

## **PRODUCING HIGH QUALITY NIOBIUM-BEARING STEELS USING THE CSP PROCESS AT NUCOR STEEL BERKELEY**

A. J. DeArdo<sup>1</sup>, R. Marraccini<sup>2</sup>, M. J. Hua<sup>1</sup> and C. I. Garcia<sup>1</sup>

<sup>1</sup>Basic Metals Processing Research Institute, Department of Materials Science,  
University of Pittsburgh, Pittsburgh, PA 15261, USA;

<sup>2</sup>NUCOR Steel-Berkeley, P.O. Box 2259, Mount Pleasant, SC 29465, USA)

**Keywords:** HSLA Steel, Niobium, Linepipe, Plate, Thermomechanical Processing,  
Microalloying

### **Abstract**

In the 16 years since the first hot band coil was rolled using the Compact Strip Production (CSP) at NUCOR Steel Crawfordsville, the product line produced by the sheet division of NUCOR and other minimills has grown in complexity and sophistication. Today, at Nucor Steel Berkeley (NSB), advanced high strength steels such as HSLA and Dual-Phase steels can be produced from electric furnace melts either as hot band or in cold rolled gauges produced on continuous annealing (CA) or continuous galvanizing (CG) lines. Similarly, with the addition of vacuum degassing, Interstitial-Free steels and AKDQ steels can also be produced on CA or CG lines. Niobium has already achieved its normal, prominent position in the HSLA steels and will soon appear in the DP and IF steels, as well. A recent, important development at NSB is the new intensified hot rolling sequence, where by heavy gauge skelp of niobium strengthened HSLA steel for X52-X70 API pipe can be rolled resulting in a uniform ferrite microstructure, impressive improvement in strength and toughness, and the absence of aberrant non-destructive test reflections. The ductility and formability of the CAL or CGL sheet grades is good, while the surface quality is more than adequate. It is now clear that high performance steels can be produced by the EAF plus thin slab casting route. This paper will review some recent advances in skelp production for ERW pipe and their underlying physical and mechanical metallurgy.

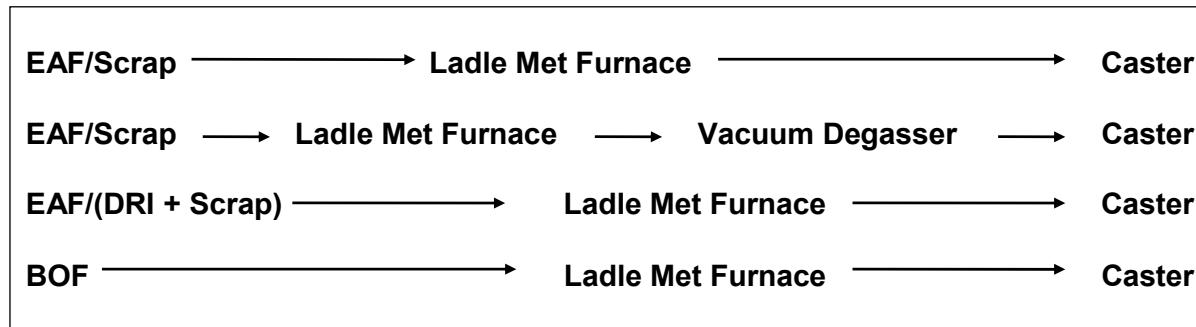
### **Introduction**

A major conference was held in Guangzhou in 2002 dedicated to thin slab cast (TSC) steel production [1]. The two major general results of that conference were: (i) the broad range of production sequences that incorporated thin slab casting, and (ii) the wide range of products that were either being produced or were being contemplated. The variation in production route was mainly in the steelmaking area, where liquid steel was supplied from electric arc furnace (EAF) or basic oxygen furnace (BOF) shops. In a traditional CSP plant the liquid is supplied from an EAF, passes through a ladle metallurgy furnace and then the caster. However, TSC is often retrofitted into integrated plants, hence the BOF steel supply. These variations are shown in Figure 1.

Obviously, the quality of liquid steel will vary depending on the steelmaking route. This varies from scrap-based EAF, through mixtures of scrap plus direct reduced iron (DRI), through by

BOF. As will be discussed below, the quality of the liquid steel entering the caster can have a large influence on the optimum alloy design, e.g., Nb or V addition, for a given product.

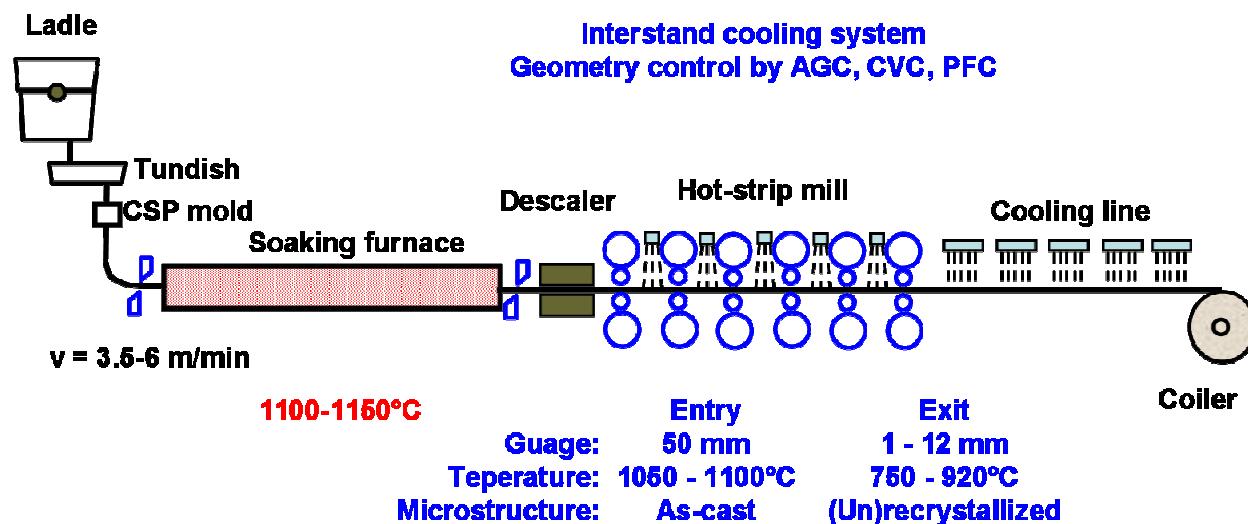
Although niobium is added to a wide range of grades of steel, including HSLA, dual-phase (DP) and interstitial-free (IF) steels, only niobium in HSLA steel will be discussed here. The benefits of niobium in HSLA are clearly established, are routinely practiced, and have been thoroughly reviewed(IMR).



