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# STRUCTURAL PRODUCT QUALITY IMPROVEMENTS THROUGH THE MICRONIOBIUM® STEELMAKING ALLOY APPROACH

BY

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## SYNOPSIS

The MicroNiobium® Alloy Approach has been applied in carbon steels exceeding 0.20% carbon such as AISI1050 grade automotive and AISI1080 long product steel applications. Developed applications involve automotive fasteners, seismic resistant rebar and pre-stressed concrete wire rod.

The MicroNiobium Alloy Approach mechanism is described and correlated to a variety of medium and high carbon steel grades and applications. The application of the MicroNiobium Alloy Approach in over 0.20% carbon steels enhances the metallurgical properties, consistency and processability of the hot rolled product. Such process and product metallurgical improvements relate to the Nb-pinning effect of the austenite grain boundaries in Nb- microalloyed steels exceeding 0.20%C steels.

The key operational attribute is the micro-addition of Nb in higher carbon steels which mechanistically pins the austenite grain boundary during the reheat furnace process, thereby minimizing abnormal grain growth in the billet or slab prior to rolling. Typically abnormal grain growth occurs when thermal fluctuations and furnace abnormalities exist in actual reheat furnace operations. This abnormal grain growth leads to inhomogeneous ferrite grains in the final hot rolled product and subsequent variations and reductions in mechanical property performance such as fatigue, fracture toughness and yield-to-tensile properties. Additional evolving development opportunities and applications span pressure vessels, automotive coil springs, eutectoid rail steels, alloy tool and die steels, and tyre rod.

**Keywords:** austenite, grain growth, niobium, reheat furnace fluctuations. properties

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# Introduction

Although the Nb-solubility is limited compared to low carbon steels, through empirical evidence and actual operating data, the MicroNiobium® Alloy Approach has demonstrated very positive results on high carbon grades such as steel wire rods and bars, eutectoid steels, and other medium carbon engineering alloy bar and sheet applications. This technology is being introduced at an accelerated pace throughout the world. The important resultant effect of the MicroNiobium Alloy Approach is the prevention of austenite grain coarsening during reheat furnace soaking of the billet, slabs or shapes before rolling.

The global steel market technological advancement known as the MicroNiobium Alloy Approach may ultimately be applied across all carbon levels to improve product homogeneity during the steelmaking and hot rolling process resulting in improved rollability, finer austenite grain size and less product variation. The concept involves the micro-addition of 0.005 to 0.020%Nb across nearly all carbon grades. Product and industrial process development results indicate that these micro-Nb alloy additions will significantly pin the austenite grain boundary and minimize the heterogeneous abnormal austenite grain growth that occurs in actual reheat furnace mill operations. This micro-approach offsets the inhomogeneous austenite grain coarsening that occurs in the slab or billet during normal reheat furnace or heat treat furnace operation when temperature excursions occur that cause overheating of the steel in normal operation. Temperature fluctuations are caused by a number of furnace and burner operational parameters.

Two Nb strategies are employed in practice depending upon the intended purpose; 1) the Micro-Niobium Alloy Approach simply minimizes the austenite grain size coarsening during reheating through the addition of .005 to .020%Nb and 2) the TMCP approach at higher Nb levels for the dual purpose of grain size stabilization, complex precipitation strengthening and thermomechanical processing. Figure 1 below schematically illustrates the key elements of the MicroNiobium Alloy Approach.

