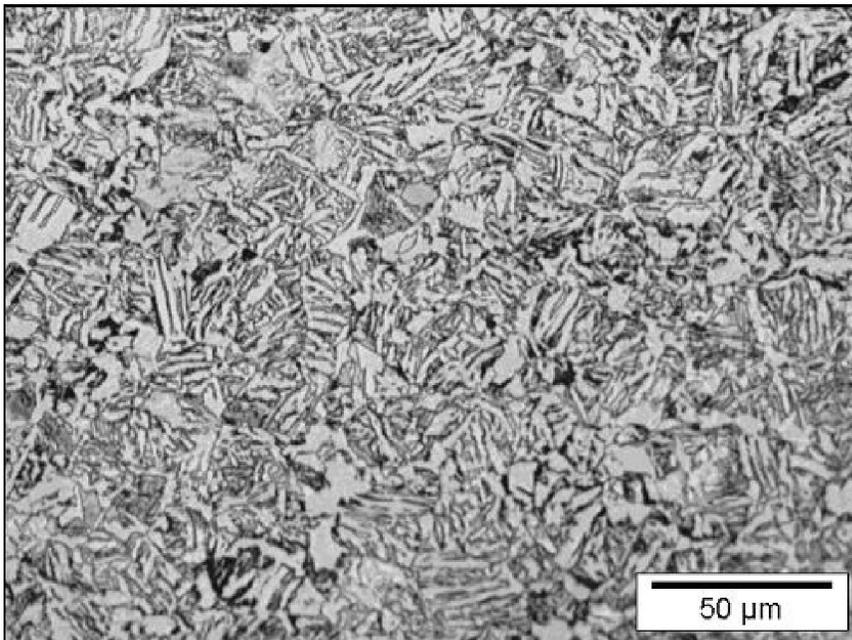


Figure 31: Photomicrograph after simulated rolling at Gleeble FT of 950 °C – 1112 °C (Bar mill) of 10 mm NH33201 (Nb + Cr) rebar etched in 2% Nital. $Z = 1.20 \times 10^{17}$

4.6.2 TEM analysis of precipitates

The precipitation behaviour of the as-rolled 10 mm Nb-Cr rebar rolled on the Bar mill was compared to both the 10 mm Nb-only rebar rolled on the Rod mill and the 10 mm V-only rebar rolled on the Bar Mill. In addition, the effect of aging was studied on the Nb-Cr rebar and the following was found: (see Figures 32-35)⁽³²⁾

- On the Nb – Cr as-rolled bar, few Nb(C,N) precipitates of the size 12-50 nm are present. This was a similar finding as for the Gleeble simulated 10 mm rebar where very few 12 nm Nb(C,N) and 50-100 nm Nb(C,N) were found.
- On the Nb only rod, few Nb(C, N) precipitates of size 10-300 nm are present.
- On the V only rod, a relatively large number of 2-10 nm TiV(C,N) precipitates together with 50-100 nm Ti, Al, V-N present.
- After aging the Nb – Cr bar, a relatively large amount of Nb(C,N) precipitates smaller than 20 nm were present.

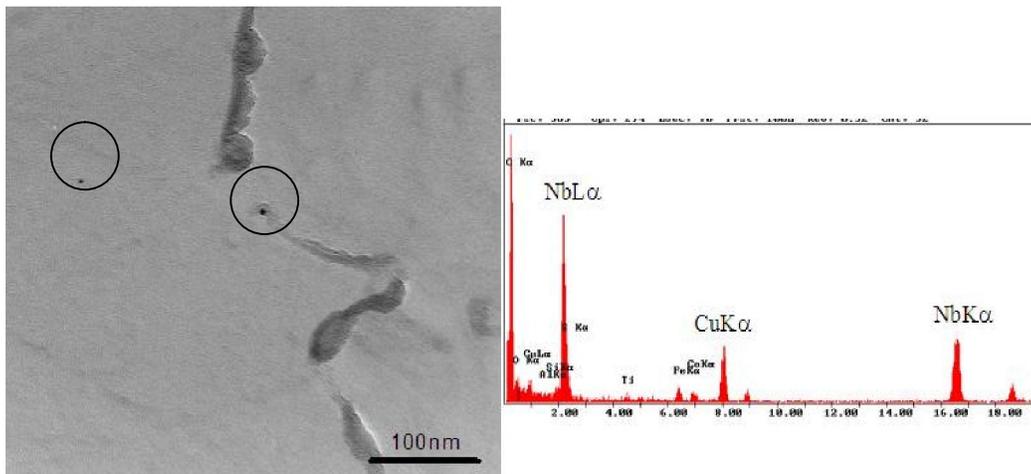


Figure 32: TEM Micrograph showing evidence of a small amount of Nb(C,N) precipitation present on the 10 mm Nb-Cr rebar rolled on the Bar mill. Similar results were obtained for the Gleeble simulated rebar.