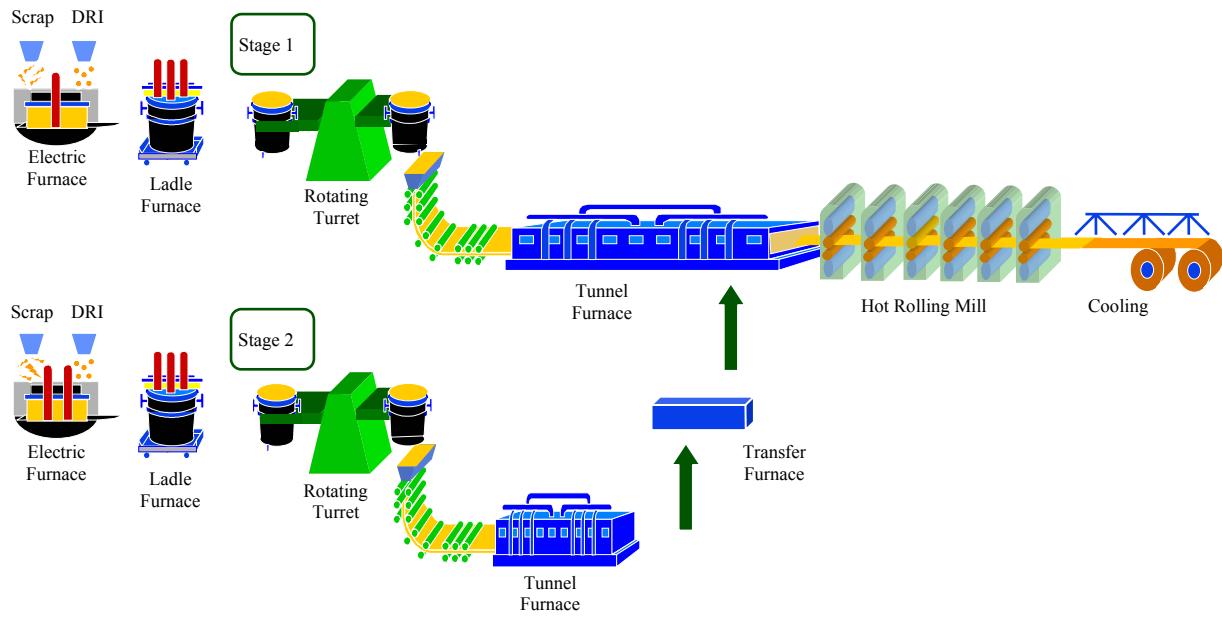
**Fig. 1 Block diagram showing different steelmaking routes up to the caster**

### The Compact Strip Production Process

The CSP process has been described in several articles, and will only be briefly described here [1-3]. After steelmaking, the liquid is teemed into the tundish of the caster, after which it is solidified to the desired thickness, approximately 50mm in this case. The slab is then sheared to the proper length and then transported to the tunnel or equilibrating furnace normally set at 1150°C. At this point, the slab exhibits an austenite grain size of 500-1000 µm [4]. After the 20 minute residency time in the furnace, the slab exits the furnace, is crop sheared, and then enters the finishing mill at approximately 1000°C. The grain size entering F1 is not appreciably different from that entering the furnace [1,4]. After the slab passes through the finishing mill of 5, 6 or even 7 stands, it enters the runout table (ROT) where it undergoes cooling to the coiling temperature, after which it is coiled and allowed to cool to room temperature. This process is shown schematically in Figures 2 and 3.



**Fig. 2 Schematic of the CSP Process**



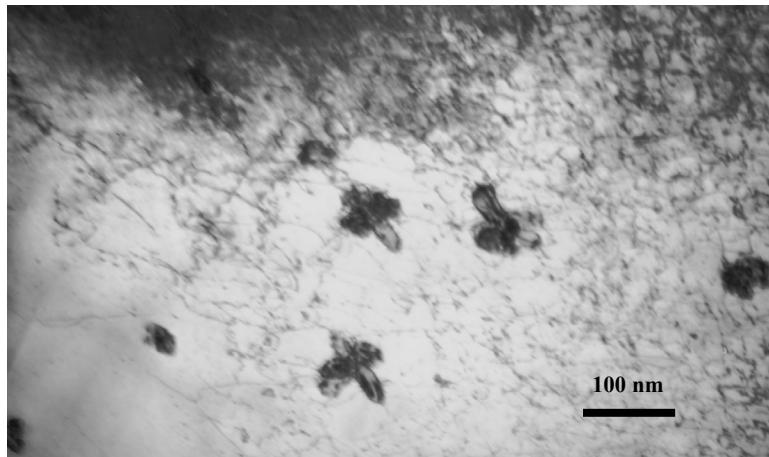
**Fig. 3 Schematic of the CSP Process**

### Steelmaking Challenges in Improving Niobium Effectiveness

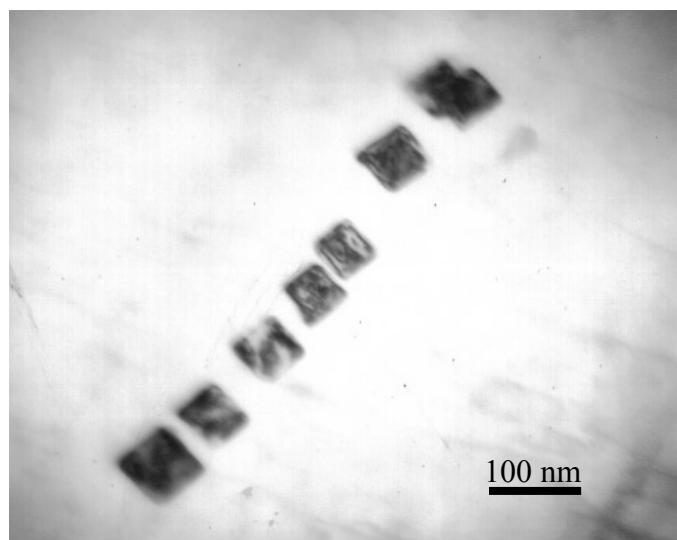
As was clearly shown in the literature [5], based on lattice parameter considerations, neither NbC nor NbCN fit well in either the austenite or ferrite lattice. The precipitation of NbC must occur by heterogeneous nucleation, i.e., on pre-existing high surface energy crystalline defects or substrates. Typically, these are subgrain or grain boundaries in the austenite. An additional very common such surface is provided by TiN that can precipitate either in the liquid, interdendritic liquid regions between the delta ferrite dendrites, or in the austenite prior to the formation of the NbC. Because of the segregation tendencies of titanium and niobium, conditions favorable for TiN precipitation exist in all but the cleanest liquid [6,7], and are often observed in both thick and thin slabs. This TiN and associated NbCN in the cruciform particles are not an issue in cold charged, thick slabs as found in the typical integrated plant, since reheating temperatures in excess of 1200°C are high enough to dissolve all of the Nb and some of the TiN in these small particles. In CSP production, however, the TiN + NbC will not normally dissolve in the tunnel furnace before entering the finish rolling train, and these particles can have a strong effect on the subsequent behavior of the steel.

