

## **DEVELOPMENT OF NB-BEARING MICROALLOYED STEELS: JSPL EXPERIENCE**

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### **Abstract**

Niobium bearing microalloyed steel has become the standard material in plate and strip for line pipe, automotive and construction grades of steel. Jindal Steel and Power Limited (JSPL) has developed various Nb-based microalloys steel grade with in-house technology to cater to the needs of such applications. The development of these grades requires significant research work both in steel-making and processing technology. The development of Nb-microalloyed steel has been found to satisfy higher strength and impact requirements of steel while saving in production costs.

This paper presents and discusses various studies conducted on the use of Nb microalloying to achieve the desired mechanical and microstructural properties both in structural and wide plates. The need for high quality, low inclusion, steels for making Nb-bearing structural beams and optimization of the API X70 plate process route are also discussed together with the benefits of applying a low carbon (<0.06%) plus Nb concept for enhanced properties and also cost advantages.

### **Introduction**

Jindal Steel & Power Limited (JSPL), part of the Jindal organization has a 3Mt capacity integrated steel plant with future plans to expand its production 6.5Mt by 2010. Steel produced at JSPL is via the Blast Furnace and Electric Arc Furnace route followed by ladle refining as per steel specification requirements capable of meeting all grades for structural steel in normal to high tensile grade from 250MPa to 450MP yield strengths in normal weldable qualities.

The development of microalloyed steels, including their alloy design, processing, and applications, has been extensively developed over the last four decades [1-4]. During this period, microalloyed, high-strength, low-alloy (HSLA) steels became an indispensable class of structural steels. Their ability to achieve final engineering properties in as hot-rolled conditions eliminated the need for heat treatments, such as normalizing. Yield strengths ranging up to 550 to 600 MPa can be attained through small additions (less than 0.1%) of selected carbonitride formers without

requiring costly alloying elements [5-6]. The resulting cost-effectiveness of microalloyed steels led to the successful displacement of heat-treated steels in applications such as truck side rails and telescoping crane booms. Recent technological developments in steel melting and hot rolling further reduced the cost and enhanced the competitiveness of microalloyed steels. Despite these improvements, the total consumption of microalloyed steel is currently estimated to be only 10 to 15% of the world's steel production (i.e., 80 to 120 million tonnes per year). This tonnage is about evenly distributed between flat and long products. As a result, there is plenty of room for growth. A major jump in the usage of microalloyed steels should have strong economic benefits for both steel producers and steel users alike.

The specific effect of niobium is so enormous that the desired improvements in properties can be achieved even with extremely low concentrations of 1 niobium atom in 10,000 iron atoms. Niobium has a high, but not too high, affinity for carbon and nitrogen and can form cubic compounds of type NbX [7-9]. It is sufficiently soluble in gamma-iron (austenite) to be capable of being precipitated again in a targeted way. The potential for precipitation in austenite and ferrite is especially important as a means of influencing the micro structural properties e.g. grain refining; retardation of recrystallization and precipitation hardening etc. The addition of niobium as an alloying element in the steelworks is especially favored by its relatively low affinity for oxygen [10]. The diffusion and precipitation processes in niobium alloyed steel are substantially determined by the relatively large atomic radius of niobium in comparison with iron. Thus, the importance of the metal niobium as an alloying element in steel is due both to its effect in solution and also primarily, to its tendency to combine with carbon and nitrogen [11].

## **Steel Making at JSPL**

### **Refining Process**

JSPL adopted the route of BF-EAF-LF-VD-CCM for the production of microalloy steel grades to accomplish steel cleanliness and desired mechanical properties [12]. The charge mix for EAF contains 100% virgin iron metallics i.e. hot metal from blast furnace and in-house made Direct Reduced Iron (DRI). The share of virgin iron metallic in the mixing is adjusted to the steel composition that is to be produced. The heat capacity at JSPL is 100 tonnes. For the production of steel with lower content of phosphorous and sulphur, the charging materials is prepared accordingly. Slag foaming is ensured in the furnace during melting and refining in order to reduce phosphorous, energy consumption and the nitrogen pick up from the furnace atmosphere. The oxygen is injected through a lance manipulator and through side lances in the steel bath. By using hot metal as virgin iron metallic, the development of CO gas in the steel bath is inevitable during oxygen blowing, which facilitates the purging of nitrogen from the steel bath and promotes the formation of slag foaming. The phosphorous content in the steel is adjusted by lime and dolomite addition during processing in addition to carbon injection that ensures foamy slag. During tapping slag carry-over is minimized by using an eccentric bottom tapping system (EBT) and hot heel technology.

