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Vanadium and niobium microalloying to increase strength of high-carbon wire steels

By Stephanie L. Miller and Emmanuel De Moor, Colorado School of Mines, USA

Abstract:
In order to investigate the effect of V, Nb, and N additions on the strength of eutectoid steels, laboratory heats were prepared and continuous cooling experiments were conducted using a Gleeble® 3500 thermomechanical simulator. V strengthening effects were observed in all microalloyed steels, and maximum strengthening was observed in the V+N steel. The V+Nb steel was found to have the greatest refinement of colony size and ILS, and subsequently higher hardness among the test alloys.

Miller

De Moor

Stephanie Miller is currently working with the Advanced Steel Processing and Products Research Center (ASPPRC) at the Colorado School of Mines, Golden, Colorado, USA, in pursuit of a doctorate in metallurgical and materials engineering, with intended completion in Spring 2015. She holds an M.S. degree in metallurgical and materials engineering from the Colorado School of Mines and a B.S. degree in materials science and engineering from the University of Illinois at Urbana Champaign. Emmanuel De Moor is a research assistant professor with the ASPPRC at the Colorado School of Mines. He is involved in advanced high-strength sheet steel, high-carbon wire steel, and abrasion plate steel research at the ASPPRC. He joined the Colorado School of Mines in 2008 as a postdoctoral research associate and has held his current position since 2010. He holds Ph.D., M.S., and B.S. degrees from the Ghent University, Belgium.

Vanadium and Niobium Microalloying to Increase Strength of High Carbon Wire Steels

S. L. Miller and E. De Moor

Advanced Steel Processing and Products Research Center
Colorado School of Mines

Abstract

The effect of increased N and Nb additions on the strength of V-microalloyed eutectoid steels was investigated using laboratory heats and continuous cooling experiments conducted using a Gleeble® 3500 thermomechanical simulator. Continuous cooling was performed from industrial austenitizing (1093 °C) and upper end laying head (950 °C) temperatures at rates ranging from 2.5-12.5 °C/s. Metallography, Vickers hardness, pearlite colony size and pearlite interlamellar spacing (ILS) measurements were used to examine the effects of these treatments. Both Nb and N additions were found to increase strength in V-microalloyed steels. Nitrogen additions were observed to decrease hardenability. Niobium additions were found to refine colony size and ILS, as well as increase hardenability. A model based on strengthening contributions of elements in solid solution, pearlite colony size, and pearlite ILS was used to investigate precipitation strengthening. Calculated versus observed hardness was compared for the microalloyed steels. N additions resulted in maximum hardness improvement, and a lesser effect was observed with Nb additions.

Introduction

High-carbon wire steels are used in a variety of applications including tire reinforcement, oil rig mooring lines, concrete reinforcement, suspension bridge cables, etc. [1–3]. The desirable combination of high strength and toughness in these alloys originates from optimized microstructures obtained through judicious alloying and thermal processing, in particular controlled cooling from the laying head temperature. Rapid cooling can produce bainite or martensite, both of which lack the necessary ductility for wire drawing, whereas lower cooling rates may produce coarse pearlite, typically resulting in lower strength. Pearlite features, such as interlamellar spacing (ILS) and colony size, dictate final properties of the wire rod.

Tensile strength of eutectoid steels can be predicted based on four major contributors: solid solution strengthening, cementite strengthening which directly correlates with cementite volume fraction, pearlite colony size, and pearlite ILS. Yield strength predictions of medium and high carbon steels have been found to follow the relationship [4]:

$$\sigma_{YS} = \sigma_{SS} + 460L^{-\frac{1}{2}} + 145(\sqrt{2}\lambda)^{-\frac{1}{2}} \tag{1}$$

where σ_{YS} is the yield strength in MPa, σ_{SS} is a term representing the combination of solid solution and cementite volume strengthening, L is colony size in μm, and λ is ILS in μm [4, 5]. A good correlation has been observed between yield strength calculated using this expression and experimental values for plain carbon steels with carbon contents ranging from 0.75-1.8 pct C [4]. As indicated by Equation (1), colony size and ILS follow an inverse square root Hall-Petch type relationship to yield strength; therefore maximum strength can be achieved by refinement of both quantities. It should also be noted that ILS is generally several orders of magnitude less than colony size, and is hence a much stronger contributor to strength.

ILS refinement is being employed extensively to improve strength levels, with measurements on the order of 10 nm for the highest tensile strength drawn wire [6]. Solution strengthening and volume fraction of cementite can be maximized by using the highest possible carbon level of ~0.8 wt pct C, beyond which there is a risk of forming brittle grain boundary cementite which limits drawability. Han *et al*. have shown that Si alloying can suppress grain boundary cementite up to 0.95 wt pct C, but the high C levels may lead to detrimental segregation in cast billets [7]. Microalloying has the potential to further increase the strength of eutectoid steels by precipitation strengthening and microstructural refinement [8, 9].

