Vol. 130 (2016) ACTA PHYSICA POLONICA A No. 4

Proceedings of the XXIII Conference on Applied Crystallography, Krynica Zdrój, Poland, September 20–24, 2015

Orientation Mapping and *In Situ* Annealing Applied for Characterization of Changes in Aluminium Alloys after Deformation

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The paper presents application of combined *in situ* annealing and orientation mapping technique for investigation the microstructural changes in the aluminium alloy 6013 with bimodal distribution of the second phase particles during recovery and recrystallization processes. Information about grain distribution, misorientation between grains, size and shape of the grains at each stage of recrystallization process were obtained. Complexity of the experimental procedure is defined and discussed in order to avoid artificial results. Although the article described the advantages and disadvantages of those method used in transmission electron microscopy and scanning electron microscopy in the work the results from scanning electron microscopy/electron backscattering diffraction *in situ* heating experiments are highlighted. Obtained data are in a good agreement with previous transmission electron microscopy and calorimetry studies.

DOI: 10.12693/APhysPolA.130.979

PACS/topics: 68.37.Hk, 81.40.Lm, 81.70.–q

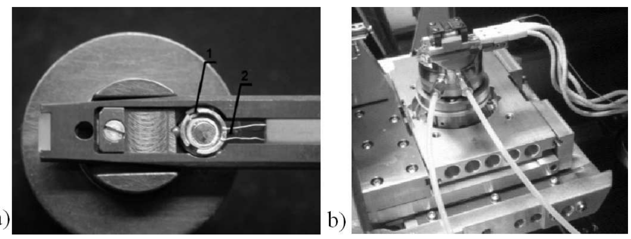
1. Introduction

Modern equipment in transmission electron microscopy (TEM) and scanning electron microscopy (SEM) as *in situ* holders and stages gave an opportunity to investigate of materials behaviour during e.g. mechanical tests, cooling or heating. *In situ* observation in electron microscopy was considered as difficult for performing and interpreting mainly because of different conditions of a sample preparation and observation which could influence on the behaviour of the material. It is crucial for those investigations whether they could be similar to the real behaviour of bulk samples. It was proved that with taking into account many factors which can influence *in situ* measurements, they can be successful source of scientific observation. New benefits from *in situ* experiments were achieved using orientation microscopy in TEM and SEM. Combined *in situ* annealing and acquisition of crystallographic data from the chosen areas of a sample together with calorimetric study improved the quality and reliability of investigations.

1.1. In situ *investigations in TEM*

Involving heating device in the study of materials enables to get additional information about behaviour during annealing. Using TEM, these phenomena can be studied in nano and microscale. Figure 1a shows heating holder, which resembles a standard holder for TEM, but has an extra heating element.

The heating process is controlled by changing the voltage and current supply. Recorded temperatures are



a) b)

Fig. 1. TEM heating holder with (a) 1 — furnace and 2 — thermocouple and (b) SEM heating stage.

repeatable for different samples. However, there was observed about 100 °C shift between the temperatures of processes inside heating devices and temperatures of the processes in the same material measured using a calorimeter [1]. The first direct observation using heating holder was carried out by Bailey [2]. It was noticed the difference in recrystallization processes between bulk materials and thin foils. Further works of Roberts and Lethinen [3] and Sztwiertnia and Haessner [4] proved that ensuring proper conditions of *in situ* experiment, assures observation of changes in the annealed material which are at least close to that occurring in a bulk sample. It turns out that in the samples cut from planes parallel to the plane of the sheet is small amount of well-defined grain boundaries, which makes both the nucleation and the migration of grains limited. An important feature of the *in situ* investigations is that the front of recrystallization is stopped in the areas of foil thinner than a certain critical value. Thin foil areas are not recrystallized due to the formation of the thermal grooving at the surface, which can inhibit the migration of boundary occurring under the influence of large recrystallization motion [3, 5]. Effect of sample thickness on the movement of grain boundaries was explained by Mullins [6]. Mullins showed that the movement of boundaries is stopped by the associated

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thermal grooving when a certain grain size is reached. The appearance of the grooves is a result of extensive surface tension at positions where grain boundaries intersect upper or lower surface of the film. Inhibition of grain boundaries in thin areas of the foil is related to the intensive growth of thermal grooves in these areas during the annealing in the microscope.

