Microalloying concepts and application in long products

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A strategy for use of microalloyed steels in long products, including bar and forging steels, is presented based on alloying and processing characteristics applicable to this class of steels. The approach, which defines in temperature/composition space specific composition and processing temperatures for different product types, helps guide selection of microalloy additions for specific applications. Results of several recent product developments characteristic of thermomechanically processed and heat treated bar steels are also presented to illustrate opportunities available for the expanded use of microalloy additions in long products.

Keywords: Microalloying, Long products, Forging, Thermomechanical processing, Heat treating

Introduction

Microalloy additions to steels have been instrumental to the successful development of new steel products with enhanced property combinations. The extensive use of microalloyed flat rolled steels in pipeline plate products and automotive sheet steels, typically with microalloy additions of less than several hundredths of a weight percent, has become commonplace. To date, use of microalloy additions in long products has been comparatively more limited and significant opportunities exist for the development of new steels tailored for specific applications and designed to reduce manufacturing costs and balance properties with performance.1–3 Thus, the focus of this paper is to present an overall alloy design approach for the development of microalloyed long products and examples of recent applications.

Perspectives on microalloy design philosophy for long products

In long products, the range of alloying and processing characteristics and applications is very broad, and the microalloying strategy depends on the specific long product class of interest. Consider, for example:

(i) direct cooled (i.e. as rolled or as forged) ferrite–pearlite steels strengthened by pearlite volume fraction, ferrite grain size and substructure, and Nb and V based precipitates

(ii) through hardened and surface induction hardened heat treated steels with primarily martensitic or bainitic microstructures

(iii) surface modified and heat treated carburized steels.4

These different material classes may involve substantial differences in carbon content and processing temperature, as well as deformation history and final microstructure. These factors fundamentally influence the applicable microalloying concepts, and it would be worthwhile to have a simple framework available to understand and develop effective microalloying and processing strategies for these products and to understand and avoid strategies that may be less likely to be fruitful in a particular application.

Microalloy elements of primary interest include the strong carbonitride formers Nb, V and Ti; interactions with Al may also be important via AlN effects. While solute effects can occasionally be important,5,6 microalloying elements usually form carbides, nitrides, or carbonitrides, and contribute by restricting the movement of either dislocations or interfaces, i.e. through precipitation strengthening or grain refinement. Both of these mechanisms are enhanced by fine particle sizes, so the design and implementation of microalloyed long products involves tailoring the composition and processing to employ the requisite precipitation mechanisms under appropriate composition and processing constraints. As a result of the number of critical elements for consideration in microalloy steel development, the alloys are often complex and the final properties are derived through careful control of both alloy content and thermal–mechanical processing history. It is well understood that the temperature ranges in which carbides, nitrides and carbonitrides form and dissolve determine whether or not a given microalloying design is appropriate for a given application,7 and the framework presented below is based partly on this understanding.

For each application or material class, the overall alloy/processing design is based on application needs (section size, strength, toughness, etc.) which drive specific microstructure preferences. For example, lower carbon as rolled steels with primarily ferritic microstructures are typically used in reinforcing bar constructional steels, while much higher carbon heat treated steels with martensitic or fine pearlitic microstructures are employed in carburised gear surfaces or high strength wire rod respectively. Intermediate carbon
levels are used in other components (e.g. heat treated springs, shafts, etc.), where a balance of strength and toughness is required. Figure 1 summarises, in schematic temperature/composition space, regions applicable to a variety of typical products and provides the framework for understanding long product microalloying approaches. From a processing standpoint, low carbon steels employed in the as rolled condition will usually be reheated to rather high temperatures (e.g. 1250°C) before rolling, while heat treated steels will usually be reaustenised at rather low temperatures (e.g. 900°C) before quenching. Forging usually involves relatively high reheat temperatures before deformation, although the allowable heating temperature is also dependent on alloy composition, particularly carbon concentration. In this context, it should be recognised that the liquidus temperature is strongly dependent on carbon concentration (decreasing at increased carbon levels) and liquation must be avoided during reheating. Similar phase stability considerations can also be important in austenitising, whereby austenite formation may be completed at much lower temperatures in higher carbon steels (such as during wire patenting of near eutectoid compositions).

