# Nb-MICROALLOYED "FIRE-RESISTANT" CONSTRUCTION STEELS: RECENT PROGRESS

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#### Abstract

Fire-resistant constructional steels have been commercialized in some parts of the world and are now poised for application in the USA, as a specification has been issued recently by the American Society for Testing and Materials (ASTM) that defines testing and material requirements. Selected metallurgical studies are reviewed, to help understand the microstructure/property relationships that control fire resistant (FR) properties in Nb-containing steels. Specific examples are cited which illustrate the apparent benefit of Mo in suppressing precipitate coarsening rates at elevated temperature and beneficial effects of microstructure refinement, microalloy precipitation, and warm working of ferrite on the FR properties. The concept of "active" fire resistance is illustrated (confirmed with both Cu and Nb containing steels thus far), whereby alloying and processing are designed to allow strengthening precipitates to form in the microstructure as a consequence of the heating encountered during a fire. The requirements of the new ASTM specification A1077/A1077M-12 are also summarized.

#### Introduction

"Fire-resistant" steels have been developed for construction applications where increased elevated temperature strength provides enhanced protection to a building structure during a fire. Improved fire protection helps to prevent building collapse caused by reduced load carrying capability of steel structures at high temperature, or provides the building occupants greater time to escape the building in the event of such a collapse. This paper is intended to provide an updated overview of some activities related to the development and implementation of fire-resistant steels (FR steels or FRS) in the USA [1]. First, some research results from the Advanced Steel Processing and Products Research Center (ASPPRC) at Colorado School of Mines will be presented to illustrate activities that have been conducted to understand the physical and mechanical metallurgy of these steels, ie. the factors controlling microstructure/property relationships under conditions relevant to FR steel applications. Second, since progress has been made recently in adopting specifications for fire-resistant steels and testing procedures in the USA, some comments will be presented to summarize the new American Society for Testing and Materials (ASTM) specification.

While interest in fire safety increased after the collapse of the World Trade Center towers following the airplane crashes and ensuing fires on September 11, 2001 in New York City, building codes and specifications for steels used in building construction have included firerelated characteristics for decades. Structural fire protection is addressed by the American Society for Testing and Materials standard E119, "Standard Test Methods for Fire Tests of Building Construction and Materials." ASTM E119 is not a material specification to designate property requirements at elevated temperature, but rather specifies a test method to assess protection from undesired thermal excursions, and thus is largely a test of heat transfer characteristics. For example, the average temperature of a steel assembly is required to remain below 1000 °F (538 °C) for vertical columns, thereby ensuring that the steel maintains "sufficient" strength. This temperature criterion, rather than a loading criterion, was apparently established in part because of the closure of important facilities for testing structural columns at elevated temperature at the National Bureau of Standards (now the National Institute of Standards and Technology) and Underwriters Laboratories (an independent, not-for-profit organization concerned with the safety aspects of potentially hazardous designs [2]). Since the specifications are most relevant to thermal characteristics and not material properties at elevated temperature, there was less incentive in the USA for constructional steels with improved characteristics at elevated temperature to be developed and implemented. This situation was in contrast to some other parts of the world, where material specifications and building designs evolved to incorporate improved strength retention at elevated temperature. In Japan, for example, fire protection was assured in a 1969 requirement that structural steels not exceed a temperature of 350 °C [2]. It was considered that the yield strength of conventional steels at 350 °C was about 2/3 of the specified values at room temperature<sup>1</sup>, and so the fire-resistant steels developed more recently and produced in Japan for the past several years guarantee a minimum yield strength at 600 °C that is 2/3 of the room temperature yield strength, ie, having a minimum yield strength ratio<sup>2</sup> of 2/3.

The temperature sensitivity of yield strength is illustrated schematically in Figure 1, comparing a "general" constructional steel and a FR steel having equivalent room temperature properties. The figure illustrates the greater strength retention of FR steel at elevated temperature, and its ability to maintain a strength level at 600 °C that exceeds 2/3 (or ~67%) of its room temperature yield strength. Some other design codes cite yield strength ratios of 50% at 600 °C [3]. While it should be noted that alloy steels have been developed with even better elevated temperature properties (creep strength, oxidation and corrosion resistance, etc.), for load-bearing applications where the steels are *routinely* employed at elevated temperature, FR constructional steel developments have necessarily incorporated a more restrictive economic constraint in the cost/performance trade off. Elevated temperature properties are only one consideration in the overall performance requirements for steels used in building construction, where cost is a critical factor and elevated temperature exposure is only a rare and essentially "accidental" occurrence.

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<sup>&</sup>lt;sup>1</sup> It should be recognized that some steels maintain their strength or may be stronger at 350 °C than at room temperature due to strain aging effects of interstitial atoms.

