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Investigation of the deformation structure in an aluminium magnesium alloy by high angular resolution three-dimensional X-ray diffraction

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The deformation structure in individual grains of an aluminium magnesium alloy deformed up to 10% in tension is characterized with high angular resolution three-dimensional X-ray diffraction. The three-dimensional intensity distribution (in reciprocal space) of all reflections investigated is rather smooth. The absence of individual sharp peaks indicates the absence of a dislocation cell structure, which was confirmed by transmission electron microscopy. Nevertheless, the formation of different orientation components associated with different elastic strains is evident within a grain.

Keywords: X-ray diffraction (XRD); Aluminium alloy; AlMg; Dislocation structure; Plastic deformation

When metals deform plastically, dislocations get stored in the material and ordered dislocation structures evolve. In pure face centred cubic metals, such as aluminium and copper, cell structures form consisting of dislocation-poor regions separated by dislocation-rich walls (e.g., [1]). The formation of such three-dimensional (3-D) cell structures is generally attributed to the mobility of dislocations in three dimensions [2]. Dislocations become confined to their glide planes either by a decreased stacking fault energy or by interaction with point defects [3]. By alloying, the structure formation is altered and the formation of cell structures eventually suppressed. Examples of non-cell-forming metals are brass and aluminium magnesium (AlMg) alloys.

Recently, we have developed a synchrotron-based technique, ''high angular resolution three-dimensional X-ray diffraction'' (3DXRD) [4], which allows for 3-D high resolution reciprocal space mapping of Bragg reflections with high angular resolution. In this manner, information on the distortion of the crystalline lattice is obtained non-destructively from individual grains deeply embedded in polycrystalline samples. Deformation structures on the scale of the dislocation structure can be monitored in situ during plastic deformation of macroscopic specimens with thicknesses up to 1 mm – a major advantage compared to the thin foils required for transmission electron microscopy.

We have shown [4,5] that 3-D maps of reflections from deformed cell-forming materials (with Cu as model material) show a remarkable structure at high resolution, consisting of sharp, bright peaks superimposed on a cloud of enhanced intensity. These peaks are interpreted as being the diffraction signal from individual, nearly dislocation-free regions in the deformation structure [4,5]. The diffuse cloud of enhanced intensity is interpreted as arising from the dislocation-dense walls [4,6].

The aim of the work reported here is to apply the same technique to almost uniform random dislocation structures. Such an investigation provides a critical test for the validity of our interpretation of the features observed in Bragg reflections from cell-forming metals. If our interpretation is correct, the reflections from a material with a uniform dislocation structure are supposed to be rather structure-free, similar to the cloud attributed to the walls in cell-forming materials. Simultaneously,

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Table 1. Chemical composition of the AlMg alloy studied as measured by optical emission spectroscopy

	Al	Mg	Fe	Mn	Si	Cr
Contents [wt.%]	95	3.9	0.29	0.53	0.14	0.091

Reporting only alloying elements with content larger than 0.05 wt.%.

information on the deformation structure of individual grains in the bulk of a non-cell-forming material is gained in situ during loading.

The sample material used was a commercial AlMg alloy with a Mg content of 3.9%. The chemical composition is provided in Table 1. The as-received material was cold-rolled to 90% thickness reduction followed by a 50% rolling reduction to a final thickness of 500 μm and then fully recrystallized by heat treatment for 1 h at 575 °C, resulting in a mean grain size of 34 μm (equivalent disc diameter obtained from EBSD).

For AlMg alloys, the cell formation ability depends on the Mg content. Cell structure formation is completely suppressed for 3% Mg (e.g., [7,8]) and dislocation tangles are observed [9,10] (frequently with microbands superimposed). The morphology of the dislocation structure changes significantly within and between individual grains [7,10]. The dislocation tangles in AlMg are often characterized as random, or alternatively as organized in general Taylor lattices with lower energy than a random dislocation structure [9]. A strong influence of the iron content on the dislocation tangles in AlMg has been found by transmission electron microscopy (TEM) and X-ray line broadening analysis [11,12]. Seemingly random tangles are formed in an alloy with an Fe content of 0.15%, whereas the dislocations appear to be organized in planar channel arrangements for an alloy with lower Fe content.