Precipitation strengthening occurs when precipitates inhibit dislocation motion through the matrix. Precipitates that contribute to strengthening are expected to follow an Ashby-Orowan relationship, with the yield strength contribution of precipitation, $\Delta\sigma_{YS}$, scaling as the square root of the volume fraction of particles, f, and being inversely related to particle diameter, X. The general equation for this contribution is given by [10]:

$$\Delta\sigma_{YS} = \frac{6Gb\sqrt{\frac{3f}{2\pi}}}{X} \tag{2}$$

where G is the bulk modulus of elasticity in shear and b is the Burgers vector. According to this relationship, maximum strengthening can be achieved by a high density of fine particles.

In order to obtain optimal precipitate sizes and distributions for maximum strengthening, full precipitate dissolution during billet reheating is desired and precipitate coarsening during subsequent processing is to be avoided. Whether or not microalloy precipitates will dissolve at a given temperature during reheating is governed by solubility. Solubility is a measure of the equilibrium thermodynamic stability of microalloy precipitates, and the solubility product, k_s, for the reaction:

$$[M] + [X] = MX \tag{3}$$

is defined by the activity ratio of reactants over products, which can be approximated as the product of the weight percent compositions of each constituent:

$$k_s = \frac{a_{[M]}a_{[X]}}{a_{[MX]}} \approx [M][X] \quad (4)$$

Table 1 lists solubility equations used to create solubility curves for V and Nb microalloy carbides and nitrides. When plotted on a graph with each axis representing the respective reacting species in wt pct, isothermal solubility curves such as the ones shown in Figure 2 are obtained. To the left side and below the curves, the species are in solution in austenite, and to the right side and above the line the precipitate reaction is thermodynamically favorable [10]. Solubility predictions based on these curves were incorporated into the alloy design for this study, with the goal of maximizing precipitation strengthening using industrial reheating and laying head temperatures.

Table 1 – Microalloy Solubility Equations [10–12]

Compound	Solubility Equation (in Austenite)	Source
VC	-9500/T + 6.72	Narita, 1975
$VC_{0.75}$	-6560/T + 4.45	Turkdogan, 1989
VN	-8330/T + 3.46	Irvine, 1967
NbC	-7900/T +3.42	Narita, 1975
$NbC_{0.87}$	-6770/T + 1.03	Turkdogan, 1989
NbN	-8500/T + 2.80	Narita, 1975

Studies have shown improved mechanical properties of eutectoid and hypereutectoid wire steels with V additions due to precipitation strengthening [7, 8, 13]. The increase of tensile strength by V additions in eutectoid steels has been reported as 1.4-1.6 ksi (9.6-11.0 MPa) per 0.01 wt pct V in the presence of 80 ppm N, comparable to the composition of the V+N alloy used in this study [8]. The high solubility of V relative to other microalloying elements allows for precipitate dissolution at industrial reheating temperatures resulting in V being present in solid solution in austenite until final thermomechanical processing steps, and may lead to small, finely distributed precipitates with significant precipitation strengthening potential. Nitrogen additions in V microalloyed steels have proved effective in increasing strength by promoting V(C,N) precipitation with improved resistance to coarsening in low carbon steels. In addition V ties up interstitial, free N that may cause embrittlement or aging effects [9, 14–16]. Nb has significantly lower austenite solubility than V, and therefore may be less effective for precipitation strengthening. However, Nb in solid solution and in precipitate form is known to retard austenite recrystallization and grain growth, which may affect pearlite characteristics and final wire rod properties [17]. Limited data are available for alloying effects on the properties of V-microalloyed eutectoid steels, particularly with Nb additions and varied N contents, which provided the impetus for the current study.

Experimental Procedure

Three microalloyed steels with compositions shown in Table 2 were designed with the goal of maximizing strength. Carbon levels were designed to be 0.8 wt pct to maximize volume percent of cementite, while avoiding formation of grain boundary cementite [7]. Two V-microalloyed alloys labeled V and V+N were proposed, one with a typical N content of an industrial electric arc furnace material (target of ~60 ppm) and one with additional N (target of ~120 ppm). As shown in Table 2, the measured N levels in the lab heats were lower than designed, at 60 ppm and 90 ppm respectively. A V content of 0.08 wt pct was chosen to be in excess of the stoichiometric V:N ratio of 4:1 to achieve maximum precipitation while tying up free N. The Nb level in the V+Nb steel was chosen based on the maximum amount of Nb that could be dissolved in austenite at 1093 °C. Manganese, silicon, and chromium levels were consistent with previous studies [7, 8].