<sup>&</sup>lt;sup>2</sup> The yield strength (YS) ratio is the ratio of elevated temperature yield strength to room temperature yield strength.

The disclosure and publication of developments in FR steels for building construction overseas led to some renewed interest and discussion in this field in the USA, stimulating initial interest in these steels in the late 1990s at the Colorado School of Mines ASPPRC. Much wider interest in FR steels resulted from activities following the catastrophic collapse of the World Trade Center towers in New York City in 2001. Interest was stimulated by the recognition that exposure of structural steel to elevated temperatures contributed to the collapse, that new opportunities may exist to develop and employ new steels, that FR constructional steels were already developed elsewhere in the world, and by the need for accurate data on elevated temperature properties for use in simulations of the building collapse [4]. As a consequence, industry in the USA has addressed this interest through the activities of an ASTM joint task force. Publishing activity indicates that fire-resistant steels and related building design are also of significant current interest in China.

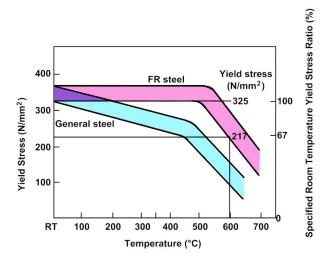


Figure 1. Schematic comparison showing improved elevated temperature strength of FR steel. (Adapted from [2], with permission).

Industry activity related to ASTM standards development considered both material requirements and the test methodology to characterize the elevated temperature properties required for fire-resistant steel. Potential testing protocols included elevated temperature tension testing, accelerated creep testing (involving determination of plastic strain rates under isothermal conditions of constant temperature and load application), along with a temperature-ramp or constant-load test (involving evolution of strain during non-isothermal conditions, ie. when heating under conditions of constant load application). Studies to examine the relationship between various test methods indicated a correlation between creep and elevated temperature tensile behavior [5]. As a result, product quality testing focused on existing methods for elevated temperature testing.

The temperatures experienced during real fires are a function of the combustible materials present and their combustion rate, the compartment geometry, ventilation conditions, and thermal properties of the structure [6]. Fire models are available to assess the required level of fire safety, and to utilize fire-resistant steels in building designs to increase fire protection, or reduce/eliminate other forms of active or passive fire protection such as sprinklers, insulation, etc. [6-8]. Implementation of FR steels has already occurred in some niche applications, and the specification of properties at a 600 °C design temperature allows these steels to be employed without fireproofing in spacious structures with lesser amounts of combustibles, such as open parking decks, atria, sports or station buildings. In other building structures where fires are more likely to become fierce and higher steel temperatures are expected, the use of FR steels should still provide a benefit in terms of reduced fireproofing [7,9-12].

## **Development of Fire-resistant Steels**

Fire-resistant steel developments have perhaps been led by activities in Japan, although publications are also available from Europe, China, Korea, and the USA [eg. 13-21]. The steels are intended to resist accelerated creep, or thermally activated deformation. The term "accelerated" creep is used here to distinguish the fire-resistant steel application from other creep-sensitive applications involving exposure to high temperatures and stresses for much longer durations (months or years) than apply to building fires, where locally elevated temperatures are more commonly encountered over time periods lasting up to a few hours. The goal in fire-resistant steel development should be to employ strengthening mechanisms that maintain greater effectiveness at elevated temperature, thus providing resistance to softening. It should be noted that long-duration creep behavior involves a different deformation regime, and some of the understanding of creep strengthening mechanisms is less applicable to the "accelerated" creep regime applicable to fire-resistant steels. Consequently, much of FR steel development has been somewhat empirical thus far, directed toward meeting specific performance attributes, and there remains an opportunity to develop further understanding of basic mechanisms to provide the tools for more efficient steel alloy and process design optimization in the future. Alloying philosophies to develop fire-resistant steels usually attempt to stabilize the initial starting microstructure and maintain effectiveness at elevated temperature of the strengthening mechanisms employed to meet the low-temperature structural requirements, by minimizing recovery, particle coarsening, grain growth, etc. An alternate approach might be to follow a "smart materials" design philosophy whereby alloying and processing are controlled to condition the initial microstructure so that additional strengthening mechanisms are activated during a fire, and some results related to this concept are mentioned below.

