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Effects of grain size on intergranular strain evolution in Ni

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Abstract

Experiments on 99% pure Ni samples have been carried out to investigate the dependence of deformation on grain size. The accumulated strains and texture changes within the grains over the sampling volume were measured by neutron diffraction as a function of (*h k l*) reflections and sample orientations with respect to the tensile axis. Inhomogeneous developments of strain are found. Large tensile lattice strain was found for reflection (2 0 0) in the direction parallel to the loading axis. Sample of smaller grain size displays larger strain magnitude. The overall trend of the lattice strain development predicted by the elasto-plastic self-consistent model agrees with the experiment to within 30% and 50% for the small grain and large grain samples, respectively.
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Keywords: Deformation; Grain size; Neutron diffraction

1. Introduction

Effects of grain size on material properties are significant. For example, the Hall–Petch relationship predicts yield stress increases as a function of decreased grain size. Similarly, improvement in fracture toughness and increase in the brittle–ductile transition temperature are also associated with small grain size.

Inhomogeneous plastic deformation leads to development of intergranular strains in polycrystalline materials has long been recognized. Microstresses develop on the scale of the grain size because distinct crystallographic orientations of grains deform plastically by different amounts since the angles which the fundamental slip directions make with the stress axis are different. Grains which are favourably oriented such that their slip systems are easily activated will undergo plastic deformation, whereas others whose critical resolved shear stress, τ_{CRSS}, is not exceeded, only respond to the load elastically. The deviations in plastic strains among grains are accommodated elastically. Since plastic deformation is irreversible, the elastic strains in grains caused by plastic anisotropy will remain upon unloading. The elastic tensile strains locked in grains with plastically hard orientations along the load axis are offset by compressive strains in plastically soft grains. Intergranular strains are defined as the elastic strains remaining in the grains because of plastic and elastic anisotropy. Intergranular strains in various materials measured by neutron diffraction have been reported [1–5]. Most of these measurements were compared with elasto-plastic self-consistent (EPSC) model based on crystal plasticity.

In this paper, a neutron diffraction experiment designed to study the grain size effect on the evolution of intergranular strain in Ni is described.

2. Experiment

The experiments were carried out on the E3 spectrometer at the NRU reactor with neutrons of wavelength 1.512 Å reflected from the (3 1 1) planes of a Ge monochromator at an angle of 88.04°. Söller slit collimators of divergence 0.36° were used before and after the sample.

A tensile sample was prepared from 99% pure Ni of average grain size 20 μm, labeled S1. Another tensile sample, labeled S2 was prepared from Ni of 200 μm grain size. Dimensions of the samples were $30(L) \times 4(W) \times 4(D)\,\mathrm{mm}^3$. Both samples were annealed in vacuum at 500 °C for 2 h before the experiment. Flow curve of the two materials are shown in Fig. 1. The yield stresses are 68 and 49 MPa for the small and large grain size materials. The samples were then pulled to 2%, 5% and 10% plastic deformation in a stepwise manner. The accu-

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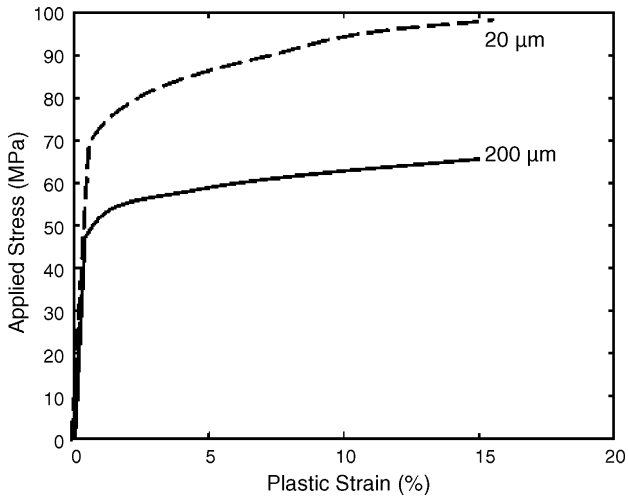

Fig. 1. Flowcurves of 99% pure nickel of grain sizes 20 and 200 μm.