As was shown in [7], scrap-based EAF steel containing 40PPM titanium and 100ppm niobium in a niobium containing HSLA steel can precipitate TiN during casting, slab shearing and transfer to the tunnel furnace in a conventional CSP plant. This TiN was very fine and delineated the interdendritic pools of liquid existing just prior to final solidification. Detailed metallography showed that NbC or NbCN had precipitated on these TiN particles during the same time/temperature interval. The resulting complex particle had cores of TiN and arms of NbC. An example of the cruciform or star-like particles is shown in Figure 4, while their distribution is exhibited in Figure 5.

Obviously, the niobium lost to the arms of the star-like particles will not be available for either conditioning the austenite in the finishing train or strengthening the ferrite. The niobium particles would not normally form in this temperature range in the absence of the TiN.



**Fig.4** TEM micrograph of star-like particles



**Fig. 5** Lines of small TiN or star-like particles

Hence, the presence of the TiN + NbCN star-like particles mean that higher bulk levels of niobium are required to reach the normal solute levels expected in these steels and that are required to achieve the desired final mechanical properties.

It is clear that the formation of TiN will depend on the composition of the liquid as it enters the caster. Steels with a high (Ti)(N) product will obviously be susceptible to their formation, while those steels with a low product will not. A summary of the precipitation potential of TiN, as influenced by steelmaking route, is shown in Table I.

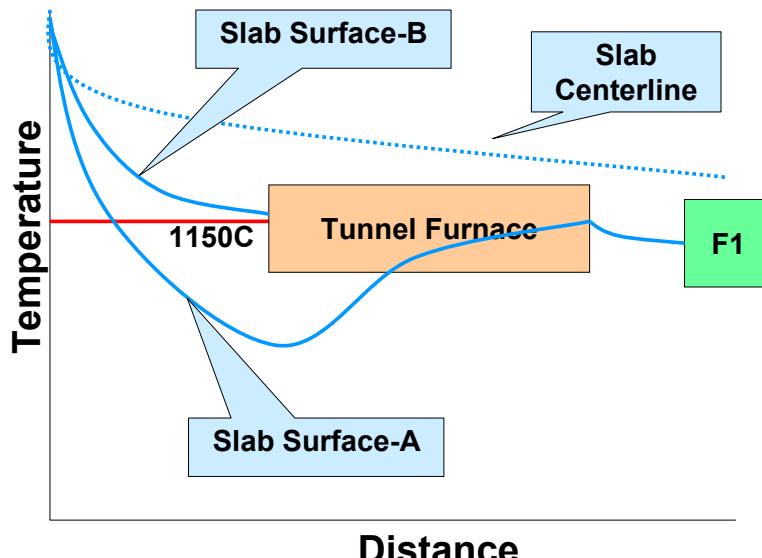
As noted earlier, a major concern is how to avoid the loss of niobium as NbC associated with the star-like or cruciform-shaped particles. The TiN will probably not be a major problem in the last three processing routes shown in Table I. The absence of the TiN in these steels will preclude the formation of the star-like particles and the loss of otherwise usable niobium. These steels will exhibit the required microstructure and properties with the normal bulk niobium levels. However, TiN will be a problem in standard CSP production using the scrap based EAF + LF practice, leading to the formation of the star-like particles and the loss of solute niobium. Higher levels of niobium will be required in these steels to achieve the required final microstructure and properties. It should be noted that similar complex precipitates have been observed in vanadium-bearing HSLA steels [8].

**Table 1 Precipitation Potential of TiN for Different Steelmaking Practices**

Steelmaking Route	Residual Ti Content	N Content	TiN Precipitation Potential at 1050°C
EAF(no DRI) + LF	M	H	VH
EAF(high DRI) + LF	L	L	L
EAF + LF + VacDegass	L	VL	VL
BOF + LF	VL	VL	L

Note: VH = Very High, H = High, M = Moderate,  
L = Low and VL = Very Low

Recent research [7,9] has shown that there are two ways of lowering the volume fraction of the star-like particles that forms prior to entering the tunnel furnace in scrap-based EAF + LF steels. The first is to raise the tunnel furnace temperature to 1200°C, at which point most of the particles will dissolve. This approach is less than satisfactory since the dissolved particles can reform during the early stages of finish hot rolling. The second approach is based on the temperature of formation of the TiN in HSLA steel typified by API X 52-80. The bulk of the TiN particles were observed to form between 1150 and 1050°C as the strand super cools between exiting the caster and entering the tunnel furnace operating at 1150°C. For example, it is not uncommon for the outer portions of the strand to fall to 1000-1050°C just before it enters the furnace. Studies have shown that this is the temperature range where much of the TiN and star-like particles form [9]. This TiN can be minimized if a cooling practice is used where the strand surface never falls below 1100 or 1150°C. Figure 6 illustrates this approach. It is estimated that perhaps 20-35% of the total niobium or vanadium might be saved by implementing these practices.



**Fig. 6 Cooling paths from Caster to Tunnel Furnace**

## Manufacture of ERW Pipe from Hot Rolled Skelp

There are three methods of manufacturing pipe from hot rolled steel: ERW, UOE-SAW and Spiral-SAW. The pipe diameters and wall thicknesses for these three routes are shown in Figure 7. As can be seen in Figure 7, ERW pipe falls into the smaller dimensions of pipe in a relative sense, i.e., smaller diameters and wall thicknesses. The ERW fabrication process is shown in Figure 8. Since it is a highly economical process, large tonnages of ERW pipe are produced.

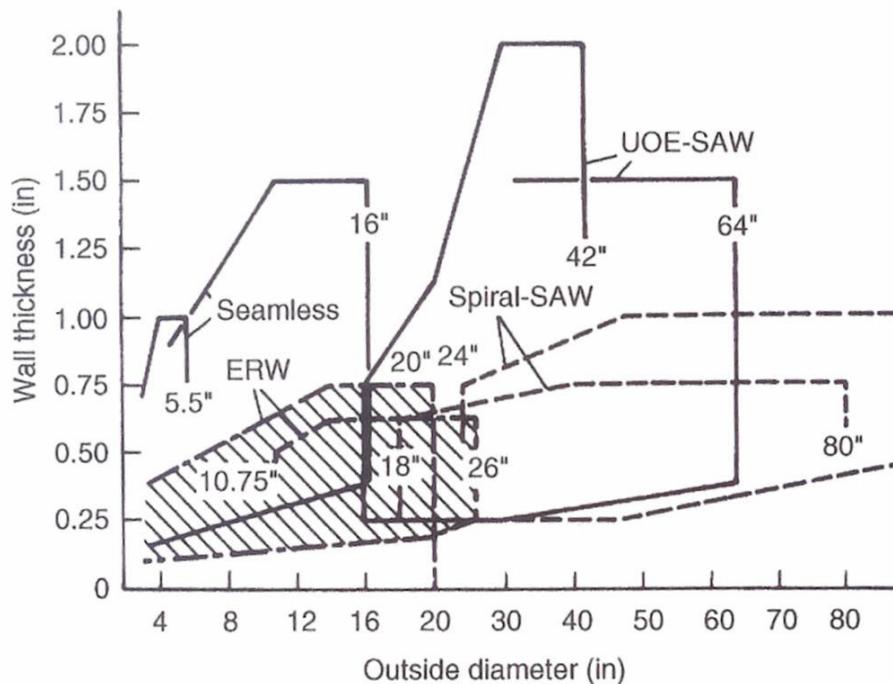


Fig.7. Pipe diameters, wall thicknesses and manufacture routes. Nara, et al., 1981