Table 2 - Compositions of Experimental Test Alloys in wt pct

	C	Mn	Si	Cr	Nb	V	Al	N	S	P
V	0.80	0.50	0.24	0.20	-	0.079	0.005	0.0059	0.004	0.004
V+N	0.80	0.49	0.24	0.20	-	0.079	0.003	0.0088	0.005	0.004
V+Nb	0.80	0.49	0.24	0.20	0.010	0.079	0.004	0.0059	0.004	0.004

Lab heat castings were assessed for carbon segregation by macroetching and carbon determination, and sectioned such that carbon levels were within ±0.02 wt pct of the nominal level of 0.80 wt pct. Hot rolling of the sections consisted of a reheating ramp to 1200 °C over approximately 2 hr, a 20 min soak, a 6-pass deformation schedule with a 15 min reheat after the third reduction, straightening, and air cooling. Approximately 20 pct reduction per pass was achieved, and the overall reduction ratio was 3:1. Round samples 5.5 mm (0.22 in) in diameter and 72 mm (2.8 in) long were machined from the hot rolled plates.

Continuous cooling tests were designed with parameters based on industrial processing temperatures as well as solubility calculations for the microalloy precipitates, with a thermal processing schematic shown in Figure 1. The tests were designed to provide insight into microstructural evolution during continuous cooling at different rates from an intermediate hold temperature. The initial soak temperature was chosen at 1093 °C (~2000 °F) in accordance with industrial billet reheat temperatures for wire rod rolling, and the intermediate hold temperature of 950 °C (~1742 °F) was selected at the upper end of the range of industrial wire rod laying head temperatures [18].

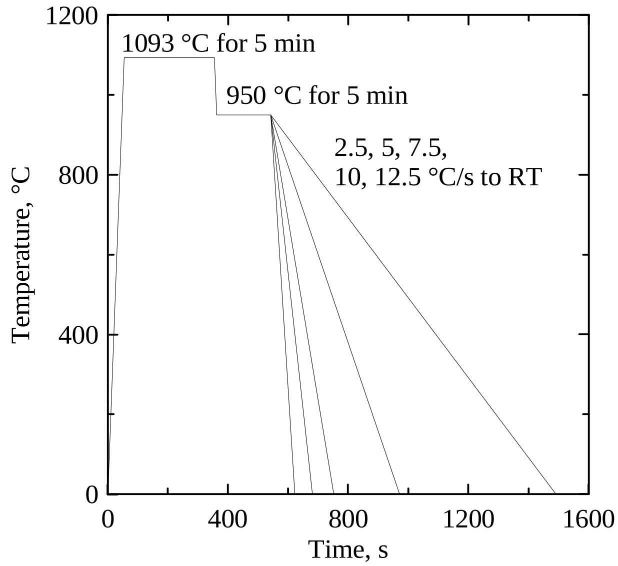


Figure 1 Thermal profile schematic consisting of an austenitizing step at 1093 °C for 5 min, followed by an intermediate hold at 950 °C for 5 min before continuous cooling to room temperature at constant rates of 2.5, 5, 7.5, 10, and 12.5 °C/s.

Microalloying element solubility was examined at these temperatures, and Table 2 lists the equations used to create solubility curves for microalloy carbides and nitrides shown in Figure 2 for temperatures of 950 °C and 1093 °C where applicable. Solubility calculations predict that the only microalloy precipitates to remain undissolved at the reheat temperature of 1093 °C are NbC as shown in Figure 2 (d). For each of the three microalloyed steels, VN, VC, and NbN remain dissolved at 950 °C as shown in Figure 2 (a) to Figure 2 (c). Thus precipitates are expected to form during cooling from 950 °C, which should promote fine dispersions of V(C,N) precipitates that may contribute to improved mechanical properties.

Testing was conducted using a Gleeble® 3500 thermomechanical simulator with a low-force setup under high vacuum conditions (< 10^{-3} torr). Microstructural analysis was performed for samples continuously cooled at rates of 2.5-12.5 °C/s. Samples were sectioned at the thermocouple wires, mounted in bakelite and metallographically prepared by grinding and polishing with diamond suspension down to 1 μm. Vickers hardness testing was conducted according to ASTM E-92 using a grid of nine measurements per sample centered along a bisecting line at one quarter of the sample diameter [19]. Following hardness testing, a 6 sec etch of 4 pct Picral was applied to the polished surfaces and a single hardness indent at one quarter of the sample diameter was used as reference mark for Scanning Electron Microscopy (SEM).

