Microalloy Precipitation in Hot Charged Slabs

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ABSTRACT
The effects of microalloy precipitation and (tunnel furnace) dissolution during direct slab production are explored relative to the position within a slab (i.e. thermal profile of the slab) and alloy content. Niobium solute contents and precipitate fractions are quantified using electrochemical extraction and inductively coupled plasma atomic emission spectrometry (ICP-AES) techniques. The locations selected for testing experimental CMn(Nb) steels incorporated the influence of temperature differences between different locations within the slab, differing solidification rates, and alloy segregation. The results show that the greatest amount of alloy precipitation occurred at the slab surface measured along the edges of the continuously cast thin slab. The extent of precipitation appeared greatest in the high niobium steel, where dissolution subsequently occurred during reheating and equalization in the tunnel furnace. There does not appear to be substantial precipitation or dissolution in the tunnel furnace for the low and medium niobium steels. The columnar region represents the bulk of the slab volume and exhibited the lowest amount of precipitated niobium. The precipitation and dissolution behaviors were generally consistent with expectations based on solubility considerations related to applicable thermal and compositional variations.

INTRODUCTION

HSLA steels are used in a range of applications including the construction, automotive, and pipeline industries. They are defined by small amounts of alloying additions such as niobium, titanium, and vanadium, at carbon levels typically less than 0.1 wt%. The alloying additions are usually added in amounts less than 0.1 wt%, known as microalloying. Micoralloying can improve mechanical properties of HSLA steel by the formation of precipitated carbides, nitrides, and carbonitrides. Precipitate size, distribution, volume fraction, and type are all prominent factors in determining engineering performance. The precipitates retard and/or prevent austenite recrystallization and encourage precipitation hardening in the final matrix. Microalloy additions retard austenite recrystallization at low finish temperatures during hot-rolling resulting in a substantially fine-grained transformation microstructure[1].

Niobium, used in amounts ranging from 0.01 to 0.10 (wt%) is usually an important component in hot-rolled linepipe steels[2]. Many studies have been completed regarding the understanding of niobium precipitation during hot-rolling deformation relative to austenite recrystallization and the dissolution kinetics of niobium during reheat processes[1-14]. In-depth research has been carried out to better understand the effects of microalloy precipitation on the mechanical properties of high strength microalloyed steels relative to niobium content and thermomechanical processing practices, where niobium is a strong grain refiner and precipitation strengthener[5]. However, studies of niobium precipitation during solidification and processing prior to hot-rolling are limited.

This research is directed towards the understanding of niobium precipitation prior to hot-rolling relative to alloy content and solidification during thin slab processing of HSLA steels. The testing of steels with low, medium, and high concentrations of niobium in conjunction with variations in sampling location (i.e. base of the caster and post tunnel furnace) will assist in understanding and optimizing niobium additions and processing for HSLA steels. Increasing niobium additions could promote premature precipitation prior to hot-rolling by increasing the driving force for precipitate nucleation and growth to occur. This would be an inefficient use of alloy additions, and thus it is necessary to better understand how compositions and processing parameters influence niobium precipitation prior to hot-rolling. Testing is specifically associated with thin slabs obtained by way of the CSP process. However, the results should be of interest in the broader context of hot-charged HSLA steels.

EXPERIMENTAL PROCEDURE

The material used for microalloy precipitation studies was provided by Nucor Steel-Hickman. Electrochemical extraction techniques were used throughout this study to quantify the amount of alloy in solution and precipitate form. Preliminary testing involved a high strength vanadium-niobium grade which was used to determine if adequate quenching occurred during sample collection. Subsequent to the quenching experiment, trial heats with low, medium, and high niobium additions were prepared. The niobium trial heats were designed to study the effects of microalloy precipitation relative to alloy content, thermal profile of the slab, and process location prior to hot-rolling. Prior to niobium precipitation studies, reproducibility testing of the medium niobium trial grade was carried out to ensure that the electrochemical extraction procedure provided accurate and repeatable results.

Electrochemical Precipitate Extraction

Quantification of microalloy concentrations in solution and in precipitate form is critical to this work. Due to the dilute alloy concentrations in HSLA steels, conventional techniques have difficulty measuring the amount of microalloy in solution. Electrochemical extraction techniques can be used to quantify the portions of microalloying addition dissolved in the steel matrix and in precipitate form in order to better understand niobium precipitation behavior. Previous studies designed a suitable electrochemical extraction technique that was used for this research and further detail is provided elsewhere[7, 13, 14, 15].