Some slag formers like burnt lime and synthetic slag are added to the ladle during tapping, together with alloying elements to reduce the processing time at Ladle Refining Furnace (LRF). Partial killing of steel bath is carried-out by addition of Al-ingots and Si-Mn in the ladle during

tapping. In order to produce clean steel with minimum gas content, the melt is subjected to degassing either by tank degassing or stream gassing in RHOB. The optimized chemistry to achieve the desired mechanical properties of microalloyed grades is highlighted in Table 1 together with the mechanical in Table 2. It is clear from the tables below that in order to get the desired mechanical properties stringent control is required with respect to sulphur and phosphorous etc [13-16].

**Table 1. Chemistries of microalloyed grades produce in JSPL.**

Grade	Range	C	Mn	S	P	Si	Al	Cr	Mo	Ti	V	Nb	N <sub>2</sub> (ppm)
API 5L X52	Min	0.05	1.40	-	-	0.25	0.025	-	-	0.010	0.020	0.035	
	Max	0.08	1.50	0.008	0.020	0.35	0.040	-	-	0.020	0.030	0.040	
API 5L X60	Min	0.05	1.25	-	-	0.30	0.020	-	-	0.015	0.020	0.040	
	Max	0.07	1.35	0.005	0.015	0.40	0.030	-	-	0.025	0.030	0.050	90
API5L X65	Min	0.05	1.45	-	-	0.25	0.025	0.100	-	0.015	0.040	0.050	
	Max	0.08	1.55	0.010	0.020	0.35	0.040	0.150	-	0.025	0.050	0.055	90
API 5L X70	Min	0.05	1.50	-	-	0.35	0.025	0.150	0.035	0.015	0.050	0.050	
	Max	0.08	1.60	0.005	0.015	0.40	0.040	0.200	0.045	0.025	0.060	0.060	90
S355	Min	0.16	1.05	-	-	0.15	0.020	-	-	-	-	0.025	
	Max	0.19	1.15	0.020	0.025	0.30	0.030	-	-	-	-	0.030	90
EN10025 E450D	Min	0.09	0.95	-	-	0.15	0.020	-	-	-	-	0.020	
	Max	0.12	1.05	0.020	0.025	0.30	0.030	-	-	-	-	0.025	90

**Table 2. Requirement of mechanical properties of microalloyed grades produce by JSPL.**

Grade	Range	YS (MPa)	UTS (MPa)	El. (%)	Impact (J)
API5L-X52	Min	380	475	32	60 at -20°C
	Max	442	575	-	-
API 5L-X60	Min	430	530	30	60 at 0°C
	Max	540	740	-	-
API5L-X65	Min	470	550	30	55 at -20°C
	Max	590	740	-	-
API 5L-X70	Min	500	585	28	40 at 0°C
	Max	600	700	-	-
EN10025 E450D	Min	430	570	30	-
	Max	-	-	-	-

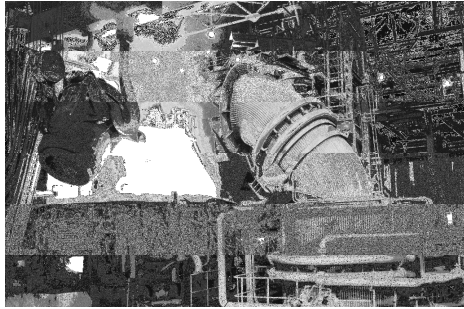


Figure 1. 100 tonne Electric Arc Furnace at JSPL.

### Comparative Performance Evaluation Between Two Degassing Facilities:

#### VD Through Tank Degassing At JSPL

Degassing as well as desulphurization is being performed in the vacuum tank where the entire melt is subjected to degassing through number of ejectors and condensers. To improve efficiency of degassing, quite often de-slagging is carried out before the melt is subjected to degassing. This ensures avoidance of flooding. The melt is also stirred with argon during the entire process of degassing. The vacuum level achieved at JSPL is below 1 mbar and the steel bath is exposed to this vacuum for at least 15 minutes. The total VD time required for degassing through this route is 30 minutes.