The schematic framework illustrated in Fig. 1 is intended to highlight the substantial variations in carbon content, processing temperatures and microstructure that are applicable to long product applications. These variations have profound implications to microalloying strategy. The reheating temperature in combination with the steel composition will control the extent of microalloy dissolution and consequently, the amount of microalloy addition available for subsequent precipitation. Fundamental solubility relationships control these behaviours. Precipitate dissolution and precipitation behaviours in microalloyed steels have been reviewed extensively elsewhere and based on equilibrium thermodynamics, the solubility of a precipitating compound such as NbC, V(C,N) or TiN as a function of microalloy and interstitial concentrations and temperature can be predicted. Figure 2 presents calculated solubility diagrams at selected temperatures for NbC (Fig. 2a), VC (Fig. 2b) and VN (Fig. 2c), based on solubility product expressions, shown in each figure, selected from the literature. Solubility isotherms are shown where applicable for reheating or austenitising temperatures in the range discussed above, i.e. from ~900 or 950 to 1250°C, for the range of carbon levels up to 1 wt-% (or 0.025 wt-%N in the case of VN). The solubility isotherms in Fig. 2a show that NbC solubility is strongly dependent on temperature and carbon content over the ranges of interest, so clearly the hot rolled low carbon reinforcing bar steels may employ substantial niobium levels if sufficient reheat temperatures are used. In higher carbon steels, Nb solubility is much lower (on the order of 0.01 wt-% in eutectoid steels such as railroad rails or high strength wire products). In such steels, Nb can be used only sparingly for the purposes of precipitation in austenite or ferrite during processing, although somewhat higher levels have also been employed to suppress austenite grain growth. Intermediate Nb levels may be viable in applications of medium carbon steels.

NbC precipitates are expected in Fig. 2a at reaustenitising temperatures in virtually all heating treating applications of long products, thereby enabling strategies for austenite grain refining (as heat treated) or thermomechanical treatment (as rolled or as forged).
remaining in the austenite may also contribute to subsequent formation in ferrite of finer strengthening precipitates. Vanadium is well understood to exhibit considerably greater solubility than Nb, and the solubility isotherms in Fig. 2b and c show that carbides and nitrides (or carbonitrides) of vanadium are only expected in austenite at the lowest temperatures in ‘richer’ alloys containing relatively high levels of V, C and/or N. Thus, vanadium may be less likely to enable grain refinement or thermomechanical processing in ‘leaner’ alloys. Nonetheless, the extensive solubility of vanadium in austenite enables substantial precipitation strengthening capabilities (via precipitation in ferrite) over a wide variety of steels across the entire spectrum of carbon concentrations and processing temperatures relevant to long products. For example, vanadium additions up to several hundredths of a weight percent can usually be considered in low nitrogen steels and nitrogen additions can also be employed to help drive precipitation in some cases, even in higher carbon steels.11 Titanium nitride solubility is shown in Fig. 2, but TiN is very stable and usually precipitates at high temperature in the austenite regime and may resist subsequent coarsening. TiN is often avoided because TiN is very hard. The stable TiN particles that form at high temperatures may be detrimental to machinability or toughness and usually, contribute little to strength, although small Ti additions are sometimes effective to suppress grain growth at high temperature.7