Based on the Fe content of 0.29% of our material, an almost uniform dislocation structure is expected after deformation. To confirm this expectation, the dislocation structure has been investigated with a transmission electron microscope, JEOL 2000FX, operated at 200 kV on a thin foil prepared from a longitudinal section of the specimen tensile-deformed to 10% strain during the 3DXRD investigation. Local orientations were determined using a Kikuchi line analysis technique [13]. Figure 1 shows the dislocation structure developed in a grain with the [1 0 0] direction close to the tensile axis. The dislocations are distributed fairly uniformly and are arranged in random dislocation tangles. Neither dislocation cells nor dislocation walls are found. The observed structure is substantially different from the well-developed cell structure in grains of similar orientation in pure copper or pure aluminium tensile-deformed to comparable strains [14,15].

The X-ray diffraction experiment was performed at the 1-ID beam line at the Advanced Photon Source (Argonne National Laboratory, Argonne, IL, USA) using the setup presented in Refs. [4,5]. The sample was illuminated by a 52 keV X-ray beam with very low relative energy spread (7×10^{-5}), low divergence (17 μrad) and high flux. The beam size was defined by slits to be $\approx$14 μm × 14 μm. The sample was mounted in a custom-made tensile rig, positioned on a three-axis goniometer. This allows for in situ deformation and orientation of the sample. A MarCCD 165 area detector was positioned 3.7 m and 0.98 m from the sample in the horizontal and vertical direction, respectively, in order to image a 400 reflection. High resolution reciprocal space maps were acquired by rocking (i.e. exposing while rotating the sample at constant speed) the sample around an axis perpendicular to the scattering plane at small consecutive intervals. By stacking the raw images, a 3-D map of the reciprocal space was constructed. A sketch of the scattering geometry is shown in Figure 2 including a definition of the coordinate system of the reciprocal space (described by the three perpendicular vectors $\mathbf{q}_x$, $\mathbf{q}_y$, and $\mathbf{q}_z$) [5].

A well-separated 400 reflection with the scattering vector close to the tensile direction was chosen from the undeformed sample, and the corresponding grain centered in the X-ray beam. By spatial scanning of the grain through the X-ray beam it was inferred that the grain is smaller than the beam size, and that the grain hence was fully illuminated. By means of the available rotations the reflection was aligned to scatter onto the detector. The sample was deformed stepwise to 1.8%, 4.2% and 10% tensile strain. At a strain of 4.2% three additional 400 reflections, corresponding to three other grains (each with the scattering vector close to the tensile axis), were identified. Each grain was centred in the

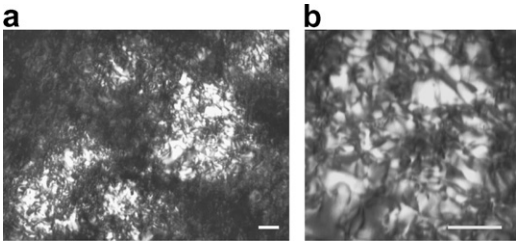

Figure 1. Transmission electron micrographs taken in the [0 0 1] beam direction on a grain having a [1 0 0] direction 9° off the tensile axis. The scale bars are 200 nm. (a) reveals a high dislocation density rather uniformly distributed over the entire image. Slight orientation differences below 2° cause the light and dark contrast in different regions. (b) shows a region with light contrast from the same area in higher magnification.

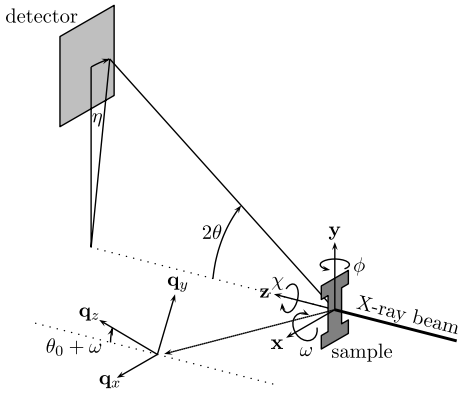

Figure 2. Sketch of the experimental setup [5] defining the laboratory coordinate system **x**, **y** and **z**, available rotations ϕ, χ and ω, scattering angles 2θ and η, and the reciprocal space coordinate system ($\mathbf{q}_x$, $\mathbf{q}_y$, $\mathbf{q}_z$).