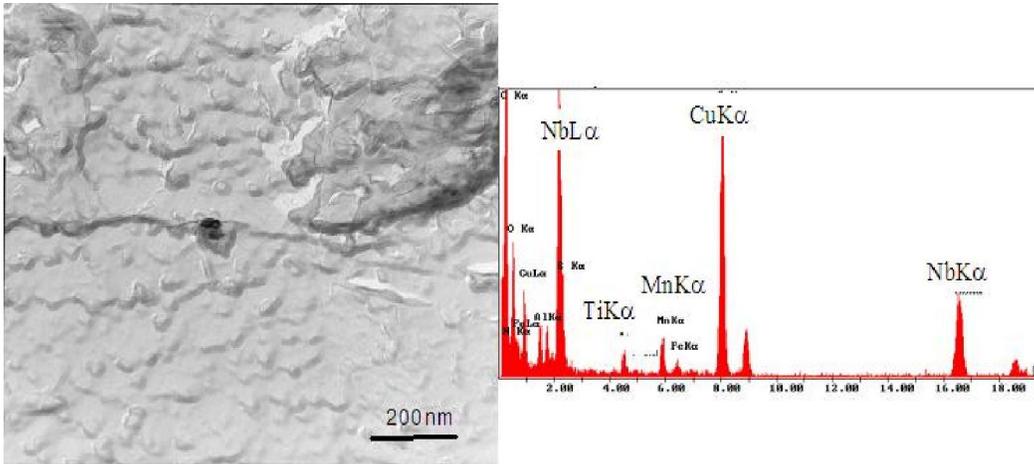


Figure 33: TEM Micrograph showing evidence of a small amount of Nb(C,N) precipitation present on the 10 mm Nb micro-alloyed rebar rolled on the Rod mill.

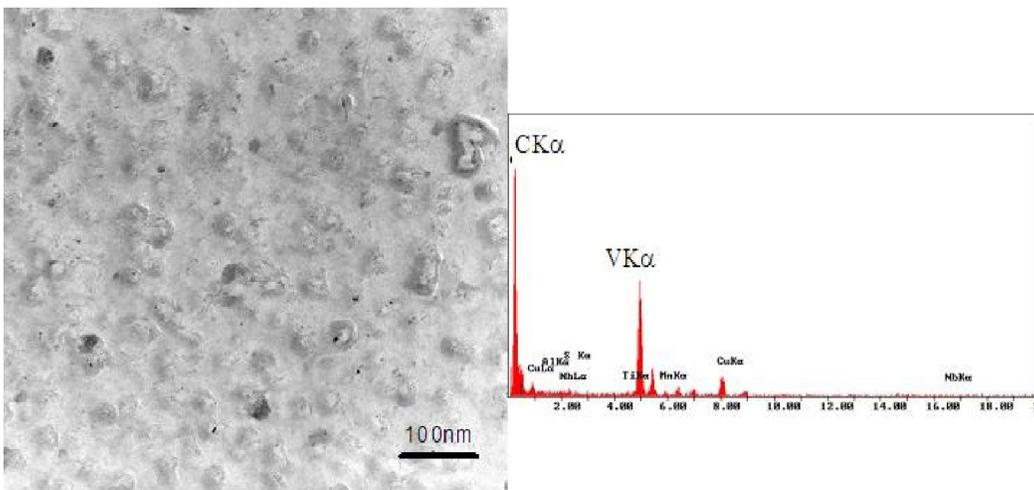


Figure 34: TEM Micrograph showing evidence of a relatively large amount of TiV(C,N) precipitates present on the 10 mm V micro-alloyed rebar rolled on the Bar Mill.

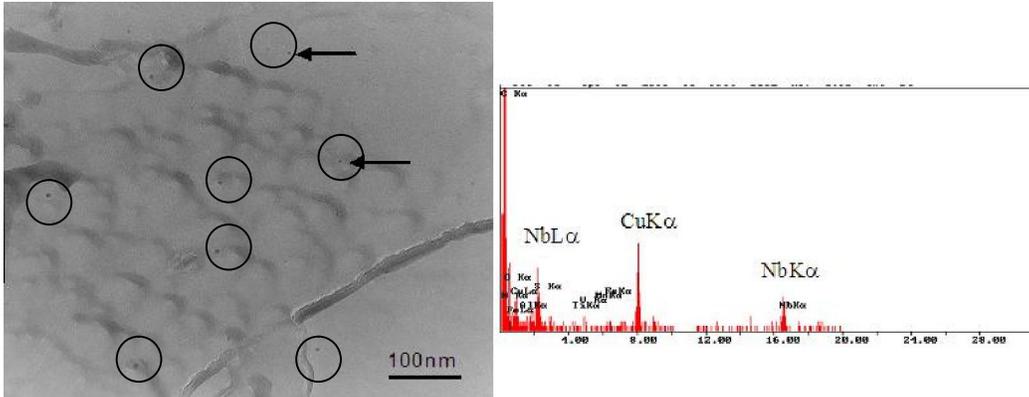


Figure 35: TEM Micrograph showing evidence of a larger amount of Nb(C,N) precipitation on the 10 mm Nb-Cr micro-alloyed rebar rolled on the Bar mill after ageing at 700 °C for 30 min.

