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Direct observation of precipitation along twin boundaries and dissolution in a magnesium alloy annealing at high temperature

Dikai Guan, John Nutter, Joanne Sharp, Junheng Gao, W. Mark Rainforth *

Department of Materials Science and Engineering, The University of Sheffield, Sheffield S1 3JD, UK

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ABSTRACT

Precipitation along twin boundaries and dissolution in a cold-rolled Mg-Y-Nd alloy was directly observed for the first time during annealing at 490 °C. Precipitation occurred concurrently with recrystallization and the combined effect of precipitation and solute segregated to twin boundaries modified the recrystallization behaviour. Precipitates later dissolved into the matrix at the point where full recrystallization was nearly complete. The precipitates and higher solute concentration along original twin boundaries hindered grain growth of newly formed recrystallized grains. Even where twin boundaries had been consumed by recrystallization, the size of recrystallized grains were still controlled by the pre-existing twin boundaries.

It is often reported that segregation and precipitation along grain boundaries (GBs) can minimise the local stored energy induced by deformation [1–5]. Grain growth kinetics are also altered due to solute drag or by particle pinning effects [6,7]. Segregation along fully coherent twin boundaries was first reported by Nie et al. [8] who observed periodic segregation of Gd and Zn atoms along twin boundaries when the sample was cold compressed and annealed at 150 °C. The solute drag restricted the movement of twin boundaries and improved the compressive strength. Later, Somekowa et al. [9] reported that segregation along twin boundaries increased hardness but degraded damping capacity in a cold compressed sample after annealing at 250 °C. Basha et al. [10] even found Zn segregation along twin boundaries during cold compression without any heat treatment. Precipitation along twin boundaries has been reported by Ye et al. [11] who found this phenomenon in a pre-strained Mg alloy after ageing at 200 °C. However, in all aforementioned reports, the segregation or precipitation along twin boundaries was only observed at low annealing temperatures, where no recrystallization occurred.

Recrystallization leading to a reduction in basal texture intensity of wrought Mg alloys has been the focus of considerable attention [12–16], since weakening the basal texture can significantly reduce anisotropic mechanical properties and improve the formability of Mg alloys. In deformed Mg alloys, grain and twin boundaries are effective sites for static recrystallization. However, recrystallization temperatures are usually higher than the temperatures used for ageing. The question therefore arises: does this segregation or precipitation happen at high temperatures? If it does, does the segregation and precipitation remain throughout the whole static recrystallization process when the original twin boundaries have disappeared due to recrystallization? How does the segregation and precipitation interact with the recrystallized grains?

To address these issues, *in-situ* TEM, *quasi-in-situ* EBSD and SEM were employed to track the entire static recrystallization process during annealing at 490 °C. We observed, for the first time, the precipitation process on twin boundaries concurrent with recrystallization and dissolution of the precipitates when full recrystallization had nearly finished.

The as-received material was a commercial WE43 alloy supplied by Magnesium Elektron as extruded T5 bar with the chemical composition listed in Table 1 of our previous study [17]. Solid solution treatments were undertaken under a continuous argon flow at 525 °C for 10 min and 1 h, followed by cold water quenching. The corresponding samples were designated as SST10M and SST1H, respectively. Subsequently, the samples was cold rolled by a reduction in thickness of 20% in one pass, with the samples designated SST10MCR and SST1HCR. Recrystallization rate is dependent on original grain size; a fine-grained sample usually recrystallizes more quickly than a coarse-grained sample. Therefore, we chose a short solid solution treatment in order to provide a fine-grained material that would recrystallize quickly, allowing us to observe precipitation and precipitation dissolution in an acceptable experimental time by using *in-situ* TEM. In contrast, a coarse-grained SST1HCR was used for quasi-*in-situ* EBSD experiments as we needed to slow down recrystallization rate to track the microstructure evolution in a large sample area.

EBSD was performed using a FEI Nano Nova 450 field emission gun SEM fitted with Oxford Instruments HKL NordlysMax^3 EBSD detector. The EBSD scans were taken after cold rolling and after 300 s, 720 s,

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* Corresponding author.
E-mail address: m.rainforth@sheffield.ac.uk (W. Mark Rainforth).

