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# Effect of thermomechanical processing schedule on the texture and microstructure of pipeline grade API X80 microalloyed steel

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## ABSTRACT

The presence of micro alloying constituents like Ti, Nb in microalloyed steel enhances its mechanical properties through grain size control and precipitation strengthening. The strengthening capability of microalloying additions can be fully utilized by choosing proper thermomechanical processing (TMP) schedule. The TMP schedule needs to be designed based on determination of critical temperatures of transformation in steel including no recrystallisation temperature ( i.e., Tnr ) of microalloyed steels. The TMP was carried out on API X80 grade pipeline steel using Gleeble® 3800 Thermal mechanical simulator. The samples were deformed in plain strain condition at three different temperatures (860, 950 and 1050°C) by keeping other deformation parameters constant. The deformation temperature was chosen based on CCT and Tnr determination studies. The deformed samples were examined in EBSD and TEM to obtain the texture and microstructural information. It is also observed that formation strain levels in the austenite matrix prior to transformation. The grain boundary misorientation angle distribution obtained from EBSD analysis can be a very important parameter to distinguish the different microstructural constituents apart from grain shape and image quality.

KEYWORDS: pipeline steel, Gleeble, Tnr, EBSD, microtexture, acicular ferrite

# INTRODUCTION

The presence of micro alloying constituents like Ti, Nb in microalloyed steel enhances its mechanical properties through grain size control and precipitation strengthening<sup>1-4</sup>. The development of pipeline steel grades with higher YS (X60 to X80 to X100) depends on achieving the grain size refinement by pancaking the austenite prior to transformation to ferrite<sup>5,6</sup>. The desired transformation is partly diffusion less leading to steel microstructure of acicular ferrite and bainite. The role of niobium in raising the no recrystallisation temperature (i.e., Tnr) significantly is well known<sup>7,8</sup>. The strengthening capability of microalloying additions can be fully utilized by choosing proper thermomechanical processing (TMP) schedule. The TMP schedule needs to be designed based on determination of critical temperatures of transformation in steel including Tnr of microalloyed steels<sup>9,10</sup>. The determination of Tnr is mostly determined by Mutihit deformation method. This paper studies the effect of deformation at different temperatures (i.e., below and above Tnr) on evolution of texture and microstructure of pipeline grade API X80 microalloyed steel.

## **MATERIAL & METHODS**

The APIX80 grade microalloyed steel was available in the form of rolled plates supplied by M/s Arcelor Mittal, USA. The chemical composition of the steel is 0.04C-0.26Si-1.75Mn-0.009P-0.034Al-0.085Nb-0.015Ti-0.0046N-0.8(Ni+Cu+Mo) in mass

%. The hot deformation by plain strain compression was carried out in a Gleeble ® 3800 thermomechanical processing simulator with Hydrawedge. The sample sizes of 20 mm x15mm x10 mm rectangular blocks were cut off from the alloy plates and their surface were smooth finished to ensure proper electrical contact for gleeble testing. The tantalum foils were used between the WC anvils and the sample to reduce friction and protect the anvils. Tnr of the steel was determined by carrying out multihit deformation method. The CCT diagram was generated using JMATPRO and performing dilatometry in Gleeble to determine the transformation temperatures. Based on the above studies, the TMP was carried out at 860°C, 950°C and 1050°C after soaking at 1200°C for 500s up to strain of 0.8 at a strain rate of  $5s^{-1}$ . The samples were immediately allowed to free cooling after deformation. EBSD experiments on the TMP samples was carried out on the transverse section using a TSL OIM data collection system attached to FEI-Nova nanoSEM microscope. The microtexture analysis was carried out using TSL-OIM analysis software. For texture analysis care has been taken to rotate the pole figure along RD by  $90^\circ$  , as only TD section was accessible in a gleeble plain strain compression tested sample.