The steel specimen is weighed prior to electrochemical extraction and subsequent testing. The sample is adequately filtered, separating the dissolved steel matrix from alloy precipitates. The precipitate residue and filter papers are dissolved in a second solution prior to chemical analysis. After chemical extraction and precipitate dissolution, each solution is diluted with deionized water prior to Induction Coupled Plasma Atomic-Emission Spectrometry (ICP-AES). The dissolved steel matrix solution is diluted to 500 mL and the dissolved precipitate solution is diluted to 100 mL. ICP-AES measurements are recorded in (liquid) concentration (mg/L). To calculate the amount of niobium measured by ICP-AES, it is necessary to multiply the concentration by the volume of the solution being analyzed:

$$Nb \ in \ solute\left(\frac{mg}{L}\right) \times 0.500L \tag{1}$$

$$Nb \ in \ precipitate\left(\frac{mg}{L}\right) \times 0.100L \tag{2}$$

The sum of the two niobium amounts in Equations 1 and 2 gives the total niobium measured, in milligrams, using ICP-AES analysis. The total value is divided by the measured mass extracted from the sample during electrochemical extraction, giving the total amount

of alloy found by ICP-AES in weight percent. The chemical analysis completed by ICP-AES (in wt %) can be compared to the reported alloy content given by the steel producer. This comparison could be represented as a percentage difference:

$$\left(\frac{ICP\ Total\ Nb(wt\%) - Nb\ reported(wt\%)}{Nb\ reported(wt\%)} \right) \times 100 \tag{3}$$

With respect to Equation 3, a negative percentage difference indicates that ICP-AES measured less alloy than expected, potentially demonstrating alloy depletion. In turn, a positive percentage difference indicates that the ICP-AES measurement is greater than the expected amount of alloy indicating a potentially alloy rich region.

Preliminary Quenching Experiment

A CMn(VNb) slab was cropped at the base of the caster. The steel contained medium niobium and vanadium levels of 0.04 and 0.047 (wt%), respectively. This steel was specifically chosen due to its vanadium alloying addition. Since vanadium is expected to remain in solution during casting, quenching of the slab crop was to be considered satisfactory if all vanadium remained in solution. In order to verify the sample collection and quenching techniques before additional trial heats were prepared, the amount of vanadium in solution and in precipitate form was quantified using electrochemical extraction. The chemical composition of the CMn(VNb) steel is shown in Table I. Each slab was approximately 1500 x 700 x 50 mm (60 x 28 x 2 inches) when cropped. The cropped slabs were quenched in water with constant agitation after shear cutting. Figure 1(a) illustrates the final dimensions of the cropped section. The 127 mm (5 in) long by 1500 mm (60 in) wide section was cut following sample collection to allow for easier handling. The 127 mm (5 in) section was then cut into five separate pieces with dimensions 300 x 127 x 50 mm (12 x 5 x 2 inches) as shown in Figure 1(b). The sample locations for preliminary tests are shown in Figure 1(c). Three separate locations within the slab were examined. The circled section (Figure 1(b)) was used to obtain the three sampling locations. Locations A, B, and C were tested from the base of the caster 305 mm (12 inches) from the edge.

Table I. Chemical Composition of CMn(VNb) Steel (wt %) for Quenching Trial

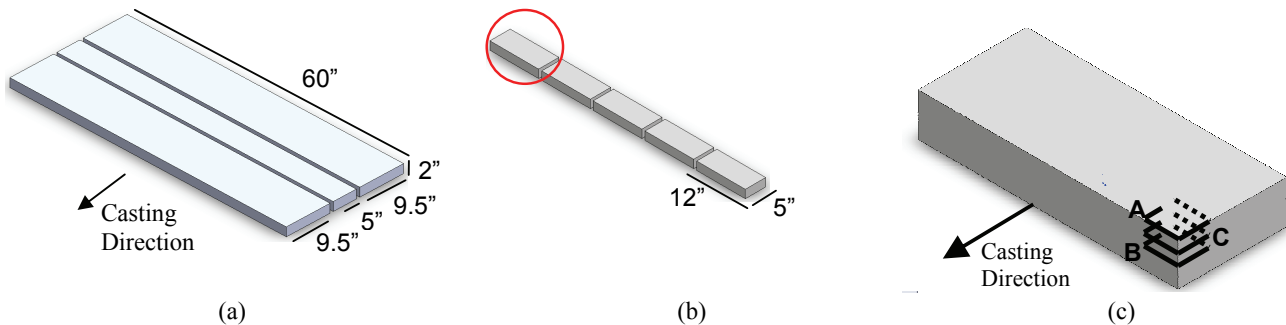|	C	Si	Mn	P	S	V	Nb	N
Med Nb	0.040	0.204	1.265	0.011	0.001	0.047	0.040	0.007

Figure 1. (a) Initial slab dimensions (in inches) after cropping at the base of the caster. (b) Delivered material and dimensions. (c) Sample locations for preliminary testing.

The specified locations of samples A, B, and C were chosen to evaluate microalloy precipitation trends relative to the thermal profile of the slab (i.e. different solidification regimes prior to hot rolling). Sample A is on the surface of the thin cast slab, B represents the centerline of the slab, while C encompasses the columnar region of the cast slab approximately 13 mm (0.5 inches) below the top surface. The preliminary tests were particularly intended to determine whether or not the method for sample collection during continuous casting was adequate.

The results from preliminary testing are shown in Table II. Niobium precipitation was not the focus of the quenching trial and only the vanadium behavior is reported for this steel. The position of each sample relative to casting direction and specimen location within the slab is shown in Figure 1. One sample was tested at each location. From ICP-AES, it was concluded that essentially all of the vanadium remained in the steel matrix for the cast specimens, and did not prematurely precipitate upon sample collection during the quenching process. Thus, the quenching method used for sample collection was considered suitable.

