



# Microstructure characteristics of the $\langle 111 \rangle$ oriented grains in a Fe-30Ni-Nb model austenitic steel deformed in hot uniaxial compression

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## ABSTRACT

The work presents a detailed investigation of the microstructure characteristics of the  $\langle 111 \rangle$  oriented grains in a Fe-30Ni-Nb austenitic model steel subjected to hot uniaxial compression at 925 °C at a strain rate of 1 s<sup>-1</sup>. The above grains exhibited a tendency to split into deformation bands having alternating orientations and largely separated by transition regions comprising arrays of closely spaced, extended sub-boundaries collectively accommodating large misorientations across very small distances. On a fine scale, the  $\langle 111 \rangle$  oriented grains typically contained a mix of “microbands” (MBs) closely aligned with  $\{111\}$  slip planes and those significantly deviated from these planes. The above deformation substructure thus markedly differed from the microstructure type, comprising strictly non- $\{111\}$  aligned MBs, expected within such grains on the basis of the uniaxial compression experiments performed using aluminium. Both the crystallographic MBs and their non-crystallographic counterparts typically displayed similar misorientations and formed self-screening arrays characterized by systematically alternating misorientations. The crystallographic MBs were exclusively aligned with  $\{111\}$  slip planes containing slip systems whose sum of Schmid factors was the largest among the four available slip planes. The corresponding boundaries appeared to mainly display either a large twist or a large tilt component.

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## 1. Introduction

It is well known that the phase transformation taking place in industrial steels on cooling from hot working temperatures precludes a direct observation of their deformation microstructure characteristics in the austenite state. In order to obtain such characteristics as a function of processing conditions, which is important for advancing the physically-based modelling of hot working of steels, Ni-30Fe and Fe-30Ni-based model alloys retaining the austenite hot deformation microstructure at ambient temperature have been employed [1–9]. The use of a Fe-30Ni-Nb model steel led to the direct confirmation of previous suggestions [10–12] that the dislocation structures introduced during hot deformation provide the preferential nucleation sites for precipitation during the multi-pass hot working of microalloyed austenite [6,13,14]. It should be noted that the above model steel not only retains the austenite hot deformation microstructure at ambient temperature but also displays similar flow curve characteristics as industrial microalloyed steels while maintaining acceptable solubility of NbC [6,11–13]. Some novel findings have recently been made by the present authors using this model steel that extend the current knowledge of the interaction between austenite hot deformation

microstructure and precipitate particles during multi-pass hot deformation [8]. Thus, the detailed understanding of the microstructure evolution during hot deformation of austenite is crucially important not only with respect to the softening processes and flow behavior, but also in relation to the strain-induced precipitation.

It has been surmised from the microstructural studies performed using Ni-30Fe and Fe-30Ni-based model alloys [1–9] that, at deformation temperatures relevant to the industrial steel finish rolling range [15,16], the austenite grains become subdivided on a coarse scale into deformation bands [17–20] and on a fine scale into “microbands” (MBs) for a majority of grain orientations. MBs are elongated microstructure features bounded by pairs of parallel low-angle planar dislocation walls, often termed “geometrically necessary boundaries” (GNBs), typically characterized by approximately opposite misorientation vectors [1–9,21–26]. The GNB/MB evolution and orientation dependence have been extensively investigated for straining at ambient temperature, especially for aluminium alloys e.g. [21–27], and the MB formation mechanisms involving dislocation multiple cross-slip [28,29] or dislocation wall splitting [30–32] have been suggested. The Risø research group have proposed that the alignment of GNB boundaries strongly depends on the crystallographic orientation of a grain for deformation in tension, compression and rolling [21,26,33–37]. Three major types of dislocation boundary structures have generally been observed [23,26,33]: Type 1, with extended planar boundaries aligned with the highly-stressed slip planes; Type 2, with an equiaxed dislocation cell structure

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without extended planar boundaries; and Type 3, with extended planar boundaries aligned with other crystallographic planes than slip planes, though still with a fixed relationship to the most stressed slip systems. Similar grain orientation dependence of the deformation substructure has recently been confirmed for Ni-30Fe and Fe-30Ni-Nb austenite subjected to hot plane strain compression [7] and uniaxial compression [8, 9], respectively. The latter studies were largely focused on the grain orientations concentrated around the stable  $\langle 110 \rangle$  fibre, with only a small number of orientations scattered around the standard stereographic triangle being studied. The findings reported in Ref. [9] have surprisingly indicated that, in contrast to the aluminium alloys [26],  $\langle 111 \rangle$  fibre grains in austenite hot deformed in uniaxial compression might contain both crystallographic and non-crystallographic MBs. Nevertheless, in the above work, only three grains having their tensile directions aligned close to the  $\langle 111 \rangle$  direction were investigated. A more statistically significant, detailed study is thus required to further elucidate the frequency of the formation of MBs of each type within the above grains and to compare the characteristics of the both types.

The aim of the present work was to carry out a detailed investigation of the microstructure characteristics of  $\langle 111 \rangle$  oriented grains in a Fe-30Ni-Nb model steel subjected to hot uniaxial compression using a strain of 0.2. The above strain level was selected as it provided a well-developed substructure, a wide range of available grain orientations and a potentially relatively short rotation path from the original grain orientation. The main emphasis of the investigation was placed on the MB characteristics.

