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MicroNiobium Alloy Approach in Medium and High Carbon Steel Bar, Plate and Sheet Products

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The recently developed application of the MicroNiobium Alloy Approach® in medium and high carbon steel long product, sheet and plate steels enhances both the metallurgical properties and processability as well as reducing the operational cost per tonne of production. The process and product metallurgy improvements relate to the Nb-pinning effect of the austenite grain boundaries. The metallurgical mechanism of the MicroNiobium Alloy Approach is related to the retardation of austenite grain coarsening during reheat furnace soaking of the billets, slabs or shapes before rolling. Variable grain size is induced by temperature fluctuations and inhomogeneity during the heating of the slabs in the reheat furnace. Such fluctuations can occur due to variations in the air to gas ratio, directly affecting the adiabatic flame temperature and heat input into the slabs. Also, in the case of heat treatment and carburizing, higher processing temperatures can be applied to the finished components, thereby reducing process time and increasing productivity. The MicroNiobium Alloy Approach (0.005-0.020%Nb) has been applied in high carbon (AISI1050 grade) automotive and long product steel and plate applications, such as fasteners, seismic resistant rebar, pre-stressed concrete wire rod and pressure vessel plate applications. The MicroNiobium Alloy Approach mechanism is described and correlated to a variety of medium and high carbon applications. Specific case examples involving pressure vessels, automotive coil springs, eutectoid steels, alloy tool and die steels and tyre rod are discussed. This approach contributes to the achievement of an ultra-fine grain, homogeneous higher carbon microstructures that exhibit superior toughness, high strength, less mechanical property variation in the final hot rolled product and reduced cost of quality. The reduced cost of quality far exceeds the additional alloy cost for the Nb addition.

Keywords: Austenite Grain Growth, Carburizing, Niobium, Reheat Furnace Variation

1. INTRODUCTION

The evolution of the MicroNiobium Alloy Approach relates to stabilization of the abnormal austenite grain growth that occurs during inhomogeneous overheating of the steel slabs, billets or blooms during the industrial reheat furnace operation. The MicroNiobium Alloy Approach mechanism relates the integration of the physical metallurgy of abnormal grain growth, the Nb carbon nitride pinning effect and the influence of the process metallurgy of reheat furnace temperature practices, variability and thermal inhomogoneity. The connection between inefficient slab, billet and/or bloom reheat performance and the consequential result of mechanical property variability in the final hot rolled product due to

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variations in grain size is often not related in both the laboratory and especially, in industrial operations. Transfer of these reheat time and temperature data from the laboratory experiment to actual industrial furnace operation are quite difficult to incorporate into the to the mill model.

Inordinate amounts of time and metallurgical resources study the chemistry or TMCP rolling regimes and thermal practices as the root cause of the problem in not achieving desirable mechanical properties. However, the root cause problem often relates to the inhomogeneity of the initial furnace reheat operation and thermal condition of the slab before hot rolling. This lack of homogeneous heating results in variations in austenite grain size which translates into increased cost of diverts and scrap during the hot rolling operation as well as variability in the final ferrite grain size and resultant mechanical properties. These additional costs of quality are enormous.

Over the past two decades, within this higher carbon steel segment, when Nb-microalloy metallurgical research studies were investigated for higher carbon steels, Nb-researchers incorporated higher Nb levels than necessary and inconclusive performance and conclusions resulted.

The mechanism of abnormal grain growth is well established and documented.[1] However, the relationship between reheat furnace temperature, solubility of the alloying elements at different carbon contents in actual industrial operations is difficult to correlate with laboratory studies and generated data. The traditional approach has been to maximize the solubility of niobium (Nb) in carbon steels to ensure the alloy's effectiveness in grain refinement, recrystallization kinetics and subsequent precipitation, especially in low carbon steels. Investigations of the effect of Nb in steel at very low concentrations has been quite limited.[2] In addition, due to more limited solubility of Nb in these higher carbon steels (exceeding 0.20%C), the application of Nb has traditionally been dismissed, considered impractical and therefore, not a cost effective microalloy addition solution. However, recent research and industrial trials have disproved this universal conclusion.

2. MICRONIOBIUM APPLICATIONS

Most recently, the MicroNiobium Alloy Approach at Nb concentrations of 0.005 to 0.020%Nb have been successfully applied to the following medium and high carbon steel grades and applications; 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, 5) eutectoid steels for rail and pre-stressed wire rod, and 6) carburized steel power transmission components such as 4130 and 6250 grades.

