

Complementary investigation of SEM and AFM on the morphology of sputtered aluminum thin films

F.M. Mwema, E.T. Akinlabi, O.P. Oladajo

Abstract—This article reports on the evolution of surface characteristics with the temperature for aluminum thin films sputtered on mild steel substrates. The films were sputtered at a constant sputtering power of 200 W and at different temperatures of the substrate; 40°C, 60°C, 80°C and 100°C. Surface microstructures were observed using a field emission scanning electron microscope (FESEM) and the results complemented by the atomic force microscope (AFM) observations. It was shown that FESEM features such as porosity, clusters and morphology can be revealed via the AFM imaging. Further analyses using AFM were then used to describe the morphology and evolution of the films at different substrate temperatures. The surface features were further related to the nanoindentation properties of the films.

Keywords—AFM; aluminum; thin films; sputtering; SEM; surface features

I. Introduction

Scanning electron microscope (SEM) and atomic force microscope (AFM) are among the most used microscopy techniques in microstructural characterizations of materials because they have high resolution (nanometric and Angstrom ranges). Extensive articles utilizing these facilities on the characterization of surfaces of thin films have been published [1]–[8]. For instance, AFM was used in characterizing the properties of Al thin films sputtered on steel substrates and information regarding surface defects and growth features satisfactorily discussed [9]. Through the SEM and AFM techniques, information regarding the distribution of the particles, grains and surface roughness can be obtained [10]. It has also been shown that with advanced SEM and AFM facilities, the crystallinity characteristics of the films can be obtained [11], [12].

Despite these extensive reports, studies reporting on the complementary characterization of SEM and AFM of thin films are few. A technical paper by Russell, Batchelor and

Thornton in 2001 [3] illustrated important aspects of complementary studies of AFM and SEM. In this paper, therefore, a complementary study of AFM and SEM on the evolution of surface properties of sputtered aluminum thin films with an increasing substrate temperature between 40°C and 100°C is presented. The AFM and SEM results were then related to the nanoindentation properties.

I. Methodology

The aluminum thin films used in this study were prepared by radio-frequency (RF) magnetron sputtering on mild steel substrates. The sputtering was undertaken using 99.99% pure Al targets of 75 mm in diameter and 3 mm thickness backed on a copper plate. The process was undertaken at a constant power of 200 W and varying substrate temperatures of 40°C, 60°C, 80°C and 100°C. Further details of the sputtering process were described in our earlier publications [9], [13].

After sputtering, the films were characterized for surface microstructure, topography and mechanical characteristics by field emission scanning electron (FESEM), atomic force microscopy (AFM) and nanoindentation facilities respectively. The details of AFM measurements for these samples were explained in our earlier work [13]. The microstructure imaging was undertaken using Zeiss Sigma FESEM whereas the nanoindentation was undertaken using a Hysitron® nanomechanical system (USA) under load control scheme up to a maximum load of 250 μ N.

II. Results and discussions

The microstructures of the aluminum thin films at different substrate temperatures obtained via the FESEM are illustrated in Figs. 1 and 2. At 40°C, the microstructure consisted of clusters of aluminum structures and very fine grains. At 60°C, the surfaces were characterized by very large and porous structures of Al. This observation was a clear indication that the sizes of the agglomerated clusters increased between 40°C and 60°C. Increasing the substrate temperature to 80°C led to a reduction in clusters and formation of dense Al structures over the substrate's surface. There was also the formation of circular structures at this substrate temperature. The presence of circular structures was an indication of grain growth and formation of grain boundaries. At this temperature, very tiny pores were also observed on the microstructure. At 100°C, there were dense and continuous structures of Al films. It was also observed that the microstructure was fully dominated by circular grains with well-defined boundaries. There were no clusters of aluminum structures as it was the case at lower temperatures.

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To further understand the evolution of the surface structures, the AFM micrographs were obtained. The representative three-dimensional (3D) AFM images for the Al thin films deposited at different substrate temperature are shown in Figs. 3 and 4. At 40°C, the AFM images showed interconnected (large) globular and fine stand-alone circular topography. The large globular topography can be interpreted as the cluster of Al structures observed from the FESEM whereas the fine circular features can be interpreted as the fine grains on the FESEM images. At 60°C, elongated features (smooth) with few globules and large pits were observed as shown in Fig. 3. These elongated features could be attributed to the increased clustering of the structures due to the increase in temperature describe from the FESEM images. The large pits observed at this temperature evidence the existence of higher porosity shown in the FESEM images. The AFM images at both 80°C and 100°C were seen to show laterally continuous features with well-defined boundaries. These images correlate with the formation of dense Al structures as shown by the FESEM micrographs in Fig. 2. Another important result from these AFM images is that the intensity of the scale bars (color bars) shown on the righthand side of each 3D image increased from 100 nm to 200 nm. This indicates that there was growth in mean average height (surface roughness) with the increase in substrate temperature between 40°C and 100°C.

