New process metallurgy developments for the production of steel wire rods

An advance in improving a 1080 grade steel by the addition of a micro-alloy was not warmly welcomed because of an incomplete understanding of its effectiveness. Tests at two plants later showed this method to be beneficial for products such as pre-stressed concrete wire rod, tire cord and medium-carbon spring steels.

By Steven G. Jansto

The MicroNiobium® Alloy Approach is applied in higher carbon steels and medium-carbon bar products exceeding 0.20% carbon to improve the mechanical property robustness of the steel via grain refinement. This mechanism allows grain refinement from billet to bar to wire rod to be more homogeneous, resulting in improved surface quality, increased productivity and reduced scrap rates throughout the process supply chain, thereby reducing the overall operational cost per ton. The niobium carbides and niobium carbonitrides also assist in pinning the austenite grains during the heating in the billet furnace, limiting abnormal austenite grain growth and inhomogeneous deformation during hot rolling. When operational combustion variations occur due to adiabatic flame temperature problems and/or air-to-gas ratio problems in the furnace, the presence of MicroNiobium in the steel often improves its robustness and minimizes the probability of austenite grain growth during processing of the billet in the reheat furnace.

A study was made to optimize the Nb composition in a 1080 steel to determine the best drawing ratio, reduction ratio, best ductility and surface quality and best final mechanical property performance at the end user wire processor. Based upon the results, it was possible to cross apply the same Nb optimization approach and procedure into lower-carbon bar products within the 0.70%, 0.50% C and 0.35% C chemistry range. The results indicate that the MicroNiobium addition of only 0.02% Nb provides an improved product robustness, reducing the cost of quality and improving productivity by as much as 10%. The results from this 1080 eutectoid wire rod study provides the fundamental metallurgical understanding that explains the improved properties. Based on these results, the same MicroNiobium methodology is applied for other carbon steels. Such a comprehensive study is not necessary as it is possible to cross apply the optimized low micro-Nb composition (0.005-0.020% Nb) for 1080 to the lower carbon steels (i.e. the 0.70%, 0.50% C and 0.35% C.) Thus, in these steels the two-fold benefit of MicroNiobium is: 1) prevention of abnormal austenite grain growth during reheat furnace aberrations, and 2) minimization of the interlammelar pearlite spacing improving mechanical properties.

Background information

Historically, Nb has not been the microalloy of choice in higher-carbon long product and wire-rod steels or even considered for that matter because of the predicted lower solubility of the Nb carbonitrides in higher carbon steels. Although there is lower solubility, current industrial applications validate the effectiveness of Nb in the grain refinement and precipitation strengthening mechanism in Nb-only and Nb-modified V-containing steels. Over the past two decades, within this higher carbon steel segment, metallurgical research studies did not typically show the positive results of Nb in high carbon grades. The reason was that in prior years, researchers incorporated Nb levels at greater than necessary concentrations leading to large carbides and consequently, found less than favorable mechanical properties. Also, the cost increased for these grades, making the approach not economically practical. Today, based upon this research, the economics favorably changed, supporting the MicroNiobium metallurgical approach globally for long products due to the optimized micro levels. Again, high-carbon Nb-metallurgy is different from low-carbon Nb-metallurgy due to the solubility differences and the process metallurgy considerations.

High-carbon steel process metallurgy (including melting and hot rolling) is infrequently reported from industrial producers. However, this research encompasses both laboratory heats and then validation with industrial heats.

What's noteworthy in this paper

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WJI: Did you start your research expecting that adding a micro-alloy would work, or were you also surprised by the results?

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WJI: Have you had any discussions with people in the industry who remain skeptical of your findings?