The framework for long products related to processing temperature and alloy composition discussed in the context of Fig. 1 may be illustrated further in the context of microalloy solubility, which is also controlled by temperature and composition. Figure 3 summarises, using the axes of Fig. 1, the relationships discussed between precipitate type, solubility, composition and temperature. For example, TiN is very stable and usually precipitates at high temperatures in the austenite regime and resists subsequent coarsening. Vanadium carbonitride, V(C,N), is much less stable and is in general easily dissolved during austenitising and precipitates at lower temperature. Niobium has intermediate solubility and may be employed as either a solute or precipitate in austenite (to influence the austenite thermomechanical processing response), or as a fine strengthening precipitate in ferrite. A direct comparison of Figs. 1 and 3 (or perhaps even better, an overlay of the two figures, not shown here) further clarifies how different microalloy design philosophies are applicable to different product types. As rolled or as forged steels employ intermediate to high reheat temperatures with moderate carbon levels for as rolled construction applications (e.g. reinforcing bar) or higher carbon levels for as forged applications requiring high fatigue strength. Higher carbon forgings are associated with reduced microalloy carbide solubility in combination with lower reheat temperatures, so Nb levels must be limited, while V additions may be more substantial. Consequently, vanadium microalloying is predominant in these steels, although dual additions of V plus low Nb levels are growing in importance. At high reheat temperatures, Nb solubility is greater and significant levels of either Nb or V may be usefully employed, particularly at lower carbon levels where Nb solubility is greater. Niobium offers added potential in as rolled or as forged applications for ‘austenite conditioning’ as part of the thermomechanical processing strategy, in which Nb may contribute by precipitating preferentially on the deformed austenite substructure, suppressing austenite recrystallisation, thereby refining the final microstructure. Warm working may further enhance properties2,13,14 and there is opportunity to develop novel thermomechanical strategies to achieve substantial additional microstructural refinement, as discussed further in an example below.12 Steels that are heat treated after rolling or forging may employ microalloying for austenite refinement, or for precipitation strengthening of the as transformed or subsequently tempered microstructure. Here, complete microalloy dissolution at the austenitising temperature may be undesired in some applications where an array of precipitates which are both fine and coarsening resistant is helpful to suppress austenite grain growth. Nb and Ti are therefore most readily applied for this purpose and their limited solubility at low austenitising temperatures precludes substantial precipitation strengthening or secondary hardening (or resistance to softening during tempering) in the final microstructure.7 Vanadium is more soluble at these temperatures and may be considered as a potential strengthenener even at low austenitising (or carburising) temperature or high carbon levels. Again, nitrogen additions may enhance the effects of vanadium and competition between aluminium and vanadium for the available nitrogen should sometimes be considered.

The preceding paragraphs have outlined a framework for understanding microalloying of long products and summarise the essential alloy and process design strategies that may be employed in various bar and forging steels to control the interactions between chemical composition and processing, and their influences on microstructure and properties that control performance. Application of these strategies to specific components involves tailoring a particular design to meet the property and processing requirements of the application.14 These microalloying strategies are increasingly employed to increase strength and performance, or to reduce the number of heat treating steps while maintaining adequate
performance (e.g. substituting as forged for heat treated microstructures). In the remainder of this paper, selected specific applications which represent the products identified in Fig. 1 are highlighted to illustrate application of the concepts summarised above. Emphasis is placed on thermomechanical processing and heat treating.

Application examples

Thermomechanical processing of microalloyed bar steels

Forgings

Thermomechanical processing (TMP) is commonly used to process flat rolled products and has greatly improved product performance, e.g. fine grained high toughness pipeline steels. With advancements in system control technology, along with use of microalloy additions, opportunities exist to also utilise thermomechanical processing to enhance long products, or components produced from long products (e.g. forgings). A recent study by Boyd and Zhao on a 0.41 wt-%C forging steel (alloy 1541) modified with 0.014 wt-%Ti and 0.114 wt-%V or 0.039 wt-%Nb illustrates the significant improvements that can be realised in TMP microalloyed forging steels. Both hot (deformation in austenite) and warm (deformation in ferrite) forging schedules were designed to assess the interrelationships between precipitation, austenite recrystallisation behaviour, substructure stabilisation and precipitation after forming. The results of this study are presented in Fig. 4 which compares as forged yield strengths and room temperature Charpy V notch energies and yield strengths for thermomechanically processed microalloyed 1541 forging steels (open symbols, Nb steel and filled symbols, V+Ti steel): data are shown for conventionally forged steels (triangles) and TMP processed steels either air cooled (diamonds) or furnace cooled (squares), along with warm forged Nb steel (circle). Adapted from Ref. 12

