Microstructure of Al-Mg thin free-standing films

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Abstract. Thin Al-Mg films were prepared by a DC magnetron sputtering on glass substrates covered with photoresist and subsequently free-standing samples were released from the substrate. The surface morphology, grain size and orientations were characterized by atomic force microscopy and transmission electron microscopy equipped with automated orientation and phase mapping software. The grain growth mechanism during sputtering is consistent with sputter deposition oblique incidence theory for growth. Strong preferred (110) orientation in direction perpendicular to the sample surface has been observed in all studied samples.

1 Introduction

Thin free-standing metallic films are commonly essential parts of microelectronic devices and micromechanical systems (MEMS) where they are exposed to a wide range of operating velocities and frequencies. Since the decrease of the sample size to micro and nanoscale results in properties considerably different from the ones observed in bulk materials, plasticity and mechanical behavior of thin films should be examined. In the case of thin films adhered to a substrate it is unclear how much the presence of the substrate influences measured properties. Therefore, a separation of the films from the substrate is unavoidable and experiments should be performed on free-standing films.

The relation between mechanical strength and grain size in bulk materials can be expressed by an inverse power law known as Hall-Petch relationship [1,2], which is a result of dislocation pileups formed in the vicinity of grain boundaries. In the case of very small grains, the validity of this relation ceases. On the contrary, a decrease of the yield stress and hardness with the decrease of grain size for sufficiently small grains has been observed in numerous studies [3,4]. Therefore an understanding of mechanical behavior of nanoscale materials is conditioned by the knowledge of their microstructure. Information about grain size and orientation distribution is crucial to determine their role in deformation processes.

Aluminum belongs to the most widely used lightweight and conductive metals in micromechanical structures. Aluminum films are often used for example as microswitches and membranes in microdevices or as interconnectors in integrated circuits. Adding Mg to pure Al could significantly increase the strength of the material [5].

In this work experimental evaluation of grain size and misorientations in Al-Mg free-standing thin films has been carried out using atomic force microscopy (AFM) and transmission electron microscopy (TEM) with automated orientation and phase mapping system (ASTAR) [6].
2 Experimental

50 nm, 100 nm and 150 nm thick films were prepared by a DC magnetron sputtering from a 2-inch target from Al – 3 wt.% Mg alloy with deposition rate 11.2 nm/min. The distance between the target and glass substrate coated in photoresist, a light sensitive polymer forming a smooth layer on the surface [7], was 7 cm. Layers were prepared using Ar gas under working pressure of 2 Pa.

Subsequently, free-standing film samples were prepared by a dissolution of photoresist in acetone and releasing of the film from the glass substrate. Afterwards, the pieces of thin film were fixed on a copper grid (diameter 3 mm, 300 mesh) and rinsed in methanol.

Surfaces of films on glass substrates were studied by AFM in tapping mode using Bruker Dimension Edge device. Free-standing samples on the copper grids were studied by a conventional TEM and ASTAR at JEOL 2200FS operated at 200 kV equipped with “Spinning Star” electron precession from NanoMEGAS.

3 Results and discussion

2.1 Atomic force microscopy

AFM images of Al-Mg 50 nm, 100 nm and 150 nm thick films grown on the photoresist covered glass substrate are shown in Figure 1. The surface morphology shows grains with size 40-60 nm for 50 nm thick film, 60-90 nm for 100 nm thick film and 80-100 nm for 150 nm thick film. Features of sample surfaces are visible in Figure 1 d) – e). The surface profile suggests a formation of columnar structures which is characteristic for thin films grown by the sputtering deposition [8]. Columns of different heights are gradually formed during deposition and a screening of shorter columns by their higher neighbors occurs [8]. When a columnar grain is screened by another one, it stops growing. Similar surface symmetries have been observed in different materials prepared by magnetron sputtering such as Au [9], Cu [10] or ZnO [11].

![AFM images of Al-Mg films](image)

**Figure 1.** AFM height sensor images of a) 50 nm, b) 100 nm and c) 150 nm Al-Mg films, d – f) corresponding topographic images.
2.2 Transmission electron microscopy

Bright field images shown in Figure 2 reveal evenly distributed polyhedral grains 20 – 70 nm large. The grain size continuously increases with film thickness, the largest grains in respective thicknesses reaching size 40 nm, 50 nm and 70 nm. In 150 nm thick films, a noticeable overlapping of neighboring grains can be observed. Comparing with the AFM images and considering anticipated above described columnar growth of the grains, the smallest grains can be ascribed to the screened columns with suspended growth, whereas the larger grains are the ones reaching the surface and therefore observable in AFM scans. The grain sizes measured in AFM were slightly larger than the ones measured from TEM images. This may have been because the radius of curvature of the AFM tip approached the measured grain size and became a limiting factor in the scan resolution.

![Figure 2](image)

**Figure 2.** a) STEM BF image of 50 nm thick Al-Mg film, TEM BF images of b) 100 nm and c) 150 nm Al-Mg films.

2.3 ASTAR

Orientations maps of the samples combined with reliability maps obtained by automatic phase and orientation mapping in TEM are shown in Figure 3. The grain size could not be directly determined from the orientation mapping due to moderately large areas of a low reliability marked by a black color in the images (Figure 4). This is most probably caused by the overlapping of the neighboring grains, which leads to ambiguity in evaluated diffraction patterns and a loss of information about the crystal orientation in these areas as a consequence.

Nevertheless, random grain orientation distribution in x and y axes of the samples is clearly visible as well as a preferential Al-(101) orientation in the z-axis corresponding to the orientation perpendicular to the film surfaces. Generally, the texture is strongly dependent on deposition conditions e.g. substrate material and orientation or deposition rate, but (110) along with (111) orientation is commonly observed in aluminum thin films [12].
Figure 3. Orientation reliability maps of samples with different thickness.
Figure 4. Original orientation map oriented in direction $z$ (notation consistent with Fig. 3) of 100 nm thick film a) compared to the map overlapped with reliability index areas marked by black color b).

4 Conclusions
The microstructure of thin Al-Mg films has been investigated. The grains in size from 20 to 70 nm are evenly distributed in all 50 nm, 100 nm and 150 nm thick films. The average size of the grains increases proportionally to the film thickness. Performed measurements suggest a columnar mechanism of grain growth where several columns are growing in a rate corresponding to the rate of deposition. The smaller grains are gradually screened out and only the largest ones continue to grow. Orientation maps revealed moderately high texture in the material with (110) orientation perpendicular to the sample surface.

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