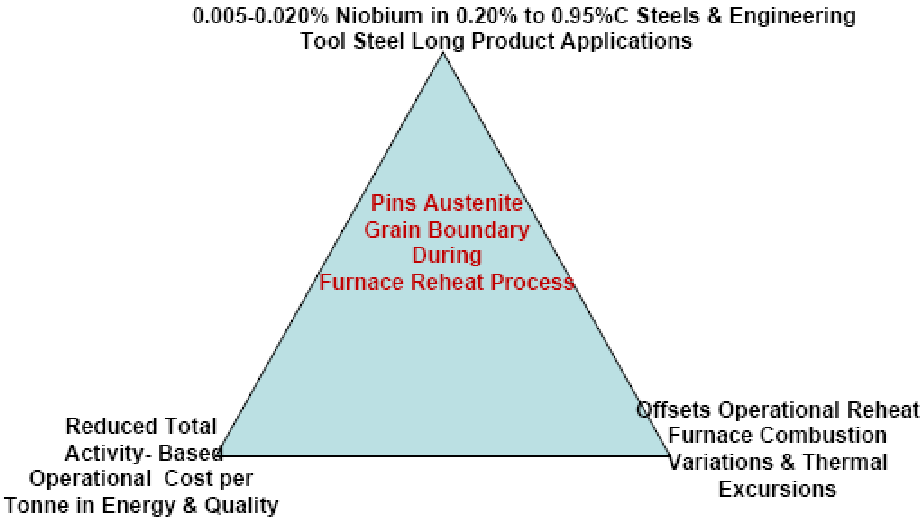

Figure 1 MicroNiobium Alloy Approach [1]

# MicroNb in Medium and High Carbon Steel Applications

Several applications that currently employ the TMCP approach for high carbon steels or the MicroNiobium Alloy Approach are shown below. The MicroNiobium Alloy practice development research and applications are highlighted (in bold print) in Table 1 [2].

Table 1 Nb-bearing medium and high carbon end user steel application via Micro-Niobium Alloy Approach® and TMCP Approach

| Shapes | Bar | Wire Rod | Structural Pipe & Tube | Rebar |
|---|---|---|---|---|
| Power plants | **9259 Spring steels*** | **1080 High carbon pre-stressed*** | Structural scaffolding | **Seismic resistant*** |
| Trailer support rails | Forging quality | Engineering | Construction | **Fire resistant*** |
| **Rails*** | **1050 Automotive fasteners*** | **Cold headed*** | Irrigation and utilities | Bridges |
| **High alloy tool steels*** | Carburized gears & shafts | **High strength bolts*** | Boiler tubing | Buildings |
|  | Quench & Temper | Wire rope | Utility power plants | Tunnels |

# Microalloy Solubility Considerations

The use of solubility equations to ascertain the solubility of different microalloying elements such at niobium, titanium and vanadium must be used judiciously. Most of the solubility data is based upon laboratory conditions approaching the equilibrium state. The problem is that in actual operations, furnace reheating conditions are certainly far from equilibrium. Since actual furnace operation is not at steady state, laboratory generated solubility data and solubility equations are only approximate guide to mathematically define a given microalloy's solubility behavior during reheating. Therefore, much of the published solubility data is difficult to directly apply to actual operational conditions in an industrial hot rolling operation. Many researchers and material designers attempt to apply this somewhat incongruent data to their actual operation and find inconsistencies in the expected performance of a given microalloy.

Typically, laboratory generated solubility equations which are part of the furnace model are a starting point and certainly the appropriate initial setting for the model. The second step involves adjustments to the furnace heating model based upon actual industrial heats, operational experience and microstructure and precipitate evaluation. Then, the furnace model solubility equations are adjusted to better characterize the true solubility of these microalloys at different carbon levels.

For example, it is given that the solubility of Nb decreases as carbon content increases. An example of the relationship between niobium solubility versus carbon content is shown in Figure 2.