Table II. Vanadium Measured in Solution and Precipitate Form in CMn(VNb) - Quenching Trial

Sample	Precipitate (wt% V)	Solution (wt% V)	Total (wt% V)	Percentage of V precipitated	Percentage of V in soluiton	Reported Alloy Content (wt% V)	Precentage Difference
Caster A	0.000	0.023	0.023	0	100	0.047	-51
Caster B	0.000	0.062	0.062	0	100	0.047	32
Caster C	0.000	0.039	0.040	0	100	0.047	-16

Material for Niobium Precipitation Studies

Research trial heats with low, medium, and high niobium additions and similar levels of carbon, manganese, silicon, aluminum, and nitrogen were prepared after validating the quenching method with the CMn(VNb) steel. The steels were designed to evaluate the extent of niobium precipitation prior to entry into the hot rolling mill relative to position within the slab (i.e. thermal profile of the slab) and alloy content. The three steels were cast sequentially, to ensure that processing parameters at the time of casting were equivalent. For each steel grade, samples were taken from both the base of the caster and prior to entry into the rolling mill. The sample collection method designed for the quenching trials was also used for the sample collection of the research trial steels. Slab dimensions were approximately 1500 x 700 x 50 mm (60 x 28 x 2 inches) when cropped. The surface temperature of the thin slab at the base of the caster was approximately 800 ºC and prior to hot rolling at the furnace exit was approximately 1050 ºC. The chemical compositions of the three CMn(Nb) steels are shown in Table III. Equilibrium Nb(C,N) precipitate fractions are shown in Figure 2 as a function of temperature, calculated using the method of K. Xu et al.[16] In the context of niobium precipitation, the influence of

Table III. Chemical Compositions of CMn(Nb) Steels (wt %)

Material	C	Mn	Si	Al	Nb	P	S	N
Low Nb	0.035	0.937	0.152	0.027	0.014	0.011	0.002	0.007
Med Nb	0.036	1.065	0.184	0.029	0.030	0.009	0.002	0.008
High Nb	0.031	1.039	0.194	0.031	0.046	0.012	0.003	0.006
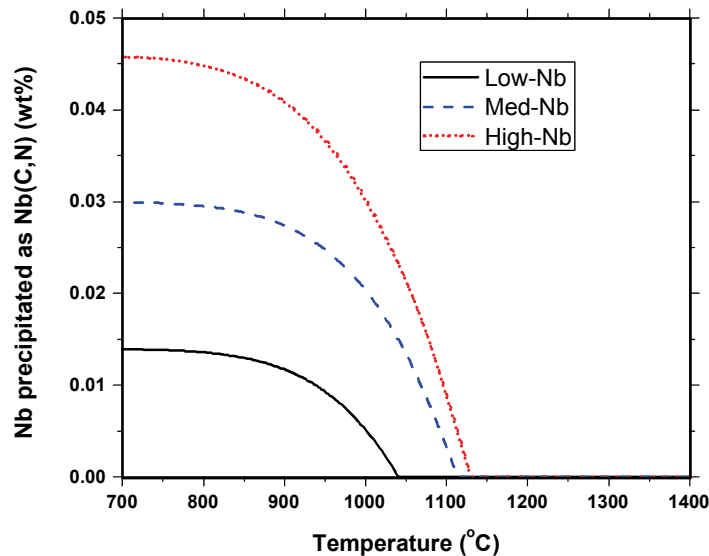
Figure 2. Calculated wt% Nb precipitated as a function of temperature for low, medium, and high niobium additions[16].

temperature differences between different locations within the slab and differing solidification rates are of special interest. Effects of alloy composition and temperatures relative to secondary cooling in the caster constitute the focus of this work. Different areas within a continuously cast slab are studied and identified with respect to niobium precipitation. These areas include the chill zone, columnar zone, and central equiaxed zone. During thin slab casting, the first solid forms along the mold wall just below the mold perimeter and acts as a shell. From the newly solidified shell, solidification continues perpendicular to the solid outer shell. Over time, the shell thickness increases as the solidification rate decreases within the slab. This decrease in solidification rate is caused by the outer shell acting as a barrier to heat flow from the core. Brimacombe has described the characteristics of different solidification regions and locations of special interest relative to continuous casting of thin slabs[17]. A schematic illustration demonstrating the different solidification regions and sample orientations in each region is shown in Figure 3. The chill zone of the slab results from fast solidification and steep thermal gradients experienced at the slab surface. The columnar zone consists of directional growth of crystals

from the outer shell perpendicularly inward toward the equiaxed zone. The equiaxed zone is the solidification region located in the middle of the slab where equiaxed crystal growth occurs. It is a result of independent solid crystals that grow within remaining liquid in the core of the slab as the thickness of the shell increases and cooling rate decreases. The presence and distribution of the different regions is influenced by casting conditions. If the casting speed and slab thickness are appropriate, the central equiaxed region may be negligible.

