



## Correspondence

## In-situ characterization of continuous dynamic recrystallization during hot torsion of an Al–Si–Mg alloy



## A B S T R A C T

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Synchrotron x-ray diffraction  
Aluminium alloys  
Hot deformation  
Continuous dynamic recrystallization  
Dynamic recovery

An extruded Al–Si–Mg alloy was deformed by torsion at 400 °C during in-situ high energy synchrotron radiation diffraction. This technique is used to prove, by analysing texture changes and the coherently scattering domain size evolution, that dynamic recovery followed by continuous dynamic recrystallization are the main restoration mechanisms. Moreover, the dislocation density types corresponding to each stage of deformation are discussed and the recrystallization grade is calculated.

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## 1. Introduction

Many metals undergo dynamic recrystallization (DRX) during hot deformation and their stacking fault energies (SFE) determine the type of recrystallization. Discontinuous dynamic recrystallization (dDRX) is linked to materials with low and moderate SFE which undergo nucleation and grain growth [1,2]. Two mechanisms have been suggested for the changes of the microstructure of high SFE materials during large plastic deformations: 1) continuous dynamic recrystallization (cDRX), and 2) geometric dynamic recrystallization (gDRX). The cDRX requires a change in texture [1–3] and involves the progressive formation and rotation of subgrains. During this process, subgrains increase their wall dislocation density provoking a misorientation increment and low angle grain boundaries (LAGBs) transform into high angle grain boundaries (HAGBs) [3–9]. In gDRX texture remains unchanged [10,11] and it fulfils the Taylor theory basis [12], requiring subgrain formation and subsequent migration and pinching off of original HAGBs [8]. Both mechanisms can occur at the same time [13,14] and the third-generation of synchrotron radiation sources can be a powerful tool to track and identify these physical phenomena occurring during thermomechanical processes [15].

The main goals of this work are to extract microstructural information from a high temperature torsion test using in-situ high energy synchrotron radiation diffraction (HEXRD) and to clarify if the formed HAGB are mainly a consequence of the transformation from LAGB (cDRX) or by HAGB migration (gDRX) [16].

## 2. Materials and methods

The AA6082 aluminium alloy, produced by hot extrusion and aged to T6 condition, showed rod-like shape grains elongated in the extrusion direction with approximately 1000 µm in length and 15 µm in diameter. The specimen was in-situ tested by HEXRD in transmission mode during hot torsion at the P07 beamline of PETRA III at the Deutsches Elektronen-Synchrotron (DESY) in

Hamburg [17]. A cylindrical sample was machined to 4 mm diameter and a gauge length of 20 mm with 2 mm transition radius to the 35 mm length and 10 mm diameter heads with its axis sample aligned in the extrusion direction. For torsion, a stepping motor driven machine with free-end movement was used at constant  $0.01 \text{ s}^{-1}$  (surface) strain rate. The test temperature was 400 °C ( $\pm 5 \text{ °C}$ ) and it was measured and controlled by K-type thermocouples close to the sample surface. The incident beam, with  $E = 87.1 \text{ keV}$  ( $\lambda = 0.01423 \text{ nm}$ ), was placed close to the non-rotating side and  $\sim 0.1 \text{ mm}$  below the outer surface of the sample. Slit dimensions were set to 0.7 mm (horizontal) x 0.2 mm (vertical) and the sample-detector distance was 1457 mm (calibrated with a  $\text{LaB}_6$  capillary). Diffracted images were collected every second in a 2-D PerkinElmer 1622 flat panel detector with an array of  $2048 \times 2048$  pixels. A schematic zenithal view of the in-situ synchrotron measurement is shown in Fig. 1.

Effective strain and stress close to the surface were calculated as:

$$\varepsilon_{\text{eff}} = \frac{1}{\sqrt{3}} \frac{2\pi N R}{1} \frac{R}{L} = \frac{1}{\sqrt{3}} \theta \frac{R}{L} \quad (1)$$

$$\sigma_{\text{eff}} = \frac{\sqrt{3} M}{2\pi R^3} (3 + m + n) \quad (2)$$

with  $N$  the number of revolutions,  $R$  the gauge radius (2 mm),  $L$  the gauge length (20 mm),  $M$  the torque,  $m$  the strain rate sensitivity (0.18), and  $n$  the strain hardening coefficient calculated as  $(\partial \ln M / \partial \ln \theta)$  that varies during the experiment. A more detailed explanation of different parameters can be found in Refs. [18,19].