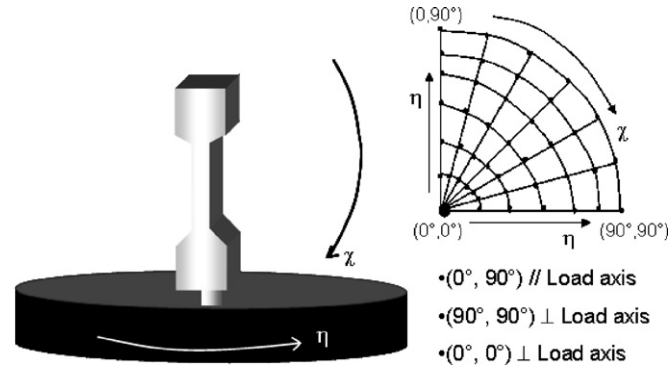

Fig. 2. The sample spherical coordinate system. η is the angle between a sample axis perpendicular to the tensile axis and the projection of the angular position of interest POI on the transverse plane. χ is the angle between the tensile axis and the POI. Lattice strain measurements were made on a (15°, 15°) grid as indicated by the intersections of the grid lines on the pole figure.

mulated strains and texture changes within the grains over the sampling volume were measured *ex situ*.

Prior to the tensile test, each sample was first characterized by determining the pole figures for the reflections (1 1 1), (2 0 0) and (2 2 0) which were used to deduce the orientation distribution function. The elastic lattice strain variations as a function of angular positions with respect to the tensile axis were measured for the reflections (1 1 1), (2 0 0), (2 2 0) and (3 1 1) by positioning the samples on an Eulerian cradle. The spherical coordinate system (η, χ) used to describe the angular position is illustrated in Fig. 2. η is the angle between a sample axis perpendicular to the tensile axis and the projection of the angular position of interest POI on the transverse plane. χ is the angle between the tensile axis and the POI. Measurements were made over a quadrant of a hemisphere on a (15°, 15°) grid. The intergranular strain states for the same four reflections were determined after each deformation step. The reference lattice parameters a_{ref} of the samples were determined by averaging measurements of all the reflections and angular positions from the undeformed samples. Pole figures for the reflections (1 1 1), (2 0 0) and (2 2 0) were measured for both samples after the tensile test to determine grain reorientations during deformation.

3. Results

Initial strain states for reflection (2 0 0) of both samples were shown in Fig. 3. The reference lattice parameter, a_{ref}, for samples S1 and S2 were determined to be 3.5265 ± 0.0001 and 3.5264 ± 0.0001 Å, respectively. Both samples show a small distribution of strains within $\pm 200 \times 10^{-6}\epsilon$ for all four measured reflections. Lattice strain in the direction of the tensile axis for reflection (2 0 0) is plotted as a function of plastic strain for both samples in Fig. 4. Both samples display a tensile lattice strain; however the magnitude is consistently smaller for sample S2.

The intergranular strain variation for reflection (2 0 0) as a function of (η, χ) for both samples after 10% strain is shown in Fig. 5. The strain is largest along the tensile axis with magnitudes of $1370 \pm 200 \times 10^{-6}\epsilon$ and $720 \pm 100 \times 10^{-6}\epsilon$ for samples S1 and S2, respectively. Except for the (2 0 0) reflection, all reflections display negligible residual strains in nearly all sample directions with no observable feature.

