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3D characterization of partially recrystallized Al using high resolution diffraction contrast tomography

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ABSTRACT

Synchrotron diffraction contrast tomography (DCT) is for the first time used to characterize recrystallized grains in partially recrystallized Al. The positions, orientations and 3D shapes of >900 recrystallized grains are reconstructed within a gauge volume. The results are compared with those obtained using electron backscattered diffraction based on a statistical analysis. It is found that recrystallized grains with size larger than 10 μm, corresponding to ~98% of the total recrystallized volume of the sample, are well characterized by DCT. The advantages of DCT for recrystallization studies and new possibilities with DCT on new generation synchrotron sources are discussed.

During recrystallization of deformed materials, nearly defect-free nuclei/grains form and grow at the expense of the surrounding deformed matrix. Recent 3D results obtained using serial sectioning have shown that the distribution of nuclei/recrystallized grains is non-uniform, and depends strongly on the local deformation microstructure [1–4]. Although both deformed matrix and nuclei/recrystallized grains can be characterized with these techniques, only limited volume or limited spatial resolution can be obtained when the sectioning is done with focused ion beam [1] or mechanical polishing [2–4], respectively. More importantly, the destructive nature of the techniques prohibits the possibility of dynamic studies to follow the recrystallization process, which is essential for a better understanding of the heterogeneities of recrystallization.

During the last 15–20 years, several non-destructive 3D characterization techniques have been invented and implemented using synchrotron X-rays, including 3D X-ray diffraction (3DXRD) [5–8], diffraction contrast tomography (DCT) [9,10], Bragg coherent diffraction imaging [11,12] and 3D X-ray Laue microdiffraction [13,14]. Among these techniques, DCT allows fast 3D reconstruction of grains in fully recrystallized samples [15,16]. Although the nuclei/grains in partially recrystallized samples are also in the recrystallized state (i.e. with very low densities of interior defects), the nuclei/grains are relatively small compared to those in fully recrystallized samples. At the same time, parts of the microstructure consist of deformed grains with significant orientation variations and strain gradients. Some of the nuclei/grains may have orientations similar to the neighboring deformed grains, from which they originate. All these factors make reconstruction of the recrystallized grain very challenging [10]. To what extend DCT can be used to characterize partially recrystallized samples is therefore still an open question.

In the present paper, we will for the first time explore the applicability of DCT for recrystallization characterization. A partially recrystallized Al sample will be used as a model material. To validate the DCT results, they will be compared with those obtained based on conventional characterization using electron backscattered diffraction (EBSD). Statistical analysis will be performed to link the 2D EBSD and the 3D DCT results and ease the interpretation of the results.

Commercial purity aluminium, Al1050, was used for the present study. The starting material had a fully recrystallized microstructure with an average grain size of ~50 μm. The material was then cold rolled to 50% reduction in thickness, followed by annealing at 325 °C for 0.5 h to obtain a partially recrystallized microstructure. For the synchrotron DCT measurement, pillar samples with cross-section of ~420 × 420 μm^2 were cut out using electrical discharge machining. The DCT measurement was conducted at beamline ID11 at the European Synchrotron Radiation Facility (ESRF). The X-ray energy was 37 keV. The direct X-ray beam was constrained by two sets of vertical and horizontal slits to 240 × 420 μm^2. Transfocators with beryllium lenses were used to focus the

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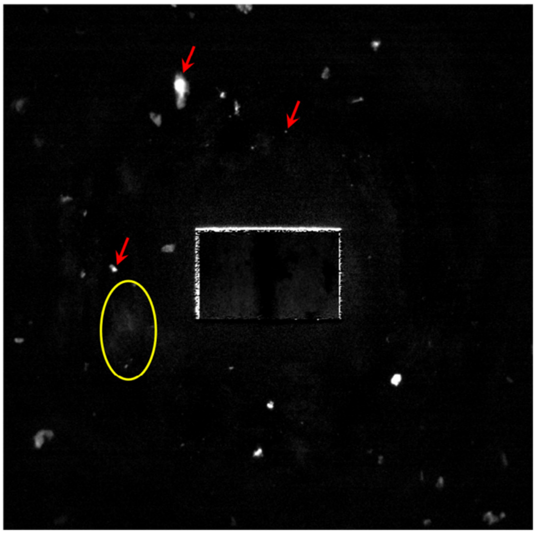

Fig. 1. A typical near-field diffraction image of the partially recrystallized Al sample, recorded using a near-field detector with 2048 × 2048 pixels (pixel size of 0.77 μm). The red arrows mark examples of diffraction spots originating from recrystallized grains, while the yellow ellipse marks diffraction from a deformed grain. The rectangle region in the middle is from the direct X-ray beam. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

beam. A uniform beam was obtained within the defined gauge. The sample was mounted on an ω rotation stage with the sample rolling direction (RD) parallel to the vertical rotation axis. A full 360° scan was performed with angular integration steps of 0.1° and an exposure time of 2 s for each projection. Both the diffracted and the transmitted beam were recorded using a near-field detector (comprising a transparent luminescent screen, light optically coupled to a CCD) with 2048 × 2048 pixels positioned normal to the incident beam at a sample-to-detector distance of 3 mm. To improve the spatial resolution, an eye-piece was used to magnify the diffraction images. The effective pixel size of the detector system was 0.77 μm. The detailed information about the DCT setup can be found in [9,10].

