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TEM study of dislocation patterns in near-surface and core regions of deformed nickel polycrystals with few grains across the cross section

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ABSTRACT

TEM investigations of dislocation structures in core and surface regions (50 μm below the free surface) were carried out on polycrystalline nickel samples of 500 μm thickness with 14, 2.5 and 1 grain across the thickness. The mean diameter of the dislocation cells was measured for different strain levels in core and surface regions of the three kinds of sample. These mean dislocation cell diameters were compared between the different samples using a statistical analysis of variance. The intragranular long-range internal backstress level was thereafter estimated in both regions for the three samples revealing a decrease in stress for specimens with few grains across the thickness. This decrease is partially responsible for the flow stress decrease of the nickel polycrystals with few grains across the thickness.

1. Introduction

Since a few decades, the industrial need of micro metal parts has considerably increased due to the trend of miniaturization of medical, electrical or mechanical devices. For these small dimensions parts, the influence of the surface effects on the mechanical behaviour is very important. The forming process of thin samples is consequently difficult due to the low values of thicknesses and the low number of grains across the thickness (ratio thickness "t" over grain size "d") and must be improved. For these samples, the anisotropy was found to be modified (Kals and Eckstein, 2000) and the sustainable strains for the sheets during the forming process were found to decrease with a decrease in thickness (Diehl et al., 2008) and in t/d ratio (Keller et al., 2009). All these modifications make the forming process less reliable (Vollertsen et al., 2006).

The effect of the free surfaces on the mechanical properties has been widely studied for single crystals during the last decades, using mechanical tests and TEM investigations (Fourie, 1968, 1970; Fourie and Dent, 1972; Mughrabi, 1970, 1971). The aim of these studies was to investigate the way of modelling the mechanical behaviour of an overall strained sample by the examination of the slip lines on free surfaces. However, the occurrence of a stress gradient and a modification of the hardening stages near the free surfaces were reported (Fourie, 1968). As a result of the escape of dislocations through the free surface, the density of primary dislocations seems to be greatly reduced. Mughrabi (1970) reported a factor 2.5 between the dislocation density near the free surface and in the core region for Cu single crystals oriented in single glide in stage II. The dislocation structures in the vicinity of the free surfaces were found to be less strongly developed than in the core regions inducing a stress decrease. The last point remains speculative without precise and quantitative measurements.

For polycrystals, numerous mechanical investigations were achieved on metallic materials with few grains across the thickness. For high purity materials and industrial alloys, the flow stress was found to decrease when the number of grains across the thickness is reduced below a critical value. This latter depends on stacking fault energy (Miyazaki et al., 1979) and strain (Keller and Hug, 2008). The strain hardening behaviour and the validity of some well-known constitutive laws are also modified (Keller et

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al., 2009). For nickel polycrystals, this mechanical behaviour modification seems to be explained by a delay of cross-slip activation for the low t/d ratio specimen which modifies the dislocation structures (Keller et al., 2009). Nevertheless, only few experiments were dedicated to the microstructural characterization of the surface effect. TEM investigations and X-ray diffraction were carried out for Cu, Al and Ni (Kolb and Macherauch, 1961; Miyazaki et al., 1979; Swann, 1966). For Al, Miyazaki et al. (1979) compared the dislocation structures between a core and a surface grain for a sample with few grains across the thickness strained to 1%. They reported a modification of the dislocation structures for the surface grains. For Cu, dissolution of the dislocation walls for distance around 5 μm below the free surfaces was reported by Swann thanks to TEM investigations (Swann, 1966). These observations were made in a coated cross-section of a sample strained to 15%. For Ni, residual stress analysis by X-ray diffraction shows that the grains on the vicinity of the free surfaces are less constrained than core ones (Kolb and Macherauch, 1961). Consequently, for polycrystals, the stress level seems to be reduced in the vicinity of the free surfaces as in single crystals. Nevertheless, the inherent heterogeneities of stress and strain in polycrystals due to the crystallographic texture and grain boundaries were not taken into account in TEM investigations by these authors. The surface effect analysis was achieved on only one or a few grains while TEM studies need a high number of analysed grains to be meaningful (see for example Feaugas and Haddou, (2007)). The conclusion given by the previous authors may depend on the selection of the analysed grains. Quantitative and statistical TEM investigations of the dislocation structures are then necessary to study rigorously the surface effect on polycrystals. The knowledge of the surface effect influence on the mechanical behaviour of metallic samples would bring new light on the way of improving the forming process.