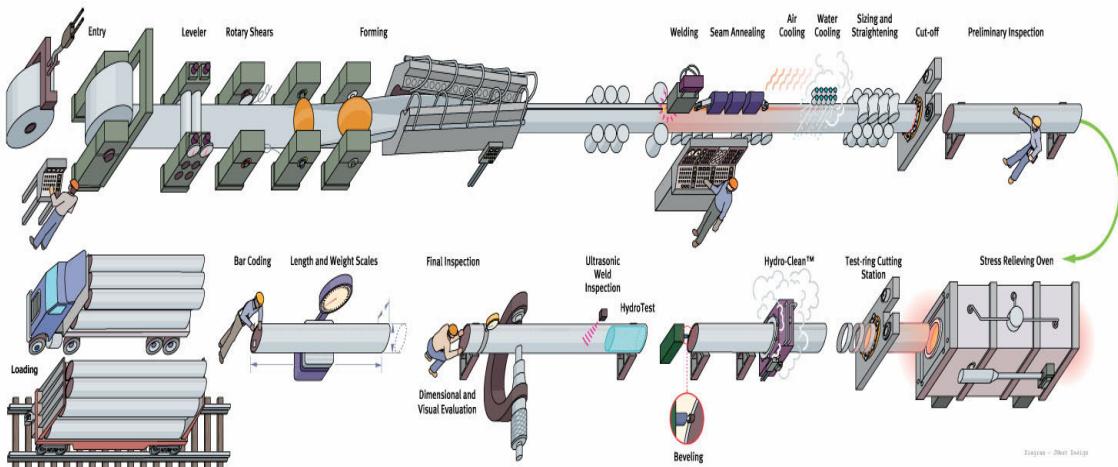


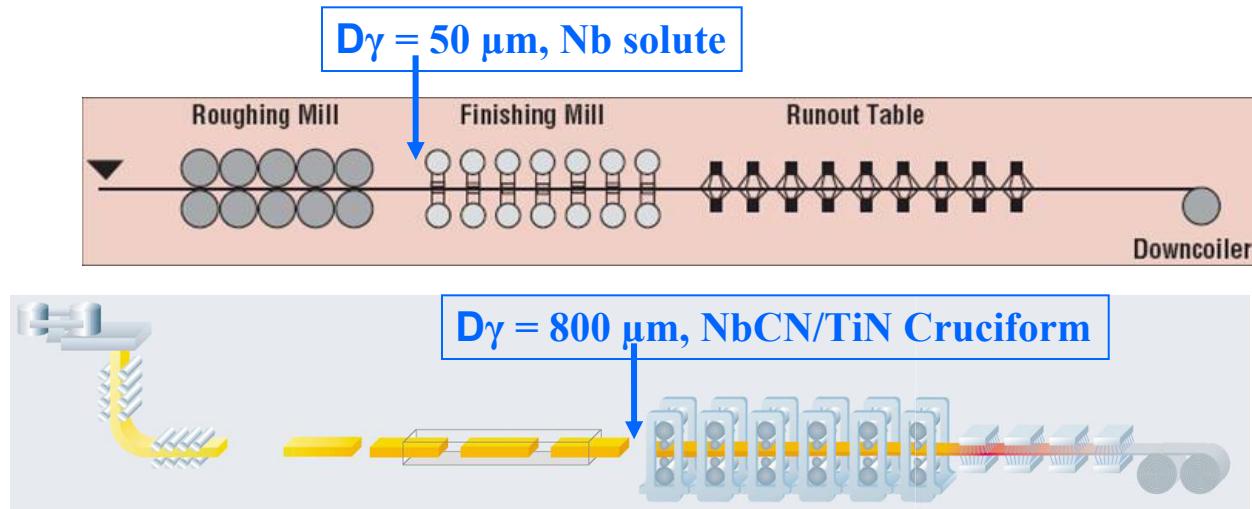
Fig. 8. Schematic of ERW pipe production.

## Challenges of Producing Hot Rolled Skelp by CSP for API-ERW Pipe

There are two major challenges concerning optimum rolling of hot band, especially in a CSP plant. The first is attempting to achieve the minimum gauge possible for either thin gauge hot rolled applications or for feed stock to the cold mill. The second is to attain a uniform microstructure in heavy gauge hot band intended for linepipe applications such as the API

grades. Only the second issue will be discussed here, since it is of immediate importance to ERW pipe manufacturing.

One major difference between rolling on a hot strip mill in an integrated mill and on a CSP mill is in the austenite grain size that exists prior to entering the finishing trains, Figure 9. One problem with niobium bearing steels produced by CSP, or similar processes, is the mixed grain structure that is often observed [6], especially in heavier gauges, over approximately 6mm. An example of this phenomenon is shown in Figure 10. Notice that there are lines of coarse, high temperature polygonal ferrite grains mixed in with the lower temperature non-polygonal/acylular ferrite expected at this cooling temperature of 565°C. This mixed grain structure is the inevitable consequence of rolling a 50 mm slab to 9.5mm in six passes while starting with an initial average austenite grain size of about 800  $\mu\text{m}$ . These mixed structures cause a deterioration of strength and toughness and spurious reflections during UT-NDT of pipes and welds.



**Fig. 9. Comparison of Integrated HSM and CSP**

When austenite undergoes hot deformation, it follows the sequence shown in Figure 11. Essentially full recrystallization occurs above temperature T95, while approximately complete suppression of recrystallization occurs below temperature T5. This diagram will change with starting grain size, composition and interpass time. Also shown on Figure 11 is the normal six stand pass sequence used at NUCOR Steel Berkeley.

This pass sequence has caused mixed grains in heavy gauge skelp intended for API linepipe applications. A typical rolling pass sequence is shown in Table 2 for a thinner product, while the resulting mill loads, flow strength of the austenite, and other rolling parameters are shown in Figure 12.