Reinforcing bars

Significant advances in the production of hot rolled reinforcing bars have been realised through the use of microalloyed thermomechanically processed steels. Property requirements for weldable reinforcing bars, critical elements in concrete structures, have evolved with the need to produce structures with improved performance, e.g. resistance to damage in earthquakes and corrosion resistance. The increased demands on final properties with tighter tolerances have necessitated that reinforcing bars be produced with improved controls of both alloying and processing, and currently, multiple reinforcing bar grades are available with yield strengths up to 600 MPa. Following development of the Temcore process, higher strength grades have most commonly been produced by partial ‘quenching’ immediately following hot rolling and self-tempering to produce products with high strength outer layers and lower strength, higher toughness cores. More recently, microalloyed reinforcing bars have been developed by direct cooling off of the hot mill to produce products with uniform properties. To maintain required toughness, microalloyed reinforcing bars are low carbon (≤0.3 wt-%) and can be produced by different routes, depending on rolling temperatures and cooling technologies employed. Reinforcing bars based on V and Nb additions have been developed. Figure 5 compares the contributions to yield strength, for two vanadium grades with those of a typical Mn+Si grade. Data are shown for steels with 0.11–0.12 wt-%V and two
Consistent with consideration of the solubility diagram in Fig. 2, VN precipitation existed in solid solution, then at lower temperatures on the runout table, VN precipitation contributes due to grain size refinement for the steel. Owing to essentially complete V dissolution at all reheat temperatures.

 Significant improvements in strength in the vanadium grades are derived by a slight increase in strength due to grain refinement, but with significant increases in strength due to VN precipitation, with an increase in precipitation strengthening increment in the high nitrogen grade. In all steels, the solid solution increment primarily reflects slight differences in Si and Mn contents. A consideration of the VN solubility diagram in Fig. 2c suggests that if the V bearing alloys were rapidly cooled from a finish rolling temperature of above 950°C where most of the vanadium existed in solid solution, then at lower temperatures on the runout table, VN precipitation would occur to produce a fine precipitate dispersion leading to the high strength increment observed. This process also produces a 30–50 MPa increase in strength due to grain refinement.

Higher strength reinforcing bars based on Nb or Nb + V additions have also been developed. Owing to the presence of Nb, the final properties depend sensitively on reheating temperature and thus, the degree of dissolution, before rolling. Figure 6 compares strength contributions due to grain size strengthening and precipitation hardening as a function of reheating temperature in a 0.25C–1.2Mn–0.5Si–0.05Nb steel with the same steel without Nb. Consistent with consideration of the solubility diagram in Fig. 2a, to realise the strength increases due to precipitation, the steel must be reheated above 1100°C to ensure sufficient Nb dissolution. Then, the steels must be rapidly cooled from 900 to 700°C, to control the subsequent precipitation potential by maintaining a sufficient concentration of solute Nb in solution to form fine Nb precipitates on subsequent cooling to room temperature. Addition of 0.045 V to the Nb modified alloy results in a ~50 MPa additional strength increase for all reheat conditions, owing to essentially complete V dissolution at all reheat temperatures.