In general, FR steels are modified versions of high strength constructional steels, usually employing microalloying technology, along with Mo additions that contribute further to the elevated temperature properties. A variety of alloys has been reported in the literature, with an emphasis on Mo- and Nb-containing low-C, low-alloy steels. Here we will review selected research from programs at the Colorado School of Mines that were conducted to understand the behavior of FR steels, with an emphasis on Nb effects and alloying/processing principles. A series of three studies began early in 2001, involving a limited number of low C steels, including a base C-Mn alloy, a Nb-containing steel, Mo + Nb and V + Nb steels, and a Cu-containing steel with further additions of Ni, Cr and Mo along with Nb and V. The Cu-containing steel was more

highly alloyed and not intended for direct comparison to the other steels, but was rather used to explore some fundamental precipitation effects that are readily controllable via Cu additions. The chemical compositions of these steels are shown in Table I.

Allov C Mn Cu Ni CrMo Nb Al N Base 0.11 1.16 0.018 0.19 0.25 0.08 0.17 0.02 0.004 0.001 0.002 0.01 0.10 1.06 0.005 0.27 0.39 0.16 0.09 0.047 0.001 0.021 0.003 0.016 Nb Mo+Nb 0.10 0.98 0.008 0.30 0.38 0.15 0.096 0.48 0.017 0.004 0.01 V+Nb 0.005 0.27 0.32 0.11 0.13 0.047 0.021 0.003 0.01 0.08 1.13 0.036 Cu 0.06 0.99 0.005 0.27 0.98 0.75 0.51 0.50 0.06 0.02 0.035 0.007

Table I. Chemical Compositions (wt.%) of Experimental Steels

Elevated temperature yield and ultimate tensile strength results for four of these steels are shown in Figure 2. The tests were run at an engineering strain rate of about 0.235 min<sup>-1</sup> (3.9x10<sup>-3</sup> s<sup>-1</sup>), with a holding time of 15 minutes for thermal equilibration prior to testing. The results show the greater low-temperature strengths in the microalloyed grades, as expected, along with greater tensile strengths at elevated temperature. Yield strength is of most significance in these constructional steels, and the Mo+Nb grade in particular sustains greater yield strengths at temperatures above about 500 °C. In some cases there is a notable increase in strength at intermediate temperatures of about 350 °C. This behavior is associated with dynamic strain aging, and is shown more clearly through examination of the full stress strain curves in Figure 3 for the base alloy and the V+Nb alloy. The rapid strain hardening and "serrated" flow curves in the base alloy at 300 °C and 400 °C, in combination with higher strengths than observed at 25 °C are clearly visible, and are indicative of dislocation/interstitial interactions associated with dynamic strain aging [22]. The V+Nb alloy also exhibits strain aging behavior, but the effects are less prominent, possibly due to reduced solute N levels resulting from vanadium nitride (or N-rich vanadium carbonitride) precipitates. It should be noted that dynamic strain aging characteristics are not usually considered a critical factor for constructional steels in the USA, and may be influenced by a variety of alloying and processing factors.

The Mo+Nb alloy used in this work was designed based on earlier development work that led to commercial FR steels with a similar chemical composition [13,20]. In this earlier work, it was shown that Nb and Mo additions to a base C-Mn steel increased the elevated temperature strength, and that a combined Mo+Nb addition provided further improved properties. Other alloying approaches may also be considered [18,24], but the Mo+Nb approach appears to have received the most attention thus far. The benefits of combined Mo+Nb additions are reported to involve precipitation strengthening by both species, and increased precipitate coarsening resistance, enhancing strength at elevated temperature. The mechanism by which Mo retards coarsening has been suggested to be associated with segregation to interfaces between Nb(C,N) precipitates and the surrounding matrix, reducing the precipitate/matrix interfacial energy. This interfacial energy provides the fundamental driving force for precipitate coarsening, although recent 3-D atom probe tomography work by Enloe (conducted on Nb-Mo containing steels processed at higher temperatures in the carburizing regime) has not identified Mo segregation to precipitate/matrix interfaces of coarsening-resistant niobium carbides, and thus has raised some doubts as to the mechanism [25]. Regardless of the applicable mechanism, however, the suppression of precipitate coarsening contributes to precipitate refinement and therefore to

strength retention at elevated temperature, and this process is considered to represent an important "synergy" between elements such as Mo and Nb in FR steels.

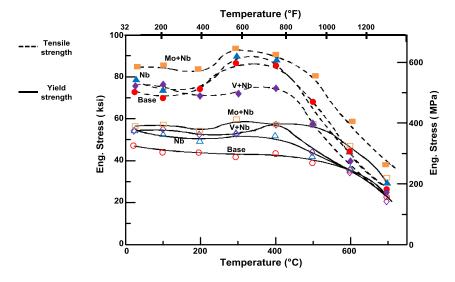


Figure 2. Yield stress (open symbols) and tensile stress (closed symbols) at  $25-700\,^{\circ}\text{C}$  for base, Nb, Mo+Nb, and V+Nb alloys, tested at an engineering strain rate of  $3.9 \times 10^{-3}\,\text{s}^{-1}$  [23].