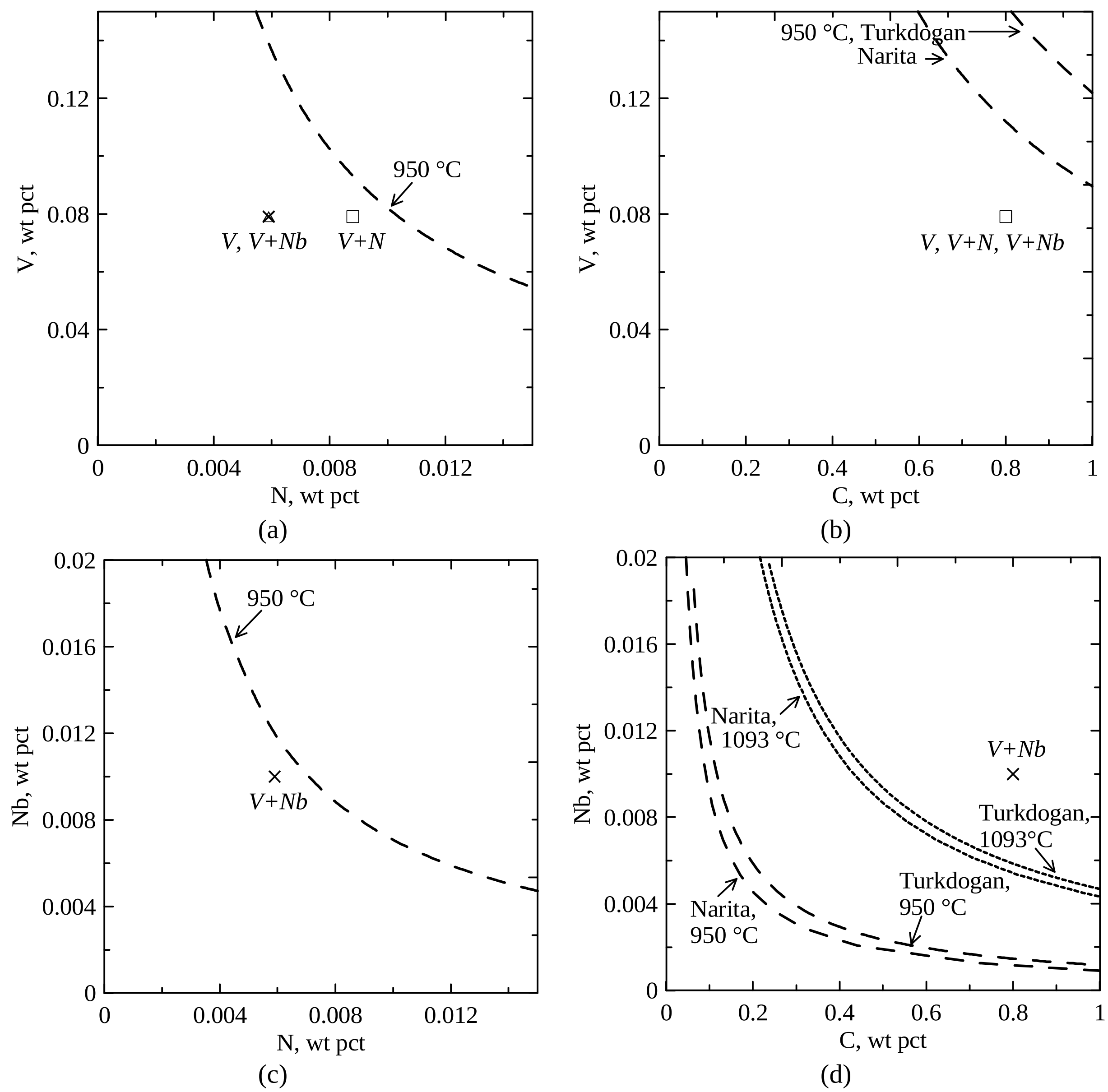


Figure 2 Solubility diagrams for (a) VN, (b) $VC/VC_{0.75}$, (c) NbN, and (d) $NbC/NbC_{0.87}$ precipitates in austenite at indicated temperatures of 950 °C and 1093 °C according to the equations presented in Table 1 [10–12].

A JEOL 7000E Field Emission Scanning Electron Microscope (FESEM) was used for microstructural characterization, including qualitative identification of constituent phases, and measurement of pearlite colony size and interlamellar spacing (ILS). A colony was defined as a region with parallel oriented cementite lamellae and colony size was measured using a circular intercept method according to ASTM E-112, at an original magnification of 5,000x as shown in Figure 3(a) [20]. Reported values for each sample represent an average of nine measurements taken from each of four different fields of view. Interlamellar spacing was also measured using the circular intercept method as a way of approximating true interlamellar spacing, at a magnification of 10,000x as shown in Figure 3(b) [21]. Reported values represent an average of nine measurements per image taken from four different fields of view. Approximate count numbers per reported average were 200 for colony size, and 1,000 for ILS and error bars reported for all plots represent the standard error of these data sets.