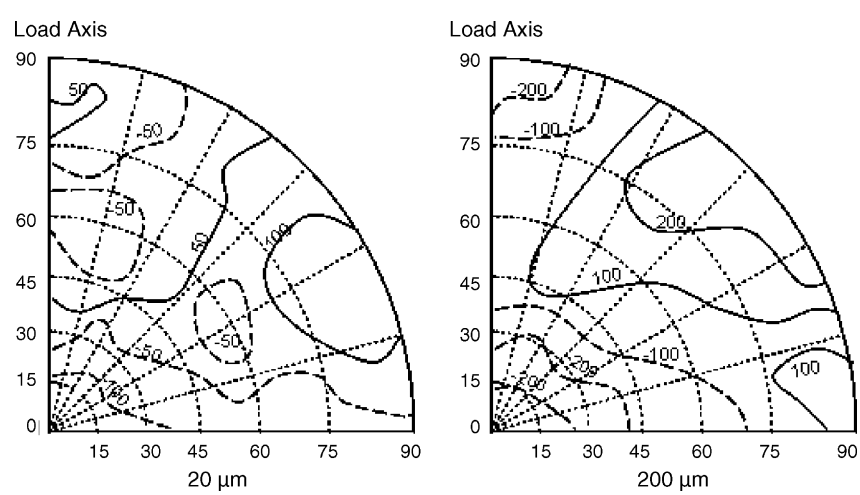

Fig. 3. Initial strain states for reflection (2 0 0) of Ni tensile samples S1 (20 μm grain size) and S2 (200 μm grain size). Compressive strains are indicated by dashed contour lines and tensile strains are indicated by solid contour lines. All strain values are in the units of 10^{-6} strain with uncertainty of $100 \times 10^{-6}\epsilon$ on average.

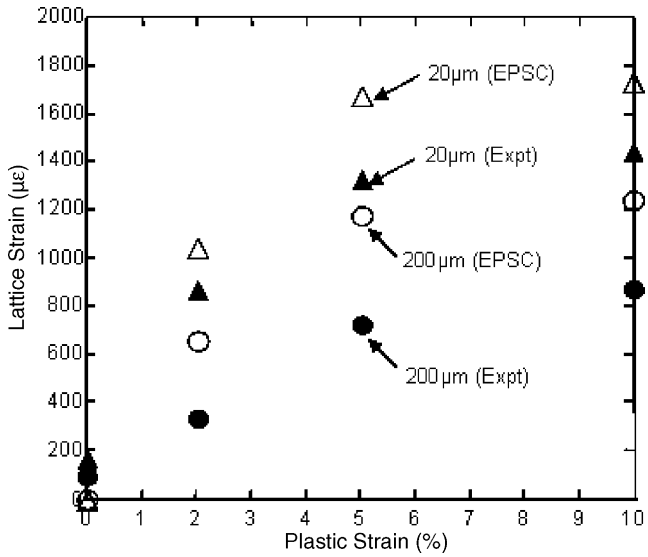

Fig. 4. Strain response in Al7050 along the axis of the tensile sample for the (2 0 0) reflections as a function of plastic strain.

An axisymmetric pattern about the tensile axis is found for the (2 0 0) strain pole figure. It is observed that strain variations are relatively independent of η as the contour lines are mostly parallel to the constant χ gridlines. The observed symmetry is not unexpected as the strain response might be expected to be axisymmetric about the tensile axis.

The results for the Ni samples with large response from reflection (2 0 0) are comparable to measurements made on other fcc materials, Inconel [6] and an austenitic steel [2], which have anisotropic elastic properties similar to nickel.

Similar behavior in the texture measurements was found for both samples S1 and S2. (1 1 1) and (2 2 0) pole figures for sample S1 before and after 10% strain are plotted in Fig. 6. Both samples exhibited random orientation distribution before deformation. The intensity distribution of the peaks changes because of grain reorientations. The (1 1 1) orientation exhibits an increase in concentration whereas a drop is found for (2 2 0) along the tensile axis. The experimental results agree with the predictions of grain rotations in an fcc alloy [7]. When the load axis of a crystal is near the [1 1 0] direction, the crystal rotates such that direction [1 1 1] is parallel with the load axis.

4. Elasto-plastic self-consistent simulations

The lattice strain and texture measurements provided the information on the statistical ensemble that can be compared with the elasto-plastic self-consistent (EPSC) calculation developed by Turner and Tome [8,9].