4.6.3 Grain size analysis

The grain size of the as-rolled 10 and 12 mm sizes for Nb-Cr rebar rolled on the Bar mill was compared to the Niobium only rebar rolled on the Rod mill and the Vanadium rebar rolled on the Bar mill which can be found in Table 10 below. Figure 36 below shows the typical microstructure achieved after etching subsequent to performing the McQuaid-Ehn test on the 10 mm trial material to reveal to a grain size of 7.0. It can be seen from Table 15 that the finer grain sizes are exhibited on the Nb – Cr trial rebar rolled at the Bar mill.

Table 15: Grain size of as-rolled material

Sample Number	Austenite Grain size	Steel grade	Mill
Cast 602134 - 10mm	7.0	NH33201 + Cr	Bar
Cast 602134 - 12mm	7.5	NH33201 + Cr	Bar
Cast 702510 - 10mm	6.5	VH33216	Bar
Cast 703723- 12mm	6.5	VH33216	Bar
Cast 700251 -10mm	6.5	NH33201	Rod
Cast 703695 -12mm	6.5	NH33201	Rod

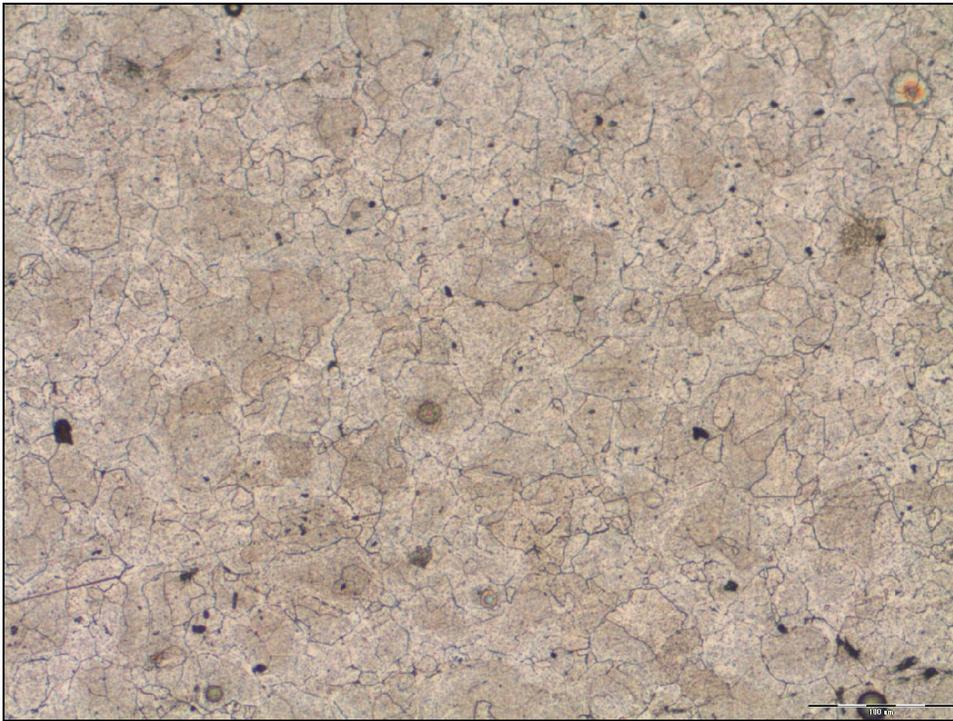


Figure 36: Photomicrograph of 10 mm NH33201 (Nb + Cr) rebar after austenitising and etched in 2% Nital to reveal a grain size of 7.0

5 DISCUSSION

5.1 Effect of the Trial Chemistry on Production and Rolling

The chemical analysis of the trial cast and rolled product analysis produced with the adjusted chemistry, i.e. increased Carbon and Silicon levels with a Chromium addition, was within the aim specification and adhered to the BS4449 standard. This grade was produced and cast on the Steel plant with relative ease during the DT. If this grade was successful in achieving the mechanical properties it would not be problem to the Steel plant in terms of full production since no additional changes in processing practices would need to be done except for alloy additions stated in the DT.

The rolling tempo of the trial rebar was the same as for standard rebar at the Bar mill incurring no additional production time losses or delays. Even with the higher hardenability of this steel grade as compared to standard rebar grades no problems such as cobbles or “kick-backs” were encountered during the rolling of this grade and almost 98 % yield was reported on the mill. Furthermore, a concern raised in the literature that Nb steels exhibit the tendency to bow on the cooling bed due to non-uniform transformation was dispelled since the lengths did not show evidence of bowing while cooling.⁽¹⁹⁾ In addition, samples for testing were cut with ease in the same manner as standard rebar, which was also an initial concern due to the higher hardenability of the trial material. Therefore, if the rebar was successful, rolling it on a full production basis on the Bar mill would also not require additional changes in processing practices or the purchase of new equipment.