### Microalloyed bar steels for heat treating applications

**Automotive springs**

Heat treated components fabricated from bar steels have recently experienced significant improvements due to the use of microalloyed steels, often in conjunction with thermomechanical processing. One specific example for automotive coil springs is considered here. New lightweight automotive designs require higher strength, lighter weight spring designs with good fracture toughness, fatigue resistance and performance at higher operating stresses. Traditionally, heat treated automotive springs have been based on SAE 5160 (0.6C–0.86Mn–0.29Si–0.83Cr) or on SAE 9259 (0.61C–0.82Mn–0.78Si–0.51Cr) steel. Recently, Head et al. developed microalloyed springs based on V (0.6C–0.81Mn–0.85Si–0.51Cr–0.1V and 110 ppm N) and V + Nb (0.51C–0.69Mn–1.31Si–0.45Cr–0.12V–0.035Nb and 120 ppm N) approaches. The steels were hot rolled, hot formed, cooled and heat treated by austenitising at ~940°C, oil quenched and tempered, and Fig. 7 compares the effects of tempering temperature on the final hardnesses of all four steels. These data show that the microalloyed steels achieved superior hardnesses and improved stability against tempering in comparison to the conventional alloys, and the highest tempered hardnesses were obtained for the Nb + V steel. The microalloyed steels also exhibited improved fracture toughness and fatigue properties, both critical to spring performance. To achieve the improved properties, TMP was employed to facilitate suppression of austenite grain growth by Nb(C,N) during rolling and heat treating leading to ASTM prior austenite grain sizes of 9–10 in comparison to 6–8 for the conventional spring steels. In addition, V precipitation as V(C,N) during cooling and tempering most likely provided an additional strength increment the Nb + V steel. Use of the TMP microalloyed steels successfully enabled a new coil spring design allowing for a 15% increase in operating stress with a 23% decrease in weight.
Heat treated components from direct cooled ferrite–pearlite steels

Components for automotive engine and transmission systems have been manufactured from microalloyed steels for over three decades and these components, used after direct cooling from forging temperatures without heat treatment, have successfully replaced low alloy quenched and tempered steels and cast irons. Figure 8 correlates ultimate strength with carbon content for plain carbon and microalloyed steels and shows the type of systematic test data that can be used to evaluate the suitability of microalloyed steels for a given application. The increased strength due to microalloy additions is due to grain size refinement, pearlitic content and precipitation hardening.

Steels with compositions characteristic of those in Fig. 8 are now being considered for selected heat treated applications, particularly involving induction hardening to replace materials in parts fabricated from alloy steels that often require independent heat treatments to control surface and core hardnesses. Successful material replacement requires a two step design process as illustrated for the following example of material replacement in a clutch race, a component in an automatic transmission fabricated from a thick walled hot rolled tube. The parts, nominally 110 mm in diameter, require a minimum surface hardness of 58 HRC after tempering with a core hardness of 24–30 HRC. To achieve the specified hardness after tempering, an as quenched hardness of 61–62 HRC is required. The required carbon content to achieve the specified surface hardness is 0.5–0.55 wt-% as predicted from a correlation of as quenched martensite hardness with carbon content. The potential for producing a 0.5–0.55 wt-% C microalloyed ferrite–pearlite steel with the desired core hardness range, which correlates to UTS values of 830–1000 MPa (Ref. 24), is obtained from Fig. 8 by extrapolation of the data as shown. The desired core hardness of a 0.5–0.55 wt-% C steel can easily be obtained in a V or V + Nb modified microalloyed steel. Following the process outlined here, a successful cost effective material substitution was made which eliminated one heat treating step.

High temperature carburising

Currently, there is significant interest (e.g. forged gears) in high temperature carburising (potentially, up to 1100°C), which has distinct advantages over lower temperature carburising with regard to potential productivity increases and cost savings. Higher processing temperatures can, however, lead to undesirable mechanical properties resulting from difficulties in controlling surface carbon content, case depth and austenite grain growth. Recently, benefits obtainable with the use of microalloy additions, along with thermomechanical processing, to produce carburising steels with greatly improved microstructures and properties have been identified. Alloying approaches for improved grain refinement via precipitates of Al, Ti, V and Nb (Refs. 7 and 26–29) have had varying effectiveness depending on alloying level, austenitising temperature, heating rate and processing history. At high temperatures (e.g. 1150°C) where only TiN is stable, TiN precipitates retard grain growth resulting in grain refinement. At lower temperatures (927°C), TiN particles are less effective as grain refiners, in part because the effects of other precipitates (e.g. AlN) may control grain growth. However, TiC is more appropriate for moderate temperature grain refinement (1000°C). V is highly soluble in austenite and as a result, is ineffective as an austenite grain refiner. For higher temperature carburising steels, new steel alloys based on microalloy additions of Ti and Nb to produce specific precipitate dispersions that are effective in minimising austenite grain growth have been designed. Alloying concepts and application in long products

