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The driving force governing room temperature grain coarsening in thin gold films

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ABSTRACT

Strong room-temperature grain coarsening in gold films on polyimide induced by cyclic uniaxial mechanical strain is demonstrated. Detailed electron backscatter diffraction analysis revealed that, in contrast to the predictions of shear-coupled grain boundary migration model, the grain coarsening is isotropic and coarsened grains do not exhibit any specific crystallographic orientations or misorientations to the neighboring grains. It is shown that a thermodynamic model where the driving force appears due to the difference in yield stresses between the grains with different sizes provides an adequate explanation of the experimental data.

Room temperature grain coarsening induced by mechanical loading has been shown to occur in Cu [1–3], Pt [4], Ni [5], Al [6–8], and Au [9] under different loading conditions such as nanoindentation [1,6], monotonic tensile loading [7,8], fatigue loading [2–4,9] as well as beam bending [5]. Despite the large number of experimental evidences, very little is known about the driving forces behind the athermal grain coarsening. In systematic investigations of nanocrystalline Al thin films [7,8] the authors were not able to explain the observed grain coarsening with “traditional driving forces” [7] and it was suggested that shear coupled grain boundary migration (SCGBM) is responsible for the grain growth. However, the SCGBM concept was developed to explain the grain boundary (GB) migration at elevated temperatures in bicrystals with clearly defined GB planes, GB types and misorientations [10–12]. In polycrystals, the grain boundaries are often of mixed type and have no coincidence site lattices. Although the SCGBM was generally observed in polycrystals [13,14], the tangential displacement of the grains with respect to the boundary must be restricted due to the constraint caused by the neighboring grains [15]. Thus, it is currently unclear to which extent the SCGBM concept can be applied to explain the grain coarsening effect in real polycrystals. It is necessary to note, that grain coarsening can also occur without GB migration as demonstrated for nanocrystalline Au films [9] where a nanotwin-assisted GB elimination mechanism was proposed.

In the present work, a detailed electron backscatter diffraction (EBSD) characterization is utilized to analyze possible driving forces governing severe room temperature grain coarsening in cyclically loaded ultra-fine grained (UFG) gold films.

The gold films were deposited on 50 μm polyimide Upilex substrates by electron beam evaporation in a Balzers BAK 550 evaporation machine with the vacuum of 2.1×10^{-7} mbar and using a deposition rate of 0.3 nm/s to a thickness of 500 nm. The samples with the width of 5 mm and length of 40 mm were cut out of the sheet using a scalpel. Cyclic tensile straining was performed on an MTS Tytron 250 tensile testing device. Sine strain function between zero strain and a peak strain was applied with the frequency of 0.1 Hz. Such a low straining rate was used in order to account for viscoelastic relaxation of the substrate and to exclude possible heating effects. Three different peak strain values were used in mechanical tests (1%, 1.5%, and 2%) but for the sake of brevity only the results for 1.5% peak strain are shown here. The strain and displacement rates for 1.5% peak strain were 0.003 s^{-1} and 60 μm/s, respectively. After 1000, 2000, and 5000 cycles, the mechanical test was interrupted to perform EBSD analysis and scanning electron microscopy of the surface. Focused ion beam (FIB) marker was used to locate the same area within the film. The points of the EBSD scans which were not indexed properly and have the confidence index of less than 0.05 were removed and appear in black in Figs. 1 and 2.

An overview of the severe room temperature grain coarsening effect in 500 nm thick gold films on polyimide substrate is shown in Fig. 1. In Fig. 1a the grain orientation map of the initial microstructure in the direction normal to the surface (ND) is shown. The film has a strong (111) texture and average grain size of 210 ± 60 nm. Fig. 1b shows the same surface area after applying 5000 cycles with 1.5% tensile strain. Strong grain coarsening leads to an increase of average grain size,

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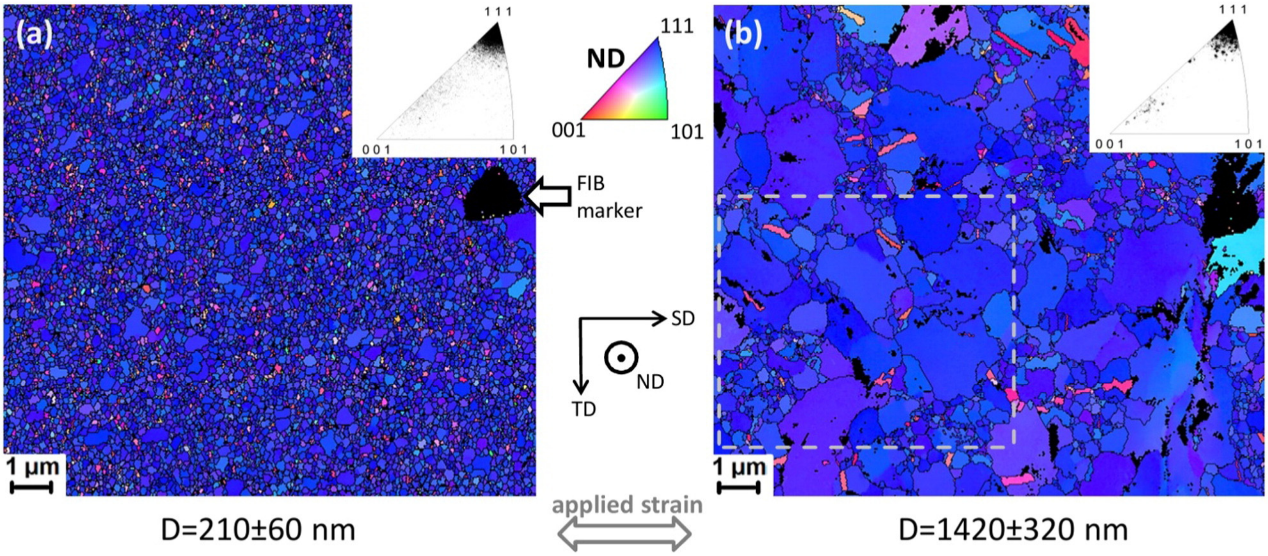