#### VD Through RH Degassing At JSPL

RH degassing is mainly used for hydrogen removal for rail steels. For cleaner steel and for ultra low sulphur steel, tank degassing is preferred. Hence degassing is carried out with the help of two snorkels and thereby exposing it to a strong vacuum. In RH degassing below one millibar is achieved much faster as shown in Figure 2. Figures 3 and 4 indicate oxygen and sulphur removal with respect to degassing time in both units respectively. The results of a comparative study carried out to evaluate the performance of both the degassing units are shown in Table 3. Figure 5 shows the photographs of newly commissioned RH degasser at JSPL.

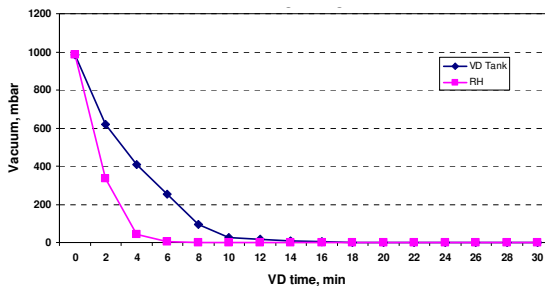
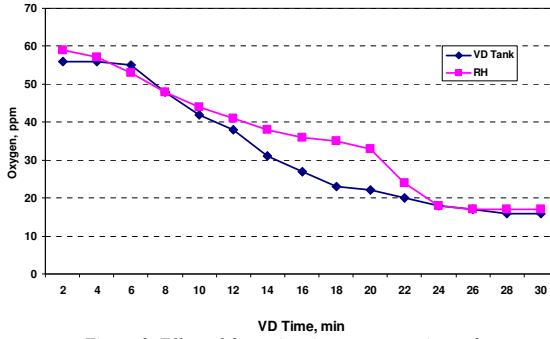


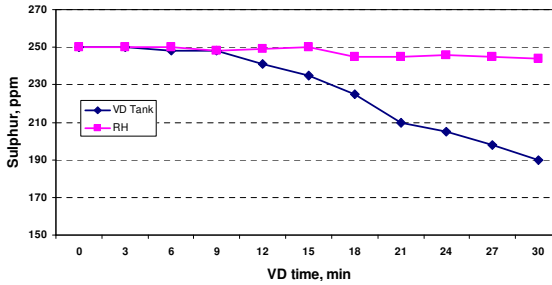
Figure 2. Effect of degassing time on Vacuum achievable.

**Table 3. Comparative study results between Tank and RH degassing units at JSPL.**

<i>Parameter</i>	<i>VD Tank</i>	<i>RH</i>
Free Board, mm	600 – 1200	200 – 300
Refractory Consumption, Kg/t	0.1 – 0.2	0.5 – 1.0
Degassing time, min	25	30
Time required to get < 1 mbar vacuum	12	4
Desulphurization, %	< 0.010	-
Dehydrogenation at 25 min degassing time, ppm	1.0	1.40
<b>Deoxidation at 25 min degassing time, ppm</b>	10 - 15	15 – 20



*Figure 3. Effect of degassing time on oxygen in steel.*



*Figure 4. Effect of degassing time on sulphur in steel.*



Figure 5. RH degassing system at JSPL.

### Continuous casting facilities for casting of slab at JSPL

The continuous casting facilities are summarized below:

- Single strand slab caster
- Machine radius 12 metres
- Slab thickness, 215, 250 and 280 mm.
- Slab width 1500 – 2650 mm
- Metallurgical length 17.3 metres
- Automatic mould level controller
- Dynamic secondary water cooling system
- Automatic marking machine
- Deburrer

The tundish of the slab caster has been designed based on water model modified design has helped to improve yield at same time improving steel cleanliness through increase in residence time as shown in Figure 6. In addition incorporation of turbo stopper along with the pouring pad improved the smooth fluid flow of steel in the tundish. By this, surface waves are reduced and high dissipation of energy in the inlet region of the tundish is obtained, whereby the separation conditions in the rest of the tundish are improved (see Figures 7-a and b). During transient casting turbulence and splashing induced by the incoming shroud jet are reduced.