## 3. Results

Debye-Scherrer rings of the collected images were processed using ImageJ [20] to produce the azimuthal angle ( $\psi$ ) - effective strain (AS) - plot [15]. Fig. 2 shows the AS-plot for the Al-220 crystallographic plane. Initial high intensity regions can be distinguished

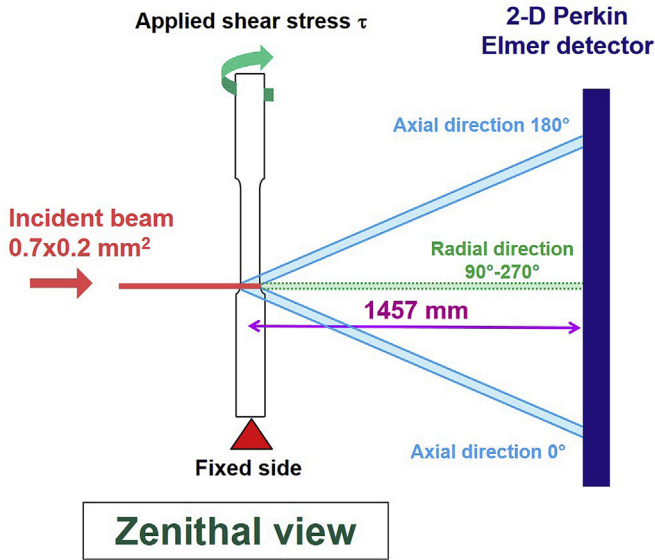


Fig. 1. Schematic zenithal view of the in-situ synchrotron diffraction experiment.

around  $\psi$  at  $\sim 35^\circ$ ,  $\sim 90^\circ$ ,  $\sim 145^\circ$ ,  $\sim 215^\circ$ ,  $\sim 270^\circ$ , and  $\sim 325^\circ$  and less intense around  $\psi$  at  $\sim 45^\circ$ ,  $\sim 135^\circ$ ,  $\sim 225^\circ$ , and  $\sim 315^\circ$  indicating a strong (111)- and a less intense (200)-fibre textures. These high intensity regions broaden monotonically up to an effective strain of  $\epsilon_{eff} \sim 0.7$  where the initial texture breakdown occurs as a consequence of misorientation formation due to dynamic recovery [15]. Up to  $\epsilon_{eff} \sim 1.1$  little variations can be observed. Finally, the strain-lines develop new changes indicative of a new texture.

The collected 2D-images were integrated in sectors as a function of the azimuthal angle using the Input4MAUD software [21] and exported into MAUD software [22] for Rietveld analysis ( $10\% \leq R_{wmb} \leq 20\%$ ). The dislocation densities were determined with the Williamson-Smallman relations [23]:

$$\rho_D = \frac{3p}{D_{eff}^2} \quad (3)$$

$$\rho_S = \frac{ke_{RMS}^2}{Fb^2} \quad (4)$$

where  $p$  is the number of dislocations on the face of the block structure ( $=1$ ) of the coherently scattering domain from a

diffraction point of view (hereafter crystallite for the sake of brevity),  $D_{eff}$  is the crystallite diameter,  $K$  depends on the lattice ( $=16.1$ ),  $e_{RMS}$  is the root-mean square from Rietveld refinements,  $F$  is the energy interaction factor of dislocations ( $=1$ ), and  $b$  is the Burger's vector.