5.2 Surface Quality

The surface quality of the cast blooms and rolled billets were of acceptable quality and were thus released for further rolling into rebar. There was no evidence of the presence of transverse cracks. The blooms were critically inspected due to a concern raised in literature relating to the sensitivity of Niobium steels to transverse crack formation during casting. Since the phenomenon of transverse cracking is more pronounced in the presence of

Nitrogen, the absence of cracks is most likely due to low Nitrogen content of the steel i.e. 0.0046 % attributed to the production of the steel via the Basic Oxygen Furnace route. ^(16, 19)

5.3 Macro-etching Results

The macro-structure of the blooms slices revealed good internal soundness with no areas of excessive segregation or porosity visible. No evidence of internal cracks was visible. This was also raised as a concern since the hardenability of the steel was increased and internal cracking due to loss of ductility was feared. The macro-etch result ratings were the same as the ratings achieved for blooms of standard Nb rebar. The macro-etch results further bear witness that no problems were encountered during the steel making and casting of the trial steel grade.

5.4 Sulphur Prints

The results obtained from the sulphur prints are not further discussed since they were unclear. This was due to the relatively low levels of sulphur present in the steel (0.009 %).

5.5 Effect of increasing the hardenability

The effect of increasing the hardenability of the micro-alloyed Nb steel via micro- and macro-alloy additions in order to compensate for the slow cooling rate experienced on the Bar mill on the mechanical properties of rebar was investigated. Increasing the hardenability of the steel had an effect on the mechanical properties in that it increased the tensile strength of the material substantially. However, the required mechanical property of minimum yield strength of 460 MPa as stipulated in the BS 4449 specification was not achieved on the 10 mm and 12 mm rebar produced from the trial cast. This could be seen by comparing the mechanical properties of the different rebar steel grades produced at the different mills in Section 4.5.1. Even though increasing the tensile strength of a material is favourable in that it offers

savings in terms of materials and fabrication, it is the yield strength that is used to guarantee the stability of a structure.

The % elongation of the trial material was much lower than the standard grades, indicating a lower ductility of this rebar compared with the other grades. Furthermore, it must be remembered that even though it appears that the trial material exceeds the required minimum UTS / YS ratio and it appears that the ratios are even greater than the standard rebar grades, it cannot be concluded that the trial material performs better in this regard. This appearance is only due to the fact that the UTS was higher and the yield strength much lower than the standard grades. This trial rebar therefore cannot provide a sufficient capacity for plastic deformation and hence cannot provide a safety margin against fracture.

5.6 Microstructure of as-rolled and simulated bar

The microstructures of the as-rolled Nb-Cr trial rebar, V-only rebar rolled on the Bar mill and Nb-only rebar was compared with the resultant microstructures obtained during laboratory simulations to determine their effect on mechanical properties. In the trial material rolled to 10 mm and 12 mm, a mixed microstructure of polygonal ferrite, pearlite and bainite was obtained. It is the relatively slow cooling after rolling at the Bar mill that produces the pearlite phase in addition to the other two phases referred to above. As mentioned previously, with this mixed microstructure the yield point is suppressed as a result of continuous yielding caused by internal stresses. ^(16, 18) This is not the case when comparing the as-rolled microstructures of the Niobium micro-alloyed rebar rolled on the Rod mill which contains acicular ferrite and bainite and the relatively coarse polygonal ferrite and pearlite microstructure obtained on the Vanadium micro-alloyed rebar on the Bar mill. Both these grades with their respective microstructures achieve the minimum yield strength requirements. The as-rolled microstructure of the trial rebar was successfully simulated on the Gleeble machine indicating an approximate finishing temperature of 1017 °C on the Bar mill.

5.7 Effect of Finishing Temperature

The finishing temperatures (FT) during simulation rolling of the trial material were varied in order to investigate its effect on the resultant microstructures and grain refinement. Since it was not practical to vary the finishing temperatures on the Bar mill during production rollings of rebar, this parameter was varied in the laboratory simulations. It was found that the microstructure changes to mostly acicular ferrite–bainite at a Gleeble FT of 950 °C (equivalent to approximately 1112 °C on the Bar mill). This was the highest FT the material was simulated at. This was the desired microstructure of the trial material since the successful as-rolled Niobium micro-alloyed rebar rolled on the Rod mill consisted thereof. The finishing temperatures immediately after the last rolling stand on the Bar mill vary between 1020 - 1060 °C on average. From the literature it is stated that in order for maximum grain refinement to be achieved in Nb steels, the finishing temperature should be low. ⁽²¹⁾ However, the results of this simulation trial require the opposite in order for the optimum microstructure to be obtained.