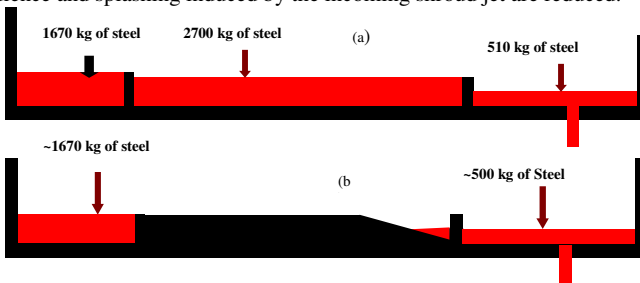


Figure 6. (a) Earlier tundish design, (b) modified tundish design.

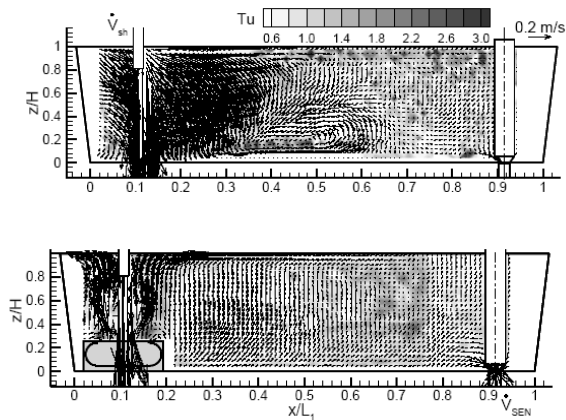


Figure 7. Fluid flow in tundish (a) Turbulent flow (b) Smooth flow with turbo stopper.

### The plate mill at JSPL

The plate mill is equipped with following facilities:

- The slab thus produced is rolled in plate mill having following features
- Level-2 controlled fully automatic top and bottom fired walking beam reheating furnace of 200mt/hour capacity
- High-pressure (200 bar) primary and secondary descalers with hydraulically controlled header height depending upon slab thickness
- Slab turning before and after 2 HI roughing mill (cross rolling)
- Automatic width control through vertical edger
- 4-High reversing finishing mill with Steckle furnace and hydraulic auto gauge control system with X-ray gauge
- Accelerated water cooling system (laminar cooling) to control metallurgical parameters with good surface finish & cooling temperature
- Heavy leveler is installed on-line to control flatness after finishing
- On line plate tilting for both surface inspection and shearing facility for cutting plates in required lengths
- On line ultrasonic testing facility.

### Thermo-Mechanical Controlled Processing (TMCP)

In order to achieve high strength properties of linepipe and some structural steels thermo-mechanical controlled processing is performed as described; rolling is carried out through a set of reduction processes where the austenite is deformed over a range of temperature for conversion of slabs into plates. This influences the final product properties to large extent

through recrystallization and precipitation kinetics [16-17]. The restoration processes has an important bearing on the developing microstructure and is associated with dynamic and static recovery and static recrystallization [18-23]. The schematic restoration process has been described in Figure 8.

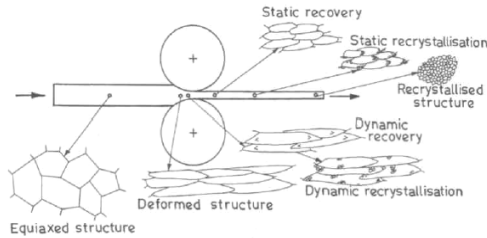


Figure 8. Schematic of restoration process during rolling.

The practice of controlled rolling below  $A_{r3}$  and accelerated cooling ( $7.8^{\circ}\text{C}/\text{sec}$ , efforts are on to increase cooling rate further to  $15^{\circ}\text{C}/\text{sec}$ ) followed by air cooling (as shown in Figure 9) ensured fine grained ferrite / bainite structure even by using lower chemistry and lower carbon [24-27]. The microstructural changes found in TMCP rolling over conventional hot rolling process is shown in Figure 10. This gives great advantage in improving toughness and weldability along with high strength [28-29]. However, Post Weld Heat Treatment (PWHT) softening requires further studies. At present this is being compensated by providing strength beyond the minimum specified yield strength. This is adding to the cost because of higher use of microalloy. Studies conducted elsewhere suggests on line heat treatment after accelerated cooling instead of air cooling. Therefore, there is a possibility of improving PWHT resistance property by using a precipitation technique [30-31].