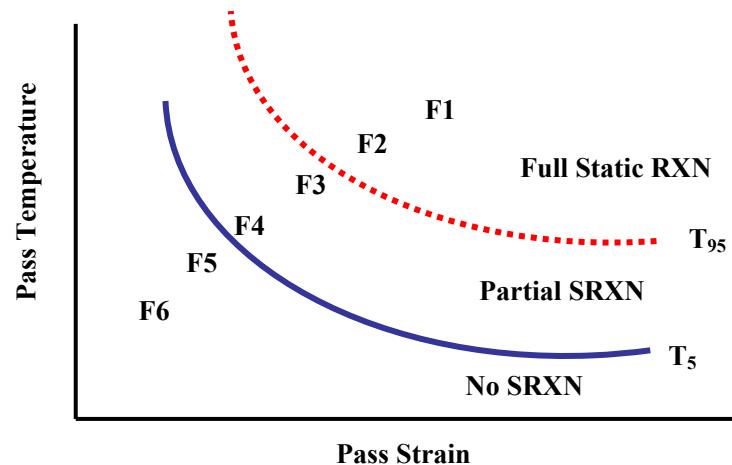
**Table 2 Rolling conditions versus pass no. for standard NSB six pass schedule.**

Pass No.	T, °C	e, %	T1, ms	T2, sec
F1	1029	40	130	
F2	1005	27	60	5.5
F3	978	25	40	4.3
F4	957	23	30	2.8
F5	936	18	20	2.8
F6	915	13	10	2.4

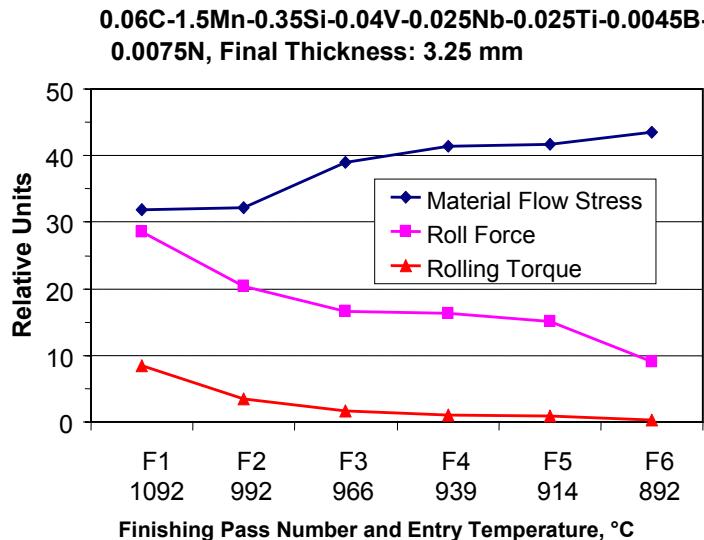
e = Reduction, T1 = Contact Time, T2 = Interpass Time



**Fig. 10 Optical micrograph of mixed grains**



**Fig. 11 Hot Deformation Behavior of Austenite**



**Fig. 12 Rolling data versus pass no. for standard NSB six pass schedule.**

A new pass sequence has been developed to eliminate this mixed grain problem in heavy gauge skelp used, for example, in API linepipe grades [6]. This innovation has resulted in U. S. Patent No. US-2005-0115649-A1 issued on June 2, 2005. It is based on achieving both grain refinement and pancaking of the austenite grains, all within the six pass finishing train. It is known from the literature and basic principles that achieving 100 percent static recrystallization of low carbon HSLA austenite in a finite time (interpass time) depends on five factors: initial grain size, composition, strain, strain rate and temperature. As the slab approaches F1, the grain size, composition, strain rate and temperature are fixed, so only strain is available to achieve compatibility with the recrystallization kinetics. It has been found that eliminating passes F3 and F4 is the key to achieving the desired metallurgical objective. The much heavier passes in F1 and F2, together with the longer interpass time from the exit of F2 to the entry of F5, are sufficient to result in complete recrystallization of the austenite in niobium-bearing HSLA steels between stands F2 and F5. These heavier early passes, together with the heavier pancaking passes, result in a very well conditioned austenite that eliminates the mixed grain final structure, even in heavy gauge steels. This new pass sequence is shown in Figure 13 using actual mill data. Note that there are three pass sequences shown in Figure 13. The standard six-pass practice is shown for coils 38-1 and 85-2. The second practice is with F3 dummied, shown as coil 68-5, while the third is with F3 and F4 dummied, shown as coils 36-1 and 38-3.

Furthermore, these heavier passes are also sufficient to overcome an important, but neglected, additional cause of the mixed grains found in this kind of product; the grains that are poorly oriented for plane strain hot deformation. Hu, in a classical study of cold rolling and annealing of single crystals of silicon-ferrite at room temperature, showed that the substructure observed after deformation was strongly dependent on the orientation of the single crystal relative to the deformation axes (as below). When the crystals had initial orientations near  $\{001\}<110>$ , they showed a homogeneous distribution of dislocations in the form of uniform cells. These crystals did not recrystallize very easily during subsequent annealing. On the other hand, crystals with orientations near  $\{001\}<100>$  exhibited a very heterogeneous dislocation structure comprised of broad, low density deformation bands separated by narrow, microbands or transition bands that contained extremely small dislocation cells and a very high local dislocation density [10]. These crystals showed rapid recrystallization during subsequent annealing because of the high local stored energy and sharp orientation change at the microbands. The reason for the difference in behavior is that the first crystal orientation had no grain interior nucleation sites for static

recrystallization, while the second had many nucleation sites. HSLA austenite is believed to behave similarly, as shown in Figure 14, where it is obvious that some grains show deformation bands and some do not [11]. As the pass strain increases, more of the poorly oriented grains will show a sufficiently high enough dislocation density that they will exhibit nucleation and recrystallization, grain refinement and subsequent uniform final microstructure.