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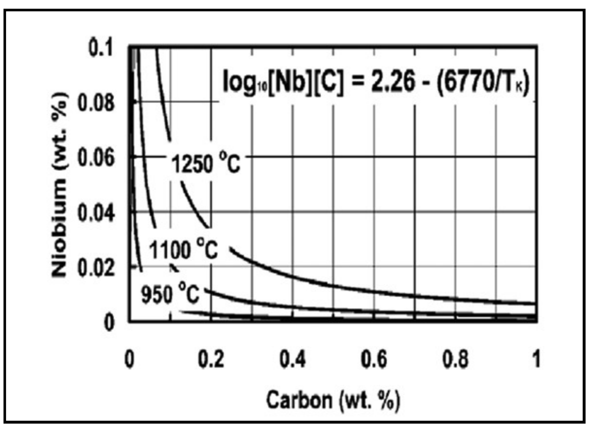

Fig. 1. Nb solubility versus C content[1].

Based upon operational experience, the optimization of the Nb content and the proper control of the reheat furnace temperatures are critical factors in producing such microalloyed high-carbon steel grades. An optimum Nb concentration has been directly correlated at a given carbon level based on the reheat furnace process metallurgy parameters, heating practices and combustion at a given mill.

The reluctance of some producers to consider the application of Nb in higher carbon steels (greater than 0.20% C) is depicted by the solubility relationship of Nb versus C content illustrated in Fig. 1.

A very short-sighted conclusion was then made: there would be no possible consideration or potential metallurgical benefit from even a very micro Nb addition approach. The conclusion was inaccurate because the Nb level was not optimized and the mechanism of Nb slowing down the diffusion of carbon in pearlite steels was not practically comprehended and correspondingly incorporated into the metallurgical operation.

This direction was stalled due to a lack of knowledge and a focus upon the powerful effect that niobium has when optimized as a micro addition. This situation blocked many researchers from realizing the prudent and positive metallurgical technological product development in this arena of metallurgy. Currently, with the optimization of concentrations at 0.005 to 0.020% Nb, several applications have been successfully applied in several medium and high carbon steel grades: 1) AISI 5160 and 9259 automotive coil springs, 2) AISI1050 automotive fasteners, 3) S500 earthquake/fire-resistant reinforcing bars, 4) 0.20% C abrasion resistant plates for heavy machinery and agricultural applications, 5) eutectoid steels for rail and pre-stressed wire rod, and 6) carburized steel power transmission components such as 4130 and 6250 grades[2].The 1080 eutectoid research work laid the foundation and groundwork and is presented in detail as an example of the Nb optimization and effect on properties.

	S0	V1	V2	VNB	NB1	NB2	NB3	NB4
C	0.829	0.778	0.776	0.781	0.811	0.770	0.805	0.791
Mn	0.756	0.707	0.731	0.730	0.760	0.635	0.736	0.723
Si	0.221	0.234	0.218	0.205	0.221	0.193	0.228	0.230
Al	0.004	0.003	0.002	0.002	0.005	0.002	0.008	0.005
Cr	0.144	0.138	0.152	0.150	0.152	0.124	0.156	0.157
Nb	0.002	0	0.002	0.021	0.023	0.038	0.087	0.119
V	0.002	0.092	0.053	0.050	0.003	0	0	0
N (ppm)	69	73	67	73	70	70	70	85

Table 1. Chemical compositions of experimental heats.

Niobium pearlitic transformation

Niobium is known to slow down the diffusion of carbon. Consequently, proper understanding of the kinetics and thermodynamics of the pearlite transformation in the presence of niobium can significantly influence the pearlite colony size, interlammelar spacing (distance between lathes) and final ASTM grain size. This inter-relationship between the grain size and mechanical properties is a core competency that a steel producer may offer to their customers. Although quite fundamental and simple, more uniform grain size and homogeneity translates into more uniform mechanical properties and more robust fabrication and ductility performance at a lower cost. Quite frequently, producers experiment with industrial trials that are higher in alloy composition than necessary which increases the cost and are thought to be required in order to improve ductility, fatigue and/or fracture toughness performance. The richer alloy compositions then become difficult to promote to the end user. Yet, grain size is often being compromised and alloys are being added to make properties. The materials engineer often over-designs the compositional changes, making more drastic changes than necessary in order to improve the properties, but in so doing, making the new materials design very costly, not competitive and both difficult to implement and convince the customer to change. However, if control of the grain size and furnace reheating practices were focused upon, properties could be achieved through implementation of a lean alloy chemistry at lower cost in these higher carbon steels. The missing link is the economics of grain size control. Fine homogeneous grain size is the main focus and quite powerful approach to reduce operating cost per ton and improved product margin for the steel mill. The MicroNiobium facilitates this grain size control.