8 Tensile strength as function of carbon content and microalloying showing extrapolation (arrow) of data for microalloyed steels to crosshatched region which indicates desired final core properties in heat treated transmission component. Adapted from Ref. 21

9 Niobium carbide solubility diagram showing NbC solubility isotherms at temperatures within austenite regime: Nb and C levels in modified 8620 steels of AIOgab et al. are shown as filled circles.
be encountered reheating before bar rolling.\textsuperscript{27} The niobium and carbon levels in the Nb added grades are shown in the solubility diagram of Fig. 9. Because of the modest carbon levels in carburising grades (e.g., \(1\%\) \(\sim 0.2\) wt-\%) combined with the potential for high reheating temperatures, sometimes approaching 1300°C, the highest Nb level selected was quite substantial. The solid lines parallel to the dashed line indicating a stoichiometric Nb/C ratio show that the carburising grades all contain ‘excess carbon’ and that solute niobium levels should be very low when NbC precipitation is complete. For these steels, NbC would be completely dissolved in the low Nb (0-0.2 wt-\%) steel at temperatures of \(\sim 1100\)°C, whereas the precipitates are stable to much higher temperatures at the higher Nb levels.

AlOgab \textit{et al.}\textsuperscript{27,32,33} characterised the precipitate dispersions and austenite grain growth behaviour in detail as a function of composition and processing, including heating rate and carburising temperature. Figure 10 shows grain growth behaviour over a range of pseudocarburising temperatures and heating rates after hot rolling. The Nb added alloys exhibited substantially finer prior austenite microstructures at low carburising temperatures and after low heating rates. The beneficial pinning effects of NbC persisted to elevated temperature for the higher Nb steels, while abnormal grain growth in the lower Nb steels was observed as a consequence of reduced particle fractions. An increase in heating rate was shown to result in finer austenite grain sizes at the onset of carburising, which subsequently contributed to enhanced grain growth.\textsuperscript{32,33} The 0.1Nb steel offers the potential for suppression of grain growth at temperatures up to \(\sim 1050\)°C, which is substantially higher than is typical for conventional gas carburising treatments. Such an increase in carburising temperature could enable productivity increases on the order of \(40\%\),\textsuperscript{34} owing to the increased carbon mobility in austenite (i.e. reduced carburising time to achieve a given case depth) at elevated temperature. AlOgab\textsuperscript{32} also examined effects of thermomechanical processing history and carburising heat treatment parameters. Significant effects of low temperature finish rolling (i.e. controlled rolling) on the NbC precipitate distribution and subsequent grain coarsening response during carburising were noted. While such effects are well known in flat rolled products, thermomechanical processing of microalloyed austenite is less common in heat treated bar products.

The significance of austenite grain size distributions on the fatigue properties of high temperature carburised steels was evaluated by Thompson \textit{et al.}\textsuperscript{28} with the same Ti/Nb modified 8620 steels. Selected bending fatigue results (applied stress \(v.\) cycles to failure) are shown in Fig. 11 for alloys processed at two different indicated heating rates.\textsuperscript{28}
steels, due to suppression of abnormal austenite grain growth during carburising. This series of studies establishes the potential for microalloying to enable elevated temperature heat treating of bar and forging steels in applications sensitive to austenite grain growth. Similar studies are underway in other laboratories\textsuperscript{5,33,35} and additional efforts are needed to establish the optimum array of pinning precipitates, although it appears clear that substantial quantities of niobium can be effective in these applications.

**Summary**

A strategy for use of microalloying in the development of new applications for long products was presented to illustrate the interrelationships between product type, carbon content, processing temperature and microalloying addition, both type and amount. Selected examples of specific developments and product application were chosen to illustrate recent applications of microalloying technology in products with properties controlled by thermomechanical processing and/or heat treating. These examples, in conjunction with several other developments not cited here, confirm that significant opportunities are available to improve long products through applications microalloying based alloy and process designs.

**Acknowledgement**

Support is gratefully acknowledged from the sponsors of the Advanced Steel Processing and Products Research Center, an NSF Industry/University Cooperative Research Center at the Colorado School of Mines.

**References**