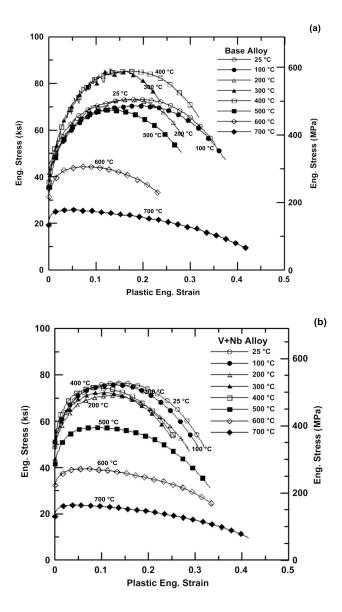


Figure 3. Engineering stress/plastic strain curves for the base (a) and V+Nb (b) alloys at temperatures between 25 and 700 °C, tested at an engineering strain rate of  $3.9 \times 10^{-3} \, \text{s}^{-1} [23]$ .

Alloying also contributes to microstructural refinement, through its hardenability effects, by reducing the temperature at which the austenite phase decomposes during cooling and by promoting bainitic microstructures. (This factor could be relevant in the microstructural design of FR steels, and is discussed further below.) While the alloy composition in the experimental steel is similar to that of FR steels reported in the literature, the steel was hot-rolled in production and it should be recognized that the thermomechanical processing characteristics, and thus microstructural details, may not be identical to commercial variants.

While the elevated temperature tension test is nominally an "isothermal" test, the heating rate to the test temperature can influence the tension test results, and of course the holding time at temperature prior to testing would be expected also to have an influence. Figure 4 shows the yield stress measured at different nominal test temperatures, plotted vs. the heating rate to the test temperature. A heating rate effect is notably found for a test temperature of great relevance to FR steel specifications, 600 °C. The sample is not under load during heating, so this response is not a consequence of creep deformation during heating, but rather illustrates an "annealing response" associated with softening of the microstructure due to longer exposures at elevated temperature during heating. Softening of the microstructure may involve such mechanisms as precipitate coarsening, dislocation rearrangement (recovery) and grain growth.

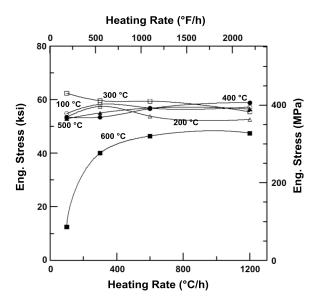


Figure 4. Influence of heating rate  $(100-1200 \, ^{\circ}\text{C/h})$  to test temperature on the yield strength of the Mo+Nb alloy. Testing was conducted at an engineering strain rate of about  $3.9 \times 10^{-3} \, \text{s}^{-1}$  following a 15 minute holding time at the test temperature [23].

Along with elevated temperature tension tests, another methodology was developed in the authors' laboratory to more closely simulate material response during exposure to a fire. In this test, a constant tensile load was applied to the specimen while heating at a nominally constant rate. As the temperature rises, thermally activated deformation mechanisms become operative, and the specimen plastically deforms when a sufficient temperature is reached, and eventually fails by "runaway" strain at higher temperatures. This test is referred to here as the "constant load" test, and has also been called a "temperature ramp" test. Test variables include heating rate and applied stress. An example of the constant load test data is shown in Figure 5 for the same four steels for which elevated temperature tension tests were presented in Figure 2. The data in Figure 5 apply to a heating rate of 1200 °C/h and an applied stress level that is half the room temperature yield stress of each alloy. While there is not a complete correspondence between the comparative results of the two tests (and this observation should perhaps be noted when considering appropriate testing and material requirements for FR steels), the figure again illustrates the superior elevated temperature performance of the Mo+Nb steel.

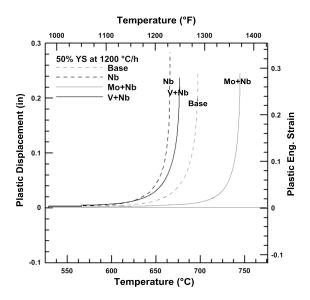


Figure 5. Constant load test results for the base, Nb, Mo+Nb, and V+Nb alloys at 50% yield strength for each alloy and a heating rate of 1200 °C/h. The reduced section of the test specimen was nominally 25.4 mm in length [23].