1.2. In situ *investigations in SEM*

In comparison to TEM there are several advantages of scanning electron microscope *in situ* investigation. The sample possesses only one free surface and is resistant to buckling [7]. It means that in spite of worse spatial resolution obtained data can be more reliable and more resemble to the analysis of bulk materials. Fast data acquisition systems which are used for electron backscatter diffraction (EBSD) analysis can provide larger scale of analysis then in TEM. For simultaneously EBSD and heating experiments performance special stage which can be tilted up to 70° is needed (Fig. 1b). Main application of this kind of stage is observation of nucleation and grain growth or phase transformation (e.g. [7–11]). The unique opportunity of comparing the same area of the sample during heating for several consecutive temperatures is given which is presented in the results part of this paper.

2. Material and experimental methods

For presented studies aluminium alloy 6013 as an exemplary material was chosen. Characteristic distribution of secondary phase particles (small ($\ll 1$ μm) and large (> 1 μm)) in this alloy is responsible for interesting behaviour during annealing and recrystallization process performance.

The samples of that alloy were supersaturated in 530 °C for one hour and then artificially aged in 165 °C for 5 days and then deformed by cold-rolling up to 90% or treated by KOBO (complex extrusion of the ingot on a press with reversibly rotating die) [12] and then reversible cold-rolled up to 90%. Such prepared material was investigated by combined *in situ* annealing and orientation mapping methods in SEM and TEM. Some previous studies in TEM and SEM were presented in [13–16] for cold-rolled samples. This article is mainly focused on the some selected results from SEM *in situ* EBSD investigation for those two deformation methods. The investigations in the SEM were performed using Gatan Murano 525 heating stage installed on SEM QUANTA 3D FEG with possibility to heating up to 950 °C. EBSD analysis were done with the TSL EDAX OIM system. Investigation were conducted on the samples planes perpendicular to the transverse direction (TD). The surfaces for EBSD experiments were electropolished. The samples were annealing inside of SEM chamber and sequence of the EBSD maps were measured in the temperature corresponding to the peaks obtained from the calorimetry studies (Fig. 2). SEM was operating with accelerating voltage 20 kV. The samples were continuously heated with heating rate corresponds to that of calorimetry heating rate 15 °C per min. Temperatures were controlled by Gatan PC software. The samples were stabilized at each temperature in order to avoid thermal drift and the EBSD maps were acquired with step size from 70 nm to 200 nm. After measuring they were cleaned up only using grain dilatation with grain tolerance angle — 2° and minimum grain size — 2 pixels.

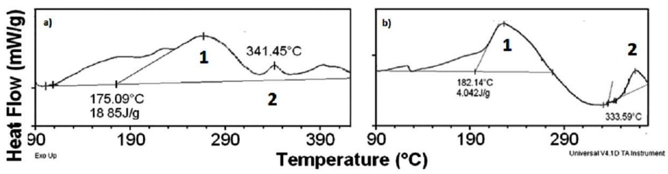

Fig. 2. Calorimetric curves obtained for AA6013 after cold rolling with reduction 90% (a) and after KOBO and cold-rolling with reduction 90% (b).

3. Results and discussion

In both analyzed cases the microstructure of the deformed aluminum alloy was composed of grains elongated in the rolling direction (RD) (Fig. 3, 25 °C). The grain size diameter did not exceed 10 µm (Fig. 3, 25 °C), but in the normal direction (ND) they were lower than 0.5 µm, and the boundaries between them were characterized as high angle grain boundaries (HAGBs). The fraction of HAGB were greater for single step deformation but the partitions of grain size lower than 5 µm were on the same level. In the areas near the large secondary phase particles, deformation zones (DZ) were identified which are responsible for nucleation of new grains at the temperature of the first (1) calorimetric peaks (270 °C and 220 °C, respectively, for different schema of deformation). Simultaneously at this temperature only small changes in the matrix areas can be observed as the arrangements of the low angle grain boundaries (LAGB) with no migration of the HAGBs. In addition, the phenomenon of particle stimulated nucleation PSN [17] was identified which corresponds with previous TEM results [13, 15]. At the temperature of the second peak (2 — about 350 °C for both cases), the migration of the HAGB in the matrix and further growth of the nuclei were observed, limited in the direction parallel to the ND with almost random grain misorientations distribution (Fig. 4, 400 °C). Moreover, in case of combined KOBO and rolling deformation, the grain size did not exceed 25 µm which needs further investigation in order to explain that fact (Fig. 5, 400 °C). Obtained results are unique in case of materials with second phases precipitations which play an important role during recrystallization process. Previous investigations were focused on one phases aluminum alloys e.g. [9]. Thanks to the analysis from SEM *in situ* EBSD studies observation from TEM and calorimetry [14] were confirmed and developed. The thesis with two stages of recrystallization process was confirmed in *in situ* annealing with larger area of observation then in TEM.