Due to solute partitioning during solidification, the last liquid within the center of the slab is expected to be alloy rich. The high alloy content can create a region of centerline segregation. This alloy rich region may also cause an inhomogeneous microstructure. The separate locations selected for testing the experimental CMn(Nb) steels incorporate the influence of temperature differences between different locations within the slab, differing solidification rates and alloy segregation. The sample locations are shown in Figure 4. Sample location A was near the slab corner and was chosen to observe the extent of microalloy precipitation on the edges of the slab. Location B is within the centerline region. This location will help identify the alloy concentration that remains in the last liquid as well as the associated degree of alloy precipitation. Sample location C is within the columnar region. A comparison of the results between samples A, B, and C addresses the influence of cooling rate and temperature differences between different locations within the slab.
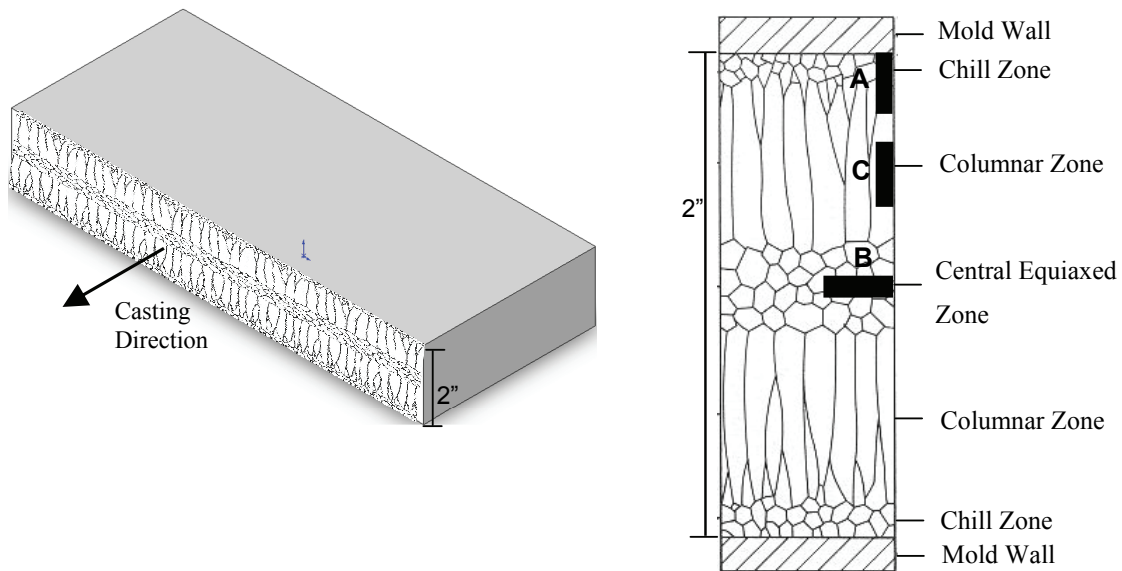

Figure 3. Solidification regions and sample orientation within continuous cast slab demonstrating the chill zone, columnar zone, and equiaxed zone[17].
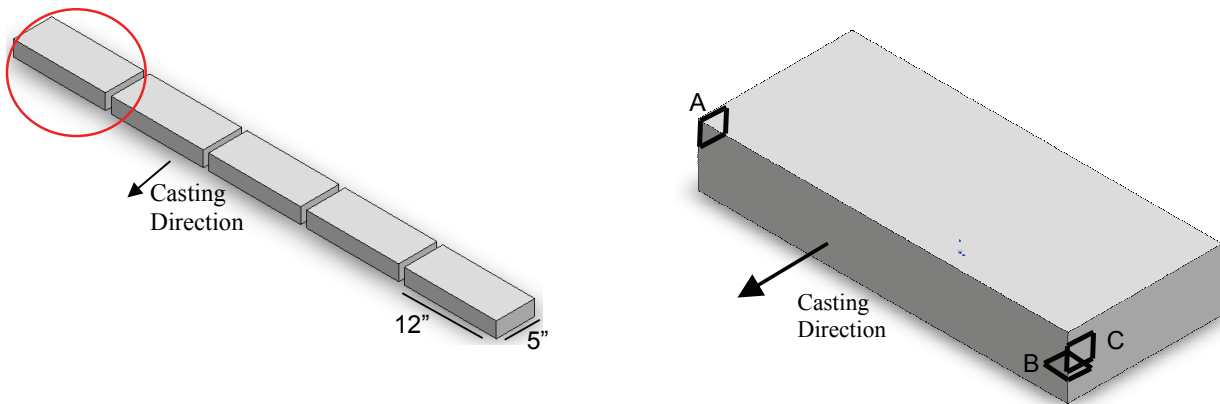

Figure 4. Sample locations for CMn(Nb) testing. The circled section was used to obtain the three sampling locations. Locations A, B, and C were tested from the base of the caster and prior to entry into the rolling mill.

RESULTS

Reproducibility testing is reported prior to in-depth precipitation studies in order to verify that the electrochemical extraction technique was repeatable. Precipitation studies are then reported relative to varying niobium additions and alloy distribution during secondary cooling prior to hot-rolling.