For example, within the eutectoid steel applications, there is an optimal concentration of Nb (between 0.010 to 0.020%Nb that refines the lamellae spacing of the pearlite in order to achieve improved fatigue and fracture toughness in rails and improved drawability in the fabrication of pre-stressed concrete wire rod. The pearlitic refinement that is experienced in actual production on the bar mill occurs at a lower Nb concentration in these higher carbon steels than under laboratory conditions. Reasons for this anomaly are being investigated, however, the billet furnace reheat conditions and uniformity of heating is a major source for this deviation based upon actual production experience compared to controlled laboratory furnace conditions.

In carburized steel power transmission engineering alloy medium carbon steel product sector, there is currently significant interest in the high temperature carburizing treatment of forged gears. The trend has been to increase carburizing temperatures (approaching 1100°C) resulting in shorter carburizing times, increased productivity and reduced overall energy costs. There is some limited research that concludes an increase in heating rate may result in finer austenite grain sizes at the onset of carburizing.[3] Niobium has been tested at different concentration levels and indicate that the NbC contribute to a more desirable austenite grain size.

The implications of these faster heating rate in a carburizing operation has direct application in changing the manner in which heating and soaking zone schedules are designed for slab and billet reheating furnaces. Essentially, a faster heating rate is proposed in the preheat and early sections of the top and bottom heating zones of the furnace. However, once the steel temperature approached 1050 to 1100°, prior to the soak zone hearth section of the furnace, the heating rate is decreased. This temperature range coincides with the onset of precipitate coarsening of the microalloy carbides. Hence, a more gradual heating rate and a lower soak zone temperature will reduce carbide precipitate coarsening of and hence, increase their effectives in the pinning of the austenite grain boundary. Industrial metallurgical research indicates that the majority of the austenite grain coarsening occurs in the soak zone of the reheating furnaces.

3. MICRONIOBIUM MECHANISM

Two Nb strategies are typically employed in practice depending upon the intended purpose; 1) the Micro-Niobium Alloy Approach which simply minimizes the austenite grain size coarsening during reheating through the addition of 0.005 to 0.020%Nb and 2) the TMCP approach at higher Nb levels for the purpose of grain size stabilization, complex precipitation strengthening and thermomechanical processing. The TMCP Approach is primarily applied to lower carbon steels (less than 0.20%C)

The focus in this paper is the influence of the MicroNiobium Alloy Approach on the austenite grain coarsening mechanism and application. In most cases to date, Nb has not been the microalloy of choice or even considered for that matter in high carbon equivalent steels because of the predicted lower solubility of the Nb precipitates in higher carbon steels. This relationship is illustrated below in Figure 1.

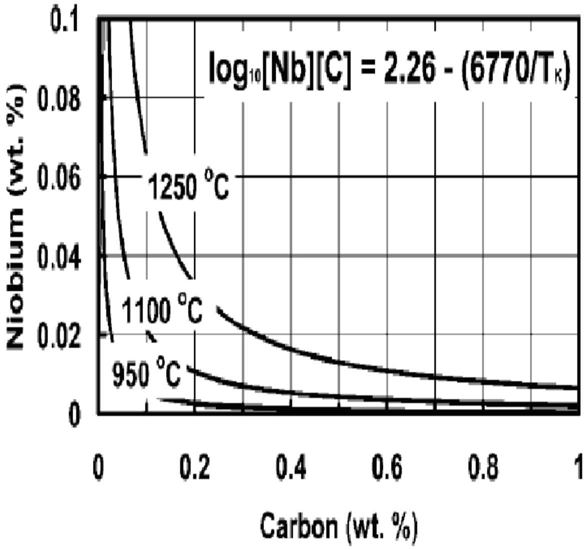

Fig 1 Limited Solubility of Nb in High Carbon Steels

Although there is lower solubility in higher carbon steels, current industrial applications have validated the effectiveness of low concentrations of Nb for the purpose of preventing abnormal austenite grain growth in Nb-only and Nb-modified-V containing steels during furnace reheating.

