#### INTERFACE SCIENCE

# Texture and grain boundary character of a Ti + Nb-IF steel after large cold rolling reductions

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Abstract The crystallographic texture and grain boundary character of a Ti + Nb Interstitial-Free (IF) steel has been studied as a function of cold rolling deformation. The results indicate that the relative proportions of high angle grain boundaries decrease as the amount of cold rolling increases. The texture of the steel became more stable as the amount of cold rolling increased.

#### Introduction

Although one of the major applications of Interstitial Free (IF) steels is in the automobile sector [1], applications of IF steels in the packaging industry is also expected to be quite substantial [2]. For this purpose it is necessary to produce the steel sheet in much thinner gauge sections than those used in the automobile industry. One way of achieving this reduced thickness is to utilize cold deformation levels much greater than what is currently practised (85–90%) now a days [3]. Furthermore, refinement of the final grain size can be used to obtain high strength steels with improved toughness and ductility. By employing large amounts of cold rolling reduction (termed "heavy cold rolling") followed by an annealing treatment, it has been possible to generate an ultrafine grained structure in a

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Ti- stabilized IF steel [4]. However, the current literature provides little information on the effect of ultra-high levels of cold rolling on the microstructural and textural aspects of these steels. In the present work, an industrially produced very low carbon Ti + Nb IF steel was controlled hotrolled, followed by high and ultra-high levels of cold rolling reduction. The resulting textural and microstructural aspects were then evaluated. A few studies [5, 6] have suggested that severe plastic deformation leads to the formation of many high angle grain boundaries. In order to verify this increase in high angle grain boundaries, the grain boundary character distribution of the hot-rolled and cold-rolled steels were also analysed. The results indicate that increase in the amount of strain may not always increase the fraction of high angle grain boundaries during large strain cold rolling.

# Experimental

The steel was melted as an industrial heat at Tata Steel, Jamshedpur, India. The chemical composition of the steel is: 0.003%C, 0.36%Mn, 0.006%S, 0.014%P, 0.012%Si, 0.047%Al, 0.0033%N, 0.011%Nb, 0.05%Ti, balance Fe (all values in wt.%). A transfer bar of this steel, was controlled hot-rolled using an instrumented laboratory rolling mill. A total deformation of 80% (reduction in thickness) was given during hot rolling in several passes. The Finish Rolling Temperature (FRT) was kept within upper ferritic range. The hot bands were then subjected to cold-rolling for a 90 and 98% reduction. It is well-known that getting reasonably accurate EBSD patterns from a heavily deformed steel is a real challenge. The quality of EBSD patterns largely depends on the quality of specimen preparation. Hence, special effort was undertaken during

EBSD sample preparation. In order to do that the heavily deformed thin sheets were cut into small pieces. These were mechanically polished and cleaned in acetone. These were then electro-polished in an electrolytic bath containing a mixture of Acetic Acid (90% by volume) and Perchloric Acid (10% by volume). Crystallographic textures were determined from the mid thickness region of the hotrolled as well as the cold rolled sheets using an FEI-Ouanta 200 scanning electron microscope (SEM), coupled with an electron back scatter diffraction (EBSD) system. During data collection, a minimum number of seven Kikuchi bands were required to index a pattern, thus ensuring very reliable information. It has been reported by Field [7] that EBSD patterns having a confidence index above 0.1 can correctly index an orientation 95% of the time. In the present experiments the average confidence index of the patterns was above 0.45 and this led to more than 96% of the patterns to be indexed correctly in every case. The average image quality of the patterns was more than 65, which also ensured that the patterns were properly indexed. The minimum angular resolution of the EBSD from alpha iron using W-filament has been found to be 1°, as reported by Humphreys [8]. In order to make accurate measurements, misorientations less than 1.5° were excluded from the data. Orientation Distribution Functions (ODFs) were measured using TSL-OIM software and  $\Phi_2 = 45^\circ$  sections (Bunge notation) were determined therefrom. Using the same software, the grain boundary character distribution of all the steel samples was determined from grain boundary misorientation maps as well as boundary character vs. number fraction plots. Optical microstructures of the rolled sheets were determined from the RD-ND section. Thin foils for Transmission Electron Microscopy (TEM) studies were prepared from the longitudinal sections of the heavily deformed sheets by dimpling and ion milling technique. The TEM work was carried out in a JEOL 2000FX-II electron microscope operated at 160 kV.

# Results

Figure 1a–c show the  $\Phi_2 = 45^\circ$  sections of the EBSD ODFs of the hot-rolled as well as the cold-rolled materials. The hot-rolled material shows a somewhat pronounced hot rolling texture consisting of the  $\gamma$  and  $\alpha$ fibres and a rotated cube component. The subsequent cold rolling of the hot-rolled material exhibits a much stronger texture after the 90 and 98% cold rolling reductions. Although both these materials also show the above texture components, the  $\gamma$  fibre is more uniform in the former as compared to the latter case. The rotated cube component became stronger as the amount of cold rolling increased. The grain boundary characters vs. number fraction plots