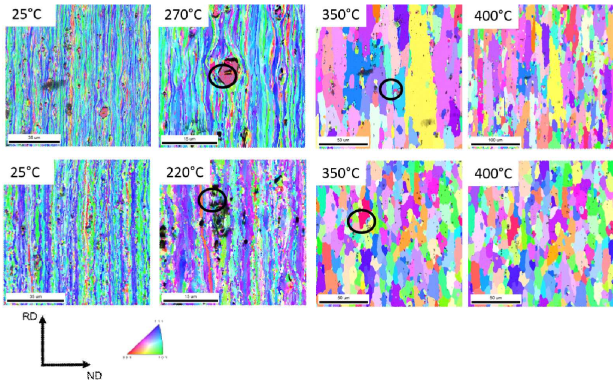

Fig. 3. Sequence of EBSD maps IPF + IQ with marked DZ which correspond with PSN behaviour for AA6013 after cold rolling with reduction 90% (upper row) and after KOBO and cold-rolling with reduction 90% (lower row) after annealing in SEM chamber by means of heating stage at each step of recrystallization.

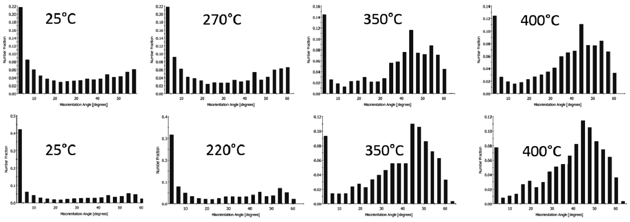

Fig. 4. Sequence of misorientation angle diagrams calculated from EBSD maps for AA6013 after cold rolling with reduction 90% (upper row) and after KOBO and cold-rolling with reduction 90% (lower row) after annealing in SEM chamber by means of heating stage at each step of recrystallization.

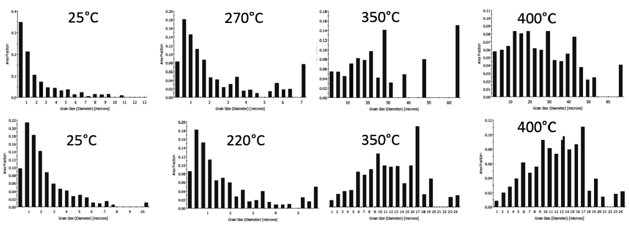

Fig. 5. Sequence of grain size (diameter) diagrams calculated from EBSD maps for AA6013 after cold rolling with reduction 90% (upper row) and after KOBO and cold-rolling with reduction 90% (lower row) after annealing in SEM chamber by means of heating stage at each step of recrystallization.

4. Conclusions

The *in situ* annealing studies in TEM and SEM are demanding and need to be carefully prepared and analysed. For the first time *in situ* SEM comprehensive investigations of microstructural changes during annealing in aluminium alloy with bimodal second phase particle distribution were presented and were in very good agreement with calorimetric and TEM studies.

Acknowledgments

This work was supported by the National Science Center (Poland) UMO–2011/03/D/ST8/04106. Author is grateful to Prof. M. Faryna, Dr. K. Berent, and Dr. P. Bobrowski from IMMS PAS and Dr. F. Brisset from Université Paris-Sud, for their assistance in SEM *in situ* experiments.

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