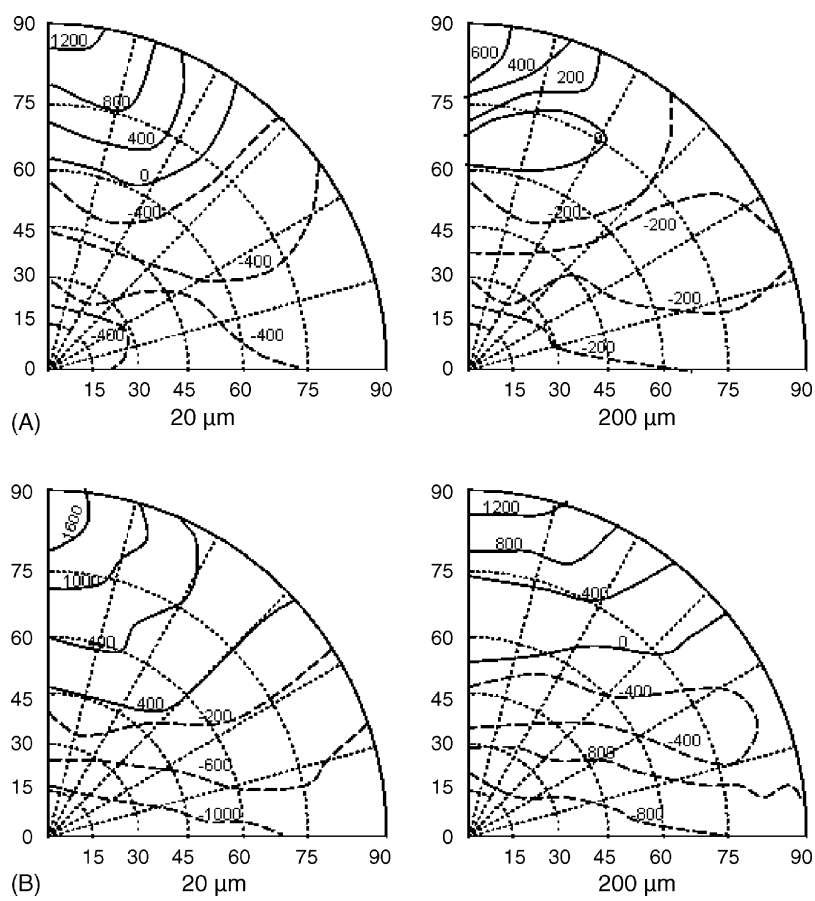

Fig. 5. Final strain states for reflection (2 0 0) of Ni tensile samples S1 (20 μm grain size) and S2 (200 μm grain size): (A) experimental results; (B) EPSC calculation. Compressive strains are indicated by dashed contour lines and tensile strains are indicated by solid contour lines. All strain values are in the units of 10^{-6} strain with uncertainty of $100 \times 10^{-6}\epsilon$ on average.