Precipitate analysis was conducted after constant load testing in the Nb and Mo+Nb alloys, using transmission electron microscopy (TEM) of carbon extraction replicas. The heating rate in this case was 300 °C/h, a relatively slow rate where there is greater time for microstructure changes to occur during heating. It appears that the carbide precipitates in the Mo+Nb alloy are finer and occur with a much higher particle number density than in the Mo-free alloy, Figure 6. This

observation is consistent with earlier work suggesting that Mo contributes to refinement of strengthening precipitates in microalloyed FR steels [13,20,26].

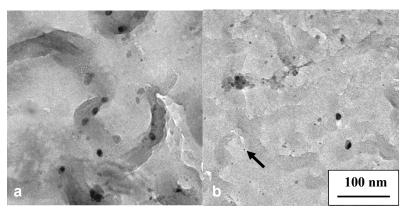


Figure 6. TEM bright field images from carbon extraction replicas showing fine precipitates in the Nb (a) and Mo+Nb (b) alloys after constant load testing at 300 °C/h [3].

It should be recognized that the improvements associated with FR steels are modest; far less than the increases in allowable temperatures of several hundreds of degrees that might be expected with (much more costly) heat-resisting superalloys. Nonetheless, the increased performance of FR steels is sufficient to justify application in some structure designs, and would be expected to contribute to fire safety whenever these steels are employed. The elastic modulus is also temperature dependent, and is generally expected to be less sensitive to microstructure, chemical composition and processing than the strength, and thus there is also a (modest) limit to the benefits achievable by increasing the softening resistance of iron-based FR steels, before elastic deflection-related issues become limiting.

The Cu-containing steel in Table I was included to determine whether precipitation *during* the heating event associated with a building fire might offer a strengthening mechanism to an FR steel, associated with the fire itself. Such a mechanism might be considered to offer a metallurgical design concept involving a form of "active" fire safety, where a potential strengthening mechanism is built into the steel itself, and only activated as a consequence of a fire. The Cu-containing steel was conditioned in three different ways prior to testing, including:

- 1. Normalizing (involving cooling from high temperature, thereby suppressing Cu precipitation and allowing the potential for strengthening precipitates to form during heating), (Cu N);
- 2. Peak aging (to provide the maximum strength at the start of testing), (Cu P);
- 3. Overaging (to reduce the strength at room temperature and preclude strengthening precipitates from forming during heating), (Cu O).

Only the normalized condition would be expected to offer the desired precipitation mechanism described above. The test results are presented in Figure 7, and confirm that improved FR properties are achieved for the normalized (N) condition. These results illustrate the potential benefit of the proposed concept, ie. controlling the solution/precipitation behavior to allow strengthening precipitates to form during the heating associated with a fire. While this "active safety" concept was explored initially using a Cu-bearing alloy, it was also considered to be potentially applicable to other strengthening precipitates such as microalloy carbonitrides in HSLA steels.

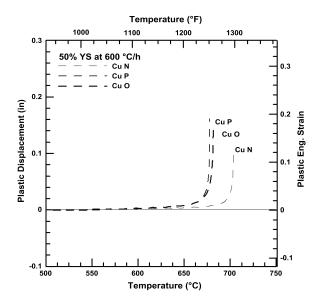


Figure 7. Constant load tests for three Cu alloy conditions: normalized (N), peak-aged (P) and overaged (O) tested at an applied stress equal to 50% of the room temperature yield strength for each condition. The heating rate was 600 °C/h and the reduced section of the test specimen was nominally 25.4 mm in length [23].

The early studies reported above led to additional work to understand better the influence of microstructure and processing variables on the fire resistant properties. This work has focused especially on the Nb-containing steel, to understand the influence of microalloying effects in the absence of the synergistic contributions of Mo. While commercial FR steels contain Mo levels of the order of 0.5% (by weight), it would be desirable to minimize or eliminate the Mo addition if that were possible, due to its high cost at present. The published literature has indicated that bainitic microstructures may exhibit enhanced fire resistant properties, and follow-up work was conducted to compare ferritic, bainitic, and martensitic microstructures in the base steel, and ferrite+pearlite with bainitic microstructures in the Nb-containing steel, also incorporating variations in the potential for NbC formation during heating. Thermal treatments were carefully

designed to separate the effects of Nb precipitation and general microstructure. The main results of this study [3] were published previously in the HSLA steels literature [27], and the details are not reproduced here. However, the results indicated that finer microstructures exhibit higher strength at both ambient and elevated temperatures, and are an important contribution of Nb microalloying which is especially prominent in the constant load test response at elevated temperature. Increased NbC "precipitation potential" during heating exhibited an unclear effect for the bainitic microstructures, but was clearly beneficial for the ferrite+pearlite microstructures, and thus may offer some potential to develop the "active" fire safety concept described above, using Nb-microalloyed steels.