The section analyses illustrated in Figs. 5 and 6 provide insights into morphological characteristics from the AFM imaging. The shape and width of these profiles indicated the growth of the surface properties with the increase in substrate temperature. It was clearly observed that as the temperature increased, there was a reduction in serrations and an increase in width of the surface profiles. These observations meant that there was the formation of dense and continuous Al thin films as the temperature increased to 100°C.

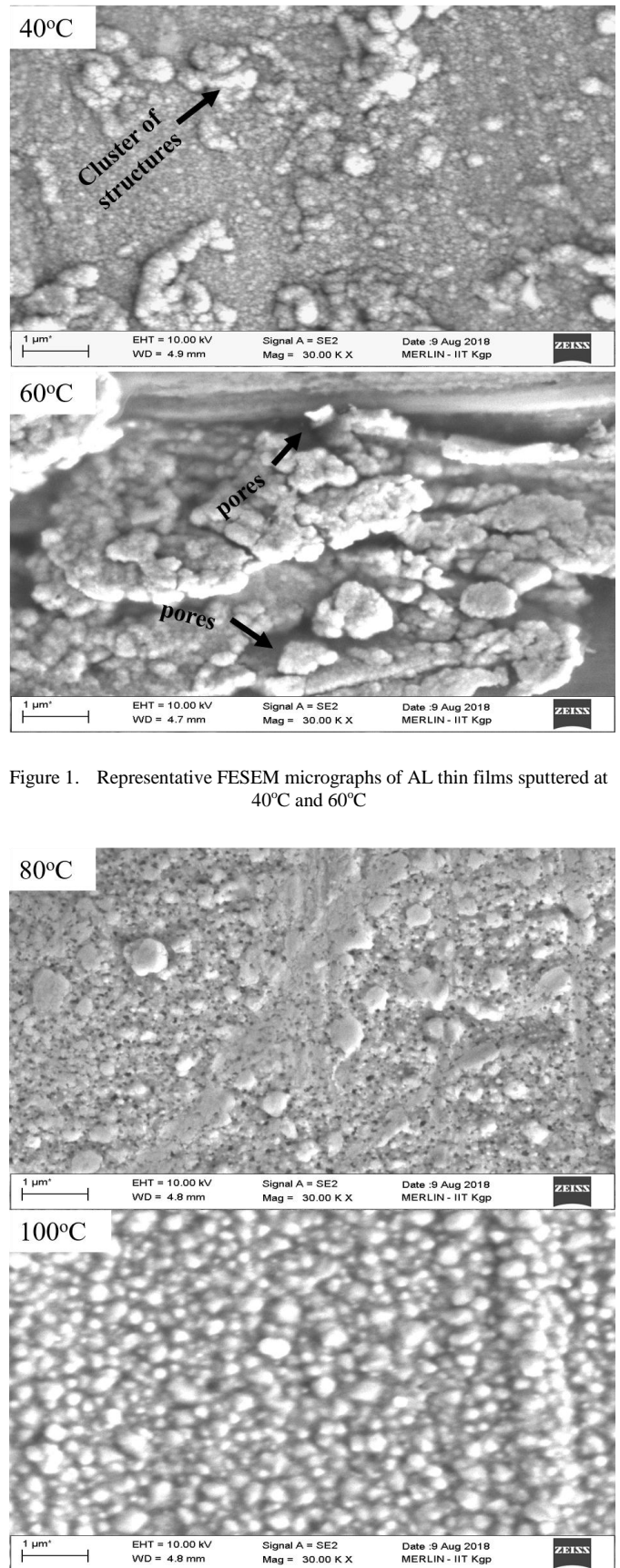


Figure 1. Representative FESEM micrographs of Al thin films sputtered at 40°C and 60°C

Figure 2. Representative FESEM micrographs of AL thin films sputtered at 80°C and 100°C