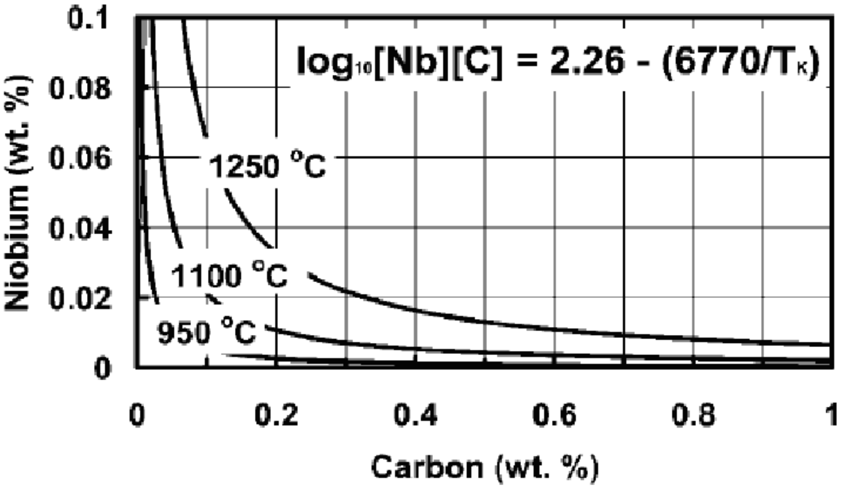

Figure 2 Niobium Solubility versus Carbon Content

For example in a 0.2% carbon steel, at 1100°C, theoretically 0.010%Nb is soluble or in a 0.4% carbon steel, at 1100°C, theoretically 0.005%Nb is soluble. As a result, many researchers and operators have adopted the conclusion that due to niobium's low solubility in high carbon steels then there is no possible benefit to the application of Nb in such steels. This situation is not the case. Recent metallurgical and industrial trial developments have disproved this conclusion. Several commercial applications are presented herein which define the optimal MicroNiobium addition in a variety of medium and high carbon steel grades.

## MicroNiobium Alloy Approach Mechanism

The MicroNiobium Alloy Approach involves a .005 to .020%Nb addition in higher carbon steels. The niobium pins austenite grain boundary and minimizes/offsets grain growth during reheating due to reheat furnace temperature fluctuations. These furnace fluctuations may initiate from a number of areas such as burner combustion problems, incorrect air to gas ratios and/or improper furnace pressure to name a few. Overheating of the entire slab or just a section of the slab translates into abnormal austenite grain growth, Evaluation of the MicroNiobium Alloy Approach effectiveness is related to less variation of mechanical properties because this abnormal austenite grain growth is retarded and the final austenite grain size before hot rolling or hot forging are smaller. The root cause for many mechanical property variations measured in the final hot rolled product relate to abnormal prior austenite grain size resulting from an inconsistent reheat furnace operation.

# Reheat Furnace Operation for Nb-Modified Medium and High Carbon Steels

The initiation point for proper austenite grain size control is in the effectiveness and consistency of the heating of the slabs, billets or profiles prior to hot rolling. Homogeneous heating and soaking of slabs is vital in order to minimize temperature gradients (ΔT) between the surface and center of the slab and the ΔT from the front end to back end of the slab. Often during the rolling of C/Mn and microalloyed steels, variability of the ΔT from the front end to the tail end and/or high ΔT's from surface to center of the slab, billet or shape translate into variable mechanical properties within a coil, bar or plate regardless of the mode of rolling. In addition to less mechanical property variation, homogeneous heating translates into flatter and straighter hot rolled product (i.e. improved flatness and shape), more uniform and finer austenite grain size, solution of the microalloy carbon nitrides and improved rollability.

Depending upon the reheat furnace efficiency and heating schedules, reheat temperatures for medium carbon and high carbon steels should generally range between 1125°C to as high as 1230 °C range. Inefficient reheating is the reason some mills overheat Nb-bearing medium and high carbon slabs, billets and sections at 1230°C, whereas efficient heating can lower the soak temperature well below 1200°C. Generally, this data varies depending on the researcher and should be incorporated into the furnace model with caution. Actual operational experience indicates that over 75% Nb-solubility is achieved at temperatures as much as 25 to 50°C lower than predicted by solubility equations.