Reproducibility Testing

The preliminary electrochemical extraction experiments with the vanadium containing steel, although very helpful in validating the quenching method, were conducted with single replicates. Additional testing was therefore conducted to assess reproducibility of the test procedures for the niobium alloys. The reproducibility tests were carried out using the medium niobium CMn(Nb) steel sampled prior to the rolling mill. A total of ten replicate samples were measured to test reproducibility and efficiency of the electrochemical extraction and ICP-AES procedures. The ten samples were taken from the columnar region, sample location C in Figure 4. The columnar region was chosen because it is likely to exhibit smaller concentration gradients than the surface or centerline positions. The results for the amount of alloy that remained in the steel matrix and that precipitated are seen in Table IV. The amount of alloy that precipitated is consistent within the set of ten samples at an average of 0.004 wt% Nb. The standard deviation for the alloy precipitation data is 0.001, which indicates good reproducibility. From these data, the average percentage of niobium in solution is 88 percent and the average in precipitated form is 12 percent. The 95 percent confidence interval for the amount of niobium precipitated is 0.004 ± 0.00045 wt% Nb. This table additionally highlights the similarity of the ICP-AES results, compared to the alloy content reported by the steel producer. This nominal percentage difference indicates that the procedure is reproducible and that the ICP-AES analysis is comparable to the alloy content reported by the producer. The results also indicate that the columnar region for the medium CMn(Nb) steel prior to hot rolling demonstrates minimal alloy segregation on a macroscopic scale. Overall, it is concluded that the reproducibility of the testing method appears sufficient.

Table IV. Reproducibility of Niobium Measured in Solution and Precipitate Form

Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
1	0.004	0.025	0.029	13	87	0.030	-3
2	0.005	0.025	0.030	15	85	0.030	-2
3	0.003	0.027	0.031	11	89	0.030	3
4	0.003	0.028	0.031	11	89	0.030	3
5	0.005	0.026	0.031	17	83	0.030	3
6	0.003	0.026	0.030	12	88	0.030	-1
7	0.003	0.025	0.028	10	90	0.030	-6
8	0.003	0.026	0.030	11	89	0.030	-1
9	0.003	0.026	0.029	10	90	0.030	-2
10	0.004	0.026	0.029	12	88	0.030	-2
Average	0.004	0.026	0.030	12	88		-0.899
Standard Deviation	0.001	0.001	0.001	2.297	2.297		2.983

Influence of Niobium Content and Slab Location on Precipitation Behavior

Slab Surface Region

The slab surface location is illustrated in Figure 4 as sample location A. Specimens were taken at the base of the caster and after exiting the tunnel furnace prior to the rolling mill for the low, medium, and high niobium steels. Ten replicate specimens were prepared for each of the alloys at each process location. The average of the ten experiments was taken to represent the microalloy precipitation behavior at the edges of the slab. The results from the edge location at the caster and prior to the rolling mill are summarized in Tables V and VI, respectively. It is observed that the low and medium niobium steels appear to exhibit some precipitation during equalization in the furnace prior to hot rolling. In contrast, the high niobium steel exhibits greater precipitation immediately after casting, followed by some niobium dissolution upon hot rolling. The results are represented graphically in Figure 5, and the error bars indicate one standard deviation about the mean.

Table V. Average Niobium Measured in Solution and Precipitate Form
Edge Location at the *Base of the Caster*

Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
Low Nb Caster	0.003	0.011	0.014	24	76	0.014	2
Med Nb Caster	0.008	0.022	0.030	28	72	0.030	-1
High Nb Caster	0.026	0.029	0.056	48	52	0.046	22

Table VI. Average Niobium Measured in Solution and Precipitate Form
Edge Location *Prior to Hot Rolling*

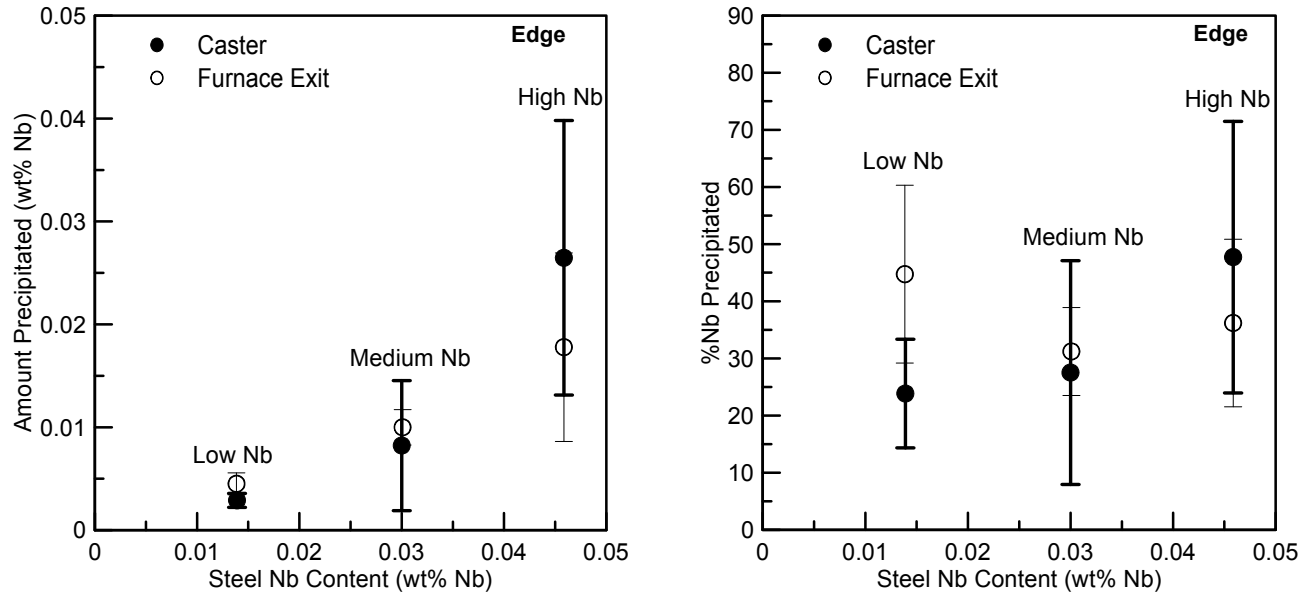| Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
Low Nb Furnace Exit	0.005	0.007	0.011	45	55	0.014	-17
Med Nb Furnace Exit	0.010	0.023	0.033	31	69	0.030	11
High Nb Furnace Exit	0.018	0.027	0.045	36	64	0.046	-3