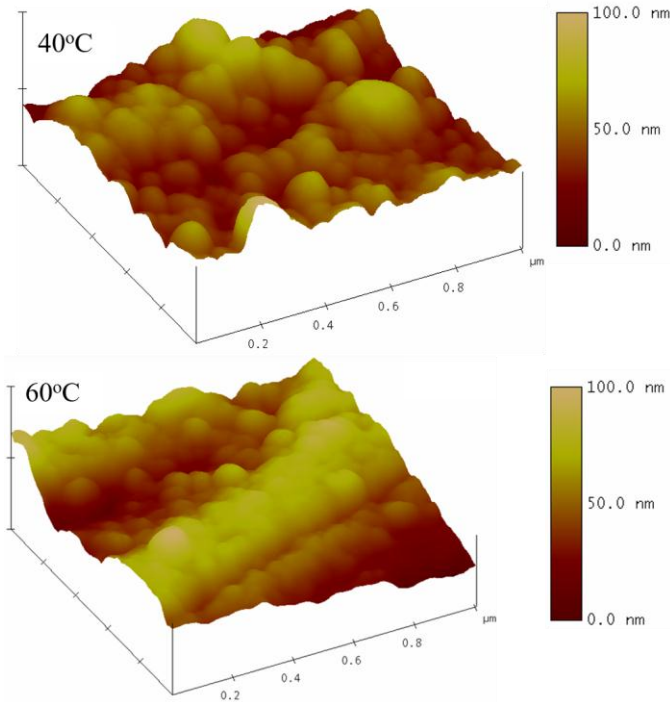


Figure 3. Representative AFM micrographs of Al thin films deposited on mild steel substrates at 40°C and 60°C (Reproduced with permission from Elsevier)

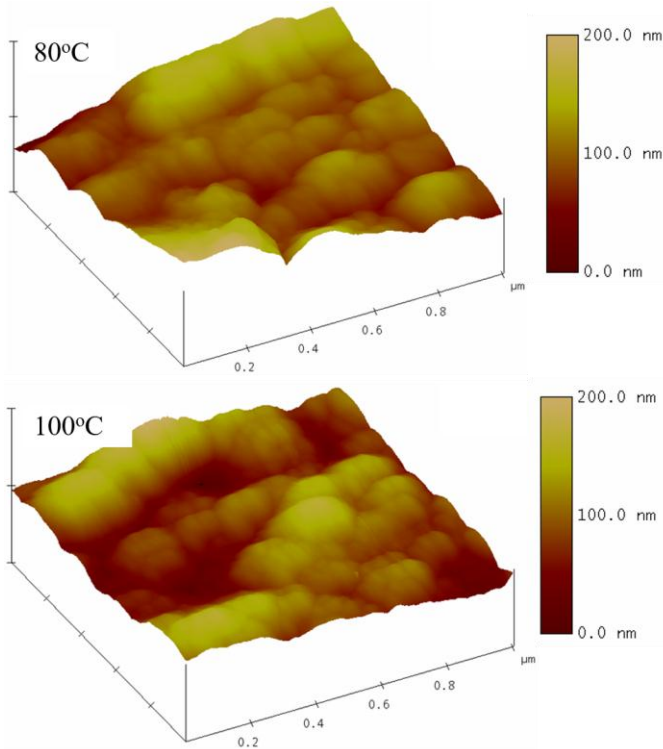


Figure 4. Representative AFM micrographs of Al thin films deposited on mild steel substrates at 80°C and 100°C (Reproduced with permission from Elsevier)

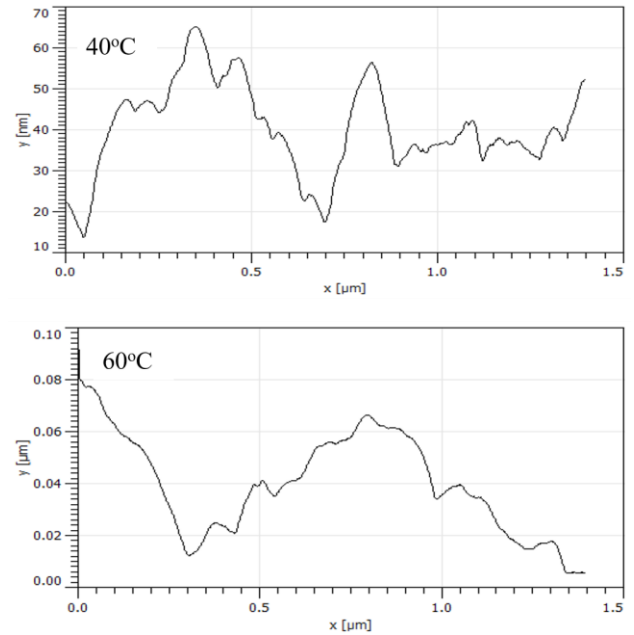


Figure 5. Section profiles taken along the diagonals of the AFM images for Al thin films deposited at 40°C and 60°C.