Based upon actual operational experience and performance, the following root causes promote the formation of coarse grain austenite in these medium and high carbon steels:

- Excessive soak zone temperatures exceeding 1230°C
- Improper furnace control cutback on operational delays, thereby overheating slabs, resulting in slab/billet sticking, abnormal austenite grain growth and lost production
- Poor combustion fan efficiency (which directly correlates to surface quality) especially on high carbon sheet production exceeding 0.50%C)
- Different air-to-gas ratios should be employed for different steel grades (i.e. low, medium and high carbon steels)
- Inefficient burner combustion at the orifice and maintenance considerations

Within the reheat furnace, the spatial distribution of heat flux from the flame remains essentially unchanged, having its maximum value downstream of the burner exit and decays with axial distance further downstream from the burner exit. The spatial distribution of heat flux in a reheat furnace is not uniform both along the flame and transverse to the flame. The maximum heat flux is found close to the burner exit and minimum farther downstream or radially displaced away form the flame axis [3, 4].

Although some mills have increased the burner flame temperature through adaptation of air preheat or oxygen enriched combustion air to enhance thermal efficiency, the problem of non-uniform heat flux distribution in the furnace and localized overheating of a section of the slabs or billets in actual operation still remains. Localized overheating of slabs and billets translates into localized abnormal grain growth.

Optimization of Combustion Air to Gas Ratio and Product Quality

The control of the optimum air to gas ratio for different carbon levels is critical and outside the scope of this paper. However, as it relates to abnormal grain growth and overheating of the steel, the combustion connection is that at the optimum air to gas ratio, the adiabatic flame temperature is the highest and most efficient for the specific energy consumption per tonne of steel and highest combustion efficiency. For example, in actual operations, the periodic checking and resetting of air-fuel ratios for burners is one of the simplest ways to get maximum efficiency out of fuel-fired process heating equipment such as reheat furnaces. Process heating efficiency is reduced considerably and product quality is negatively affected if the combustion air supply is significantly higher or lower than the theoretically required air. Air-gas ratios are determined by flow metering of air and fuel or flue gas analysis.

The role of the MicroNiobium Alloy Approach in the reheat furnace operation provides some processing flexibility in retarding the austenite grain growth due to several of the aforementioned operational and heating issues experienced in actual operating conditions. Several industrial examples and applications of the MicroNiobium Alloy Approach will illustrate its effect.

## Nb Microalloy Design Considerations

In some instances, Nb has not been the microalloy of choice or even considered for that matter in high carbon equivalent steels because of the theoretical lower solubility of the Nb carbonitrides in higher carbon steels previously discussed. Although there is lower solubility, the effectiveness of Nb in the grain refinement and precipitation strengthening mechanism in Nb-only and Nb-modified V containing steels. Within the higher carbon steel segment, much of the Nb-high carbon microalloy metallurgical research over the last two decades incorporated higher Nb levels than necessary. Consequently, results were inconsistent and many incorrectly concluded that there is no place for Nb in high carbon alloy designs.

These higher Nb levels (exceeding 0.040%) were thought to be necessary in order to obtain proper grain refinement, microstructural control and strength in higher carbon equivalent steels. Experience to date has in fact indicated that the higher Nb levels in high carbon steels certainly make the processing more challenging and the resultant properties are not optimized. It is important to consider the synergistic precipitation behavior effect between the Nb and V and, in some cases, Mo which can contribute to the improved mechanical performance. This duplex or triplex microalloy complex precipitation behavior requires further study [5].

Based upon actual commercial product applications, recently a richer understanding of the Nb-high carbon technology mechanisms, metallurgy and processing parameters has been achieved. This information is invaluable for the implementation process to successfully incorporate low levels of Nb in existing high carbon equivalent steels to improve fatigue, fracture toughness, ductility and overall product performance. The development of the coil suspension spring is an excellent example of such a successful application.

Other results from product applications reveal the fact that in some cases more Nb is not always better. Based upon operational experience, the optimization of the Nb content and the proper control of the reheating furnace are critical. An optimum Nb concentration may be directly correlated to a given carbon level depending upon the reheat furnace process metallurgy parameters, heating practices and combustion conditions at a given mill. For example, the quality and consistency of reheating high carbon (>0.50%C) billets and slabs can be enhanced through the incorporation of combustion practices resulting in an air to gas ratio less than 1.00.