As the grain size is reduced hardness (and yield stress) increases through the well-known Hall-Petch equation[3]. As yield / hardness increase, usually ductility decreases since dislocations have a higher number of boundaries to pile up into and the volume available for their movement is greatly reduced. Dislocations travel inside specific slip systems of the crystal lattice so in most cases when they meet a grain boundary they are blocked and stopped by the incoherent interface between the two grains. The only cases in which dislocations travel from one grain to the next is when there is a coherent interface between the two grains. Hall-Petch is the only classic strengthening mechanism that does not reduce ductility. Other mechanisms like strain hardening, mixed crystal or precipitation hardening can cause a reduction and reduce the ductility significantly[4-5]. For nano-grained materials the ductility can significantly increase, when the deformation behavior of the grain boundary dominates entirely over the bulk deformation. The improvement can often be expressed in an equation of the Hall Petch form. However, the appropriate use of grain refinement requires an understanding of the effective grain size that actually governs the mechanism of yielding or failure.

Several examples are presented in this paper where very minor chemistry changes can result in significant end user benefits, This MicroNiobium Approach has achieved this objective via only slightly modifying the chemistry and in some cases reduces the material and operational cost via the grain refinement mechanism resulting in more homogeneous ferrite-pearlite microstructure and improved mechanical properties.

Niobium optimization analysis in eutectoid pearlitic steel

Experimental laboratory heats of eutectoid high carbon steel were produced with Nb, V and Nb+V and then cast in a vacuum furnace. The major goals were to improve ductility of the eutectoid steel and realize increased productivity, improved reduction in area, less lamination and improved drawability. The objective is to compare the mechanical properties and drawability during wire rod production of a single microalloy addition of Nb against a V addition against a dual Nb+V combination. The compositions melted are shown in Table 1.

Laboratory ingots were then welded to industrial billets of similar carbon grades and hot-rolled at an industrial operation into 11-mm diameter wire rods following stan-

Wire Rod Sample	Yield Strength 0.2% (N/mm^2)	Tensile Strength (N/mm^2)	Reduction of Area (%)
SO	627	1086	30.4
V1	733	1154	26.8
V2	719	1154	22.9
VNB	740	1169	38.0
NB1	648	1139	45.1
NB2	680	1102	41.4
NB3	666	1115	38.4
NB4	709	1150	35.8

Table 2. Mechanical properties based on tensile tests.

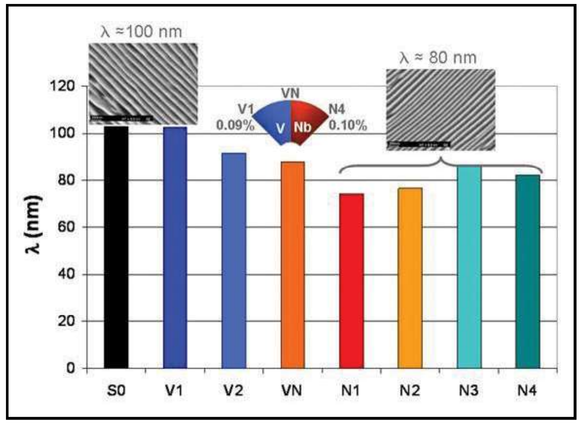

Fig. 2. Interlammelar spacing of pearlite in microstructure of as rolled wire rod

dard operational reduction and thermal practices for the production of pre-stressed concrete (PSC) wire rod. The wire rods were characterized by optical microscopy, scanning electron microscopic (SEM) examination and tensile tests. The pearlite interlammelar spacing was measured using the line intercept method procedure with the SEM. The austenite grain size was measured utilizing the NFA 04-102 standard method. The hot-rolled wire rods were then cold drawn in the laboratory. Their mechanical behavior was investigated through tension and torsion tests after each drawing pass.