The observed initiation of grain growth at approximately 1150°C, for both normal and abnormal grain growth, is related to the coarsening and dissolution of precipitates. The time and temperature dependence of the Nb, V and or Ti precipitate pinning forces to the dissolution kinetics of the microalloy system directly affects the grain size before hot rolling. There are basically two modes of grain growth: normal and abnormal.[1]

During normal grain growth, sometimes referred to as continuous grain growth, the distribution of grain sizes will remain approximately constant. Continuous growth is typical for plain carbon-manganese (C-Mn) steels. The grains increase their diameter continuously and gradually, displaying a monomodal grain size distribution. The grain coarsening of C-Mn steels occurs between 900 and 1200°C following the well known continuous grain-growth law:

$$D = kt^a \exp(-b/T)$$

where k, a and b = constants

D= average grain diameter; t = time; T = temperature

In contrast, during abnormal grain growth or secondary recrystallization, sometimes called discontinuous grain growth, particular grains grow significantly while others remain small. Discontinuous growth is typical for aluminum killed and microalloyed steels. The grain growth process is suppressed up to a certain temperature and then, a sudden increase in austenite grain size takes place at a given temperature. It is this discontinuous grain growth condition where the MicroNiobium mechanism is operable exhibiting the Nb-pinning effect of the austenite grain. The Nb-carbonitride precipitates that are dissolved in atomic clusters or exist as Nb(C,N) nano-precipitates in the austenite grain, will suppress the grain boundary motion in these higher carbon steels. Niobium, titanium and vanadium are the most popular microlloys of choice in the steel industry in lower carbon steels, with titanium and vanadium traditionally added in the higher carbon steels historically.

Vanadium bearing steels typically resist grain coarsening up to approximately 1000 to 1050°C. Niobium bearing steels resist grain coarsening at temperatures between 1100-1150°C. Titanium is reported to prevent coarsening up to 1200°C. However, the problem experienced with titanium is that the grain boundary pinning is through the precipitation of the cuboidal titanium nitride precipitates formed during the casting process. Thus, although effective in pinning the austenite grain growth, these large cuboidal TiN precipitates create stress risers in the steel during hot rolling. In the final medium and higher carbon products, they also result in lower toughness and fatigue properties compared to Nb or V-containing high carbon steel. In contrast, the Nb and/or V-carbonitrides are spherical and more coherent with the austenite grains during the reheat furnace process and with ferrite grain matrix during and after hot rolling. For example, Figure 2 below compares the cuboidal Ti,Nb(C,N) precipitate morphology with the spherical Mo, Nb(C,N) precipitates in medium carbon steel.

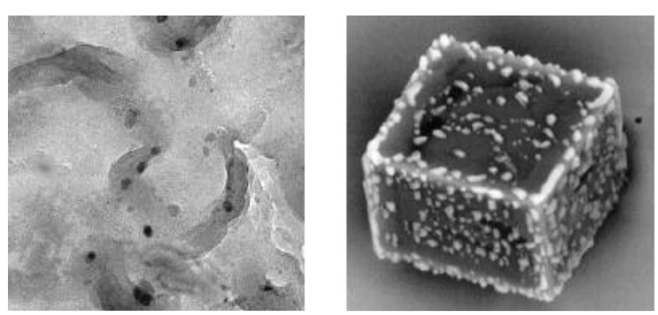

Fig 2 Mo.Nb(C,N) (left) and Ti,Nb (right) Precipitate Shape

The Mo,Nb(C,N) precipitates are spheroidal and typically 3-5 nanometers versus the cuboidal TiN which is approximately 30-40 nanometers with very fine Nb(C,N) precipitates (in white) in the TiN cube.

The sharp corners of the TiN precipitates act as a stress riser in the ferrite matrix of the final hot rolled product. The implications are that under conditions of tensile stress-strain, impact loading at low temperature and/or low/high cycle fatigue conditions, the sharp corners of the cuboidal TiN precipitates act as a stress riser within the ferrite matrix. Under such loading conditions, the stress field around the corner initiates a crack. Under higher stress and fatigue cycles cracks will propagate. Certainly, with spherical nano-precipitates pinning the austenite grain, the stress field around the precipitate-ferrite interface region is lower.

Certainly, this methodology of analysis is under further study in medium and high carbon steels as the literature and research in this topic is limited and offers opportunities for the scientific community to further study. However, the pinning of austenite grains via titanium additions is a concern due to its propensity to create higher strain fields around the precipitate than spherical precipitates.