## MicroNiobium Effect in 1050 Medium Carbon Automotive Fasteners

The most recent application involves a 0.02%Nb addition to flat rolled steel fasteners in the gauge range of 1.9 to 3.0 mm. A one month industrial trial was conducted in which 0.02%Nb was added in all 1050 heats. Quite simply, a cost of quality comparison was made between the historical quality cost and the Nb one month trial cost of quality. The cost of quality included internal hot mill diverts for gauge, cobbles and excessive mechanical property across the sheet and from the head to the tail of the coil. All three quality attributes improved reducing the overall cost of quality compared to historical 1050 performance without Nb. The improvement in the cost of quality far exceeded the additional alloy cost for the Nb. The improvement was validated by less variation in the prior austenite grain size. Also, no specification change is necessary as the 0.02%Nb meets the chemistry specification.

## MicroNb Effect on SAE9259 Automotive Coil Springs

Niobium grain refinement enhances toughness and fatigue resistance in high carbon and alloy grades at concentrations as low as 0.01%. Nb to as high as 0.030 to 0.040% range. This has been applied to some degree in Japan, Canada, United States and Europe via the TMCP approach. Currently, both Nb high carbon steel research and commercial applications have gained significant momentum. This re-kindling of interest and acceptance is building upon much of the excellent research and development performed over two decades ago in the bar and wire rod sector.

Mechanical properties improve with the addition of Nb in rebar, structural shapes and automotive structural components, such as springs. For example, a North American vehicle front suspension coil spring of 0.51%C with Mo-V-Nb was developed and commercialized with improved mechanical properties compared to conventional springs. A similar effect was observed when adopting 0.035%Nb in a 9259 engineering alloy spring steel grade. The improved properties are attributed to the grain refinement, microstructure, inclusion morphology and precipitate strengthening provided by Nb [6]. The chemistry of the Nb-modified spring steel is shown in Table 2.

Table 2 Nb-Modified 9259 spring steel heat analysis

| **Grade** | **C** | **Mn** | **P** | **S** | **Si** | **Cu** | **Ni** | **Cr** | **Mo** | **V** | **Nb** | **Ti** | **N ppm** | **ASA** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **SAE 9259** | 0.61 | 0.86 | 0.014 | 0.021 | 0.78 | 0.008 | 0.008 | 0.51 | 0.008 | 0.005 | 0.002 | 0.003 | 55 | 0.028 |
| **V-SAE 9259** | 0.60 | 0.81 | 0.020 | 0.017 | 0.85 | 0.007 | 0.009 | 0.51 | 0.003 | 0.100 | 0.002 | - | 110 | 0.002 |
| **Nb-V-Mo 9259** | 0.51 | 0.69 | 0.016 | 0.020 | 1.31 | 0.007 | 0.012 | 0.45 | 0.040 | 0.120 | 0.035 | - | 120 | 0.002 |

The improved hardness at temper temperature has translated into increased strength, better fatigue endurance limits and good fracture toughness, thereby allowing for a lighter weight design coil spring. This Nb-V modified coil spring steel has resulted in the reduction in weight of a coil spring by approximately 15%. The fatigue performance comparison is illustrated below in Figure 3.