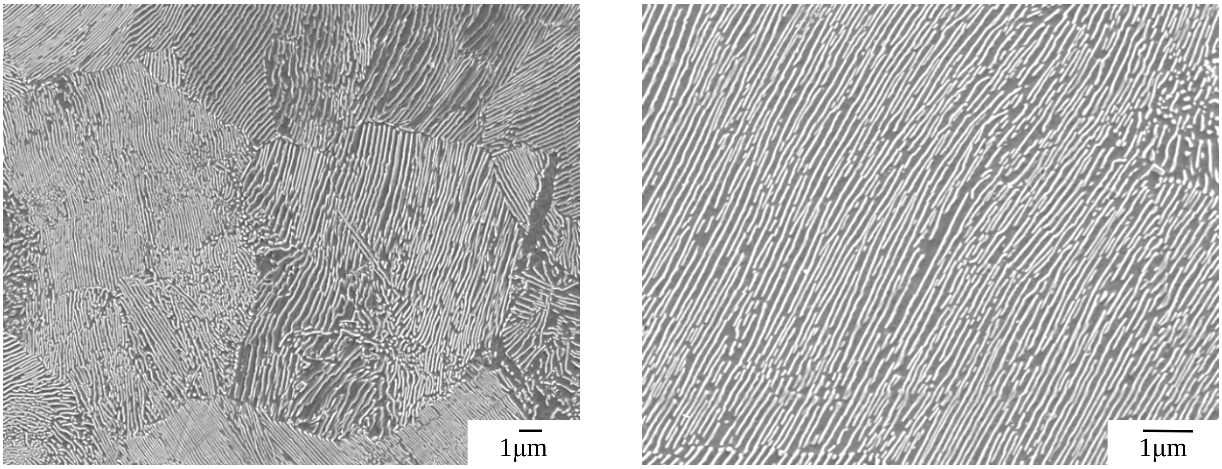


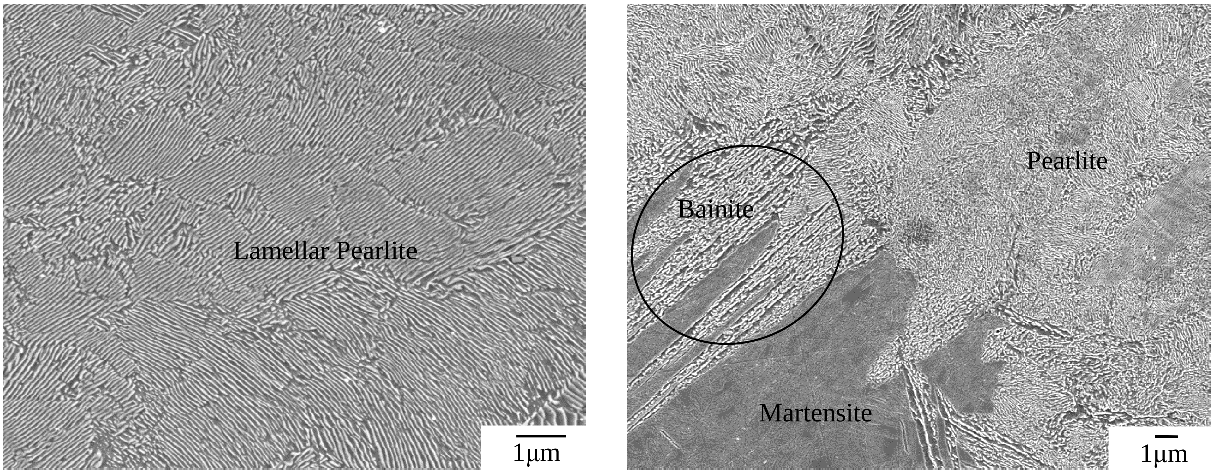
Figure 3 Representative FESEM images used to measure (a) pearlite colony size and (b) pearlite interlamellar spacing.