4. MICRONIOBIUM 1050 EXPERIMENTAL INDUSTRIAL METHODOLOGY CASE STUDY

The adaptation of the MicroNiobium Approach in these higher carbon steels has been implemented on an industrial scale. A 1050 sheet steel was selected which represented a high volume product for the automotive fastener industry. Figure 3 below outlines the industrial relevance and cost-benefit considerations to justify the experiments.

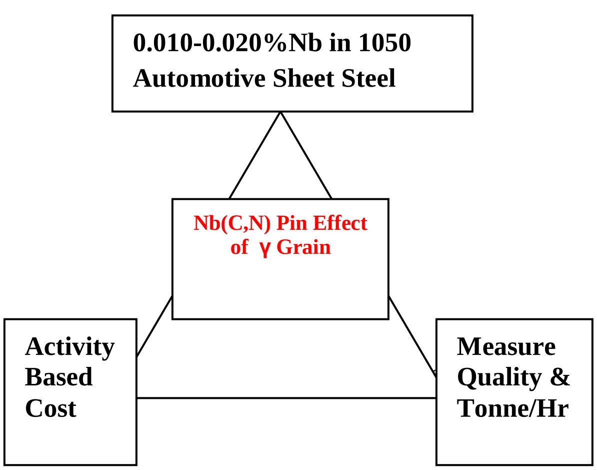

Fig 3 Industrial 1050 MicroNb Automotive Sheet Implementation Methodology

A 30 day time period was selected to introduce MicroNb alloy additions in the range of 0.010-0.020%Nb in all AISI 1050 steel industrial heats. No change was made in the melting, reheating furnace or hot rolling mechanical or process metallurgy practices.

The performance assessment executed an Activity-Based Cost (ABC) comparison methodology which measures the tonne per hour hot rolling production rate, the percent of diverts for flatness, shape and mechanical properties and the cobble scrap rate for the MicroNb heats versus the historical non-Nb heats' performance. This ABC methodology is highly recommended for all MicroNb medium and high carbon experimental validation and justification. ABC has proven to be highly effective in terms of gaining an accurate understanding of the cost reduction, implementation time and quality impact.

In the comparison of the MicroNb to the non-Nb historical performance, the steel operation experienced a minimization of abnormal austenite grain growth which translated into a finer ferrite grain microstructure. The measured improvement parameters were:

1. Improved processability and rollability at the mill
2. Reduced cobble rate
3.Reduced diverts for poor quality
4. Increased production rate (i.e. tonne per hour)
5. Reduced mechanical property variability

These attributes resulted in an approximate 3-5% reduction in operational cost per tonne and a lower Total Activity Based cost of production for the MicroNb 1050 steel compared to the non-Nb 1050 steel grade.

The Industrial 1050 MicroNb Automotive Sheet Implementation Methodology described is a case study for global research and operational metallurgical community to analyze the cost-benefit in medium and high carbon steels. The critical success factor is that no change in heating or rolling practices (such as reduction schedules, discharge temperature, roughing, finishing and coiling temperatures were made. Each mill performs their respective metallurgical and operational analysis. Since the %Nb levels are so low, the recrystallization mechanism is just initiating (≤5%) or not even measurable in these higher carbon grades. Thus, no change in reduction schedules is necessary making this development quite easy to implement into operations.

5. REHEATING FURNACE OPERATION AND ITS EFFECT ON AUSTENITE GRAIN SIZE

The quality and efficiency of the reheating process has a profound effect on the austenite grain size and uniformity of grain size along the entire length of the slab. This step in the steelmaking process often receives low priority in the evaluation of product quality and mechanical property performance. The homogeneity and efficiency of heating is highly influenced by the air to gas ratio of the furnace burner combustion condition. The optimum air to gas ratio of 1.10 yields the highest adiabatic flame temperature. However, often in actual operations, cracked burner orifice plates, poor burner tuning and inefficient combustion fan performance contribute to variations in the air to gas ratio. These situations have a huge effect on the optimal adiabatic flame temperature performance. Effectively, variable adiabatic flame temperature means variations in the heat input to the steel and the austenite grain size. For example, in the case of a furnace firing with natural gas (CH_4), the adiabatic flame temperature is shown as a function of the air to gas ratio in Figure 4.