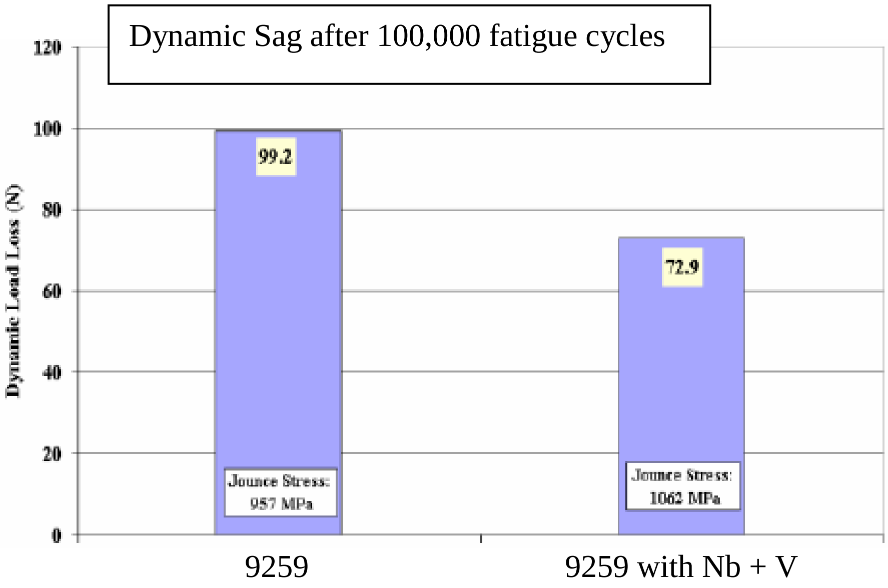

Figure 3 Fatigue Cycle Test [6]

The fracture toughness ($K_{IC}$) measurements show a 27% improvement in fracture toughness of the Nb + V modified grade compared to the V-modified grade as illustrated in Figure 4.

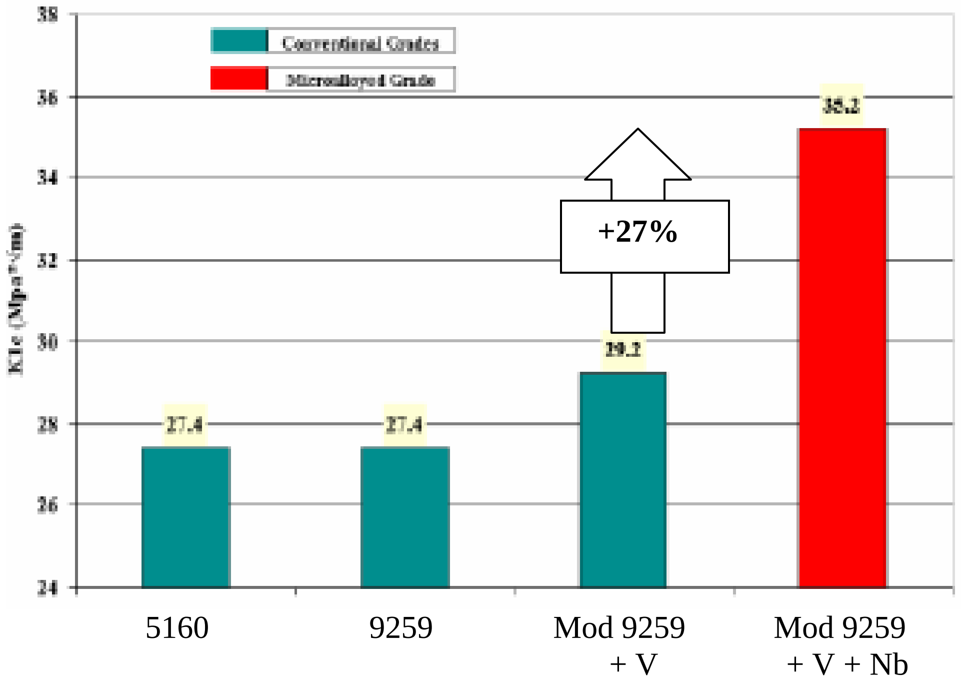

Figure 4 $K_{IC}$ Fracture toughness comparison of Nb-V bearing coil spring [6]

The resultant Nb-V modified grade exhibited improved yield and tensile strength which translates into better cyclic fatigue life and improved fracture toughness. The adjusted steel chemistry, grain refinement, Nb-V(CN) precipitation strengthening and overall lower volume fraction of hard oxide inclusions resulted in the better properties. This application illustrates the complimentary synergy between Nb and V in these higher carbon engineering tools steels. In actual operation, the Nb to V stoichiometric ratio has been reduced to further reduce the cost of the V and Nb additions.

## MicroNiobium Alloy Approach in Pressure Vessel Plate

The production of ASTM A516 Grade 60 plate steel has successfully incorporated 0.02%%Nb for the dual purpose of 1) reduce Charpy V-Energy variation between heats and 2) increase the minimum Chary threshold, thereby reducing diverts. Figure 5 illustrates the increased mechanical property scatter and minimum threshold for Nb-bearing pressure vessel plate versus non-Nb pressure vessel plate.