Figure 5. Slab surface/edge location; niobium in precipitate form measured by ICP-AES in wt % (left) or % of total niobium (right) plotted against the reported alloy content given by the steel producer.

Columnar Region

The columnar location is shown in Figure 4 as sample location C. Specimens were taken from the caster and exiting the tunnel furnace prior to the rolling mill for the low, medium, and high niobium steels. Five replicate specimens were prepared for each of the alloys at each process location and presumably represent the behavior of most of the slab volume. The number of samples was reduced from that of the slab surface on the basis of the good reproducibility of results from the columnar region reported earlier. The average results from the columnar region for the caster and prior to hot-rolling are summarized in Tables VII and VIII, respectively. Figure 6 provides a graphical representation of the amount of niobium precipitation for each alloy and process location. The results obtained from the columnar region indicate that less precipitation occurred at the base of the caster and through the tunnel furnace in comparison to the edge location presented above. The variability in the results appears to be reduced for the columnar region in

Table VII. Average Niobium Measured in Solution and Precipitate Form
Columnar Location at the *Base of the Caster*

Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
Low Nb Caster	0.003	0.010	0.013	24	76	0.014	-7
Med Nb Caster	0.004	0.027	0.031	14	86	0.030	3
High Nb Caster	0.005	0.045	0.050	10	90	0.046	9

Table VIII. Average Niobium Measured in Solution and Precipitate Form
Columnar Location *Prior to Hot Rolling*

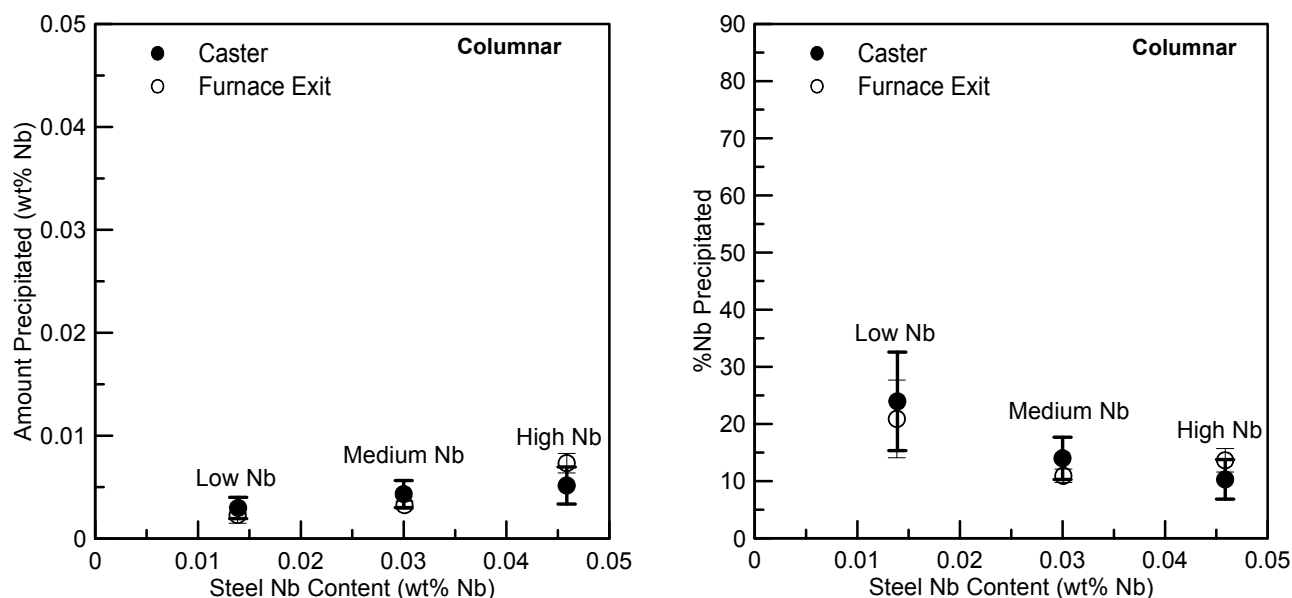| Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
Low Nb Furnace Exit	0.002	0.009	0.012	21	79	0.014	-16
Med Nb Furnace Exit	0.003	0.026	0.030	11	89	0.030	-1
High Nb Furnace Exit	0.007	0.047	0.054	14	86	0.046	18