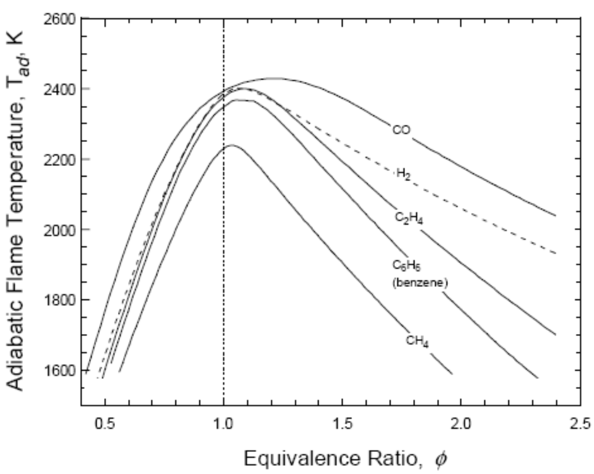

Fig 4 Air to Gas Equivalence Ratio versus Adiabatic Flame Temperature

The highest adiabatic flame temperature translates into higher throughput and maximum furnace efficiency. The optimum air to gas ratio also develops an atmosphere in the furnace that is optimal for good surface quality and scale formation. As the air to gas ratio decreases, the adiabatic flame temperature decreases and then, the iron oxide scale thickness increases, which acts as an insulating layer on the slab surface, reducing the slab heat conduction efficiency. This variation in the heating process will significantly affect the resultant thermal homogeneity and gradient from the surface of the slab to the center of the slab, as well as the austenite grains size and distribution.

Periodic checking and resetting of air-fuel ratios for burners is one of the simplest ways to get maximum efficiency out of fuel-fired process heating equipment such as furnaces, ovens, heaters, and boilers. Most high temperature direct-fired furnaces, radiant tubes, and boilers operate with about 10% to 20% excess combustion air at high fire to prevent the formation of dangerous carbon monoxide and soot deposits on heat transfer surfaces and inside radiant tubes. For the fuels most commonly used by U.S. industry, including natural gas, propane, and fuel oils, approximately one cubic foot of air is required to release about 100 British thermal units (Btu) in complete combustion. Exact amount of air required for complete combustion of commonly used fuels can be obtained from the information given in one of the references. Process heating efficiency is reduced considerably if the combustion air supply is significantly higher or lower than the theoretically required air.

Therefore, discontinuous austenitic grain growth is directly influenced by such thermal variation conditions within the furnace caused by variable air to gas ratios. The adiabatic flame temperature variation with the air to gas ratio (Fig. 4) and the effect of temperature on the prior austenite grain size is illustrated below in (Fig. 5).[4] The connection between the process metallurgy and the physical metallurgy is made.

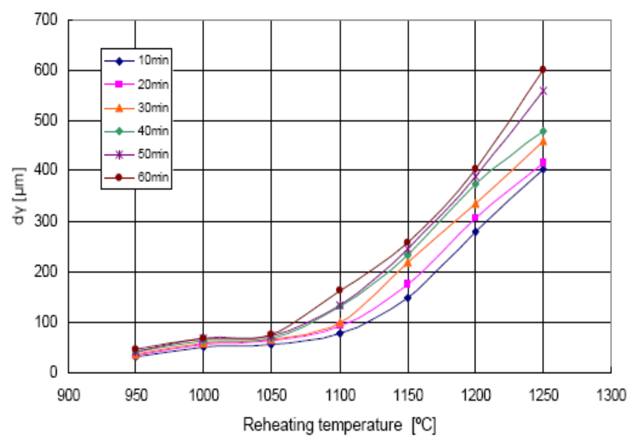

Fig 5 Austenite Grain Size vs. Reheating Temperature

For example, the relationship between the air to gas ratio and the resultant austenite grain boundary may be correlated with the integration of these two figures (Fig. 4 and Fig. 5). The furnace operational process metallurgy can be converted to the reheating temperature of the slab and then into the estimated austenite grain size. For example, if one section of the slab is at 1200ºC and the adjacent section is 1225°C due to an air to gas variation 0.05; then it follows that the austenite grain size would be approximately 325µm for the 1250ºC section versus the adjacent section at 280µm grain size for the 1225°C region. Such differences in prior austenite grain size due to such thermal variations in combustion lead to a variable ferrite size in the final hot rolled product and hence, variable mechanical properties.