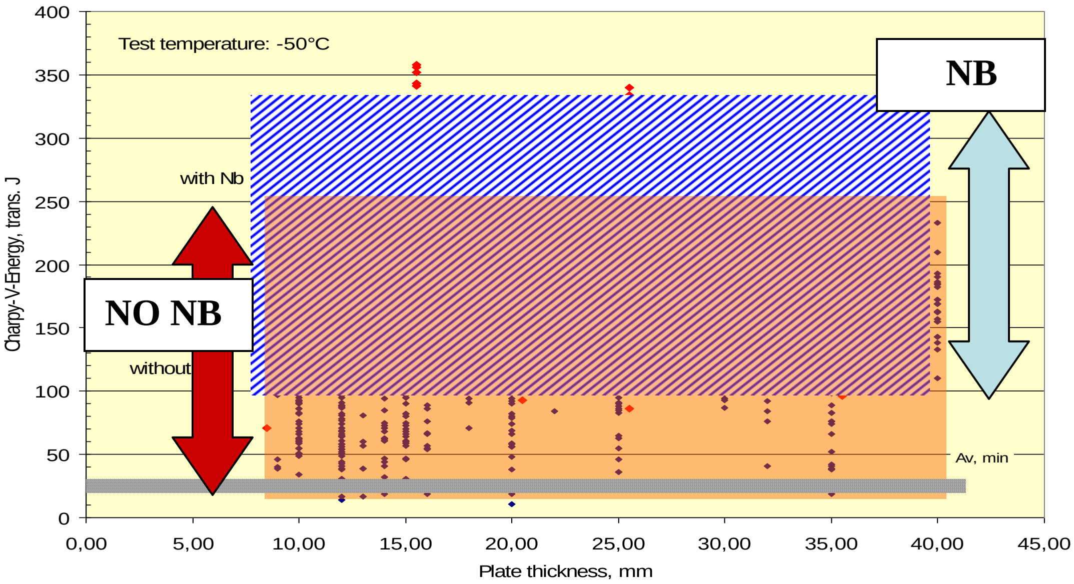

Figure 5  Nb versus Non-Nb pressure vessel plate [7]

The minimum toughness specification at -50ºC is 27 Joules. Note several scatter points below the threshold and consequently, the material was downgraded at a cost. With Nb, the minimum toughness raised to 100J nearly a 3.5 time improvement over the specification minimum of 27J.

## MicroNiobium Alloy Approach in Eutectoid Steels

Another rapidly developing area of research involves the application of micro Nb-modified eutectoid steels. It has been demonstrated that the process and physical metallurgy for this 1080 steel can be cross applied to both rail steels and pre-stressed wire rod since many of the grades are produced from similar high carbon compositions (approximately 0.80%C). This cross-application concept was introduced a few years ago for other plate and sheet applications [8].

For eutectoid and near-eutectoid rail steels, the wear resistance is related to both hardness and the pearlitic interlammelar spacing. The interlammelar spacing controls the strength, but several other factors need to be considered in this project as it relates to wear and rolling contact fatigue on rails. These factors include; 1) the grain boundary volume fraction coverage of pro-eutectoid cementite, 2) the steel internal cleanliness and 3) non-metallic inclusion chemistry, morphology, volume fraction and distribution. Figure 6 shows the effect of the pearlitic block size and reduction in area of patented 2%Cr versus Nb-modified 2%Cr steel wire rod [9].