Figure 6. Columnar region; niobium in precipitate form measured by ICP-AES in wt % (left) or % of total niobium (right) plotted against the reported alloy content given by the steel producer.

comparison to the surface location. The absolute amount of niobium in precipitated form is greatest in the high niobium steel, while the relative amount is greatest in the low niobium steel in the columnar region. There does not appear to be substantial precipitation or dissolution during equalization in the tunnel furnace for the low and medium niobium steels; some precipitation is indicated for the high niobium steel. It is also observed that the variability between the columnar samples is generally less than the variability observed in the edge samples, based on a comparison of standard deviations (error bars).

Centerline Region
The centerline sampling region is depicted in Figure 4 as sample location B. Specimens were taken from the caster and exiting the tunnel furnace prior to the rolling mill for the low, medium, and high niobium steels. Five replicate specimens were prepared for each of the alloys at each process location. The average results from the centerline sampling region for the caster and prior to the rolling mill are summarized in Tables IX and X, and graphically in Figure 7. The results obtained from the centerline region indicate less precipitation at the base of the caster and through the tunnel furnace in comparison to the edge location presented above. However, precipitation observed within the centerline location is greater in comparison to the columnar region. It is also observed that the variability is similar to the columnar region, and less than the variability observed at the edge location. The results may indicate some precipitation in the tunnel furnace, perhaps associated with local regions of alloy enrichment.

Table IX. Average Niobium Measured in Solution and Precipitate Form
Centerline Region at the *Base of the Caster*

Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
Low Nb Caster	0.004	0.007	0.011	40	60	0.014	-23
Med Nb Caster	0.005	0.025	0.031	17	83	0.030	2
High Nb Caster	0.009	0.044	0.053	17	83	0.046	15

Table X. Average Niobium Measured in Solution and Precipitate Form
Centerline Region *Prior to Hot Rolling*

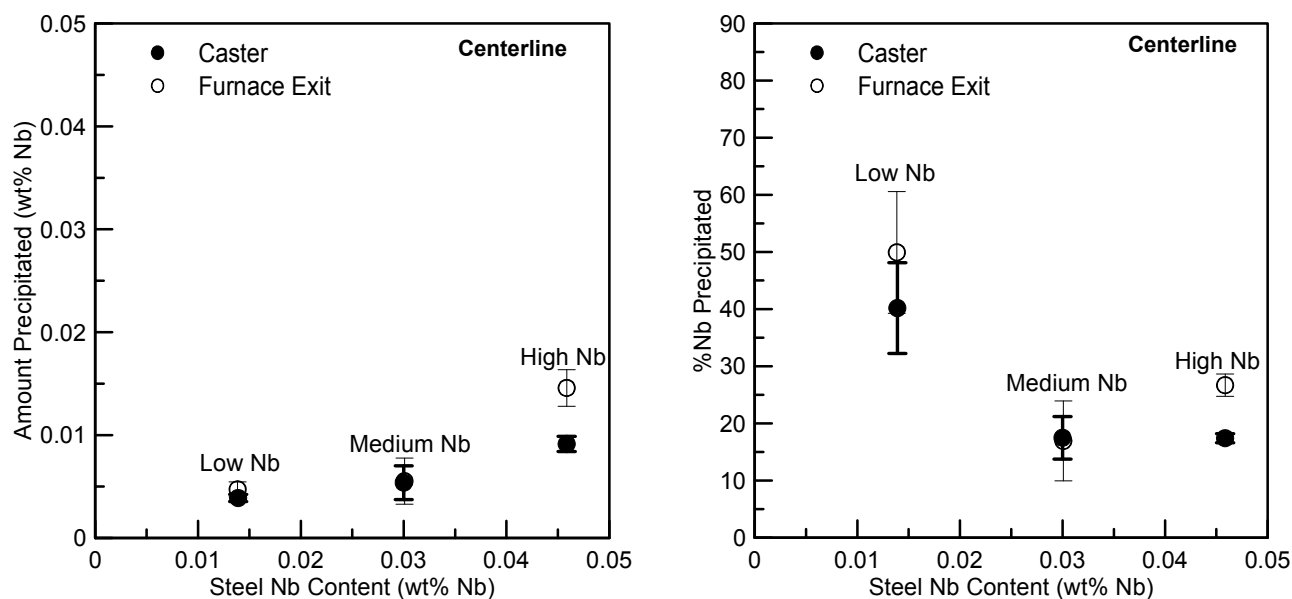| Sample	Precipitate (wt% Nb)	Solution (wt% Nb)	Total (wt% Nb)	Percentage of Nb precipitated	Percentage of Nb in soluiton	Producer's Reported Alloy Content (wt% Nb)	Precentage Difference
Low Nb Furnace Exit	0.005	0.006	0.011	50	50	0.014	-24
Med Nb Furnace Exit	0.006	0.028	0.033	17	83	0.030	11
High Nb Furnace Exit	0.015	0.040	0.055	27	73	0.046	19

Figure 7. Centerline region; niobium in precipitate form measured by ICP-AES in wt % (Left) or % of total niobium (right) plotted against the reported alloy content given by the steel producer.