Results

The microstructure of the laboratory produced wire rods are pearlite and homogeneous for all of the experimental steels. Optical observation did not reveal any negative influence form the Nb addition on the degree of segregation, decarburization and/or microstructure homogeneity. The mechanical properties of the wire rods are shown in Table 2.

Generally, the tensile strength levels are of the same order for the V-containing or Nb-containing steels, however, the Nb steel trend is a slightly lower yield strength. Nevertheless, all Nb-compositions exceeded the base steel S0 yield strength minimum of 627 N/mm^2 and the 1086N/mm^2 tensile strength. As expected, the combination of Nb+V exhibited the highest yield and tensile strength. However, the important attribute is the consistently higher % reduction of area (%RA) for the Nb-containing grades compared to the V-containing grades. Review of the mechanical property data shows the best elongation with Sample NB1 which is the 0.02%Nb MicroNiobium Alloy design at 45.1% RA. The yield strength, tensile strength and reduction of area is met as defined by SO in Table 2. The mechanism of this optimization of the Nb concentration in this high carbon eutectoid steel is the refinement of the lamellar spacing. The Nb is affecting the diffusion rate of the C and hence delaying the pearlite transformation. As shown in Fig. 2, the refinement of the lamellar spacing is the finest at 75 nm with 0.02%Nb directly linking the superior elongation. The optimized composition of NB1 exhibits nearly double the %RA. Fig. 2 illustrates the significant refinement of the pearlite. The NB1 composition exhibits the finest interlammelar spacing at 72 ɥm. **or 72 µm?**

The master rods were then drawn into the final pre-stressed wire rod product. Fig. 3 relates the reduction of area to the strain ratio of the initial diameter to the final product diameter. The optimum strain ratio for the industrial wire draw operation is between 1.50-1.80. The consistent 55% reduction of area (µ-Nb with the 0.02%Nb eutectoid chemistry) over this strain ratio range is excellent (i.e. flat) and results in increased productivity at the wire rod mill.

The evolution of the reduction of area with the strain is highest for the Nb-containing steels. This result indicates the improvement in ductility due to the Nb addition.

Industrial trials. Based upon the laboratory results an industrial heat was produced at a BOF operation. Subsequent to the evaluation of the laboratory heats and excellent results, an industrial heat was then produced with the optimized 0.018% Nb similar to the laboratory heat NB1 composition. The Nb heat was continuously cast into billets with no problems and the billet quality was excellent. Nineteen billets were hot-rolled into 7.5 mm wire rods. The chemical composition of the industrial heat is shown in Table 3.

Two sets of samples were taken from two different wire-drawing plants after each drawing pass. The drawing pass data were used to establish the work-hardening curves of the steel. The wire rods produced from the industrial heat have a higher Cr content, and lead to a higher tensile strength. The wire rods were shipped to two plants to manufacture strands for PSC applications. The mechanical properties of the wire rods were obtained from the beginning and end of each coil to check mechanical property consistency. The mean values of the ultimate tensile strength and the % reduction of area are listed in Table 4.

The 7.5-mm wire rod quality was evaluated. No axial segregation, surface damages or decarburization as observed.

C	Mn	P	S	Si	Cr	Nb
0.830	0.773	0.011	0.012	0.237	0.270	0.018

Table 3. Chemical composition of industrial heat

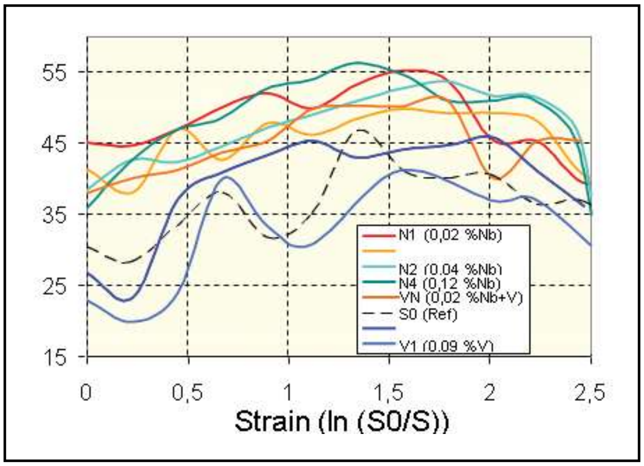

Fig. 3. Reduction of area versus strain.