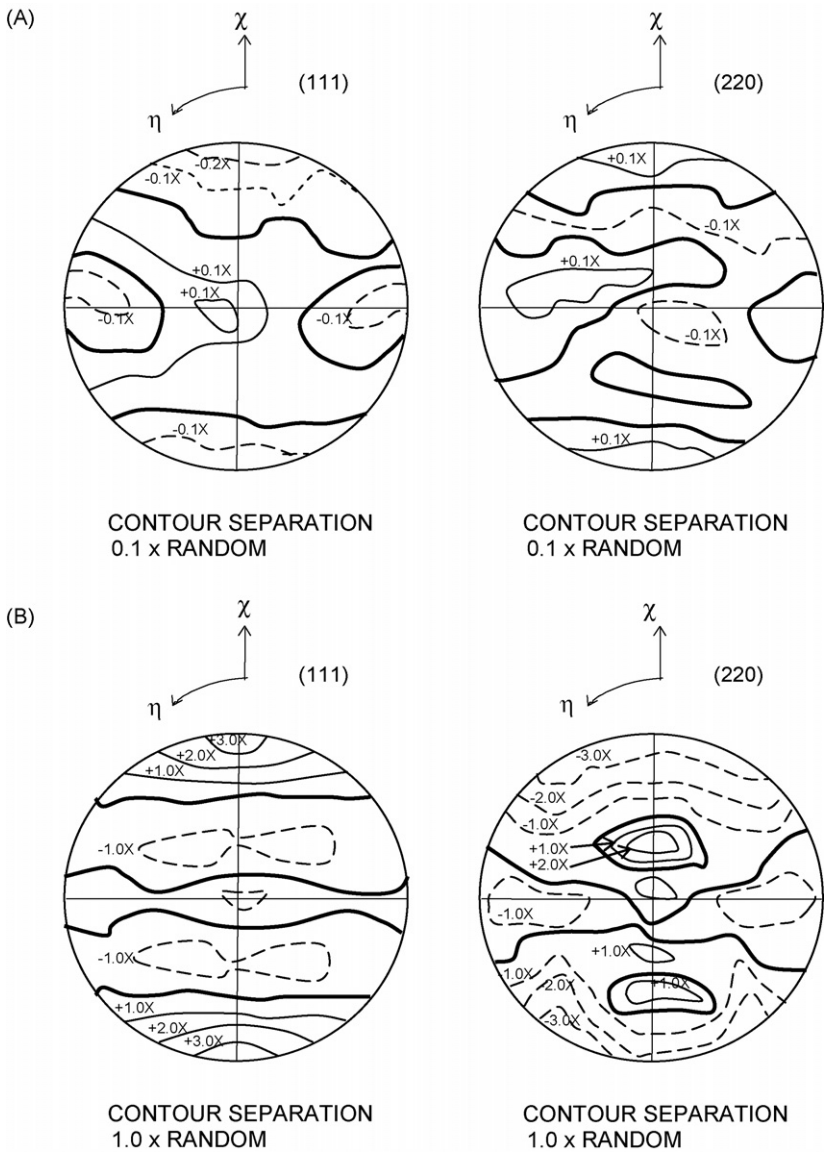

Fig. 6. (1 1 1) and (2 2 0) pole figures of sample S1 measured (A) before tensile test; (B) after 10% uniaxial deformation. Heavy solid lines are the random contours. Contours above random are indicated by solid lines, and dotted lines represent contours below random.

The texture of the samples prior to the tensile test is described by a set of 2032 grains. The single crystal elastic constants of Ni with $c_{11} = 161.54$, $c_{22} = 69.23$ and $c_{12} = 132.00$ GPa are used. The 12 potential slip systems were those of type $\{1\,1\,1\}\ \langle 1\,0\,\bar{1}\rangle$. Self-hardening was assumed and the onset of each slip system is independent of the others. The experimental flow curve was simulated closely by setting the parameter τ_{CRSS} to 14 and 22 MPa for samples S1 and S2.

The simulated lattice strain response parallel to the tensile axis for reflection (2 0 0) is plotted alongside experimental data in Fig. 2. The model predicts an increase of the tensile lattice strain as measured, with sample S1 increases at a faster rate than S2. However, the model predicts larger strain magnitude ∼30% larger for sample S1 and ∼50% large for sample S2.

The model calculation for the lattice strain variation as a function of angular position for reflection (2 0 0) after 10% strain is plotted in Fig. 3 together with the data. The overall pattern—changing from tensile strain along the loading axis to small compressive strain as the angular position moves towards the transverse plane, is predicted correctly. Overestimation of the strain magnitudes was observed. The model also predicts a larger response for other reflections, although reflection (2 0 0) was found to have the most pronounced effect as shown in the experiment.

5. Discussions

The causes of the discrepancies in the comparison of the intergranular strains may be inadequate descriptions of the hardening or the slip systems. Definition of hardening of each slip difficulty is a major difficulty with the model. An attempt has been made to change the mode of hardening. Instead of self-hardening, it was assumed that the activation of a slip system has the same hardening effects on all the other slip systems. However, sim-

ilar results were obtained to those with self-hardening except that the overall magnitudes of the strains have dropped by about 20% for both samples.

The experimental results suggest that grain size does not alter the overall pattern of the lattice strain evolution but smaller grain size does lead to larger lattice strains. This is also proved by the model calculation. The model is in better agreement with the small grain size sample, S1. The lower critical resolved shear stress of the large grain sample may have affected the intergranular strain magnitude as the applied stress required for a particular plastic strain is smaller, and hence reduced the elastic lattice strain required to balance the inhomogeneous plastic activities between grains. Effects of grain size on deformation texture and mechanical behavior have been reported. However, the deformation texture of the two samples studied are similar, this may be attributed to the relative small deformation (10%ϵ) applied in this experiment. Because deformation modes are expected to be dominated by grain boundary activities as grain size reduces to submicron size, measurements on smaller grain size samples may give a completely different result.

Grain size effect on mechanical properties is believed to be contributed by the increase in density of grain boundary; it will be interesting to determine variations in strains and misorientations within grains, subgrains and grain boundaries for the two samples. Neutron diffraction does not yet have the resolution to perform such measurements. Experiment using synchrotron radiation with submicron beam will be carried out to study the two samples in the near future.

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