The purpose of this paper is first to carry out a quantitative and statistical study of the dislocation structures on surface (50 μm below the free surface) and core regions for three nickel polycrystals with 14, 2.5 and 1 grain across the thickness. Secondly, a mean value of local intragranular internal stress level is estimated from the cell dislocation diameters in both regions as a function of plastic strain. These intragranular stress levels are thereafter compared to other values found by mechanical tests by the authors in a previous paper (Keller et al., 2009). The overall purpose is to explain the stress decrease and strain hardening modification for nickel samples with less than four grains across the thickness.

2. Experimental procedure

2.1. Sample preparation and characterization

High purity polycrystalline nickel (99.98 wt.%) consisting of 0.5-mm thickness rolled sheet was employed. Choosing a simple face centred cubic (fcc) metal with high stacking fault energy (125 mJ/m^2) is suitable for TEM investigation of the free surface effect. In such metals, the deformation mechanisms are well-understood and the typical dislocation cell structures taking place with strain are more practical than Taylor lattices (typical of low stacking fault energy fcc metals) to estimate the mechanical properties by TEM observations (Kuhlmann-Wilsdorf, 1989).

Dog bone shape samples were first cut parallel to the rolling direction and then annealed into secondary vacuum (10^{-6} mbar) to annihilate the rolled texture and obtain different grain sizes without oxide surface layer formation. Three types of microstructure were obtained (Fig. 1) with grain sizes of 36 μm, 200 μm and 500 μm (calculated from surface grains analysis) inducing t/d ratios (thickness over grain size) equal to 14, 2.5, and 1. All samples present a weak texture, with a low maximal density pole around 2 mrd (Multiple Random Density), revealed by neutron diffraction. More details about the heat treatment, the microstructure and the crystallographic texture analysis can be found elsewhere (Keller et al., 2009).

2.2. Mechanical tests

The three kinds of samples were deformed up to strains of 0.025 (0.03 for $t/d = 14$), 0.05 (only for the specimen with $t/d = 1$), 0.1 and up to fracture by a uniaxial tensile machine strain controlled by an extensometer mounted on the gauge section. The initial rolling direction of the samples was kept parallel to the tensile axis. The strain amounts were chosen as a function of the strain hardening stages: end of the second hardening stage for $\varepsilon = 0.025$ (0.03 for $t/d = 14$) and third stage for the other strain levels. The strain rate, $\dot{\varepsilon}$, was set up to 2.4×10^{-4} s^{-1}.

2.3. Sample preparation for TEM analysis

Thin foils were cut parallel to the free surface of the samples and prepared for TEM investigations. The foils were first mechanically grinded to a thickness of 100 μm and were then electrolytically polished (25% nitric acid, 75% methanol, 8.5 V, 268 K) by a Struers TenuPol® to be observable by TEM. Two different kinds of thin foils were observed. The first one proceeds from core regions of the samples and was taken in the middle of these latter (≈250 μm below the free surface) by grinding the two lateral surfaces. The second one was taken near the free surfaces by grinding the opposite side of the free surface. The observed region is located around 50 μm below the free surface (Fig. 1). Hence, for specimen with 14 and 2.5 grains across the thickness, core and surface grains were observed. For specimens with one grain across the thickness, most surface and core regions observed by TEM proceed from central grains with no horizontal grain boundaries. The investigations were carried out on a side entry Topcon 02B transmission electron microscope, working at 200 kV (tilt +/− 10°).

2.4. Statistical interpretation of TEM observations

An image analysis of the TEM pictures was carried out to estimate the mean cell diameter in the interior region of grains to avoid the contribution of the strain incompatibilities between grains on the experimental results. For each sample and each region, more than 10 grains were analysed with a number of cells ranging between 50 and 220 depending on the samples (see Table 1).