UTS (MPa)		%Reduction Area	
Coil Head	Coil Tail	Coil Head	Coil Tail
1241	1253	44.0	43.7

Table 4. Mean values of ultimate tensile strength (UTS) and % reduction of area.

The 7.5 mm wire rod exhibited a homogeneous pearlitic microstructure, even at the core. The entire heat of coils was processed successfully without any rolling process modifications. The required tensile strength in the final product of 2160 N/mm^2 was achieved. The rolling process formed 2.4-mm drawn wires from the 7.5-mm wire rods. However, at one plant an extra drawing pass was applied, which led to even higher tensile strength without any surface quality, cracking or forming issues.

As discussed before, this transformation corresponds to a strain in the region of approximately 1.5 to 1.8. As a result of the higher Cr content, the work-hardening curves established from the plant-drawn rods shifted to the upper part of the graph compared to the NB1 curve. The drawing plant B curve nicely coincides with the laboratory produced NB1 curve. The difference between the curve for plants B and Plant A is due to the extra 20% reduction pass. A comparison of the % RA improvement with Nb versus V shows a 30% improvement for Plant A and a 20% improvement for Plant B, respectively. Both plants experienced a 10% improvement in productivity due to the higher % RA.

Wire rod production observations and overlooked trends

The consistent manufacture of high-quality finished wire rod products begins with the reheating of the billets at the reheat furnace. This paper does not discuss continuous casting that directly affects internal and external billet quality. For the purpose of this work, assuming a consistent incoming billet, the authors will focus on a few operational areas that are a priority to facilitate high-quality, cost-effective rolling of the billets. The reheating process has a profound effect on the final hot-rolled steel quality and mechanical property consistency. The uniformity of heating applied across the entire width and length of the slab or billet is critical to achieve customer properties regardless of the chemistry. The resultant ferrite grain size in the final hot-rolled product is significantly governed by the initial prior-austenite grain size. There are numerous reheat furnace process metallurgy and combustion parameters in the actual operation that affect mill productivity, scrap rate and diverts. This reheating step in the steelmaking process often receives low priority in the evaluation of product quality and mechanical property performance, especially the toughness through the plate thickness. In laboratory studies, the furnace-heating step is typically quite uniform, resulting in a homogeneous and fine prior austenite grain size. Unfortunately, in industrial operations, it is much more difficult to control the uniformity of heating along the entire length and through the thickness of the work piece. This factor is often not given high priority when evaluating mechanical property diversions on an industrial scale. The furnace conditions are correlated to product quality via furnace process variables such as the air-to-gas ratio, furnace burner condition, furnace pressure, energy efficiency, adiabatic flame temperature (AFT) and furnace refractory condition. Corrective actions and operational practice recommendations are presented in order to minimize inhomogeneous heating that results in inferior product quality and toughness variations in the through-thickness-direction.

This inhomogeneity of heating becomes the root cause for variations in the final hot0rolled microstructure, grain size and mechanical properties, especially toughness. Hence, since such inconsistencies in furnace heating are often ignored in research studies, many researchers incorrectly assume and conclude that the composition or role of a given microalloy is the problem if poor mechanical property performance is experienced, when in fact the furnace-heating practice is the root cause. Furnace heating practice relates: 1) reheat top and bottom zone set points, 2) soak-zone temperature set point, 3) actual reheat top and bottom zone and soak zone temperature, 3) times in each zone related to push rate, 4) furnace pressure, 5) combus-