**Fig. 1**  $\Phi_2 = 45^\circ$  sections of the EBSD ODFs of (a) hot-rolled steel, (b) 90% cold-rolled steel, (c) 98% cold-rolled steel

for both the cold-rolled steels are shown in Fig. 2a, b. Interestingly, there is a progressive decrease in the number fraction of high angle boundaries as the amount of cold rolling deformation increases, the number fraction becoming as low as 0.26 after 98% cold-rolling. These results are also corroborated by the misorientation maps in Fig. 3a, b. The quality of the maps also indicates that the patterns were indexed properly. The red line indicates the presence of low misorientation boundaries ( $<15^\circ$ ) and blue line indicates high misorientation boundaries ( $>15^\circ$ ). These figures show that both the low and high misorientation boundaries are present in both cold rolled steels. The fraction of high misorientation boundaries decreases



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Fig. 2 Grain boundary character vs. number fraction plots of the (a) 90%, (b) 98 % cold rolled steels

to some extent as the amount of cold rolling increases from 90 to 98%. This observation differs from the previous results for steel that had been finish rolled at 964 °C and subsequently cold rolled [9]. Figure 4a, b show the longitudinal section TEM micrographs of the 90 and 98% cold rolled steels. Both the micrographs clearly indicate the usual dislocated substructure structure. The tendency for recovery at a few places is also evident.

# Discussion

The heavily cold-rolled IF steel samples are characterized by the presence of a large number of grain boundaries and dislocation boundaries arranged in a lamellar fashion. Many of these boundaries are of small to medium misorientation angles, whereas others are high angle boundaries with misorientation angles greater than 15-20°. Severe deformation is known to induce a high density of high angle boundaries which is significantly larger than the number of original grain boundaries in the undeformed metal.

Fig. 3 Grain boundary misorientation maps of the (a) 90% and (b) 98% cold-rolled steels

Although the dislocation structures and the character of the grain boundaries formed in different face centred cubic (fcc) metals and alloy during deformation at room temperature have been extensively studied in the last two decades [10, 11], much less work on the deformation structures and grain boundaries in body centred cubic (bcc) metals and alloys such as steels has been reported to date. Reis et al. [4] studied a Ti-IF steel that had been commercially rough-rolled and then laboratory finish-rolled to a thickness of 20 mm with finish rolling temperature (FRT) of 930 °C. The steel was subsequently cold-rolled to a maximum reduction of 95%. They noted that increasing the rolling reduction from 70 to 95% leads to a significant increase in the proportion of high angle boundaries. A similar observation was reported by Saha and Ray [9] for a Ti + Nb-IF steel, which was cold rolled by different amounts (90 and 98%) while keeping the finish rolling temperature in the austenite recrystallization range. In the present work the Ti + Nb-IF steel had a composition similar to that reported by Reis et al. [4] and also to that used by Saha and Ray [9]. The results for the present 635 °C FRT material in this investigation clearly show an opposite trend whereby the number fraction of high angle



Fig. 4 TEM micrographs of the (a) 90% and (b) 98% cold rolled steels

grain boundaries has been observed to decrease progressively as the amount of cold rolling increases from 90 to 98%. The present results clearly indicate that the previously reported trend for IF steels (Reis et al. [4] and Saha and Ray [9]) are not solely a function of cold rolling deformation but are a function of the FRT during hot-rolling. The results of their 930 °C FRT material and our previous 964 °C FRT material are similar because both the steels have been in the austenite range at these temperatures. Although the texture intensity of the hot-rolled material is not very sharp, a large number fraction of low angle boundaries (and subgrain boundaries) are formed [12, 13].

This difference in the deformation response of the two materials can be explained in the following manner. A close look at the  $\Phi_2 = 45^\circ$  sections of the EBSD ODF plots (Fig. 1) clearly indicates that a very sharp texture has already built up after 90% cold rolling and there is very

little change in the intensity of the texture even after 98% cold rolling. This means that effectively a stable texture has formed in this material after 90% cold rolling. A greater rolling reduction beyond this level does not change the texture, which means that grain rotations have become insignificant after 90% cold deformation (reduction). A very sharp [14] and stable texture [10] leads to the decrease in the high angle grain boundary fraction.

It has been suggested [10] that the low and high angle boundaries produced during deformation originate from (1) grain subdivision by dislocation accumulation (microstructural mechanism) and (2) grain rotation (textural mechanism). The probability distributions of misorientation angles based on the above mechanisms [10] indicate that the low misorientation angle grain boundaries are mainly formed by mechanism (1) whereas the high misorientation angle grain boundaries are primarily formed by the mechanism (2). Therefore, it is expected that when textural stability is achieved, the chances of grain rotation will be minimized and, as a result, not many high angle boundaries will be created during further deformation. This eventually will lead to a lower density of high angle boundaries as the amount of cold rolling increases. The misorientation maps (Fig. 3) clearly show that significant number fractions of low misorientation boundaries have been created in between the high misorientation boundaries. Formation of such high fraction of low misorientation boundaries is related to the development of substructure (Fig. 4) [12, 13] as well as strong texture [14, 15] due to severe cold rolling deformation.

# Conclusions

Increasing the amount of deformation via cold rolling from 90 to 98% in a Ti + Nb-IF steel leads to decrease in the proportion of high angle grain boundaries. The formation of a stable crystallographic texture after 90% cold rolling is suggested to be the reason for the observed behaviour.

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