Results and Discussion

Microstructural Features

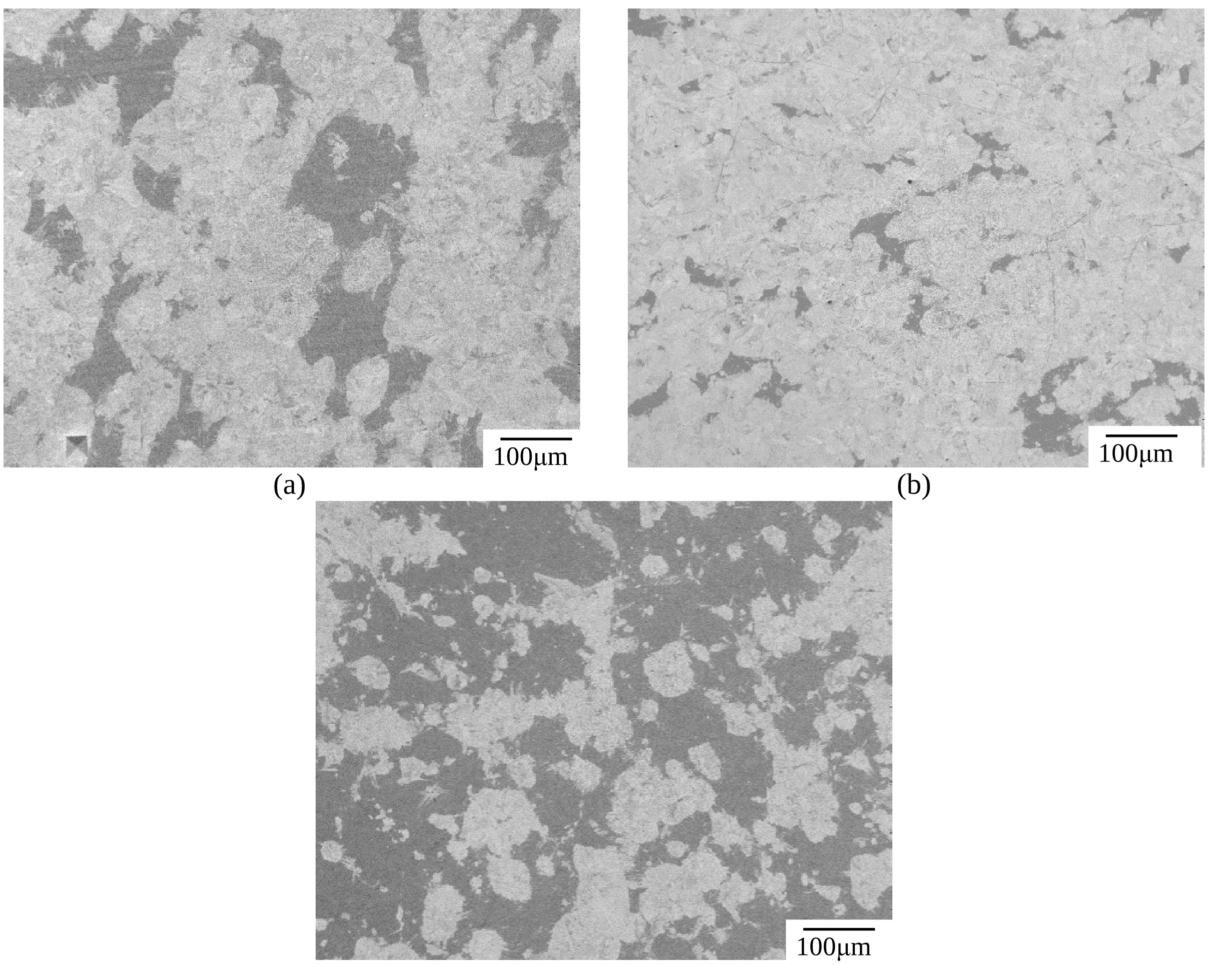
Metallography was conducted to identify microstructural constituents present after continuous cooling at various cooling rates. Figure 4 shows representative FESEM images of the V steel samples cooled from 950 °C. Controlled cooling at a constant rate of 2.5 °C/s produced a fully pearlitic structure, absent of martensite or bainite as shown in Figure 4 (a). Slow cooling rates produced lamellar pearlite in all alloys, and as cooling rate increased so did the propensity for formation of other transformation products such as bainite and martensite. Figure 4 (b) shows an image for a sample cooled at 12.5 °C/s where bainitic and martensitic regions are clearly visible.

Alloying effects on hardenability were qualitatively observed. Figure 5 shows low magnification FESEM images of samples continuously cooled at 12.5 °C directly from 1093 °C for the V, V+N, and V+Nb steels. Varying levels of martensite characterized by the dark gray etching response are observed. For a single cooling condition, the V+Nb steel showed the most martensite, followed by the V, and V+N steels. This suggests reduced hardenability in the V+N steel and a delayed or slower pearlite transformation in the V+Nb steel. Continuous cooling transformation (CCT) curves generated for these alloys agree with these hardenability observations, but are beyond the scope of this paper [22].



(a) (b)

Figure 4 Representative images of a (a) fully pearlitic and (b) mixed microstructure. Image (a) taken from the V steel cooled from 950 °C, at a rate of 2.5 °C/s and (b) at 12.5 °C/s. Samples etched with 4 pct Picral.



(c)

Figure 5 Low magnification FESEM taken from samples continuously cooled from 1093 °C at 12.5 °C/s for (a) V, (b) V+N, and (c) V+Nb steels. Samples etched with 4 pct Picral.

Vickers Hardness and Quantitative Metallography

Figure 6 shows the effects of alloying content and cooling rate on microhardness measurements for the samples exhibiting fully pearlitic microstructures. All steels show greater hardness with increased cooling rate. The V+N steel shows consistently greater hardness than the other microalloyed steels, except at a cooling rate of 2.5 °C/s. The V+Nb steel data are limited due to formation of bainite and martensite at cooling rates greater than 5 °C/s. A hardness comparable to the V steel was obtained for a cooling rate of 2.5 °C/s, whereas cooling at 5 °C/s resulted in intermediate hardness compared to the two other steels.