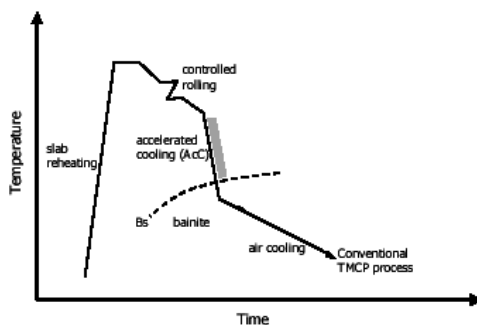


Figure 9. Schematic temperature profile of TMCP process.



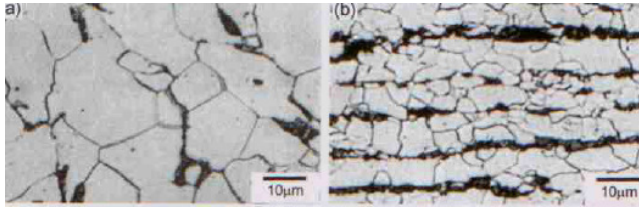


Figure 10. Microstructures of different rolling processes  
(a) Conventional Hot Rolling (b) TMCP Rolling.

### Development of API X-70 Grade by TMCP rolling

Ten experimental API X-70 heats were made with the capacity of 100 tonnes each heat with varying chemical analysis to get desired mechanical properties and these heats were rolled in plate size of 26mm thickness. Table 4 shows the chemistry of various trials taken. To evaluate the mechanical properties 2 tensile test sample prepared according to ASTM E8 specification from plates made of individual slab.

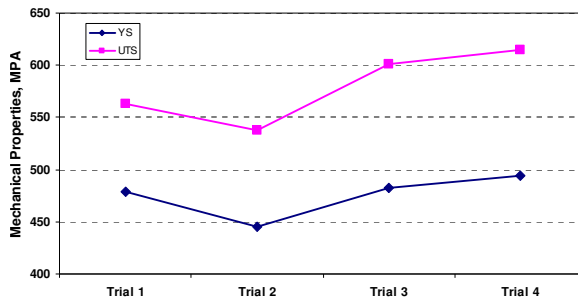
Table 4. Chemistries of various trials taken for development of API X70.

		%C	%Mn	%S	%P	%Si	%Al	%Ti	%Nb	%V	%Cr	%Mo	N <sub>2</sub> (ppm)
Trial 1	Min	0.07	1.45	-	-	0.25	0.030	0.010	0.040	0.050	0.10	0.03	-
	Max	0.09	1.60	0.005	0.015	0.35	0.045	0.015	0.050	0.055	0.15	0.04	60
Trial 2	Min	0.05	1.25	-	-	0.30	0.025	0.015	0.050	0.040	-	-	-
	Max	0.08	1.35	0.005	0.015	0.40	-	0.025	0.060	0.050	-	-	80
Trial 3	Min	0.06	1.50	-	-	0.35	0.025	0.015	0.050	0.050	0.15	0.035	-
	Max	0.08	1.60	0.005	0.015	0.40	0.040	0.025	0.060	0.060	0.20	0.045	80
Trial 4	Min	0.06	1.50	-	-	0.35	0.025	0.015	0.060	0.050	0.15	0.035	-
	Max	0.08	1.60	0.005	0.015	0.40	0.040	0.025	0.065	0.060	0.20	0.045	80

The results obtained from tensile tests are reported in Table 5. Data presented in this table are the average properties of different testing specimens prepared from individual heats. Analysis of these results indicates that the addition of microalloying elements significantly increases the yield and tensile strength. In the trial 1 Cr was maintained between 0.10-0.15% to the base chemistry and in second trial heats were cast without Cr and Mo content. Nb% was gradually increased to understand the effect on strength in trials 3 and 4. Results of trial 2 showed lesser mechanical properties compared with trial 1 where Cr was added. Trials 3 and 4, as expected, resulted in increase in strength with increasing Nb content of steel. Niobium individually has increased the yield and tensile strength to the extent of 12MPa and 15MPa respectively. Chromium and molybdenum additions, however, resulted in a substantial increase in yield and tensile strength (trial 2 and 3) by 37 and 73MPa respectively as shown in Figure 11. Elongation, on the other hand, reduced with increasing microalloying additions but it remained well with in the specification limit. The reason for increase in strength has already been outlined earlier in this paper.