Published results in the literature, as well as the results indicated here, have shown that Nb is an effective alloying element for increasing high temperature strength, and this is particularly true in FR steel applications where its contribution is often enhanced through Mo additions. Nb forms carbonitride precipitates at higher temperatures than are usually associated with Mo-carbide precipitation, and further work would still be helpful both to understand the controlling mechanisms better, as well as to identify the optimum levels of Nb and Mo, or the Nb-to-Mo ratio. Weldability is an important consideration with respect to FR steel developments, and the FR steels developed to-date (590 MPa class) are low C steels with typical additions of 0.02-0.03%Nb along with modest levels of Mo and Mn that reportedly provide excellent HAZ toughness [10,28]. These alloying elements thus provide attractive combinations of room temperature strength, resistance to softening at elevated temperatures up to approximately 600 °C and good HAZ toughness.

Benefits of refined microstructure were considered potentially to be associated with the substructure present in steels transformed at lower temperatures. Consequently, the effect on elevated temperature properties of thermomechanical processing to produce warm-worked ferrite was examined in later studies of the Nb-containing alloy. Warm working is well known to increase the strength at room temperature, and the objective was to evaluate whether stable substructures produced during thermomechanical processing at relatively high temperatures (ie. above the temperatures frequently employed in FR steel testing) can enhance the fire resistant properties. The thermomechanical processing details in this study are summarized in Figure 8. The reheating temperature of 1100 °C was selected to dissolve NbC and to avoid substantial austenite grain growth prior to laboratory rolling. Total rolling reductions were limited due to the small difference between the thickness of the available starting material and the test specimen, so a single pass 25% reduction was applied at 1000 °C for ferrite grain refinement, and then a 10% finishing pass was taken at different temperatures in the austenite, intercritical, and ferritic phase fields, respectively, using an embedded thermocouple for temperature control.

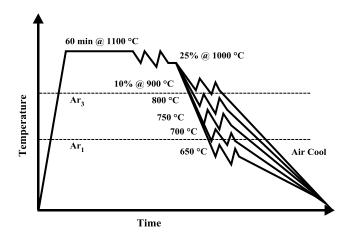


Figure 8. Schematic illustration of thermomechanical processing variations used to examine the influence of warm working in the Nb-containing steel [29].

The microstructures for each condition contained primarily equiaxed ferrite and pearlite along with some Widmanstätten ferrite [29,30]. Each of the laboratory rolled samples was characterized further using electron backscatter diffraction (EBSD) to investigate the presence of ferrite substructure. Image quality maps showed an increasing presence of "darker" features as the finishing temperature was reduced, indicating more deformation (ie. stored dislocations) within the structure. EBSD misorientation maps also confirmed the presence of increased amounts of ferrite substructure at low finishing temperatures. The EBSD results were quantified and Figure 9 displays the distribution of misorientation angles for the as-rolled conditions, for approximately 10 degree intervals. The fraction of each boundary type was determined relative to the calculated total length of the ferrite/ferrite boundaries, and the results show a greater fraction of low-angle boundaries (with misorientation angles between zero and ten degrees) at the lowest finishing temperatures, with the greatest fraction noted at 650 °C. The boundary fractions include uncertainty associated with low-angle boundaries from the pearlite and Widmanstätten ferrite constituents present within each sample, which are not characteristic of the warm worked substructure. However, these constituents of the microstructure are similar for the different processing conditions, so the comparisons in Figure 9 should be qualitatively correct.

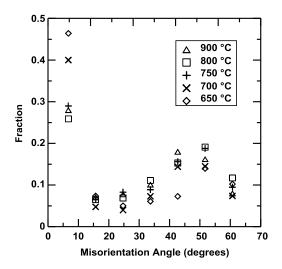


Figure 9. Distribution of misorientation angles for the Nb steel, laboratory rolled using finishing temperatures between 900 °C and 650 °C [30].

Overall, the microstructure analysis confirms that low temperature finish rolling enhanced the development of ferrite substructure. The room temperature and 600 °C tensile properties are summarized in Figure 10 [29]. The tensile results indicate that sub-critical (ferritic) rolling increases the strength both at room temperature and at 600 °C. Most importantly, the yield strength ratio is increased by about 5% for the steel finished at 650 °C in comparison to the other conditions [29].