5.8 Effectiveness of Niobium as a Strengthener

Niobium is said to increase the strength and toughness of the material by simultaneously grain refinement and precipitation hardening, as well as phase transformation control. From the TEM analysis of the precipitates, it was found that in the Niobium-Chromium trial rebar and the Niobium rebar rolled on the Rod mill, a very low volume fraction of Nb(C, N) was contained in the steel. It was only upon ageing the steel that some Niobium precipitation occurred. This suggests that during rolling and cooling of the Niobium rebar on both the Rod and Bar mill, the Niobium remains largely in solution and therefore it was not utilised effectively as a precipitation strengthener in the steel. This result was in contrast to the Vanadium rebar where copious precipitation of TiV(C, N) was evident and hence the Vanadium was used effectively as a precipitation strengthener. In the literature studied, contrasting views with regards to the presence of Nitrogen were found. Therefore in this trial, it was decided not to make an intentional Nitrogen addition to the steel. It should be

noted that Nitrogen is added to the Vanadium steel grade but not on the Niobium Rod mill grade. This lack of Nitrogen could contribute to the low density of carbonitride precipitation observed in the Niobium grades. ^(12, 15, 32)

Niobium is said to be an effective strengthener when the carbon content of the steel is low, since it allows for increased solid solubility in the austenite phase. Billet soaking time and temperature also affects the solubility of the niobium and hence its grain refining effectiveness. However, in the trial cast, the carbon content of the steel was increased, which initially indicate a possibility that not all the added niobium was taken into solution. In addition to that it was also initially thought that the reheat temperature was too low. Currently the reheat temperature is between 80-100 °C lower than the actual furnace temperature, therefore for the Bar mill, the actual reheating temperature is approximately 1180 °C. By reading off from Figure 37 below, which was plotted by IMMRI for the trial rebar, it is evident that all the Niobium was in solution prior to rolling. ⁽³²⁾ The grain sizes for the 12 mm trial rebar was found to have the finest grain size of 7.5. The grain size found on the other samples evaluated was of similar magnitude and ranged from 6.5 – 7.5.

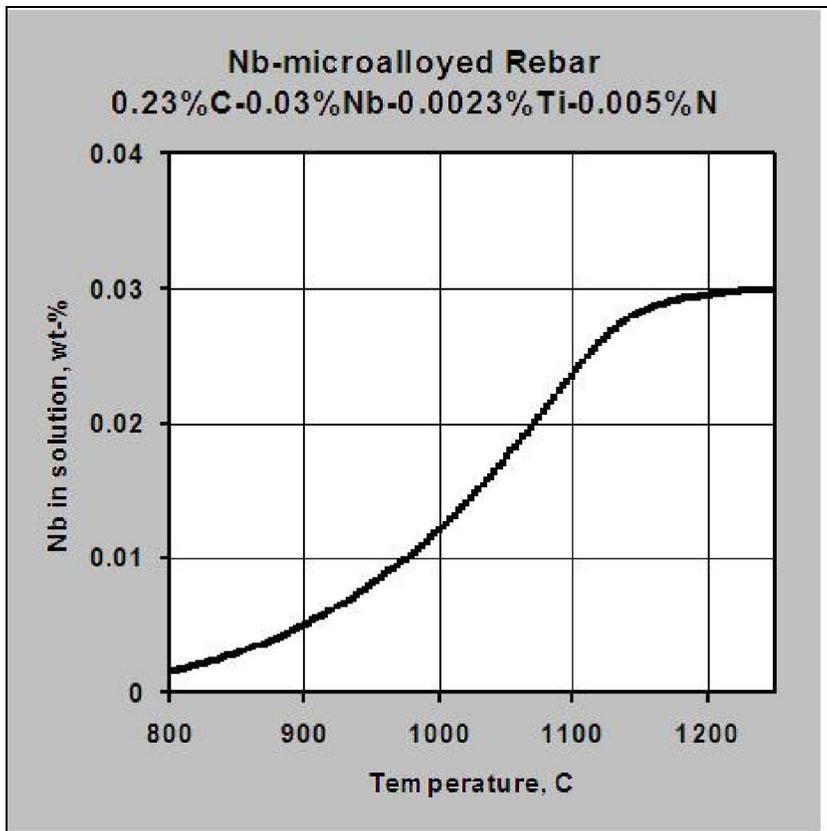


Figure 37: Solubility of Niobium (Graph supplied by IMMRI) ⁽³²⁾

5.9 Hardness Tests

The hardness tests performed on the material were done for comparison purposes, especially in the event where the actual tensile tests were not performed on the material. Hardness does not form part of the rebar specifications. The 10 mm Niobium grade rolled on the Rod mill exhibited the highest average hardness value, while the 10 mm Vanadium grade rolled on the Bar mill showed the lowest values. This can be related to their respective microstructures. The hardness of the Gleeble simulated Niobium-Chromium trial rebar correlated well with the as-rolled hardness, i.e. 237 HV vs 230 HV. Furthermore, the hardness values correlate well with the ultimate tensile test results.

6. CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

- The primary objective to use both the Bar and Rod mill at ArcelorMittal South Africa Newcastle Works to produce rebar complying with specifications thereby introducing the advantage of interchangeability was not met.
- The hypothesis that it would be possible to produce reinforcing bar with a minimum yield strength requirement of 460 MPa successfully at the Bar mill using suitable micro- and macro-alloying element additions was proved untrue. The trial cast with the adjusted Rod mill Niobium micro-alloyed chemistry rolled at the Bar mill to 10 mm attained a yield strength of 429 MPa and 12 mm attained a yield strength of 436 MPa.
- After technical and economic investigations of the current process routes available for the production of the trial rebar on the Bar mill, it was decided that the optimum route was that of micro-alloying.
- The effect of different microstructures on mechanical properties was investigated to design an optimum microstructure needed to achieve the required minimum strength specification. The desired microstructure was acicular ferrite and bainite. Different micro- and macro-alloying elements were investigated in order to design a suitable steel grade for the production of the rebar. Niobium was added as the primary strengthener. To compensate for the slow cooling rate experienced on the Bar mill, the hardenability of the trial steelgrade was increased by the additions of Carbon, Silicon and Chromium. However, this desired microstructure was not attained during the trials. Instead a mixed microstructure consisting of polygonal ferrite, pearlite and bainite was obtained on both the 10 and 12 mm rebar. This microstructure was not optimum in terms of promoting higher yield strength. The yield strength was found to have decreased while the ultimate tensile strength increased.
- The effect of finishing temperatures, via Gleeble simulated rolling, to obtain the optimum microstructure and hence the required yield strength, was investigated. The desired microstructure of acicular

ferrite and bainite on the trial rebar could be obtained by rolling about 50 – 60 °C higher in order to finish closer to 1080- 1120 °C on the Bar mill.

- From the Transmission Electron Microscopy it was evident that the Niobium was not effectively utilised as a precipitation strengthener in the trial rebar rolled on the Bar mill and the Niobium only rebar rolled on the Rod mill, even though it was in solution prior to rolling. The Vanadium was however found to be an effective precipitation strengthener in the Vanadium micro-alloyed rebar.
- Niobium does contribute to the formation of the microstructures during rolling which improves the yield strength.

6.2 Recommendations

- Since the desired microstructure of acicular ferrite and bainite could successfully be attained during the Gleeble simulations it is suggested that an industrial trial be conducted on the Bar mill using higher reheat and rolling temperatures for the 10 –12 mm Niobium-Chromium rebar in an attempt to reproduce the simulation results.
- From the trials performed, it became evident that the Niobium contributed minimally to precipitation strengthening on not only the Niobium-Chromium rebar but also the Niobium-only rebar. It is therefore recommended that a Niobium trial cast with a Nitrogen addition be produced and the effect on the density of Niobium carbide precipitation be investigated.
- Since the effect of using a combination of Vanadium and Niobium as micro-alloy additions to steel for the production of rebar at the Bar mill was not done in these trials, it is recommended that this claimed synergistic effect be further investigated.

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APPENDIX A: DEVELOPMENT TRIAL SCHEDULE

DEVELOPMENT TRIAL
No. 06-001

AIM: To successfully increase the yield strength of 10 and 12 mm Niobium micro-alloyed reinforcing bar (NH33201) to be rolled at the Bar mill.

COMPILED BY: Kim Rajkumar (x8483, x6715)

1. Background

Niobium is used instead of Vanadium in reinforcing as a cost saving initiative. Customers prefer 10 and 12 mm Rebar lengths rolled at the Bar mill as opposed to straightened Rod mill lengths, due to its surface quality. However, because of the vast differences in cooling between the Rod and Bar mills, the chemistry needs to be adjusted in order to achieve a minimum yield strength of 460MPa.

2. Production Planning

J. De Witt

2.1 Schedule the production of one trial cast, steel grade NH33201

STEEL PLANT

3. Scheduling

Scheduler

3.1 The DT is to be executed on the last cast in sequence.

3.2 Notify the standby metallurgist and K. Rajkumar (Tel: x 8483/ sc: 6715) an hour before the start of the blowing process of the sequence.

4. Hot Metal Pre-treatment

Hot Metal Operator

4.1 Follow standard practice instructions for production of this steel grade.

5. BOF Production

BOF Operator/Standby Metallurgist

5.1 Follow standard practice instructions for production of this steel grade, with the following aim changes/additions on the last cast of sequence:

- **Carbon to 0.21 – 0.25 %, aiming for 41= 0.20% C;
44 = 0.21% C**
- **Silicon to 0.45 – 0.55 %, aiming for 41= 0.50% Si;
44 = 0.50% Si**
- **Chromium to 0.15 – 0.20 %, aiming for 0.18%**

6. Ladle Furnace Production LF Operator/Standby Metallurgist

6.1 Follow standard practice instructions for production of this steelgrade, with the following necessary trimmings on the last cast of sequence:

- **Carbon to 0.21 – 0.25 %, aiming for 0.23%**
- **Silicon to 0.45 – 0.55 %, aiming for 0.53%**
- **Chromium to 0.15 – 0.20 %, aiming for 0.18%**

7. Concast Production CC Operator/Standby Metallurgist

7.1 Follow the standard practice instructions for production of this steel grade.

7.2 The last six blooms of the trial cast will be put with AH33203 as per schedule.

BILLET MILL

8. Billet Mill Production Scheduler

J. De Witt

8.1 Schedule 20 tons from the trial cast for rolling into **101mm billets** for the Bar Mill

8.2 Once the cast is produced, the cast number will be communicated

8.3 The remainder of the trial cast blooms will remain on hold until the results of the DT is known. If successful the material will be released for the production of rebar, if not, the material will be regraded to a suitable structural steel quality.