**Fig.1:** Schematic of plain strain compression testing in Gleeble

## **RESULTS AND DISCUSSION**

The Tnr of the experimental steel was determined by multihit hot deformation in a

plain strain compression facility. The samples were soaked at  $1200^{\circ}$ C for 10 min followed by cooling to first deformation temperature of  $1150^{\circ}$ C at a rate of  $10^{\circ}$ C/s followed by soaking time of 5 sec prior to deformation. The deformation was carried out for 0.1 strain at a strain rate of  $2s^{-1}$  followed by interpass time of 5s between successive deformation passes.

The deformation was carried out at temperature intervals of 25°C up to 900°C followed by free cooling. The true stressstrain curves of the multihit deformation is shown in Fig.2. The analysis of the multihit deformation data was carried out by several researchers by examining the changes in mean flow stress over the deformation temperature range<sup>11-13</sup>. The mean flow stress was calculated by integrating the stress strain curve over the strain range using Origin. The Tnr of 1000°C was determined for the steel based on the deviation in the slope of mean flow stress vs. temperature plot.



**Fig.2:** True stress-strain curves of multihit deformation for Tnr determination



#### Fig.3: MFS Vs 1/T plot

The simulated transformation temperatures of the experimental steel was found to be 828°C for austenite to ferrite transformation. Ac3 :598°C for Bainite transformation; and 427°C for martensite start Ms. The CCT dilatometry in gleeble was carried out at cooling rate of 20°C/s is shown in Fig.4. The free cooling in the hot deformation tests correspond to a cooling rate of 20°C/s. The bainite transformation temperatures obtained via gleeble CCT dilatometry correlate well with the predicted transformation temperatures obtained via JMATPRO. The Bainite start temperature was ~613°C and the transformation complete at 523°C From the studies, it is clear that bainite forms from austenite on continuous cooling at 20°C/s.(Fig.5)



Fig.4: CCT Diagram of experimental steel



**Fig.5:** CCT dilatometry of experimental steel at 20°C/s

Based on the above results, After soaking at  $1200^{\circ}$  C for 500s , three deformation schedules up to 0.8 strain at a strain rate of  $5s^{-1}$  was carried out at: (i)  $50^{\circ}$ C above Tnr- $1050^{\circ}$ C ;(ii)  $50^{\circ}$ C below Tnr -  $950^{\circ}$ C and (iii) well below Tnr and ~just above Ac3 temperature-  $860^{\circ}$ C. The deformations was carried out to understand the effectiveness of Tnr on the development of texture and microstructure of the steel under investigation.



Fig.6: Flow curves at different deformation temperatures.

deformation after The samples were sectioned along TD plane and samples were prepared for optical metallography and EBSD. PAG etching was performed on the samples to estimate the austenite grain size and shape prior to transformation to microstructures<sup>14</sup>. bainite/acicular ferrite The etchant composition was 1.35 gms of picric acid in 100ml distilled water and mixed with 0.5ml of sodium alkylsulfonate (Extran) and 1ml HCl. The pancaking of austenite grains will be clearly evident from the PAG micrographs. The average grain size was measured using ImageJ, public domain image processing software.

Fig.7a shows that austenite grain sizes after deformation at  $1050^{\circ}$ C shows large equiaxed recrystallized grains ~24 $\mu$ m taken from the centre of deformed region. There is

no sign of pancaking due to dynamic recrystallization. Fig.7b shows pancaking of austenite with decreasing grain size  $\sim 7\mu$ m, with the deformation band widths  $\sim 25 \mu$ m containing mixed microstructure at 950°C. Fig.7c shows complete pancaking with very fine austenite sizes $\sim 2\mu$ m and smaller width of deformation bands $\sim 10 \mu$ m for 860°C sample.