**Table 5. Trial Result of Mechanical Properties of API X70.**

Trial	YS (MPa)	UTS (MPa)	El. (%)	Impact (J)
Trial 1	479	563	42	207
Trial 2	445	538	50	190
Trial 3	482	601	32	218
Trial 4	494	615	41	227



*Figure 11. Results of various trials taken on mechanical properties in API X70 graded steel.*

Cooling after finish rolling had a profound influence on the grain refinement during the  $\gamma / \alpha /$  bainite - transformation and the precipitation of niobium carbonitrides suppressed grain growth which ensured a fine grain structure and subsequent high mechanical properties of the finished product [33-34].

### **Development of E450D Microalloyed steel**

Buoyed by the success of line pipe steel plant took up the challenge of developing 63 mm thick plates requiring 450MPa minimum yield strength. Reportedly there is no other Indian manufacturer able to guarantee high yield strength in plates of such thicknesses. These plates were processed from continuous cast slabs of 215x2020mm and alloyed with micro alloying element like Nb & V. These slabs were reheated and soaked at 1,200°C and were subsequently rolled into 63mm plate for TMCP technology. The microstructure of plates is shown in Figure 12.

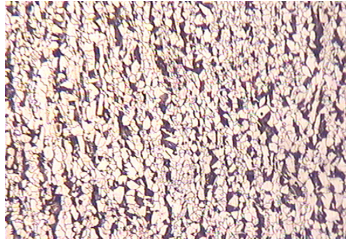


Figure 12. Microstructure of rolled E450D product at 100X.

### Comparative Study

A comparative study has been carried out to find the influence of various microalloying elements on the end properties. For this investigation a few heats were treated with vanadium and some with niobium in EN10025 E355 grades. It can be seen that with relatively smaller addition of niobium in comparison to vanadium helped in achieving desired strength keeping all other parameters intact that led to desired cost reduction. The present practice is to cast EN10025 E355 grade with Nb as the only microalloying element. Figure 13 indicates the consumption pattern of microalloying element over a period of time.

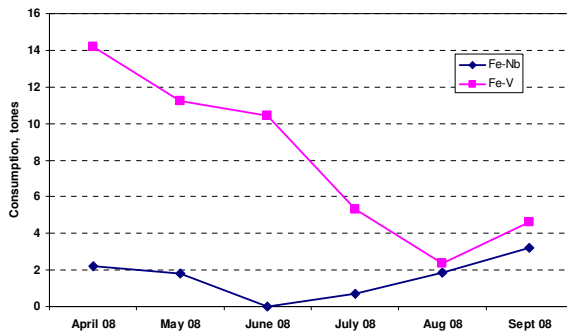
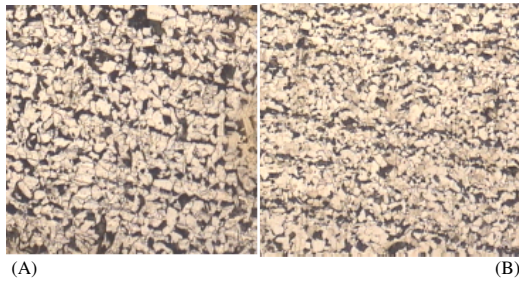


Figure 13. Monthly trend of micro alloy consumption at JSPL

Microstructural examinations were undertaken on samples prepared by standard metallographic techniques and etched with 2% nital. Etched samples were studied using LICA microscope. An image analyzer was used to measure the area fraction of pearlite and ferrite. There has not much change in the microstructures observed as indicated in Table 6 and Figure 14 except the finer grain size observed in Nb steels.

**Table 6. Summary of microstructural results of microalloyed steels.**

Parameter	V Heats	Nb Heats
V	0.035	-
Nb	-	0.025
ASTM Grain Size No.	7.5	8.5
% Pearlite	40.6	39.6
% Ferrite	59.4	60.4



*Figure 14. Microstructure after rolling (A) 'V' Steel (B) 'Nb' Steel.*

### **Challenges encountered during rolling of microalloyed steels**

Following challenges were experienced during rolling of micro alloyed steels:

1. low YS and UTS;
2. transverse cracks originated from the oscillation marks of the cast slabs, and;
3. blister formation.