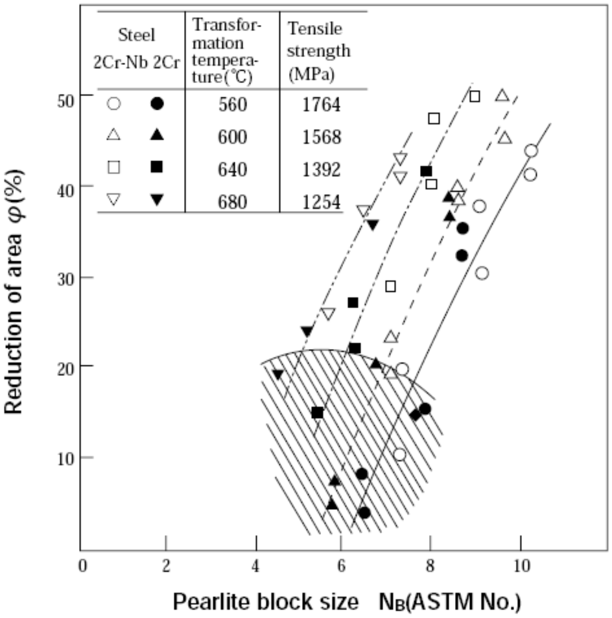

Figure 6  2Cr-Nb vs. 2Cr wire rod reduction of area (%) comparison

The reduction in area will increase with an increase in pearlite block size number (which is a decrease in the pearlite block size). The MicroNiobium Alloy Approach of only 0.02%Nb reduces the pearlite block size by reducing the austenite grain size. As a result, ductility measured in % reduction in area is improved.

The increase in demand for high speed rail systems and improved rail performance has initiated research into Nb-modified rail steels. Improved rail performance is measured by the following attributes: 1) wear resistance, 2) rolling contact fatigue resistance, 3) ductility and 4) weldability. The finer pearlitic microstructures in Nb-bearing 1080 steels result in better wear resistance than martensitic and ferrite-spheroidized cementite microstructure at similar hardness levels. Only 0.02%Nb is needed to refine the pearlitic colony size in rail steels [10].

The metallurgical and operational knowledge gained from the eutectoid steel development in rail may be cross applied to other products in the 1080 carbon family such as wire rod for tyre cord and prestressed concrete wire rod for construction.

# References

[1] S. Jansto, "Current Development in Niobium High Carbon Applications," *MS&T Conference,* October 16-20, 2011, Columbus, Ohio.

[2] S. Jansto, "21st Century Niobium-Bearing Structural Steels," *HSLA2011International Microalloy Conference*, May 31-June 2, 2011, Beijing, China.

[3] M. Katsuki and T. Hasegawa, “The Science and Technology of Combustion in Highly Preheated Air,” *Proc. 27th Symposium (Intl.) on Combustion*, The Combustion Institute, PA, 1999, pp. 3135-3146.

[4] A.K. Gupta and Z. Li, Z., “Effect of Fuel Property on the Structure of Highly Preheated Air Flames,” *Intl. Joint Power Generation Conference*, Proc. IJPGC-97, Denver, CO, November 3-5, 1997, ASME EC-Vol. 5, 1997, pp. 247-257.

[5] S. Jansto, “Seismic and Fire Resistant Niobium-Molybdenum-Bearing Long and Plate Products,”*1st International Symposium on Fundamentals and Applications of Mo and Nb Alloying in High Performance Steels*, November 6-7, 2011, Taipei, Taiwan.

[6] M. Head, T. King and A. Radulescu, “Development of New Microalloy Steel Grades for Lightweight Suspension Systems,” presented at *AISI Great Designs in Steel Seminar*, 2005, Livonia, Michigan (www.autosteel.org).

[7] A. Kern, “High Performance Steels for Pressure Vessels,” *International Symposium on Niobium-Bearing Structural Steels*, New Delhi, India, April 12-14, 2011.

[8] S. Jansto, “Cost Effective Microalloy Structural Steel Balance of Process Metallurgy and Materials Engineering,” *MS&T*, Pittsburgh, PA, October 2008.

[9] T. Takahashi, M. Naguma and Y. Asano, *Journal Japan Society of Technological Plasticity*, v19, 1978, p726.

[10] Y. Tamura, M. Ueda, T. Irie, T. Ide, J. Fukukawa and M. Muraki, *NKK Technical Report*, No. 79, 1978, p 335.