## 2. Experimental procedures

An austenitic model steel with a composition of 0.11% C, 29.59% Ni, 1.77% Mn, 0.3% Si, 0.098% Nb, 0.001% Mo and Fe balance (in wt.%), supplied by the Corus Technical Centre, Swindon, UK, was used in the present investigation. The as-received hot rolled slab was machined to make cylindrical compression samples, having a length of 15 mm and a diameter of 10 mm, with the compression axis parallel to the rolling direction. The samples were subjected to solutionization treatment performed at 1300 °C for 1 h in an argon gas atmosphere to dissolve any pre-existing precipitates and bring all the available niobium into solid solution. This resulted in the starting mean grain size with an equivalent circle diameter of  $\sim 340 \mu\text{m}$ . The hot deformation was performed in uniaxial compression using a computer controlled servo-hydraulic deformation simulator. The samples were heated to 925 °C, at a rate of 10 °C/s, held for 20 s and subjected to single-pass deformation carried out at a strain rate of  $1 \text{ s}^{-1}$  using a true strain of 0.2. The boron nitride paste was used as a lubricant, which ensured negligible barreling of the specimens. The deformation was followed by immediate water quenching to preserve the austenite deformation microstructure and suppress NbC precipitation.

Microstructural examination was performed in the central regions of the hot compressed samples on a plane containing the compression axis (CA) using the electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) techniques for twelve grains for which the CA was oriented close to the  $\langle 111 \rangle$  direction. The sample preparation methods for the above techniques are detailed elsewhere [8,9]. The EBSD study was conducted using a Zeiss LEO 1530 FEG SEM operated at 20 kV. The instrument was equipped with a fully automatic HKL Technology (now Oxford Instruments) EBSD attachment. Data acquisition and post-processing were performed using both the Channel 5 and Aztec software including the modified Kuwahara filter [38] for the orientation averaging. Some EBSD data post-processing was also performed using the V-MAP software kindly provided by Prof. Humphreys of The University of Manchester. To characterize the grain substructure, detailed EBSD maps were acquired using a step size of 0.2  $\mu\text{m}$ . For the characterization of the overall grain structure and crystallographic texture, an acquisition step size of 8  $\mu\text{m}$  was employed and an area of about  $5 \times 5 \text{ mm}^2$  was scanned. Pattern solving efficiencies

were typically above 99% and 97% for the fine and coarse step sizes used, respectively. The TEM investigation was performed using a JEOL JEM 2100F microscope operated at 200 kV. The compression axis was marked as a pair of fine notches on the rim of each TEM foil and the notches were aligned along the main goniometer tilting axis labelled X. The crystal lattice orientations and misorientations were determined using convergent-beam Kikuchi diffraction patterns [39].

Oriented stereographic projections were constructed for selected crystallite orientations, obtained using both the EBSD and TEM techniques, on the basis of the specimen axis vectors expressed in the crystal lattice coordinate system. These vectors were obtained from the corresponding orientation matrices [40]. In the case of EBSD, this made it possible to assign individual poles of  $\{111\}$  slip planes on the pole figures their concrete indices and, thus, to determine the Schmid factor values for the slip systems on these planes. Additionally, misorientation angle/axis pairs for selected larger-angle MB boundary segments were determined from the orientation matrices and the corresponding misorientation vectors were added to the oriented stereographic projections. This made it possible to determine the angles between the above vectors and normals of the slip planes the traces of which were aligned close to the MB boundary traces and, assuming that the MB boundaries indeed coincided with the above slip planes, to approximately evaluate the ratio between the tilt and twist components of these boundaries. In the present work, the MBs are classified as “crystallographic” and “non-crystallographic” when their boundary traces are deviated less and  $> 10^\circ$  from the nearest  $\{111\}$  plane trace, respectively.

## 3. Results

### 3.1. Grain/twin structure and crystallographic texture

Fig. 1a shows an example of the microstructure of the starting material obtained after the solutionization treatment. The corresponding misorientation histogram for the high-angle boundaries displayed a pronounced peak centered at about  $60^\circ$  (Fig. 1b). The misorientation axis vectors of the above boundaries were strongly clustered around the  $\langle 111 \rangle$  direction in the standard stereographic triangle (see Fig. 1b). This demonstrates that  $\Sigma 3$  first-order twin boundaries, characterized by  $60^\circ/\langle 111 \rangle$  misorientation, represented a significant volume fraction of the pre-existing high-angle boundaries. The crystallographic texture of the starting material was close to random, being characterized by a maximum of only 1.4 times random (Fig. 1c).

The EBSD orientation map presented in Fig. 2a illustrates that the original austenite grains became slightly elongated and the pre-existing annealing twin boundaries locally lost their coherency as they became curved and their angle/axis misorientation relationships became deviated from the original exact  $\Sigma 3$  twin misorientation (i.e.  $60^\circ \langle 111 \rangle$ ). The latter is demonstrated more quantitatively in Fig. 2b. The figure shows that the peak at the higher end of the misorientation spectrum for high-angle boundaries became markedly broader compared to the starting material. Furthermore, the distribution of the corresponding misorientation axis vectors changed from that originally strongly clustered around the  $\langle 111 \rangle$  direction to that extended from the  $\langle 111 \rangle$  towards the  $\langle 101 \rangle$  directions. This demonstrates the tendency for the pre-existing coherent twin boundaries to become converted to general high-angle boundaries relatively early in the deformation process. Fig. 2c shows that the crystallite orientation distribution changed from an essentially random starting distribution to the relatively well-defined  $\langle 011 \rangle$  fibre texture with the maximum of about 2.8 times random. This confirms the expected tendency for the austenite grains to rotate towards the  $\langle 011 \rangle$  direction during uniaxial compression [20].

### 3.2. Examples of the microstructure characteristics

The average grain orientations subjected to a detailed EBSD study are plotted in the inverse pole figure for the CA shown in Fig. 3. Some

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