6. REHEAT FURNACE OPERATIONAL THERMAL VARIATIONS INFLUENCING AUSTENITE GRAIN GROWTH AND FINAL GRAIN SIZE

The process metallurgy link between reheat furnace operational variables, resultant efficiency of heating and soaking, prior austenite grain size and final hot rolled ferrite grain size is a critical quaternary relationship. Based upon mill industrial mill trials and operational implementation studies, the following diagram is presented to link the process metallurgy to the physical metallurgy. Often, successfully making the connection between industrial process metallurgy operational variables and the resultant physical metallurgy of the microstructural characteristics, precipitate morphology, precipitate chemistry and final properties is considered to be highly complex. It is certainly a challenge, but is quite simplified when one connects the process metallurgy to the physical metallurgy. Recent product development activity comparing different pinning elements such as Nb, V, Ti and Al have been studied. Figure 6 below schematically represents several of these process/physical metallurgy connections.

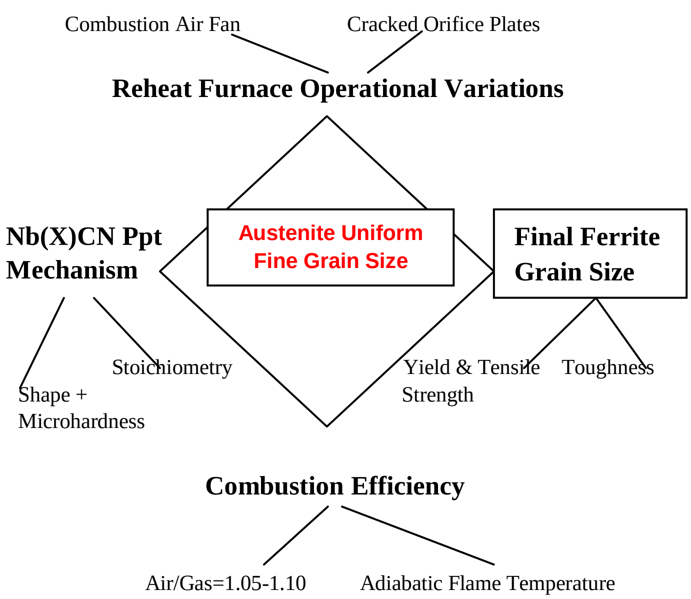

Fig 6 Physical Process Metallurgy Connection

This process metallurgy-physical metallurgy connective methodology, represented as PM$^{2®}$, must be carefully considered when relating the precipitate pinning elements and the desired mechanical properties such as toughness, fatigue strength and fracture toughness. Although an element such as titanium can effectively pin the austenite grain during furnace reheating, one experiences deterioration in toughness due to the cuboidal morphology of a TiN or a cuboidal TiN with spherical Nb(C,N) precipitating on the cuboidal TiN. The root cause of the lower toughness is due to the cuboidal TiN and not the Nb(C,N) spherical precipitation on the TiN cubes. Therefore, future alloy design must closely consider the influence of these different microalloy carbonitride precipitates on the through thickness toughness properties of steels, especially on the intermediate and heavy thickness applications.

New research is being conducted in multi-microalloy precipitate pinning combinations such as Nb,V(C,N) in traditional high carbon V-bearing long products at very low Nb concentrations (as low as 0.005%Nb). It is this specific process metallurgy-physical metallurgy relationship that is retarded in the presence of a MicroNb Alloy Approach mechanism based on actual operational hot roll performance. The Nb(C,N) precipitates (and/or atomic clustering the instant before precipitation of these 3-5 nanometer precipitates) provide the pinning action delay in austenite grain growth in this specific region of the overheated slab.

7. MICROALLOY AUSTENITE PINNING ELEMENT OF CHOICE

The relationship between reheat furnace temperature, furnace heating and soaking time, solubility of the alloying elements at different carbon contents in actual industrial operations is very difficult to correlate with laboratory studies and generated data. It is well known that addition of Al [5], Nb [6] and Ti [7] is effective to depress abnormal grain growth because of the presence of their carbide or nitride as pinning particles. These elements have been traditionally used for this purpose. However, often many research studies to-date will change the austenite condition simultaneously as the precipitation condition changes. Thus, the independent influence of the precipitation condition on abnormal grain growth has not been comprehensively studied with conclusive evidence.

