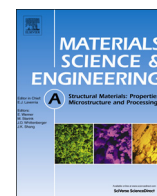




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# Toward interplay between substructure evolution, dislocation configuration, and yield strength in a microalloyed steel

P.K.C. Venkatsurya<sup>a</sup>, R.D.K. Misra<sup>a,\*</sup>, M.D. Mulholland<sup>b</sup>, M. Manohar<sup>b</sup>, J.E. Hartmann<sup>b</sup>

<sup>a</sup> Laboratory for Excellence in Advanced Steel Research, Institute for Materials Research and Innovation, University of Louisiana at Lafayette, LA 70503, USA

<sup>b</sup> ArcelorMittal, Global Research and Development Center, 3001 E. Columbus Drive, East Chicago, IN 46312, USA

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## ABSTRACT

We focus our attention here on the directional dependence of yield strength in high strength microalloyed steel using transmission electron microscopy and x-ray diffraction. The primary objective is to study the interplay between substructural evolution, notably cell size, dense dislocation walls (DDWs), dislocation tangle zones (DTZs), lamellar boundaries, crystallographic texture, and yield strength. The study elucidates for the first time the strong impact of thermo-mechanical deformation-induced dislocation and lamellar structures, which are likely to modify the slip pattern, leading to directional dependence of yield strength. Majority of the dislocations tend to pile along the {110} slip planes as dense dislocation walls. At low strains, grains are first divided into cell blocks that are nearly dislocation-free. At higher strains and with progress in thermo-mechanical processing dislocation tangled zones and lamellar boundaries develop. It is hypothesized that the differences in dislocation configurations, dislocations cells and cell blocks, and lamellar boundaries synergistically contribute to directional dependence of the yield strength in the high strength ferrous alloy. The presumption is envisaged on the basis of observations that the microstructural constituents were similar in the entire plane of the hot rolled strip and the crystallographic texture was weak.

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## 1. Introduction

Heterogeneities occurring at the grain-scale and within the grain, inclusive of substructure evolution, have an important role in defining the mechanical properties of metals and alloys [1–3]. Deformation substructures enable us to better understand processing-structure-property paradigm. It is surprising that there are relatively few comprehensive studies on the evolution of deformation substructures in thermo-mechanically processed microalloyed steels in comparison to the non-ferrous alloys. This subject area appears to have been less explored in-depth and the understanding continues to be fragmented. In contrast, the texture has been widely studied in microalloyed steels, motivated by the desire to understand the recrystallization behavior [4–6]. Recently Misra's group examined the effect of alloy chemistry and texture in influencing mechanical properties of microalloyed steels [7,8]. It was observed that at similar yield strength, the characteristics of texture depend on the microstructure, i.e. polygonal ferrite versus bainite.

In the context of up-and-coming spiral welding of microalloyed steels for the fabrication of linepipes, the variation in yield

strength along different orientations with respect to the rolling direction is an aspect of serious concern and is not understood. The underlying reason of this concern is that the circumferential direction of the linepipe (direction exposed to the main stress) depends on the forming angle, diameter, and width of the strip [9]. Typically, during the fabrication process, the angle between the original plate rolling direction and the resulting hoop direction in the pipe, i.e. the direction of maximum tensile stress varies and depends on the pipe diameter [9]. Thus, it is important to ensure that the thermo-mechanically processed steel has near-uniform yield strength in the plate along different orientations to the rolling direction.

Motivated by the above background, the objective of the present study is to fundamentally understand the interrelationship between the general microstructure, substructural evolution (cell structure, dislocation structure, texture components), and yield strength along different orientations with respect to the rolling direction. It is important that we consider deformation-induced dislocation and cell structure, together with texture evolution. Dislocation and cell structure developed during thermo-mechanical processing may also directly impact the yield strength, particularly if the texture is weak.

Furthermore, given that the strain rate sensitivity to flow stress (or yield strength) is an important material parameter whose sign or magnitude may govern texture [2], we have conducted depth-sensing

\* Corresponding author. Tel.: +1 337 482 6430; fax: +1 337 482 1220.  
E-mail address: [dmsira@louisiana.edu](mailto:dmsira@louisiana.edu) (R.D.K. Misra).

**Table 1**  
Chemical composition of the experimental microalloyed linepipe steel (in wt%).

Element	C	Mn	Si	Mo	Nb+Ti+V	Ni	Fe
555 MPa (80 ksi) Steel	< 0.1	< 2	< 0.4	< 0.5	< 0.15	< 0.5	Balance

nanoindentation experiments to explore strain rate sensitivity of the microalloyed steel. Nanoindentation approach enabled us to understand the material behavior using identical specimen geometry and loading configuration as a function of strain rate by studying the dependence of hardness (which is  $\sim 3$  times the flow stress) as a function of strain rate.

## 2. Experimental material and procedure

The experimental material was a hot rolled niobium-microalloyed steel with nominal chemical composition summarized in Table 1. The microalloyed steel was continuously cast as  $\sim 250$  mm thick slab and thermo-mechanically processed to  $\sim 12$  mm in ArcelorMittal, 84 in. hot strip mill, East Chicago, USA.

The yield strength was measured along  $0^\circ$ ,  $45^\circ$ ,  $75^\circ$ , and  $90^\circ$  with respect to the rolling direction (RD) using flat tensile specimens of 25 mm gage length machined according to ASTM E8M specification. The tensile tests were conducted at room temperature using a servohydraulic MTS system.

