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Spatial correlation between local misorientations and nanoindentation hardness in nickel-base alloy 690

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ABSTRACT

Misorientation increases with plastic strain in metals, and this observation has been used as an empirical assessment of plastic strain in recent years. The method has been validated for a sample area corresponding to a 100 μm × 100 μm square, but on the micrometer scale misorientations no longer seem to correlate with plastic strain. Misorientations are however not dependent on plastic strain but rather on dislocation density, which means it should also be related to hardness. Therefore, we have in this work compared maps of predicted hardness calculated from misorientation determination with maps of actual hardness measured by nanoindentation.

It was shown that the predicted and measured hardness maps do indeed correlate spatially in nickel-base Alloy 690, although the measured values have a significantly smaller hardness variation. This is explained by a presumably high and uniform density of statistically stored dislocations, which contribute to hardness but do not affect the misorientation determination from electron backscatter diffraction. Thus local misorientation can be used to qualitatively map the local effective plastic strain distribution, for example to identify regions of increased hardness.

1. Introduction

In recent years electron backscatter diffraction (EBSD) has been used to characterize changes in polycrystalline metals that result from plastic deformation [1–12]. Specifically the change in misorientations, i.e. the change in crystal lattice orientation between different points in the material, has become popular to focus on. A scalar misorientation quantity can be defined in different ways, and the most common methods have been summarized in literature [13]. No matter how misorientations are defined though, when averaged over a larger area they tend to increase with average plastic strain, and this empirical observation has allowed the use of EBSD for plastic strain estimation.

This misorientation based plastic strain assessment technique has been validated for quantification of plastic strain using a sample area corresponding to roughly a 100 μm × 100 μm square [1,2]. Spatial variations in local misorientation values can be seen on a micrometer scale as well, and attempts at correlating these local misorientations with strain measurements based on digital image correlation (DIC) have been performed, although with unsatisfactory results in copper [14,15] and aluminum [16].

Sasaki et al., however, discussed that DIC measures the actual strain, while misorientations between neighboring points reflect the local dislocation density, and that these two quantities do not necessarily correlate on a micrometer scale [15]. They illustrated that plastic deformation can accumulate where dislocations had passed through, but that misorientations only increased where dislocations accumulated. Misorientations may thus be closer related to *effective* plastic strain, which is a measure of strain hardening, i.e. dislocation density buildup, rather than plastic strain, which is a measure of geometric shape change.

Polycrystalline metals are plastically inhomogeneous at the microscopic size scale, thus an inhomogeneous hardness distribution can be expected as the material deforms. As such, maps of local hardness would be relevant for processing and monitoring degradation of materials where local material properties are important, e.g. recrystallization, stress corrosion cracking (SCC) and fatigue.

If misorientations do indeed correlate with effective plastic strain, it can be used as a tool to predict the hardness profile. Therefore, nanoindentation hardness mapping has been used in this work as a means to investigate the correlation with those predicted by misorientation mapping.

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An inherent uncertainty with the misorientation technique is that misorientations are in principle only dependent on the inhomogeneous straining, which induces geometrically necessary dislocations (GNDs), whereas the total dislocation density also consists of statistically stored dislocations (SSDs), which are induced by homogeneous straining [17]. Since homogeneous strains do not contribute to misorientations, but do contribute to strain hardening, misorientation distribution does not necessarily reflect the local hardness distribution accurately on the micrometer size scale, which necessitates a clarification of the actual relation between misorientation and hardness.

In practice, strains on a larger scale are often estimated from misorientation maps (acquired with EBSD), which suggests that a verified extension of this method to the micrometer scale would constitute a significant advance.

In this work we have shown the existence of a correlation between local hardness and local kernel average misorientation (KAM) which appears useful, but since deformation mechanisms can differ between materials, and even for the same material if deformation temperature or rate is significantly changed, we cannot *a priori* infer that the findings in this work are generally applicable.

2. Experimental

2.1. Test material

Alloy 690, a solution strengthened high chromium nickel-base alloy with a face-centered cubic (FCC) crystal structure, was used in this work. It is highly ductile, has significant strain hardening, and deforms by dislocation slip at the temperature and strain rates involved in this work.