Fig. 1. Grain orientation maps of 500 nm thick gold film (a) before and (b) after application of 5000 cycles with 1.5% strain. The insets display the corresponding distributions of crystallographic orientations in stereographic triangles. The arrows marked with acronyms SD, ND, and TD show the strain direction, normal direction, and transverse direction, respectively. The dashed rectangle in (b) corresponds to the area which is considered in detail in Fig. 2. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

extracted from the same area, to 1420 ± 320 nm. The total number of grains decreased from 9400 to 1400 and there are virtually no surface areas which conserved the initial microstructure. The orientation distributions within the stereographic triangles shown in the insets demonstrate that no significant texture transition occurs during grain coarsening.

The most straightforward way to uncover the mechanism of the observed grain coarsening is to determine the common features the coarsened grains have in comparison to non-coarsened grains. To consider the evolution of the microstructure in more detail the grain orientation maps in the straining direction before cyclic loading, after 1000 cycles, and after 5000 cycles are shown in Fig. 2. The grain boundaries are separated into three groups according the misorientation angle. The low angle grain boundaries (LAGB) appear in yellow, the general high angle grain boundaries (HAGB) are shown in black and the twin boundaries are highlighted by the red colour. The numbers from 1 to 10 depict the same grains at each stage of the mechanical loading. The first observation which should be made is that the coarsened grains do not have any specific crystallographic orientation with respect to the loading direction. Secondly, the grain coarsening is isotropic, meaning that there is no preferential direction of the grain extension although the applied strain is uniaxial. Third, there is no clear correlation between the grain coarsening and GB misorientation angle. By comparing Fig. 2b and c one can find numerous examples of non-migrating LAGBs (e.g. GB surrounding grain 6), HAGBs (e.g. GB between grains 7 and 9) and twin boundaries (e.g. bottom-right GB of grain 5). At the same time, there are many examples of migrating LAGB, HAGB, and twin boundaries. Moreover, the misorientation of the GB of a growing grain changes during coarsening as soon as a neighboring shrinking grain disappears. Thus, the GB misorientation cannot be a decisive factor which promotes the grain coarsening. The only common feature of the coarsened grains, which was determined using the current experimental method, is the initial grain size. Almost all numbered grains have initial sizes, which are more than two times higher than the average grain size. The only exception is grain 9, which has an initial equivalent diameter of 350 nm.

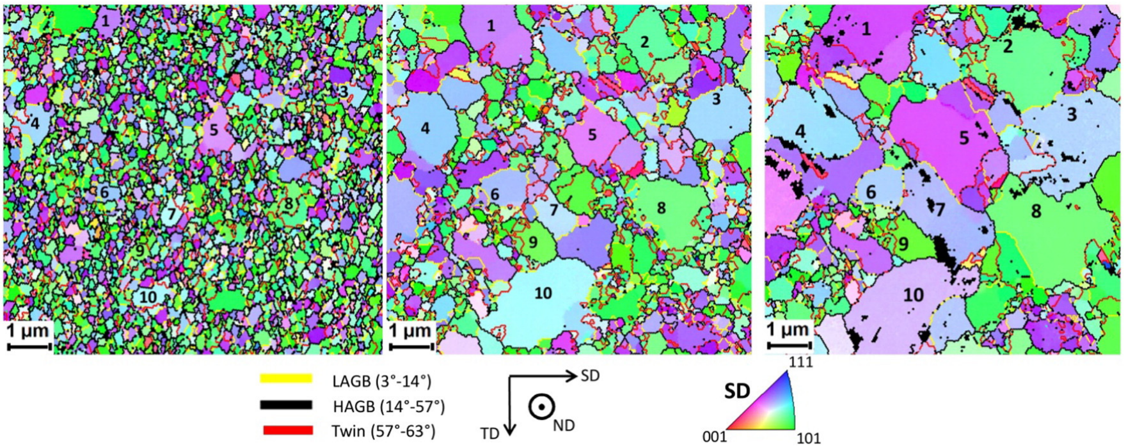

Fig. 2. Detailed evolution of the microstructure during cyclic loading. The grain orientation maps in the strain direction (SD) are shown for 500 nm thick gold film (a) before straining, (b) after 1000 cycles (c) and after 5000 cycles with 1.5% peak strain. Selected grains which coarsen during the deformation are marked with the numbers 1 to 10. The low-angle, high-angle and twin grain boundaries are coded by different colors as stated. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)