The observed initiation of grain growth at approximately 1150°C, for both normal and abnormal grain growth, is related to the coarsening and dissolution of precipitates. The time and temperature dependence of the Nb, Al and or Ti precipitate pinning forces to the dissolution kinetics of the microalloy system directly affects the grain size before hot rolling. Especially, for Nb, there are only few reports about the effects of precipitation condition although Nb is known as an effective element to depress abnormal grain growth for high temperature carburizing, and recently, the practical use is being spread widely. Future research will be focused upon the tertiary synergy between the inherent characteristics of the microalloy of choice behavior, the cost of alloy and production improvement savings and the variables affecting furnace reheat efficiency (shown below in Figure 7).

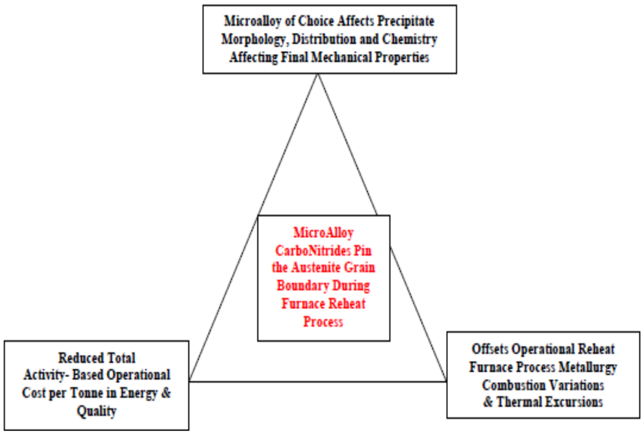

Fig 7 Microalloy choice, cost and furnace variation

The Activity Based Cost (ABC) methodology is a subset of the comprehensive ABC methodology applied to the entire steel operation. In the comprehensive ABC methodology, the primary drivers of the total operating cost per ton of steel are comprised of the following cost components: 1) alloy, 2) BOF/EAF conversion, 3) reheat furnace gas consumption, 4) plate mill/hot strip mill conversion, 5) cobbles, 6) diverts and 7) electrical energy consumption. [8] The BOF/EAF costs are linked to the charge model which will calculate forecasted cost and then make an adjustment based upon an actual operating data variance report resulting in an adjusted cost/tonne. The natural gas cost (mmbtu per tonne) is the actual consumption of the given hot rolled steel order. In this grain growth analysis, subset elements of the comprehensive ABC methodology are utilized.

The reduction in the total activity based cost through the grain boundary pinning mechanism with the proper chosen microalloy and an increased emphasis on furnace control and practices translate into energy savings and reduced emissions at the reheat furnace. Through a reduction in the variations of the furnace operational parameters previously discussed, the improved efficiency at the furnace offers the opportunity to consider lower soak zone temperatures. Industrial experience has revealed as much as a 20 - 35°C reduction in furnace operating temperatures with disciplined furnace practices. With such a reduction in furnace soak zone temperature, the specific energy consumption can be reduced by approximately 0.05 to 0.10 mmbtu/tonne. For example, at a 2.0 million tonne per year operation, at today's energy prices, operational energy cost is reduced by $1.0 to 2.0 million per year. The intangible benefits of reduced carbon, NOX and SOX emissions are experienced. Also, at these lower furnace temperatures, less scale forms and yield improves.

7. CONCLUSION

The validation of the application of 0.005-0.020%Nb in the 0.20-0.95%C steel segment is a metallurgical approach to assist industrial operations in minimization of the random abnormal grain growth experienced during adiabatic flame temperature fluctuations due to air to gas ratio variations.

The influence of furnace operational parameters and root cause analysis of the factors contributing to these aberrations significantly affect energy consumption, cost and product quality. The PM^2 methodology is vital in the root cause analysis of process metallurgy parameters and deviations which directly affect the physical metallurgy characteristics, such as austenite and ferrite grain size, the mechanical properties (i.e. strength, fatigue, fracture toughness and impact properties) and the overall production cost.

On a micro-scale, the function of the Nb(C,N) nanoprecipitation (and/or atomic clustering) pins the austenite grain for a sufficient time during the reheat process such that the ferrite grain size is more uniform in the final product. The results are validated by the 1050 industrial automotive fastener experiments and the economics are proven through the Activity Based Cost analysis methodology. The MicroNb Approach has been introduced into several other medium and high carbon industrial plate and bar products and is projected to grow substantially as a result of its very favorable cost benefit value added relationship, thereby reducing producers' operational cost per tonne and providing a more consistent performing product to the end user.

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