BAR MILL

9. Bar Mill Production Scheduler

Sailesh Murilal

9.1 Couple the trial cast to order numbers to be scheduled and rolled at the Bar Mill as follows:

NLP6030841	10 mm	10 tons
NLP6030842	12 mm	10 tons

9.2 Please note that **no other material except** the trial cast material should be coupled to the above order numbers.

9.3 Clearly note on the schedule that the material will be rolled according to DT 06-001 and that Kim Rajkumar (Tel: x 8483/ sc: 6715) must be contacted at least 1 hour prior to the rolling of the material.

10. Bar Mill Production

Bar Mill SQC'S

10.1 Inform Kim Rajkumar (Tel: x 8483/ sc: 6715) one hour before both rollings of the trial material.

10.2 Roll the trial material into 10mm and 12 mm nostra standard lengths, according to normal nostra rolling practice.

11. Bar Mill Inspection

Bar Mill SQC'S

11.1 Cut samples from the DT material and label it clearly with cast number and bundle number.

11.2 The DT material must be put on hold.

11.3 Send the samples to the Test House for attention of Kim Rajkumar.

QUALITY MANAGEMENT

12. Test House / Kim Rajkumar

12.1 Mechanical testing must be done on the DT samples.

12.2 Report the results to Kim Rajkumar

APPENDIX B: ZENER-HOLLOMAN PARAMETER

On the Gleeble Machine at IMMRI, the highest achievable strains are about 1-10 s⁻¹. However, during rolling on a Bar Mill, the strain rates are in the order of 50-100 s⁻¹ and for rolling on the Rod Mill these rates increase to about 100 - 1000 s⁻¹. Therefore in order for the simulation to correlate with the actual strain rates, the Zener - Holloman parameter, Z, is used. This parameter combines the relationships between temperature, applied strain rate and resulting grain size for a hot rolling process. The calculation of this parameter is given as:

$$Z = \dot{\epsilon} \exp (Q_{\text{def}} / RT) \dots\dots\dots(1)$$

and

$$d = AZ^{-p} \dots\dots\dots(2)$$

- where d = grain size,
- A and p = material constants
- $\dot{\epsilon}$ = strain rate
- Q_{def} = activation energy of deformation
- R = gas constant
- T = absolute temperature

$Q_{\text{def}} = 400$ kJ/mol was selected after consulting some texts ^(38, 39) in order obtain the value for Niobium steels. Figure B1 below shows the equivalent deformation temperatures of the Gleeble machine for 1 s⁻¹ and Bar Mill strain rate of 100 s⁻¹ for similar austenite grain sizes to be obtained. Currently, the finishing temperatures for the 10-12 mm immediately after the last rolling stand on the Bar Mill varies between 1020 - 1060 ° C on average. This implies that for a strain rate of 100 s⁻¹, the equivalent deformation temperature on the Gleeble machine operating at 1 s⁻¹, would range 875 – 915 ° C. ⁽³²⁾