Fig. 3 shows the evolution of a)  $\sigma_{eff}$ , b) crystallite size and Taylor factor  $M$  (calculated as in Ref. [24]), and c)  $\rho_D$  and lattice parameter  $a$ . The lower limit of the dislocation density can be calculated as [23]:

$$\rho = (\rho_D \rho_S)^{1/2} = \sqrt{\frac{3pk}{F} \frac{e_{RMS}}{bD_{eff}}} \quad (5)$$

The crystallite size after heating at  $400^\circ\text{C}$  and before deformation is around  $3500 \text{ \AA}$ , decreasing to  $\sim 1500 \text{ \AA}$  up to  $\epsilon_{eff} \sim 0.25$  (same order of magnitude that in Ref. [25]) due to the formation of cells and subgrains in the early stages, while the dislocation density  $\rho$  increases from  $\sim 4 \times 10^{12} \text{ m}^{-2}$  to  $\sim 4 \times 10^{13} \text{ m}^{-2}$  (Fig. 3 c). By increasing the strain, the crystallites increase their size up to around  $4500 \text{ \AA}$  at  $\epsilon_{eff} \sim 1.1$  by wall sharpening, re-arrangement of dislocations, and a total decrement of  $\rho$  down to  $\sim 1 \times 10^{12} \text{ m}^{-2}$ . Simultaneously,  $M$  decreases continuously, evidencing texture variations. The crystallite size decreases to  $3500 \text{ \AA}$  at  $\epsilon_{eff} \sim 1.5$  with negligible variation in  $\rho$ .

#### 4. Discussion

The total dislocation density according to the Kocks-Mecking equation [26] reads as:

$$\rho_{K-M} = (\sigma_{eff} / \alpha M b \mu)^2 \quad (6)$$

where  $\sigma$  is the effective stress,  $\alpha = 0.5$ ,  $M$  is taken from Fig. 3, and  $\mu$  is the shear modulus equal to  $21.6 \text{ GPa}$  [27]. From Fig. 2 can also be inferred that a new (200) texture starts to develop from  $\epsilon_{eff} \sim 1.1$  with high intensity regions between  $\sim 40^\circ$  and  $\sim 140^\circ$  and between  $\sim 220^\circ$  and  $\sim 320^\circ$ . This texture is roughly similar to that formed during rolling at high temperature in a 6082 aluminium alloy [28] and it is used to validate, in first approach, the use of the Kocks-Mecking equation in this study. The calculated  $\rho_D$  and  $\rho$  define the upper and lower limits of the total dislocation density, respectively (Fig. 3c). During the early stages of deformation when both  $\rho_D$  and  $\rho$  increase (up to  $\epsilon_{eff} \sim 0.25$ ),  $\rho_{K-M}$  is close to the average of the internal dislocation density  $\rho$  due to the cell and subgrain formations. When dynamic recovery occurs  $\rho_{K-M}$  approaches to  $\rho_D$ , which represents the wall dislocation density, becoming both roughly equal between  $\epsilon_{eff} \sim 0.7-1.1$ . Above  $\epsilon_{eff} \sim 1.1$   $\rho_{K-M}$  slightly