**Fig.7:** PAG micrographs for samples deformed at (a) 1050°C; (b)950°C; (c) 860°C



(b)



**Fig.8:** IPF maps for samples deformed at (a) 1050°C; (b)950°C; (c) 860°C

EBSD Maps were acquired at 500X with average confidence index > 0.6 with a step size of  $< 0.15 \mu m$ . Fig.8 shows the inverse pole figure maps along with high angle grain boundaries clearly shows the grain size

variation obtained after reduction up to 0.8 true strain at three different temperatures. The presence of pancaked elongated grains is very clear from the 860°C TMP sample with most grains oriented with <111> plane normal to the rolling section.



**Fig.9:** Misorientation angle distribution of samples deformed at (a) 1050°C; (b) 860°C.

The misorientation angle distribution shows the presence of large fraction of low angle grain boundaries in case of sample deformed at 1050°C than the sample processed at 860°C (Fig.9). The low angle boundaries evolves between the parallel plates of bainite formed at intergranular sites of large recrystallized strain free austenite grains. The absence of peaks in the mid band of misorientaion angle distribution(i.e., 20° - 45°) is possible only if the phase transformation from austenite to ferrite occurs following Bain or N-W orientation realtionship. The acicular ferrite forms from

intragranular nucleation within the prior austenite grains and hence forms multiple oriented non-parallel plates with high angle boundaries between them. This lead to higher fraction of high angle boundaries in case of sample processed at 860°C as the deformation strain storage in the form of dislocation tangles, interface dislocations, etc., is more, which can serve as site for nucleation of intragranular ferrite transformation leading to acicular ferrite microstructure. This implies that strain in the austenite prior to transformation can favor formation of acicular ferrite rather than bainite.

The ebsd data was analysed for texture and represented by ODF (orientation distribution function) sectioned at  $0^{\circ}$  and  $45^{\circ}$  as shown in Fig.10. These sections contain all possible major texture components that can evolve during deformation of steel. The rotated cube component was present at all deformation temperatures and becoming weaker with decrease in processing temperature. The evolution of typical fcc deformation texture for samples with  $T_{def}$  < Tnr evidences the pancaking and redeformed austenite grains orientation of along the rolling direction without recrystallization<sup>15,16</sup>. The presence of strong ND {(111)[1-10] to(111)[0-11] to(554)[-2-25]  $and RD{(114)[1-10]} to (332)[1-10]}$ fiber is observed for 860° processed sample. The observed RD fiber component can form from the Cu texture of rolled austenite. The observed ND fiber component can evolve from the Brass texture of as rolled austenite.





**Fig.10:** ODF sections at  ${}^{\phi}_2 = 0^{\circ};45^{\circ}$  for samples deformed at (a) 1050°C; (b)950°C; (c) 860°C; (d) at  ${}^{\phi}_2 = 45^{\circ}$  section with important texture components in Bunge notation<sup>17</sup>.

The TEM examination of the sample deformed at 860°C shows bainite plates with heavy dislocation structure (Fig.11a). The dark field imaging at a higher magnification shows the presence of very fine niobium precipitates(Fig.11b) carbide at the dislocation sites. The niobium carbide precipitation was responsible for the solute drag effect and hence raise the Tnr of the given steel providing a large deformation window that can pancake the austenite at lower rolling loads and thus favors formation of high strength acicular ferrite and bainite microstructures.



**Fig.11:** TEM micrographs of sample deformed at 860°C showing bainitic ferrite plates and Niobium carbide precipitates under dark field.

#### Conclusion

The given microalloyed steel of APIX80 grade was deformed in plain strain condition at three different temperatures (860, 950 and 1050°C) by keeping other deformation parameters constant. The Tnr of the steel was determined by mean flow stress method and found to be ~1000°C. The CCT dilatometry at 20°C also shows that it favors bainite transformation at ~600°C as predicted by JMATPRO. The EBSD examination of samples deformed below Tnr showed pancaking of austenite leading to fine grain sizes and large equiaxed grains with bainitic ferrite plates for sample deformed above Tnr. It is also observed that

formation of acicular ferrite and bainite microstructures in these steel is very much dependent on the deformation strain levels in the austenite matrix prior to transformation.

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