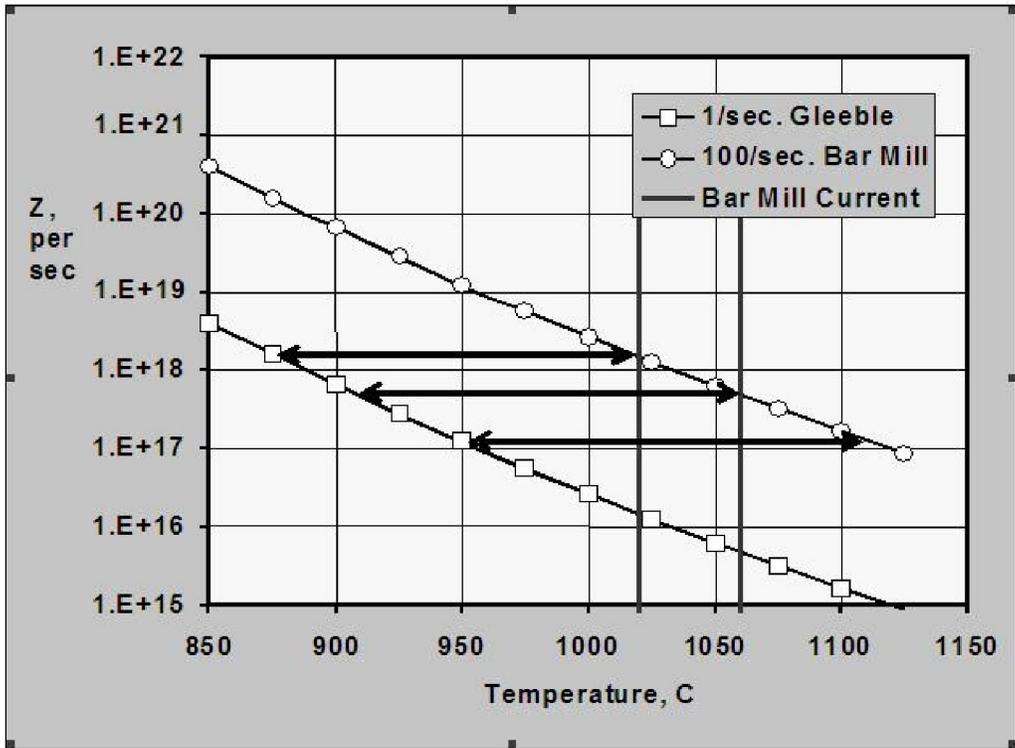


Figure B1: Equivalent deformation temperatures for $Q_{def} = 400$ kJ/mol⁽³²⁾

APPENDIX C: ADDITIONAL MICROSTRUCTURES

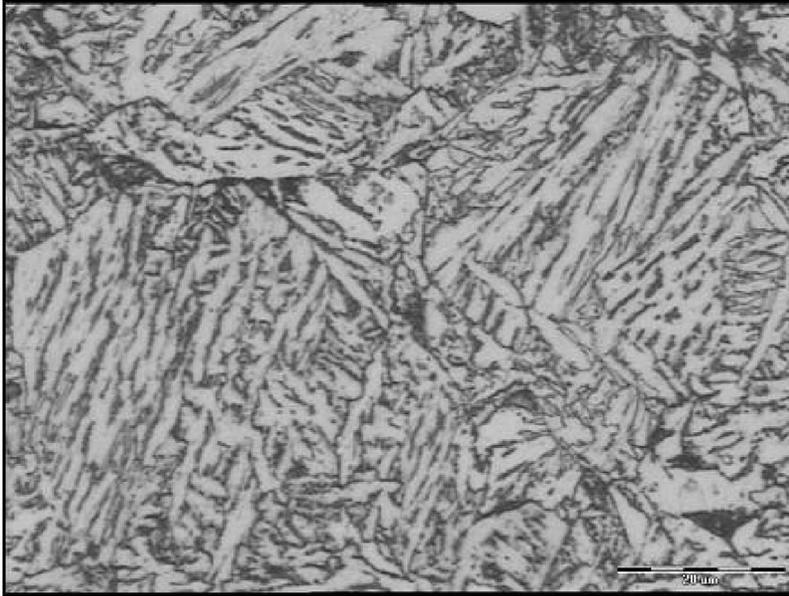


Figure C1: Photomicrograph showing a microstructure containing acicular ferrite and bainite on 12 mm niobium micro-alloyed rebar rolled at the Rod mill

Magnification: x 500

Etchant: 2% Nital

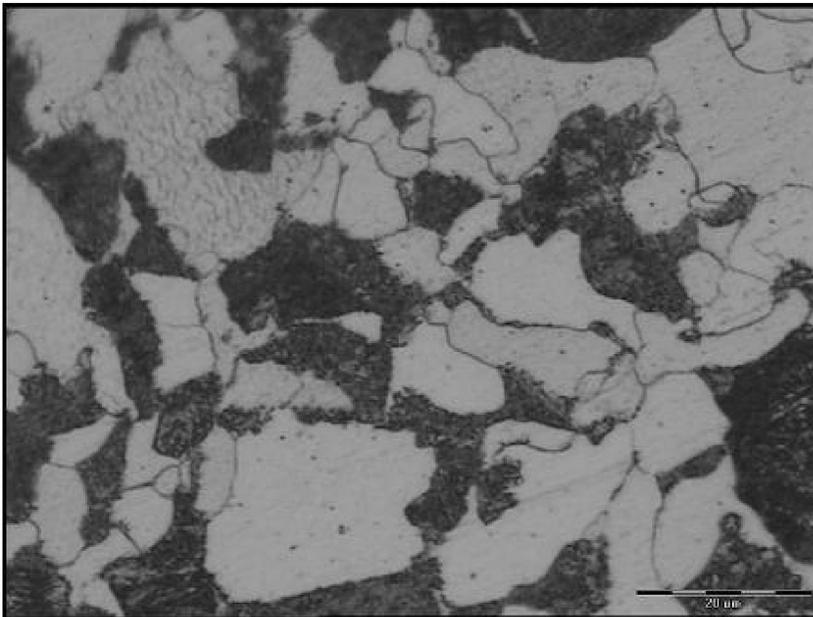


Figure C2: Photomicrograph showing a microstructure containing polygonal ferrite and pearlite on 12 mm vanadium micro-alloyed rebar rolled at the Bar mill

Magnification: x 500

Etchant: 2% Nital