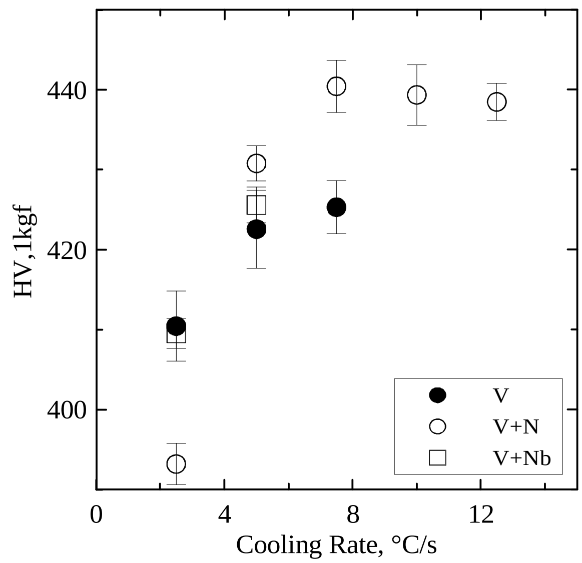


Figure 6 Microalloying effects on Vickers hardness for samples continuously cooled at different rates from 950 °C.

Quantitative metallography included pearlite colony size and pearlite ILS measurements. The strengthening model presented earlier indicates that ILS should be the primary contributor to strength, and hardness trends should hence correlate to ILS. Figure 7 (a) presents the effect of cooling rate on pearlite ILS. In general, a decrease in ILS was observed with increased cooling rate. The V+Nb steel showed the greatest ILS refinement, especially at a cooling rate of 5 °C/s which may help explain the increased hardness observed compared to the V steel. The V and V+N steels exhibit similar or greater hardness levels despite larger microstructural features, implying other contributing strengthening mechanisms. Although the increased N level resulted in greater hardness levels in general, ILS for the V+N steel was found to be larger in most cases compared to the V steel.

Figure 7 (b) shows the effect of cooling rate on average pearlite colony size, with smaller colony sizes observed in samples cooled at faster rates. The V+N steel exhibits the smallest colonies. The effect of the Nb additions appears to be strongly dependent on cooling rate with the greatest cooling rate of

7.5 °C/s resulting in the most refinement compared to the V steel, whereas similar or greater colonies are observed at slower cooling rates. It should be noted that colony size is considered to play a secondary role in strengthening.

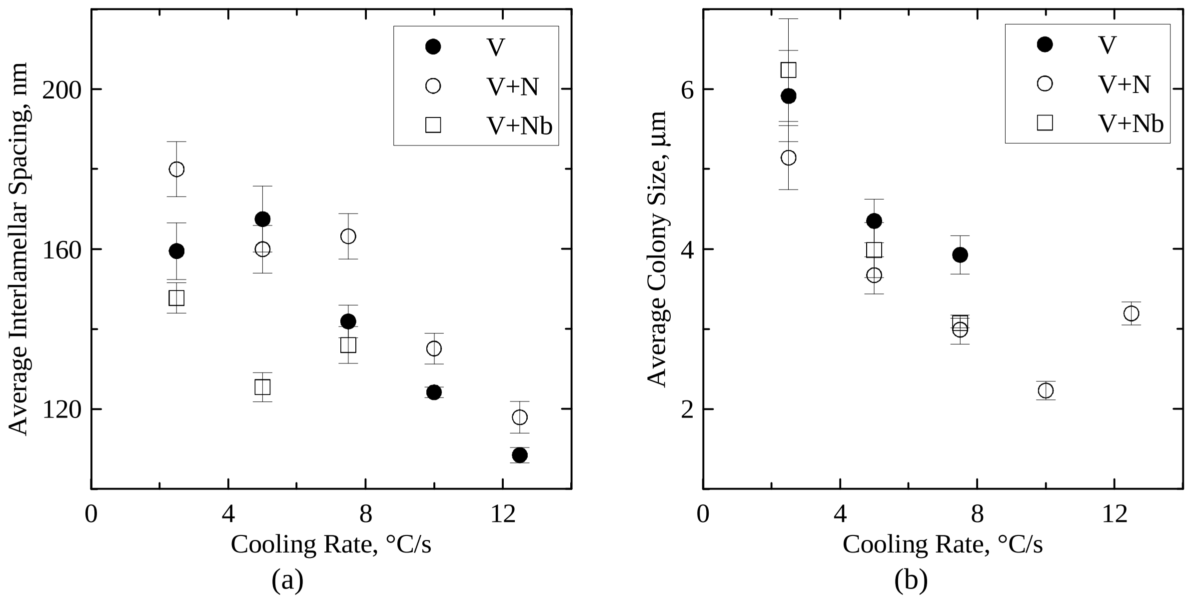


Figure 7 Microalloying effects on (a) pearlite interlamellar spacing and (b) pearlite colony size measurements as a function of cooling rate for samples continuously cooled from 950 °C.