A typical diffraction image is shown in Fig. 1. Most of the diffraction spots, e.g. those marked by the red arrows, are sharp and well-defined, appearing typically at 1–2 consecutive angular steps. These diffraction spots originate from recrystallized grains. Only few blurry, large and weak diffraction spots, as the one marked by the yellow ellipse, are visible and appear typically over a large number of angular steps. These originate from the deformed matrix.

Table 1
Microstructural parameters for recrystallized grains calculated based on DCT and EBSD data. V_V, D_{ESD}, N_V and N_{total} are volume fraction, average grain size, number of grains per unit volume and total grain number, respectively. For EBSD data, the D_{ESD} was calculated based on the statistical analysis.

	V_v	D_{ESD} (μm)	N_V (10^{-5} μm^{-3})	N_{total}
DCT	24%	18.4	2.7	929
EBSD	28 ± 4%	14.6	4.1	469

The diffraction images were analyzed with the DCT software (http://sourceforge.net/projects/dct) available at the beamline. A recently developed algorithm allowing 6D reconstruction (3D for position + 3D for orientation) [17,18] is used to reconstruct the grain shapes. In total 929 recrystallized grains are found within the gauge volume. The 3D volume is shown in Fig. 2a. The recrystallized grains are typically not spherical but elongated along the RD and the transverse direction (TD). Also the recrystallized grains are not randomly distributed, but clustered in bands parallel to the RD and TD. For clear visualization, a typical 2D section is shown in Fig. 2b. The volume fraction, V_V, of the recrystallized grains is about 24% and the average grain size (defined as equivalent spherical diameter (ESD)) is about 18 μm. The number of grains per unit volume, N_V, (determined by N_{total}/V, where V is the gauge volume) is about 2.7×10^{-5} μm^{-3} (see Table 1).

To verify the 3D results, the microstructure of the sample was characterized using a Zeiss Supra 35 field emission gun scanning electron microscope equipped with a Channel 5 EBSD system. EBSD measurements were conducted on the longitudinal section (defined by the RD and normal direction (ND)) using a scanning step size of 0.5 μm, covering two areas of ~6.7×10^5 μm^2.

An example of the EBSD maps is shown in Fig. 3a. The recrystallized grains are selected from the EBSD maps using the following criteria: i) grain size (equivalent circular diameter, ECD), larger than 3 μm, ii) surrounded at least partly by high angle boundaries to the deformed matrix, iii) the internal misorientation angle is <1°. Fig. 3b shows the recrystallized grains only. Similar to the 3D results, clusters of recrystallized grains are seen in bands parallel to the RD. In total, 469

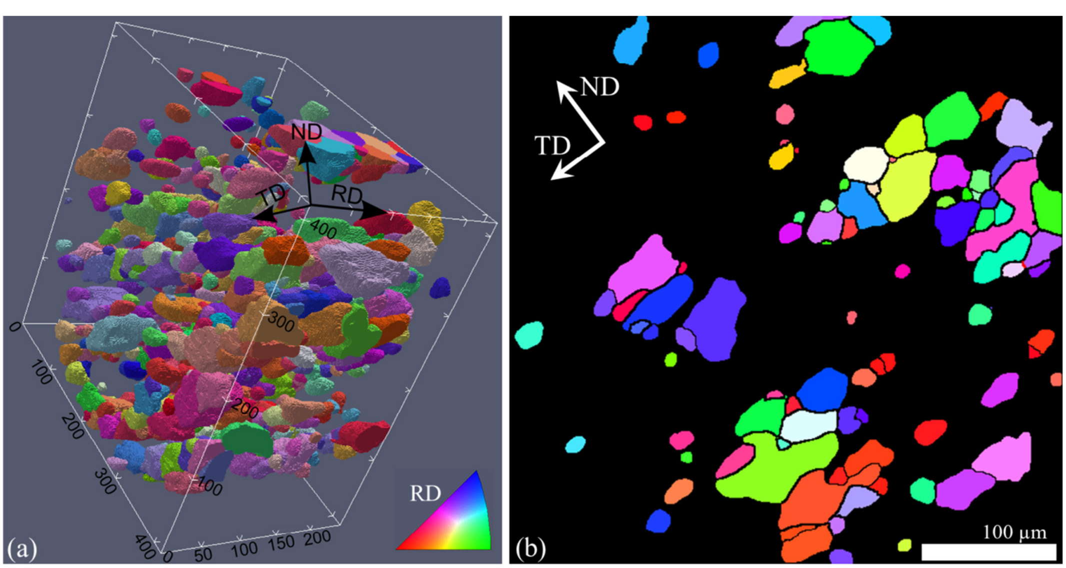

Fig. 2. 3D reconstruction of recrystallized grains measured by DCT. (a) The 3D volume of 240 × 420 × 420 μm^3; (b) a 2D section. The colors in both (a) and (b) represent the crystallographic orientation along RD (see the inset in (a)). In (b) thin and thick black lines represent boundaries with misorientation >2° and >15°, respectively. The voxel/pixel size is 0.77 μm. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)