Light and scanning electron microscopy studies were carried out on specimens that were mounted and polished using standard metallography procedures and chemically etched with a solution of 2% nital for the measurement of grain size and to identify the microstructural constituents at low magnification. The recorded light micrographs were subsequently analyzed by Image J-analysis software to measure the grain size distribution. Transmission electron microscopy (TEM) was carried out to study substructure, dislocation structure, and precipitates. Electron transparent foils for TEM were prepared by cutting thin wafers from the steel samples, and grinding them to  $\sim 50$   $\mu\text{m}$  thickness. Three millimeter discs were punched from the wafers and twin-jet electropolished with a solution of 10% perchloric acid in ethanol as electrolyte. Carbon extraction replica approach was also used to study the distribution of strengthening precipitates. This involved etching the metallographically polished surface with 2% nital and sputter-coating with carbon onto the etched surface. Next, the surface was scored to  $\sim 3$  mm squares and the sample etched first with 10% nital and then with 2% nital. Subsequently, the extracted replicas were rinsed with distilled water and placed on the copper grid and dried. Foils and carbon extraction replicas were examined with a Hitachi 7600 TEM operated at 120 kV.

Texture measurements were carried out on metallographically polished mirror finish samples of dimensions  $20 \times 20$  mm<sup>2</sup>. The Mo  $K_\alpha$  radiation was used for X-ray diffraction and pole figures were measured in the back reflection mode. Pole figures corresponding to (110), (200), (211), and (310) were obtained. The orientation distribution function (ODF) was calculated using a series expansion method ( $l_{\text{max}}=22$ ) from the pole figure data and plotted in contour lines in the Euler space (Bunge's notation).

As mentioned in the introduction section that strain rate sensitivity may govern the texture components. Thus, we have conducted depth-sensing nanoindentation experiments using a MTS Nanoindenter-XP. An accompanying reason for conducting indentation experiments was to study the relationship between strain rate sensitivity to substructural evolution. Prior to nanoindentation experiments, the samples were electropolished to remove the residual stresses that are induced during mechanical

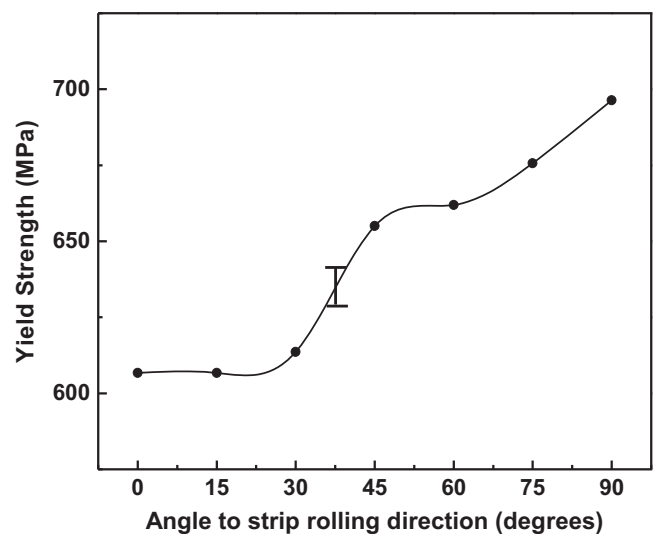
polishing to mirror-finish. The nanoindenter system consisted of a Berkovich three-sided pyramidal diamond indenter with a nominal angle of  $65.3^\circ$  and indenter diameter of 20 nm. The experiments were conducted at low strain rates in the range of  $5 \times 10^{-3}$  to  $1 \text{ s}^{-1}$ . Low strain rates were selected to eliminate the possibility of any adiabatic heating effect. The maximum depth of the indentation was fixed at 500 nm. More than 20 indentations were made for each test condition and the indentations were separated by a distance of 50  $\mu\text{m}$ .

## 3. Results and discussion

The sequence of presentation of results and discussion is as follows. We first describe the yield strength data, followed by description of the general microstructure. It is important to demonstrate that the general characteristics of the microstructure (grain size, precipitation) were similar in different orientations with respect to the RD. Second, we describe in detail the results of texture measurements. Next, the substructural evolution is presented and discussed, which involves a step-by-step comparison for different orientations with respect to the RD to fundamentally understand the differences in yield strength. The experimental observations are presented with explanation to the observed differences and ensure connection between the different aspects, and to also facilitate the reader. Finally, substructural evolution and texture are correlated to strain rate sensitivity in the context of variation in yield strength along different orientations with respect to the RD.

### 3.1. Mechanical properties

The yield strength (YS) of Nb-microalloyed steel obtained from the tensile tests for test directions of  $0^\circ$ ,  $45^\circ$ ,  $75^\circ$ , and  $90^\circ$  with respect to the rolling direction (RD) is summarized in Fig. 1. There is significant variation in the yield strength. The yield strength was similar from  $0^\circ$  to  $30^\circ$  with an average value of  $\sim 606$  MPa and then increases to a maximum value of 696 MPa at  $90^\circ$  to the RD (transverse direction), with elongation being similar in all the directions at  $\sim 18$ – $20\%$ . It is pertinent to mention here that similar variation in yield strength was also obtained in the lower strength microalloyed steel (490 MPa) under similar conditions of thermo-mechanical processing. Thus, based on the yield strength data,



**Fig. 1.** Yield strength of Nb-microalloyed steel as a function of orientation angle with respect to the strip rolling direction. Bar indicates the maximum range of deviation from the mean value for the tested specimens.

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