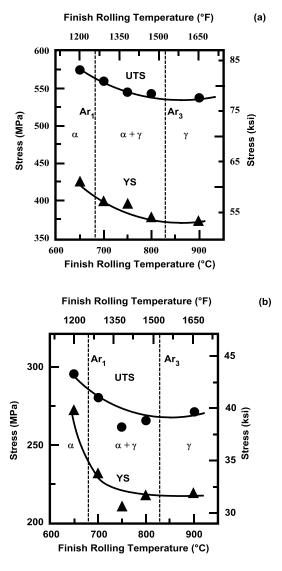
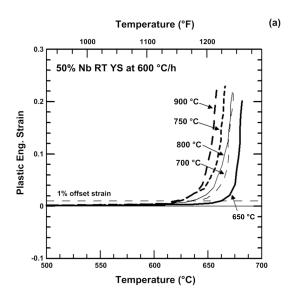


Figure 10. Yield and tensile strengths at room temperature (a) and 600°C (b) for Nb-steel finish rolled (10%) at different temperatures, tested at an engineering strain rate of 3.9x10<sup>-3</sup>s<sup>-1</sup>[29].

Corresponding constant-load tests are shown in Figure 11 for loading conditions involving either: (a) the same applied load for each specimen, selected to apply 50% of the Nb alloy room temperature yield strength (374 MPa) prior to thermomechanical processing, or (b) 50% of the room temperature strength of each material after finish rolling at the temperature of interest. The trends in Figure 11(a) correspond closely with the elevated temperature tensile properties presented in Figure 10, with the onset of plastic deformation and final failure during heating both increasing in relation to the elevated temperature strength, and the sub-critically rolled (650 °C) steel reaching the highest temperatures among the different conditions. In the results of Figure 11(b), where the applied stress varied based on the room temperature strength of the material tested, some interesting similarities and differences are noted. First, the onset of plastic deformation (based on intersection with the dashed 1% strain-offset horizontal line) is again at the highest temperature for the specimen finish-rolled at 650 °C, confirming that this microstructure may offer improved FR properties. However, it is also important to note that the specimen finish-rolled at the highest temperature (900 °C) exhibited the highest failure temperature (where runaway strains are encountered), perhaps as a consequence of the applied stress being relatively lower for this steel. Additional testing conducted at even higher applied stress levels, showed some similarities [31]. At an applied stress level of 2/3 the room temperature strength of the as-received material (identical for each condition), the sub-critically rolled specimen exhibited the highest temperatures for both onset of plastic deformation, and ultimate failure. When the applied stress was varied based on the room temperature strength of the condition of interest, however (ie. at a value representing 2/3 of the yield stress), the onset temperature for plastic deformation in the warm-rolled specimen was similar to the other steels, and the failure temperature (onset of runaway strain) was the lowest among the different conditions.



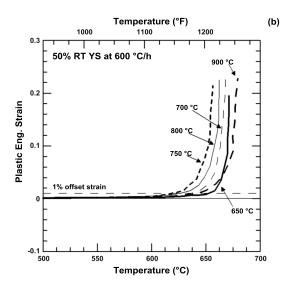


Figure 11. Constant load tests for the Nb-alloy finish rolled 10% at different temperatures (indicated on the figures). The results in (a) are for a constant applied stress (187 MPa), while those in (b) were conducted at an applied stress 50% of the room temperature yield stress for each condition.

The smaller difference in temperature between the onset of plastic deformation and failure for the sub-critically rolled (650 °C) specimen needs further attention, but could perhaps reflect a breakdown of the stable substructure developed during warm rolling, once the test temperature exceeds the temperature used during warm rolling. In any case, this work suggested that warm worked ferrite may be an effective and important strengthening mechanism in FR steels, due to the stability of the dislocation substructure created during warm working of ferrite at relatively high temperatures. Details related to specific loading and testing conditions may be important to consider, however, it should be noted that warm working process technologies are currently more applicable to plate steel production in comparison to structural shapes, as low finish temperature rolling is inherently more difficult for rolled sections where the cross-section geometry is more complicated. Also, warm working has a greater potential to develop anisotropic properties. The properties reported here were measured in the longitudinal direction, and additional work would also be helpful to characterize the elevated temperature transverse properties. In addition, detailed studies of microstructural evolution are needed to confirm the hypothesized changes with temperature.

## **Adoption of Specifications**

Following several years of investigation and discussion, a specification for FR steels has been adopted recently by ASTM, designated A1077/A1077M-12, "Standard Specification for Structural Steel with Improved Yield Strength at High Temperature for Use in Buildings." This specification, published in March 2012 [32], covers alloy steel in bars, in plates up to 4 inches (100 mm), and structural shapes for use in bolted or welded steelwork for buildings or for general structural purposes. Coiled plates are not excluded, but require additional testing. Separate values are given in Imperial or SI units, as indicated herein. Two grades are specified, having minimum yield strengths of 36 ksi (250 MPa) or 50 ksi (345 MPa). Killed steels are specified, with compositions and maximum P<sub>cm</sub> carbon equivalent values as shown in Table II.