### **Low YS and UTS**

In spite of substantial microalloy addition with Nb, Ti and V as desired, mechanical properties could not be reached. The attention was focused in reducing the soaking zone temperature where normally ranges between 1,250-1,300°C. Too lower soaking zone temperature created the problem of turn-up at the mill though the properties could be achieved [35-39]. After extensive study the reheating temperature has been optimized at 1,200-1,220°C this has helped in achieving desired mechanical properties. In addition it was ensured that fining temperature below 700 and coiling temperature around 500-550°C is maintained [40].

### **Transverse cracks**

In the initial period of development, presence of transverse cracks were observed in few heats, however, by suitably designing the casting powder and regulating the casting speed the zero

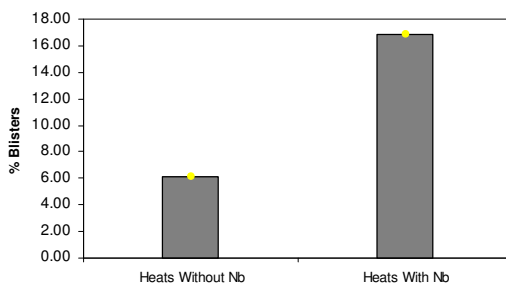
ductility temperature (ZDT) 700-900°C was avoided at unbending area. Because of high width of the slab the casting speed was reduced to have a temperature below 700°C at unbending. These, however, increased the load on the machine and demanded for more segment down time at unbending area. Effort is being made to maintain carbon continuously below 0.07% and search is on for an even better casting powder so that the speed can be increased and the load on the unbending segment can be reduced.

#### **Tackling the problem of blister formation:**

The high strength and thicker plates were found to have blisters primarily on the loose side of the plates in the heats where Nb was added as alloying element as shown in Figure 15. These blisters needed considerable rework before dispatch of these plates. At JSPL steel, as earlier indicated earlier the steel is made from coal based sponge iron and hot metal the nitrogen level has been found to be considerably high (above 90ppm) in comparison to the BOF steel making and gas based DRI in EAF. the presence of higher amount of nitrogen in spite of vacuum degassing has found weak Nb-carbonitrides there by weakening the grains and ultimately caused blisters that can be seen only in thicker plates (above 40mm). In order to combat the problem of formation of brittle Nb-carbonitrides, the one of the efficient nitrogen fixer [41-43], Ti has been used in small measure in addition to rising the aluminium level in the steel. This adjustment in steel making as practically eliminated the problem of blister from our product.

#### **Techno-economics aspect of using Nb as micro alloying element**

In the initial period structural mills and in lower range of high tensile strength steel (YS 355MPa) the use of vanadium was preferred [44-47]. It has been experienced that the requirement of V in steel has been much higher to get the similar kind of properties in the end products. There by raising the cost of production of steel structures and plates. The replacement of V by Nb helped to achieve properties in a similar line with significant reduction in consumption of ferro-vandium. The details of techno-economic aspects using Nb is shown in Table 7.



*Figure 15. Effect of Nb on blister formation.*

**Table 7. Techno-economics of using Nb as microalloy.**

<i>Aspect</i>	<i>With V addition</i>	<i>With Nb addition</i>
Requirement of element in steel to get similar properties	0.030 – 0.035	0.020 – 0.025
Requirement of Ferro-alloys, Kg/Heat	160	90
Cost, Rs/Kg of steel Ferro alloys	1,800	1,300
Recovery, %	Poor	Better
YS, MPa	418	399
UTS, MPa	544	552
Elong, %	28	27

### Conclusions

- The potential of Niobium as micro-alloying element in production of high strength steel has been well established at Jindal Steel and Power Limited, Raigarh.
- Vacuum degassing of micro-alloyed steel helps in achieving high toughness, ductility along with high strength.
- The comparative study of degassing units indicates usefulness of tank degassing over RH degassing unit in production of clean steel.
- The Tundish design based on water model study has contributed significantly towards yield improvement and cleaner steel.
- The success of micro alloyed high strength steel depends to a large extent on thermo mechanical controlled processing followed by accelerated cooling.
- The work is going on to arrest PWHT softening through incorporation of online heat treatment.
- The various problems encountered during making of micro-alloyed plates and coils, like inconsistent strength, blisters and cracks have been tackled successfully through technological modifications.

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