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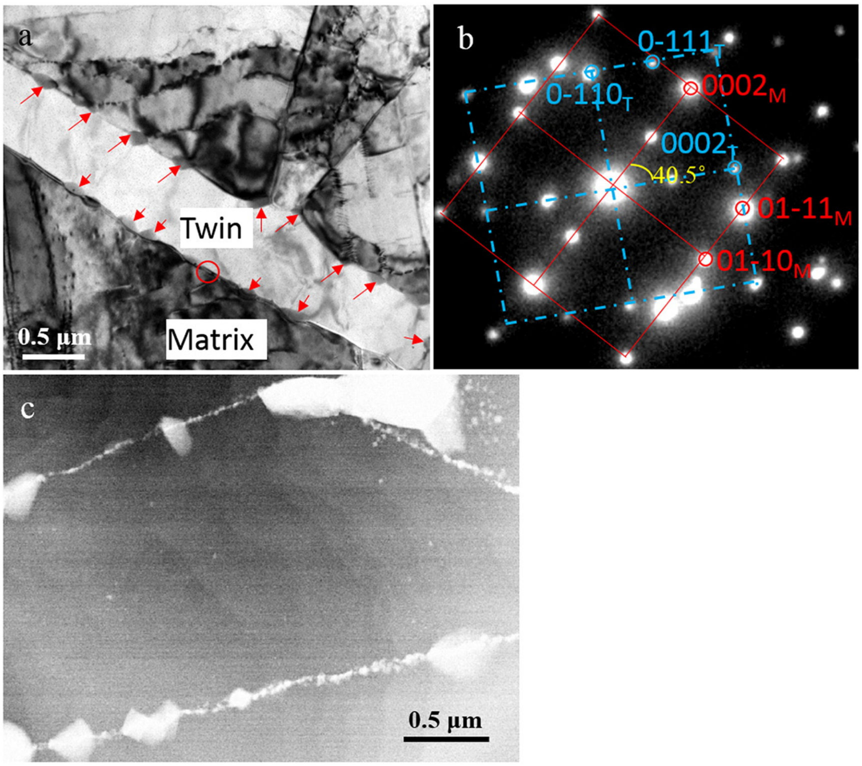

Fig. 1. (a) TEM BF image, (b) corresponding SADP image from interface between twin and matrix (see red circle shown in (a)) and (c) STEM HAADF image of sample SST10MCR after annealing at 490 °C for 5 min. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

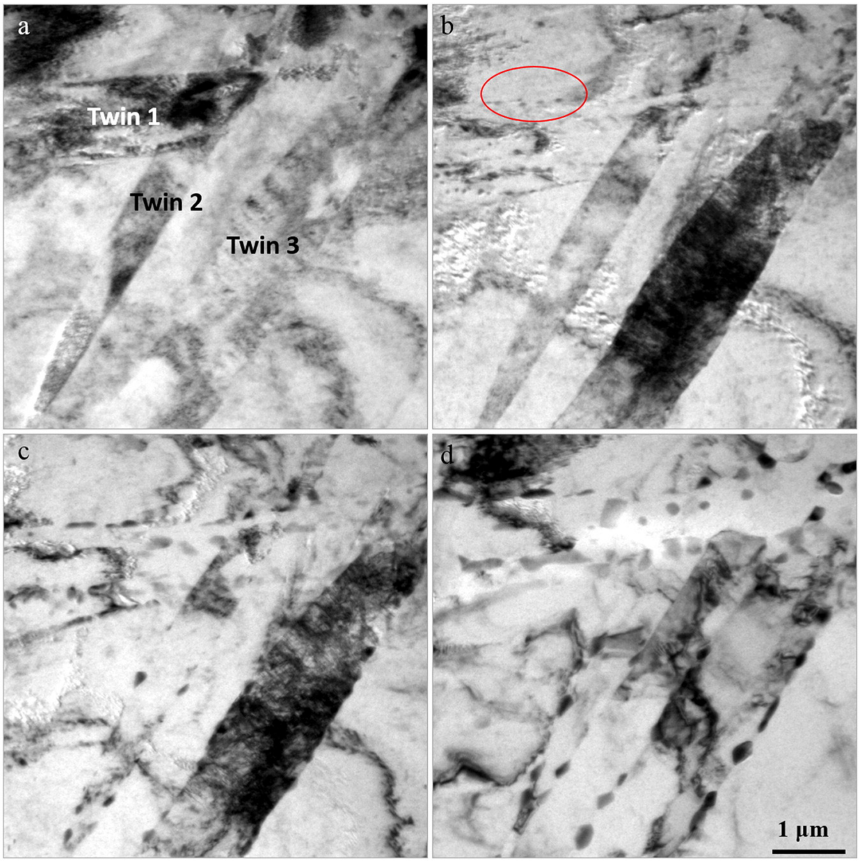

Fig. 2. *In-situ* TEM BF images of sample SST10MCR: (a) as-cold rolled, (b) after heating 362 s when the temperature was up to 350 °C, (c) after heating 471 s when the temperature was up to 400 °C, and (d) after heating 793 s and the temperature held at 400 °C for 322 s. All the temperatures were controlled by hot stage heating unit instead of actual values of TEM sample temperature.