Table II. Chemical Composition Requirement of ASTM A1077 [32]

	Heat Analysis in wt.% (maximum or range)	
Element	36 ksi (250 MPa) Minimum Yield Strength	50 ksi (345 MPa) Minimum Yield Strength
Carbon	0.15	0.15
Manganese	0.5 - 1.40	0.5 - 1.60
Phosphorus	0.035	0.035
Sulfur	0.035	0.035
Silicon	0.35	0.55
Nickel	0.50	0.50
Chromium	1.00	1.00
Molybdenum	0.20 - 0.70	0.20 - 0.90
Copper	0.50	0.50
Vanadium	0.15	0.15
Niobium	0.05	0.05
Titanium	0.03	0.03
Boron	0.002	0.002
P <sub>cm</sub> *	0.26	0.29

 $P_{cm} = C + Si/30 + (Mn + Cu + Cr)/20 + Ni/60 + Mo/15 + V/10 + 5B$ 

Along with the minimum 0.2% offset yield strength, mechanical property requirements specify minimum tensile strength and total elongation, and allow a provision to specify the maximum yield-to-tensile strength ratio ( $\sigma_y/\sigma_t$ ) at room temperature as 0.80. A Charpy V-notch impact requirement of 20 ftlb (27 J) absorbed energy at 32 °F (0 °C) is specified. Most importantly, elevated temperature tension tests are required, following ASTM E21. The 0.2% offset yield strength is to be measured at 1100 °F (600 °C), following thermal equilibration for at least 20 minutes at the test temperature. The strain rate is specified as 0.005  $\pm$  0.002 min $^{-1}$ . The elevated temperature minimum yield strength is specified as 24 ksi (165 MPa) or 33 ksi (230 MPa) for the 36 ksi (250 MPa) and 50 ksi (345 MPa) grades, respectively. Thus, the yield strength at 1100 °F (600 °C) must exceed about 2/3 of the minimum level specified at room temperature.

The adoption of standards for materials and testing now provides structural engineers engaged in building design for the USA and internationally with a narrowly defined set of options for utilization in new building designs specifying fire-resistant steels, and the performance levels will provide a significant improvement over current steels via increased design temperatures to 1100 °F or 600 °C. These steel performance characteristics are similar to the levels that have been employed elsewhere, and may be achievable with relatively "minor" adjustments in the alloying and processing practices. Initial implementation might be most likely in applications where fire-related aspects are included in detailed design calculations (eg. high-rise construction, bridges or tunnels with Class II flammable material transport), as well as in some applications where thermal protection (spray-applied rock wool) is undesirable, too costly, or can be eliminated or reduced in thickness as a result of improved steel characteristics [4,7,9,10]. Fire resistant properties undoubtedly could contribute further structural redundancy, resistance to local collapse [11,12], and improved safety performance in many other applications, although widespread general application in steel construction would require further experience. Thus it is likely that the FR steel and its applications will evolve as the costs and benefits of different approaches become more clear through interactions among the steel manufacturing, structural engineering, and architectural communities. In the meantime, steel research should continue to be focused on understanding the elevated temperature strengthening mechanisms in these steels, and in developing efficient alloying and thermomechanical processing strategies for commercial production.

#### **Conclusions**

The background "landscape" in which fire-resistant constructional steels are being addressed in the USA has been reviewed, along with some metallurgical research studies to understand the microstructure/property relationships that control fire resistant (FR) properties. Importantly, specification A1077/A1077M-12 has recently been issued by the American Society for Testing and Materials, defining requirements for elevated temperature testing and steel properties, and thus enabling design and construction using these steels. Research was presented to illustrate elevated temperature properties of some different steels tested using either an elevated temperature tension test, or a constant-load, temperature-ramp test intended to simulate behavior of structural members in a fire. Important effects of strain-aging at low temperature, and of the loading conditions and heating rate to temperature in the elevated temperature test are observed. The constant-load test results illustrate differences between steels, with a Mo+Nb steel exhibiting better FR properties than comparative CMn, V, or Nb steels tested identically. The Mo+Nb steel was designed to have a similar chemical composition as reported for some FR steels, and exhibited finer precipitates after elevated temperature exposure, consistent with a retarding effect of Mo on NbC coarsening kinetics. The potential to develop "active" fire resistance was demonstrated for both Cu and Nb containing steels, by conditioning the steel to provide sufficient "precipitation potential" before heating, so that strengthening precipitates can be formed during the heating associated with a fire. Finally, the potential for warm-worked ferrite to enhance FR properties was demonstrated, wherein the strengthening substructure may remain relatively stable up to temperatures near the warm deformation temperature.

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