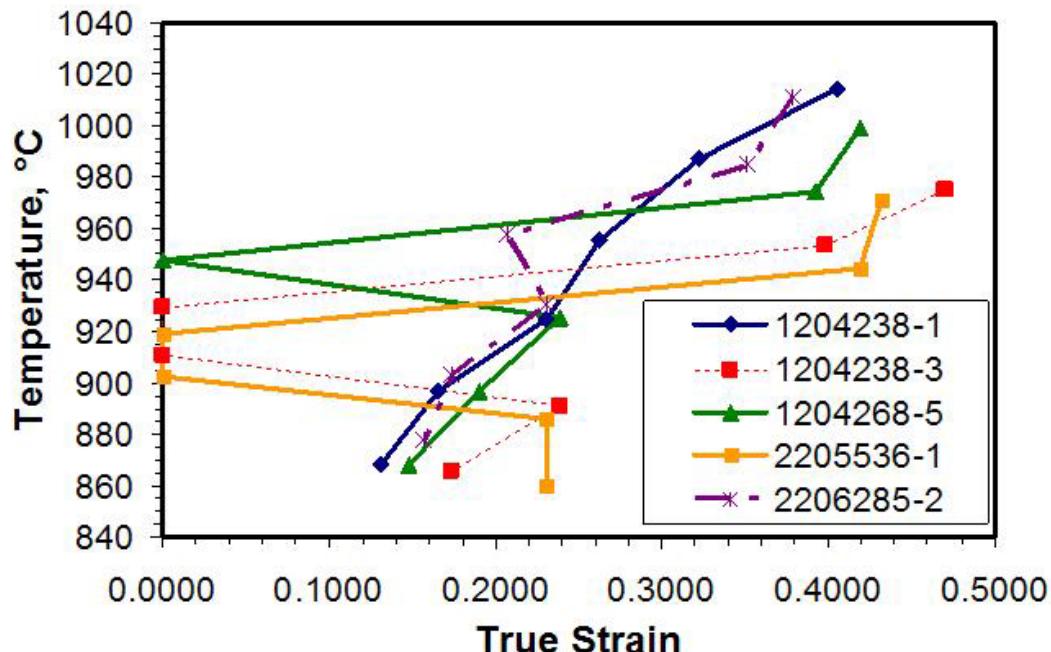


Fig. 13 T –  $\varepsilon$  pass sequence data

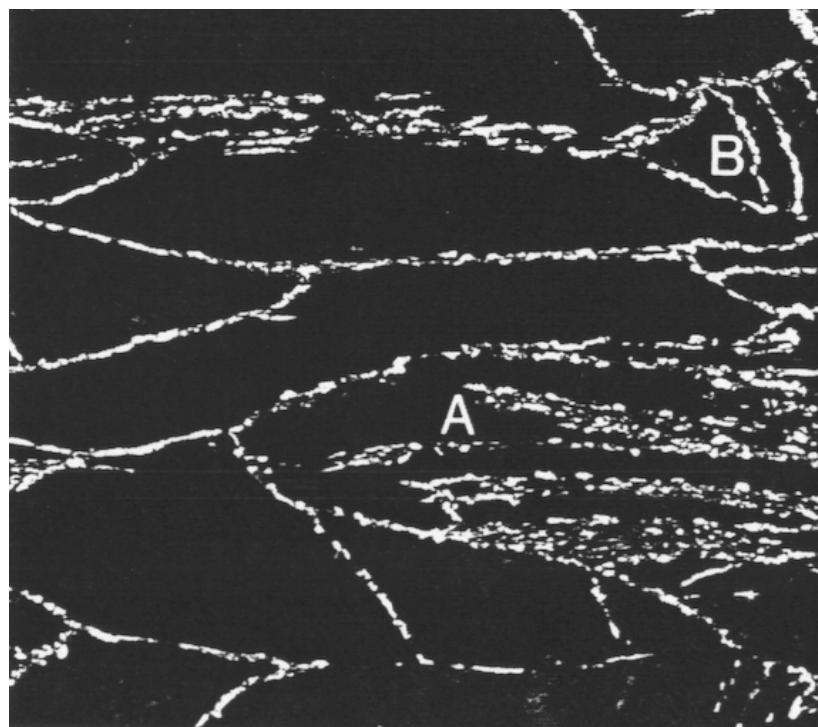
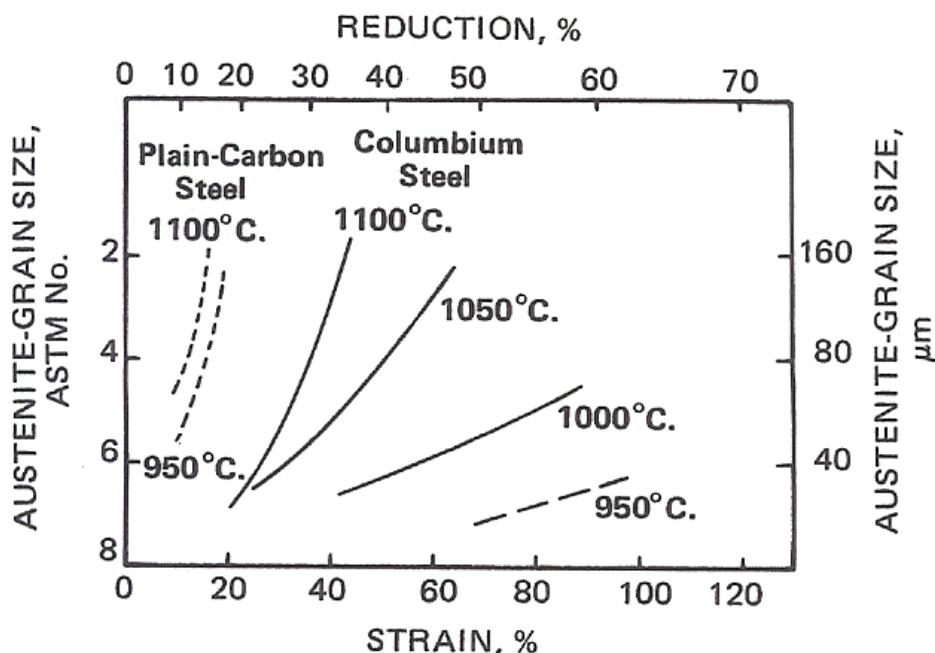


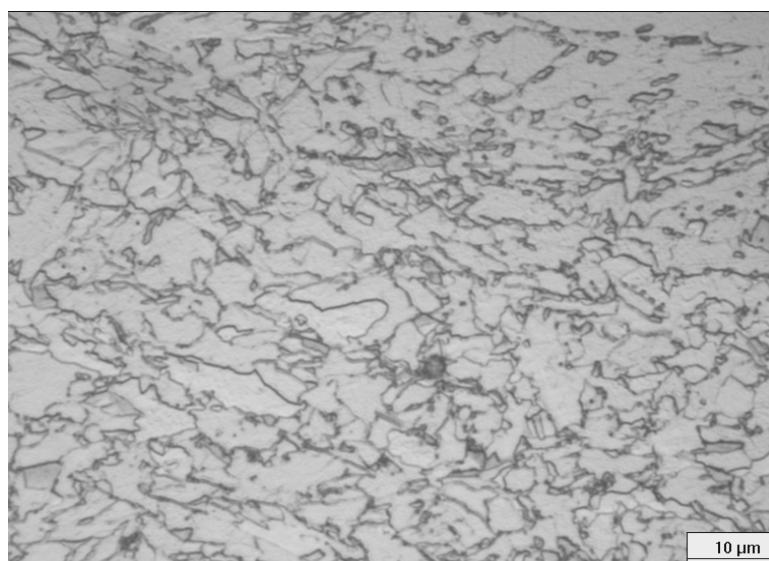
Fig. 14 Dark field optical micrograph of nucleation of ferrite at deformed austenite grain boundaries, deformation bands (A), and annealing twins (B).

Kozasu, et al., studied the recrystallization of coarse grained austenite in the early 1970s [12]. Figure 15 [12] shows that recrystallization of coarse grained austenite can occur only with very large reductions, of the magnitude found when stands F3 and F4 are dummied, as shown schematically in Figure 13. The final microstructure that results from this new rolling practice is shown in Figure 16.

The uniformity of the microstructure results in higher strength and toughness and the absence of spurious UT reflections [6]. This new rolling practice is used for all heavy gauge HSLA strip and skelp produced by NSB.