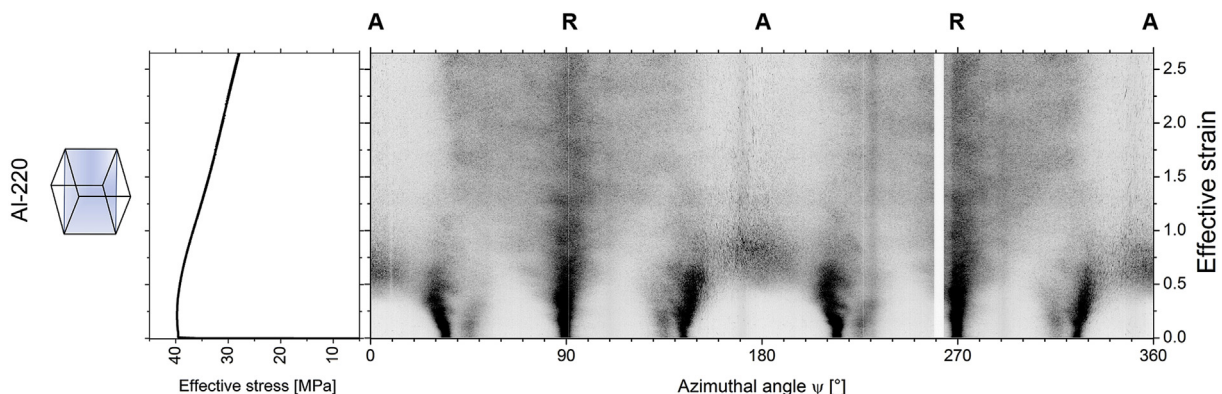
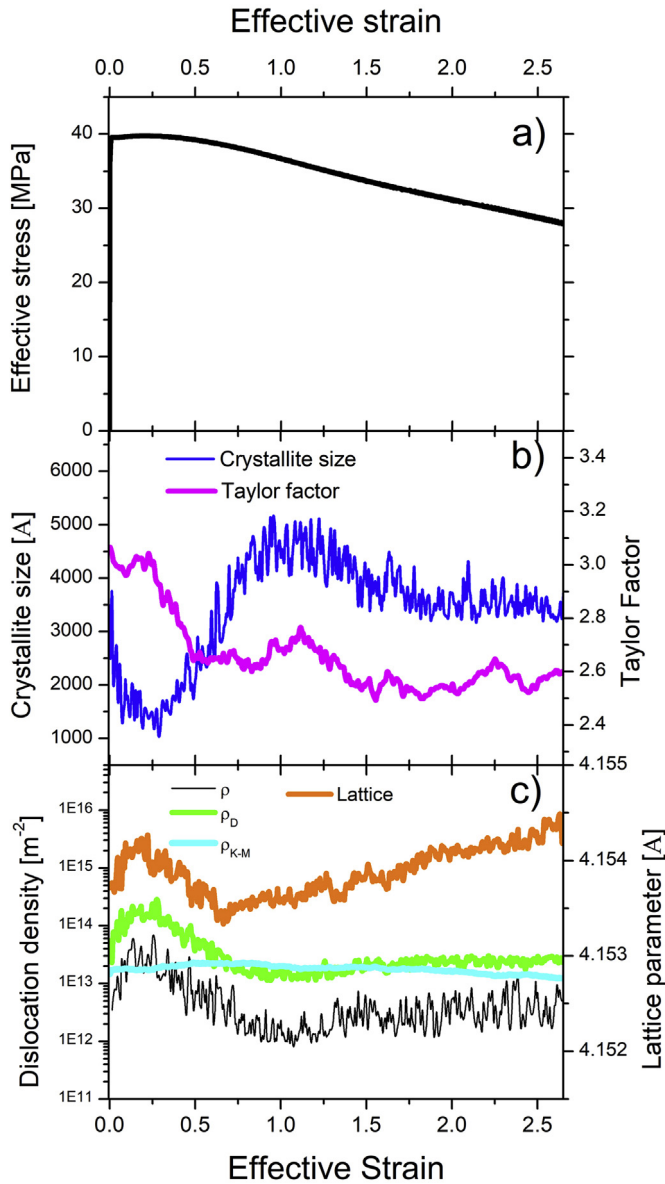


Fig. 2. AS-plot for the Al-220 reflection. The azimuthal angle is indicated at the bottom and axial (A) and radial (R) directions at the top.