The material used in this work came from a tube that has been characterized in a previous work [18]. In the as-delivered condition, the microstructure consisted of an austenitic matrix phase, fine intergranular $M_{23}C_6$ carbides, and large intragranular Ti(C,N) precipitates. This particular tube was chosen for having shown a homogeneous microstructure. The chemical composition of this tube is given in Table 1.

In order to minimize the overall dislocation density in the material, it was recovery heat treated at 1050 °C for 4 h and slowly cooled down to room temperature inside the furnace. This heat treated material has been considered as being free from effective plastic strains in this work. The heat treatment caused the average grain size, measured by the intercept method, to increase from around 50 μm to 70 μm. The Ti(C,N) precipitates remained unchanged, but the intergranular $M_{23}C_6$ carbides had changed from a semi-continuous network of fine carbides to coarse and discreet ones, but were still located exclusively at the grain boundaries. No intragranular $M_{23}C_6$ carbides were observed either before or after the heat treatment.

Cylindrical uniaxial tensile specimens were manufactured from the heat treated material along the tube's axial direction. The nominal diameter was 5.00 mm, and the gauge length was 12.55 mm. All specimens were strained at room temperature using a servo-hydraulic testing machine at a strain rate of approximately $3 \cdot 10^{-4}\ s^{-1}$, with strain, ε, defined as the logarithmic strain as

$$\varepsilon = \ln(L/L_0), \tag{1}$$

where L_0 was the length of a region before deformation, and L is the current length. The specimens were unloaded before failure and retained a plastic strain, ε_p, of 0.029, 0.062, 0.087, 0.116 and 0.144 respectively. The heat treated material exhibited a yield strength of 273 MPa.

2.2. Prediction of local effective plastic strains

Material from both the unstrained and strained specimens was used to prepare specimens for EBSD. The material was carefully sectioned along a plane normal to the tube's axial direction using a Struers Accutom-5 precision cut-off machine. The sampled material was mounted in conductive resin and ground using SiC paper, followed by polishing using diamond paste. Final polishing was performed using 0.06 μm colloidal silica.

An EBSD system from *HKL* Technology was used within a LEO 1530 Gemini field emission scanning electron microscope (SEM) to obtain crystal orientation maps on the sampled materials. The accelerating voltage was set to 15 kV, and the aperture was opened to 120 μm. The working distance differed slightly between samples, but was between 8–10 mm. On each specimen, an area of 610 μm × 460 μm was mapped using a square grid with step length 0.9 μm. *HKL* software was used for acquisition of the orientation maps and phase identification, but in-house scripts were used for post-processing of the orientation data. The indexing rates for the specimens were between 98.0–99.9%, and no cleanup of non-indexed points was performed.

An orientation smoothening filter proposed by Kamaya was applied to each orientation map [19]. This filter is essentially a grain-wise moving average of the orientation, and tends to increase the precision in the orientation map at the cost of a slight loss of spatial resolution. A square kernel of 3 × 3 data points was used for the moving average. A grain was defined as a region enclosed by boundaries containing more than 10 points, and a boundary was defined between two neighboring points when the misorientation between them exceeded 5°. This threshold was in general high enough to allow deformation-induced misorientations, but low enough to identify grain boundaries.

Each point in the EBSD map is directly connected to four neighboring points, and diagonally connected to four additional points. In this work, KAM with a kernel consisting of only the four closest neighboring points was used to assign a scalar value to each point, representing its local misorientation level. The KAM were lognormally distributed, and the average KAM, KAM_{ave}, over a sampled region was calculated as

$$KAM_{ave} = \exp\left[\frac{1}{N}\sum_{i=1}^{N}\ln(KAM_{L,i})\right], \tag{2}$$

where $KAM_{L,i}$ is the local KAM at point i, and N is the number of points in the sampled area. Such an average was calculated for each of the specimens with different level of strain. As shown in Fig. 1, KAM_{ave} increased approximately proportionally with uniaxial plastic strain, and a linear function was fitted through the data points to serve as a calibration curve. The level of misorientation uniformity in the specimens was assessed by splitting the full sampled area into 15 subregions of the same size. Their

Table 1
Composition of the alloy 690 tube.

Element	Ni	Cr	Fe	C	Si	Mn	P	S	N	Ti	Al
Weight-%	Bal.	29.5	10.0	0.020	0.28	0.31	0.007	0.001	0.040	0.35	0.18