**Fig. 15 Effect of deformation temperature and initial grain size on critical amount of deformation required for completion of recrystallization in the plain-carbon and niobium steels**



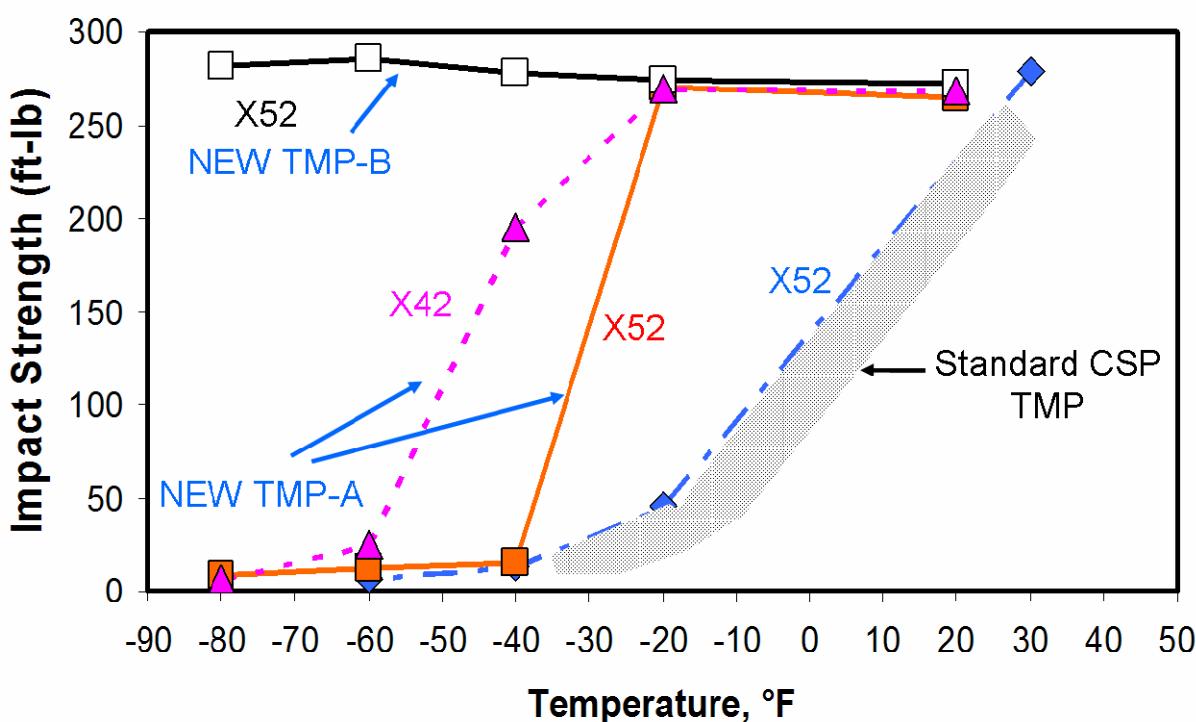
**Fig. 16 Optical micrograph of uniform acicular ferrite via the new TMP path.**

## HSLA Steel Skelp Production for API-ERW Pipe at NUCOR Steel Berkeley

As shown in Figure 16, the use of the new rolling practice with stands F3 and F4 dummied has led to impressive improvements in the as-coiled microstructure. It has also resulted in markedly improved strength, Table 3, and toughness properties, Figure 17. A more complete list of HSLA grades produced at NSB is shown in Table 4. The performance of HSLA X70 skelp produced by NSB and evaluated in pipe for by the ERW pipemaker is presented in Figure 18. Clearly, the CSP process is capable of producing HSLA skelp acceptable for API-ERW pipe. The Hot Strip Mill Division of NUCOR Steel, including Berkeley, SC, Blytheville, AR, Crawfordsville, IN, and Decatur, AL, together produced in excess of 500,000 tons of skelp for ERW pipe in 2005.

**Table 3. Influence of Thermomechanical Rolling Path on Tensile Properties of Selected HSLA Steels Produced at NUCOR Steel- Berkeley**

Steel	Rolling Practice	Yield Strength		Tensile Strength		% Elong	HRB
		MPa	ksi	MPa	ksi		
HSLA-Nb-4	Standard	355.8	51.5	441.6	64.1	36.1	78.8
HSLA-Nb-4	New	420.3	61.0	508.5	73.8	34.3	82.0
HSLA-Nb-5	Standard	402.4	58.4	485.7	70.5	32.7	81.7
HSLA-Nb-5	New	456.1	66.2	547.1	79.4	32.2	87.0



**Fig. 17. Charpy V-notch impact data for 12.7mm (0.50 in.) thick X42 and X52 linepipe skelp produced at NUCOR Steel-Berkeley's CSP Plant, Charleston, SC.**

	Heat Number	Slab Number
	1200924	1200924-3
Vendor	NUCOR-Berkeley, SC	

Item	20" (X70) 0.375"	
Run	Coil	Date
10-2	1	3-12-02

TRIAL NUMBER		
NUCB-021		

#### LADLE CHEMISTRY

C	.040	Nb	.069	Cu	.090	Al	.036
Mn	1.280	V	.003	Ni	.300	B	-
P	.014	Ti	.011	Mo	.300	Ca	.002
S	.002	Si	.310	Cr	.030	N	.008

LOW COILING TEMPERATURE  
WITH TITANIUM --> (1:1.4 Ti/N<sub>2</sub> RATIO)

SRA = Stress Relief Annealing

#### MECHANICAL TESTING

	Skelp	Pipe	PIPE - SRA
Strap Yield	81.0	82.0	80.5
Ring Yield	77.5	81.0	
Tensile	94.0	92.0	92.5
Y/T Ratio Strap	.86	.89	.87
Y/T Ratio Ring		.84	.88
Weld Tensile		92.5	97.5
% Elongation	31.5	29	30.5
YPE (Luders)	X	X	X
SRA Temp			350