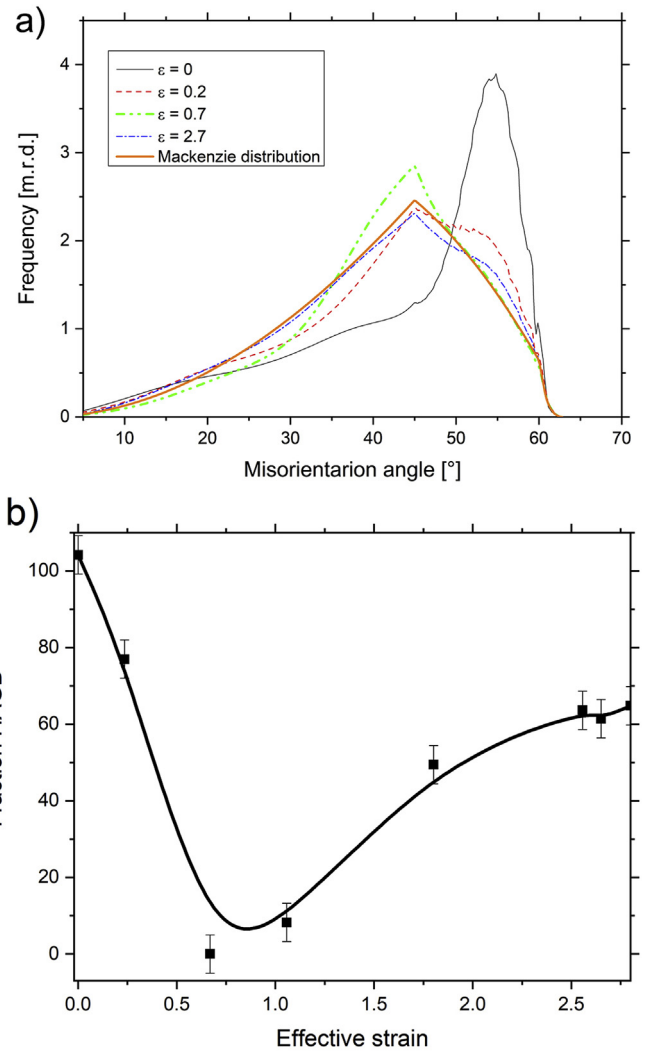


**Fig. 3.** Evolution of the different parameters as a function of the effective strain: a) effective stress, b) crystallite size and Taylor factor, and c) dislocation densities and lattice parameter.

decreases when compared to  $\rho_D$  due to the wall dislocation absorption by the existent and new created boundaries. Consequently, the stress softening is produced mainly by the texture change up to  $\sim 0.7$  of strain, and due to the annihilation of dislo-

cations by cDRX above  $\sim 0.7$  of strain.

The variation of the lattice parameter  $a$  is directly connected to the Swift or texture effect [29]:  $a$  initially increases up to  $\epsilon_{eff} \sim 0.25$ , then it decreases until the initial texture breaks down at  $\epsilon_{eff} \sim 0.7$ ,



**Fig. 4.** a) Misorientation distribution at different effective strains and the Mackenzie distribution. b) Fraction of HAGBs.

and finally it increases up to  $\epsilon_{eff} \sim 2.7$ . The crystallite size and dislocation densities are constant from  $\epsilon_{eff} \sim 1.5$  while  $a$  monotonically increases, interpreted as grain rotation and accommodation of misorientations by changing the lattice parameter, ascribed to the cDRX.

Pole figures produced during Rietveld refinements were exported to the software MTEX [30] to obtain the misorientation angle distribution (Fig. 4a). Only the evolution of the HAGBs is analysed (the maximum resolution of MTEX is set to 5° which is assumed as the error). The fraction of HAGBs, considered as a trend,

$$\% \text{ HAGBs} = \frac{[\text{Area total} - \text{Area}_{15-62.8}]_{\epsilon_{eff}} - [\text{Area total} - \text{Area}_{15-62.8}]_{\epsilon_{eff} \sim 0.7}}{[\text{Area total} - \text{Area}_{15-62.8}]_{\epsilon_{eff} \sim 0.7}} \times 100 \quad (7)$$

is calculated as the relative area variations at different  $\epsilon_{eff}$  compared with the condition at  $\epsilon_{eff} \sim 0.7$ :

During the early stages of deformation and up to  $\epsilon_{eff} \sim 0.7$  the HAGBs decrease from  $\sim 100\%$  to  $\sim 0\%$  (Fig. 4b), in agreement with

the formation of LAGBs by dynamic recovery. Subsequently dislocations arrange at LAGBs increasing their misorientation constantly and increasing the fraction of HAGBs [31] up to ~65% at  $\varepsilon_{eff} = 2.7$ . The fraction of HAGBs can be related directly with the % of recrystallization.

## 5. Conclusions

The following conclusions are drawn:

- In-situ synchrotron radiation is a powerful tool to quantify the evolution of texture and the size of coherently scattering domains in an Al–Si–Mg alloy undergoing continuous dynamic recrystallization.
- Up to ~0.25 of strain, subgrain formation occurs while the crystallite size is reduced. As deformation increases up to ~1.1 of strain, the internal dislocation density decreases while the crystallite increases by dynamic recovery remaining onwards constant.
- Grain rotation of crystallites was proved by studying the lattice parameter evolution.
- The ratio formation of high angle grain boundaries is calculated by analysing the misorientation angle distributions.
- The dislocation density types corresponding to each stage of deformation are characterised. Initially, the internal dislocation density prevails and wall dislocations become relevant when recovery takes place.

## Declaration of competing interest

None.

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