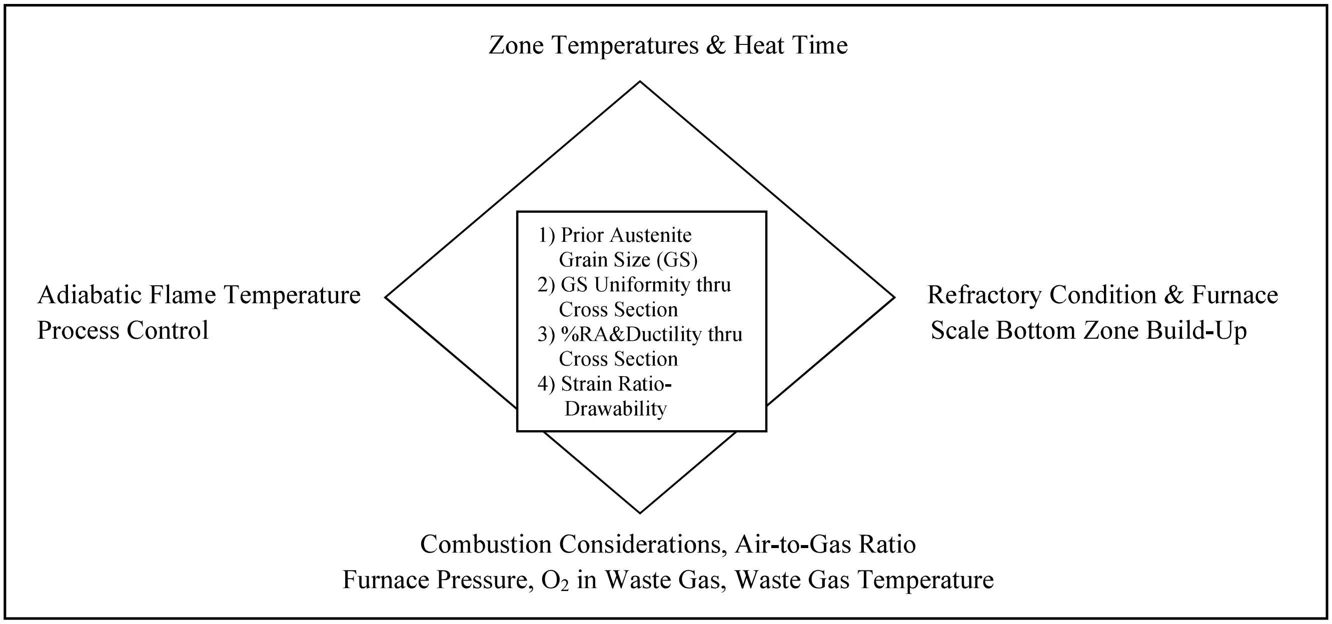

Fig. 4. Furnace process control variables, prior austenite grain size and properties[6].

tion air flow, 6) air-to-gas ratio and 7) overall refractory and furnace maintenance[6].

This process metallurgy link between reheat furnace operational variables, resultant efficiency of heating and soaking, PAGS and final hot-rolled ferrite grain size is a critical quaternary relationship that warrants more in-depth study and analysis. See Fig. 4. Finally, this connection and methodology between combustion effectiveness, metallurgy and its effect on austenite grain size is emphasized with the objective of improving quality and mechanical property consistency.

The reheat procedure for slabs, billets and blooms before hot rolling is a fundamental and critical step in the hot-roll process. The combustion and the heat transfer process influence the deformation schedule, recrystallization and the resultant mechanical properties of the final hot-rolled product. In the reheat furnace, the slab is actually heated via radiation off the refractory walls of the roof and side-walls. The roof and sidewall refractory absorb heat from the flames emitted from the combustion burners. The AFT is affected by the fuel type, burner efficiency and air-to-gas equivalence ratio. The highest adiabatic flame temperature translates into higher heat input, higher production throughput and maximum furnace efficiency. The optimum air-to-gas ratio also develops a furnace atmosphere that is conducive for good surface quality, high-heat penetration into the slab and optimal scale depth and viscosity. Fig. 5 shows the effect of different air-to-gas ratios (i.e. equivalence ratios) on the adiabatic flame temperature for different gases.