DISCUSSION

The average results from all steels and sampling locations are plotted in Figure 8 for comparison. The results show that the greatest amount of alloy precipitation occurs at the slab surface along the edges of the continuously cast thin slab. The extent of precipitation appears greatest in the high niobium steel, where dissolution subsequently occurs during reheating and equalization in the tunnel furnace. The columnar region represents the bulk of the slab volume and exhibits the lowest amount of precipitated niobium. The high percentage of niobium precipitation along the slab edges can be understood in terms of the slab surface temperature and solubility
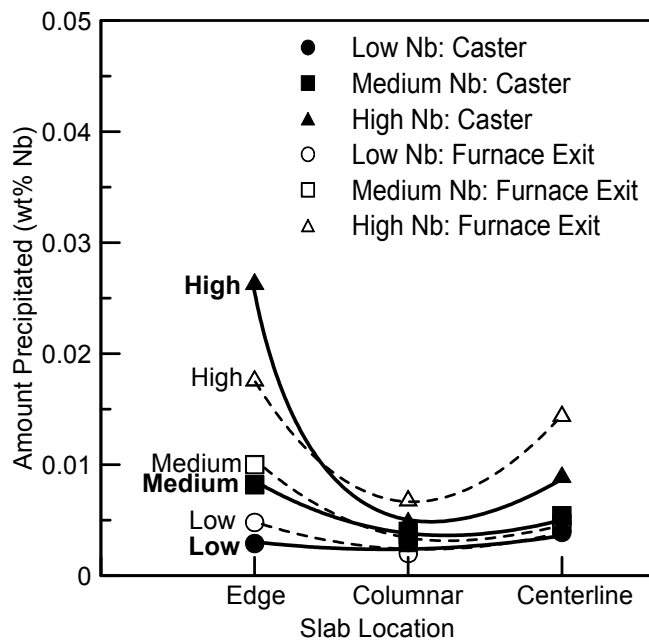

Figure 8. Niobium in precipitate form measured by ICP-AES (in wt %) plotted against the sample location through the thickness of the slab.

of niobium carbonitrides. The lower temperatures of the slab surface at the base of the caster are associated with an increasing driving force for alloy precipitation, and therefore conducive to precipitation in the surface/edge/corner regions. Increasing the niobium content also increases the supersaturation and allows niobium precipitation to occur at higher temperatures. Both of these behaviors are consistent with the trends expected from the equilibrium solubility characteristics illustrated in Figure 2[15]. The results also suggest that some niobium dissolution for the high niobium alloy occurs during processing through the tunnel furnace. The soaking time and temperature within the tunnel furnace "equalizes" the temperature profile of the slab, increasing niobium solubility in the cooler regions of the casting, i.e. at the surface. This effect is beneficial because the re-dissolved niobium in the austenite is available to enhance the thermomechanical processing response and final properties. An increased amount of niobium is also seen to precipitate within the centerline region (relative to the columnar region), presumably caused by the alloy rich liquid present during the final stage of solidification. The medium niobium alloy also demonstrates higher precipitation along the slab surface with less precipitation within the columnar and centerline regions. In this alloy, there is little difference between the results from the base of the caster and prior to hot rolling. In the low niobium steel it appears that some niobium precipitation may occur during equalization in the tunnel furnace, prior to hot rolling.

SUMMARY

This research was directed towards understanding niobium precipitation in hot-charged slabs relative to alloy additions and position within the slab. Steels containing low, medium, and high concentrations of niobium were tested at multiple slab locations. Preliminary results from a vanadium-containing steel confirmed that the quenching method used for sample collection during CSP processing was adequate. Replicate testing also confirmed that the electrochemical extraction method was reproducible.

Three steels with different niobium additions were used to determine the extent of niobium precipitation prior to entry into the hot rolling mill relative to position within a slab (which affects thermal history) and alloy content. The areas of interest included the chill zone (i.e. slab surface), the columnar region, and the centerline of the slab. The slab surface/edge results were associated with greater variability, and increased precipitation in the high and medium niobium alloys. In the high niobium alloy, some niobium dissolution occurred during equalization in the tunnel furnace subsequent to casting. This behavior was consistent with the lower solubility associated with higher niobium levels and lower temperature. The three alloys demonstrated that the absolute amount of niobium in precipitated form was greatest in the high niobium steel, while the relative amount was apparently greatest in the low niobium steel. The columnar region that comprises the bulk volume of the material demonstrated less variability between samples in comparison to the edge location. Especially for the high niobium alloy, the amount of alloy precipitated on the slab surface was greater than observed at the other sampling locations. The amount of precipitation in the centerline region was slightly greater compared to the columnar region.

ACKNOWLEDGMENTS

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