Microalloying Contribution to Hardness

While hardness measurements provide a direct strength comparison of the microalloyed steels, an effort was made to separate strength contributions of the microalloying precipitates from anticipated strengthing of the observed microstructural features using the model proposed by Taleff [4]. The model correlates pearlite colony size and ILS directly to yield strength according to Equation (1). The steels used to derive Equation (1) were not microalloyed, and it is reasonable to assume that deviation from the values predicted in the equation, or deviation from a 1:1 line, may reflect contributions from microalloying elements. The $\Delta\sigma_{SS}$ term was assumed to be 170 MPa from linear regression analysis of steel in a similar composition range, specifically 0.78-0.89 wt pct C, 0.88-1.56 wt pct Mn, 0.25-0.10 wt pct Si [5]. Yield strength values were converted to Vickers hardness measurements according to [23]:

$$\sigma_{YS} = -90.7 + 2.876(HV) \tag{5}$$

where σ_{YS} is yield strength in MPa and HV is Vickers hardness at 1 kgf. This expression was obtained from a regression analysis conducted by Pavlina *et al.* using the experimental data of over 150 hypoeutectoid steels with yield strengths ranging from of 300 to1,700 MPa [23].

Figure 8 shows the results of the Taleff analysis for samples cooled from 950 °C on a measured versus calculated hardness plot. Measured hardness values were significantly larger than calculated hardness values for all alloys. It is reasonable to assume that V effects, including precipitation strengthening, occurred in all of the alloys, and that differences in the microalloyed steel data reflect the contributions of Nb and N. Comparing the V steel data to the V+N and V+Nb steel data, both Nb and increased N improved strength. The V+N steel showed the greatest strength improvements compared to the V steel, with a maximum hardness improvement of $\Delta HV_{V+N} \approx 27$ as indicated by the arrow in Figure 8. The magnitude of that improvement translates to a yield strength increase of approximately 78 MPa according to Equation (5). This implies enhanced V strengthening effects with a higher N content, consistent with the tendency of N to promote fine precipitates that resist coarsening. A peak in hardness observed for the V+N steel implies a range of microstructural feature sizes where precipitation strengthening was most beneficial. It should be noted that in each of these alloys, a portion of the bulk vanadium content may have precipitated in austenite or remained in solid solution in ferrite or cementite, reducing the volume fraction available for precipitation strengthening [24]. Therefore reported microalloy strengthening contributions may not reflect maximum achievable values. Furthermore, industrial Stelmor® deck cooling may allow for more precipitation compared to continuous cooling, in particular at high cooling rates.

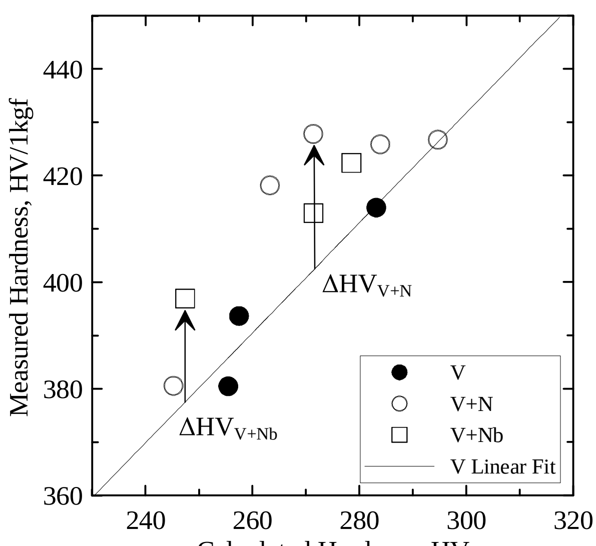


Figure 8 Comparison of measured hardness values to those predicted by Equation (1) using experimental pearlite colony size and ILS data from samples cooled at 950 °C [4]. The ΔHV terms indicate maximum hardness improvements observed for the microalloyed steels compared to the V steel data.

The V+Nb steel data showed consistent hardness improvement over the V steel values, but lesser in magnitude. The largest improvement indicated by $\Delta HV_{V+Nb} \approx 19$ HV translates to a yield strength improvement of approximately 55 MPa according to Equation (5). The enhanced hardenability and greater microstructural feature refinement observed for the V+Nb steel suggest that the increase in strength may be due to a delay in the kinetics of the pearlite transformation due to Nb additions. A study of the effects of deformation in the austenitic temperature region prior to pearlite transformation is a fruitful area of further work, in conjunction with thermal processing using Stelmor® deck cooling profiles. Nb in particular may refine prior austenite grain sizes which may influence the pearlite transformation, and the Stelmor® cooling profile will reduce the presence of martensitic and bainitic constituents.

Conclusions

In order to investigate the effect of V, Nb, and N additions on the strength of eutectoid steels, three lab heat castings were produced. Continuous cooling experiments were conducted incorporating an industrial austenitizing (1093 °C) and upper end laying head (950 °C) temperature. Metallography, Vickers hardness, pearlite colony size and pearlite ILS measurements were used to examine the effects of these treatments. Nb additions to a V alloyed eutectoid steel resulted in increased hardenability and greater refinement of microstructural features. Increased N levels were found to decrease hardenability. Additions of Nb and N were shown to increase hardness of V alloyed eutectoid steels.

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