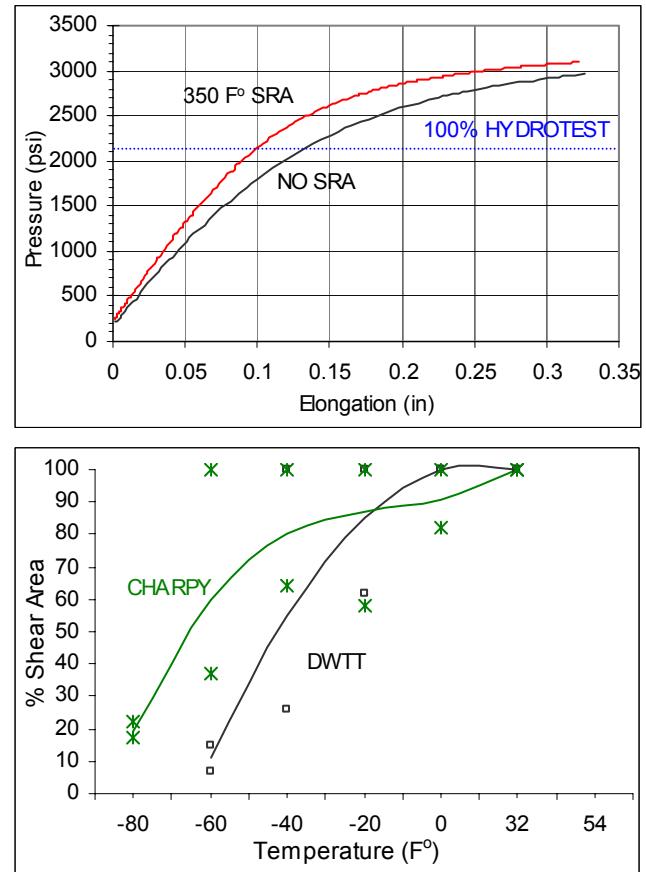
#### IMPACT TESTING

#### PIPE 8

	DWTT		Charpy		Size	2/3
	Shear Area		Shear Area, %		Energy, J	
54	X	X	X	X	X	X
32	100	100	100	100	159	144
0	100	100	100	82	136	114
-20	100	62	100	58	100	67
-40	100	26	100	64	114	102
-60	15	7	100	37	91	17
-80	X	X	17	22	10	11

#### STEEL MILL ROLLING PARAMETERS

Furnace Time and Temp. =	Finishing Temp High/Low =
Finish Entry Temp. =	Average Finish Temp. =
Reduction Below Reference =	Coiling Temp High/Low =
Cooling Rate =	Avg. Coil Temp. = LOW



ESTIMATED TRANSITION TEMPERATURES				
TEST	→	85% SA	50% SA	
DWTT	→	-20°F		-45°F
CVN	→	-30°F		-65°F

#### Comments

- \* TENSILE RESULTS - Coil met minimum x70 requirements & was slightly over 78 ksi yield target after SRA.
- \* IMPACT RESULTS - MET TRIAL TARGET VALUE OF (80%-85% SHEAR AREA MINIMUM @ 32°F)
- \* ULTRASONIC RESULTS= no problems reported.
- \* IMPROVEMENT SUGGESTION= improved toughness / increased shear areas @ lower temperatures.

**Figure 18. Charpy V-notch and DWTT impact data taken from 510mm (20 in.) diameter pipe X 9.5mm (0.375 in.) wall made from X70 skelp produced at NUCOR Steel-Berkeley's CSP Plant, Charleston, SC using the new TMP path.**

**Table 4. HSLA Strip and Linepipe Skelp Compositions  
Produced at NUCOR Steel- Berkeley**

	C	Mn	Nb	Mo	Ni	N**	Grade
<b>HSLA-Nb-4</b>	<b>0.045</b>	<b>0.5</b>	<b>0.025</b>			<b>0.009</b>	<b>X42</b>
<b>HSLA-Nb-5</b>	<b>0.055</b>	<b>1.10</b>	<b>0.045</b>			<b>0.009</b>	<b>X52</b>
<b>HSLA-Nb-6</b>	<b>0.045</b>	<b>1.30</b>	<b>0.07</b>			<b>0.009</b>	<b>X60</b>
<b>HSLA-Nb-Mo-1</b>	<b>0.045</b>	<b>0.85</b>	<b>0.07</b>	<b>0.15</b>		<b>0.009</b>	<b>X60</b>
<b>HSLA-Nb-Mo-2</b>	<b>0.045</b>	<b>0.85</b>	<b>0.07</b>	<b>0.30</b>		<b>0.009</b>	<b>X65</b>
<b>HSLA-Nb-Mo-3</b>	<b>0.045</b>	<b>1.30</b>	<b>0.07</b>	<b>0.30</b>		<b>0.009</b>	<b>X65/70*</b>
<b>HSLA-Nb-Mo-Ni-1</b>	<b>0.045</b>	<b>1.30</b>	<b>0.07</b>	<b>0.35</b>	<b>0.25</b>	<b>0.009</b>	<b>X70</b>

Note: \* Depends upon gauge \*\* 90 ppm Max

### New Product Development at Nucor Steel Berkeley

Nucor Steel Berkeley (NSB) has been producing microalloyed HSLA sheet and skelp for nearly a decade in yield strength levels up to 560 MPa and in gauges up to 12.5mm. These product lines have recently been reviewed [6] and discussed. Since the Guangzhou Conference in 2002, NSB has expanded its high strength steel product line to include dual - phase (DP) and experimental complex phase (CP) steel. Furthermore, the addition of a vacuum degassing unit has enabled NSB to produce interstitial-free (IF), motor lamination and enameling steels.

The DP steels are produced either in the hot rolled or GI condition, and at UTS strength levels of 580 MPa. A 780 grade is under development in both conditions.

### SUMMARY

Nucor Steel Berkeley has demonstrated that the CSP process is a very cost -effective method of making a broad range of high quality steel products. As mentioned above, steels ranging from niobium-bearing HSLA, DP, CP, IF, motor lamination and enameling steels can be successfully commercially produced using the CSP process. Practices that optimize the use of niobium in the appropriate members of this product line are being implemented.

Niobium-bearing HSLA steel skelp for high performance API ERW linepipe can be successfully produced by the CSP process. API X-70 properties can readily be obtained by NUCOR using niobium-bearing HSLA steels and CSP processing in gauges up to 12.5mm(0.5 in.) and widths to 1575mm(62 in.), resulting in ERW pipe diameters of 500mm(20 in.).

## References

1. International Symposium on Thin Slab Casting and Rolling (TSCR' 2002), Guangzhou, China, December 3-5, 2002, Chinese Society for Metals.
2. Continuous Caster Roundup-2001, Iron & Steelmaker, ISS, Warrendale, PA, Vol. 28, No. 11, 2001, pp. 36-46.
3. G. Flemming et al., Metall. Plant and Tech., Vol. 11, 1988, pp. 16-35.
4. Y. Li, et al., Proc. Materials Solutions Conference 2002, Columbus, OH, ASM International, (2002), 5-15.
5. A. J. DeArdo, "Nb in Modern Steels," International Materials Reviews, Vol. 48, No. 6, (2003), 371-402.
6. A.J. DeArdo, et al, in ref. [1], 194-210.
7. C. I. Garcia, et al., in ref. [1], 386-396.
8. Y. Li, et al., in ref [1], 218-234.
9. A. Ruiz-Aparicio, MS Thesis, University of Pittsburgh, 2004.
10. H. Hu, Recovery and Recrystallization of Metals, Interscience, NY, 1963, 311.
11. A.J.DeArdo, Microalloying '95, ISS-AIME, Warrendale, PA, 1995, 15.
12. I. Kozasu et al., Microalloying'75, Union Carbide Corporation, New York, NY, 1977, 120-135.