Since most furnaces consume natural gas, the maximum adiabatic flame temperature of 2250°K occurs at approximately 1.10 equivalence ratio. Under these conditions, approximately one-half of the heat generated from the combustion of the fuel heats the steel. As the equivalence ratio increases, combustion efficiency declines and AFT decreases. There are several operational reasons that adversely increase the equivalence ratio. The following furnace factors can create higher equivalence ratios:

- cracked burner orifice plates leading to sub-optimal flame temperature;
- refractory cracks in furnace roof and/or sidewall leading to air infiltration into the furnace;
- low furnace pressure due to inefficient combustion fan mechanical performance (bearings, out-of-balance, component wear);
- reduced working volume in the bottom zone of preheat and reheat section due to scale buildup;
- improper dilute oxygen enrichment at combustion burner tip;

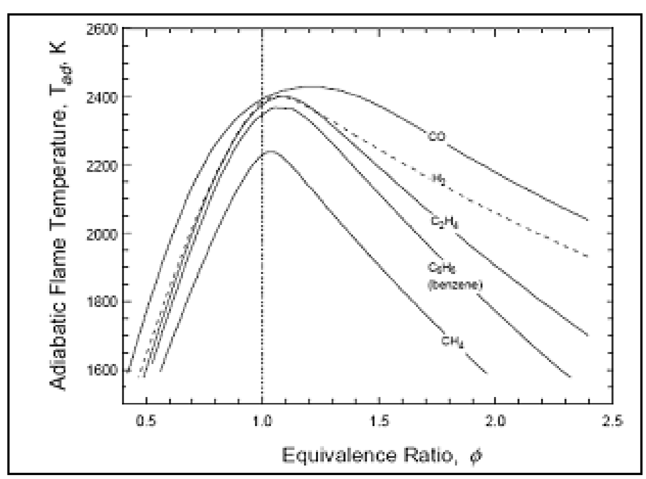

Fig. 5. Air-to-gas equivalence ratio versus adiabatic flame temperature[7].

- Scale formation and viscosity;
- Numerous furnace operations throughout the world operate at both high reheat (>1150°C) and soak zone furnace temperature (>1225°C), thereby overheating both plain carbon steels and microalloyed steels leading to abnormal grain growth. Observations made at numerous mills around the world find high-temperature furnace operation even more prevalent on higher carbon steels exceeding 0.20%C. The cause-and-effect relationship of these poor furnace-heating practices have a detrimental effect on steel quality due to abnormal and variable grain size and inhomogeneous heating through the slab thickness.

Most mills perform the heating and soaking of the billets in pusher-type furnaces, with the balance heated in walking-beam furnaces. Although in some cases, these are correct reasons for problems, there are occasions where these causes are secondary or tertiary and can be offset via some operational adjustments. These adjustments can compensate for these problems and assure that quality steel is still heated and rolled despite these obvious problems until the appropriate maintenance can be scheduled and executed. Often, such operational adjustments are often ignored or not even considered, thereby costing the mill productivity, quality and increased cost of production until the maintenance is executed.

Conclusions

The application of niobium (Nb) in high-carbon steels enhances both the metallurgical properties and processability of products. Such process and product metallurgical improvements relate to the Nb-pinning effect of the austenite grain boundaries in microalloyed 0.25 to 0.95%C steels minimizing abnormal grain growth during the reheat furnace process prior to rolling. Consequently, Nb microalloyed medium- and high-carbon, long-product steel applications have been developed. The MicroNiobium Alloy Approach is described and correlated to a variety of medium- and high-carbon steel grades and applications. Development opportunities and applications span automotive coil springs, eutectoid rail steels, alloy tool and die steels, automotive fasteners, reinforcing bars, wire and tyre rod. The detailed analysis of the optimization at .018% Nb in 1080 eutectoid pre-stressed concrete wire rod provided the basis for this metallurgical strategy. This approach contributes to the achievement of desired ultra-fine grain, homogeneous high carbon steel microstructures that exhibit superior toughness, strength, fatigue performance, less mechanical property variation in the final hot rolled product, reduced cost of quality and improved weldability. Cross application of 0.005 to 0.020% Nb provides for more robust long products via austenite grain boundary pinning during reheating and very fine interlammelar pearlite spacing during hot rolling. The improvement in the cost of quality far exceeded the additional alloy cost for the MicroNb addition.

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