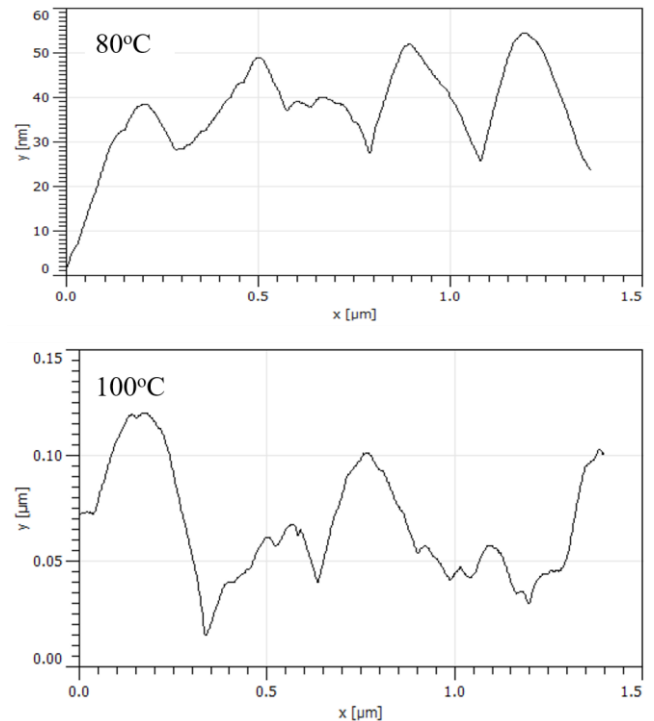


Figure 6. Section profiles taken along the diagonals of the AFM images for Al thin films deposited at 80°C and 100°C.

To characterize the lateral roughness characteristics and distribution of the surface structures with the increase in substrate temperature, two dimensional fast Fourier transform

(2D-FFT) was applied to the FESEM micrographs (Figs. 1 and 2) and their power spectra obtained as represented in Figs. 7 and 8. The use of this technique in surface analyses has been described in the literature [7], [9]. As seen in Figs 7 and 8, the average radii of equivalent circles (R) of the high-frequency region of the spectra (bright feature) were determined as $1.08 \pm 0.043 \mu\text{m}$, $1.06 \pm 0.169 \mu\text{m}$, $1.03 \pm 0.102 \mu\text{m}$, $0.42 \pm 0.001 \mu\text{m}$ for films sputtered at 40°C , 60°C , 80°C and 100°C respectively. As seen, the values (R) decreased with the increase in substrate temperature. A very small value of R indicates uniform, smooth and continuous microstructure with less porosity and defects whereas large values of R means that the microstructure is rough and exhibiting pits and serrated surface topography as was discussed from the section profiles. Furthermore, a small value of R is an indication of small correlation length across the surface of the film; which means that the films deposited at 100°C were laterally homogenous [14]. These results can be well related to the AFM observations described above and other previous publications [14].

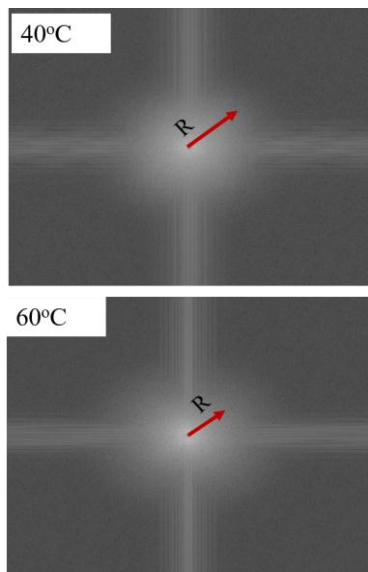


Figure 7. Representative power spectra for the FESEM micrographs of the Al thin films deposited at 40°C and 60°C . The red arrow marked R indicates the radius of the equivalent circle of the bright feature of the FFT process.

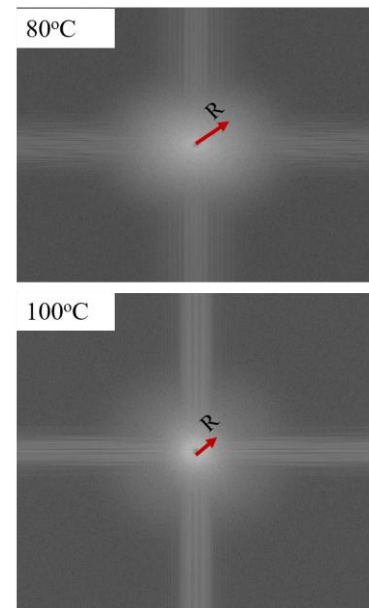


Figure 8. Representative power spectra for the FESEM micrographs of the Al thin films deposited at 80°C and 100°C . The red arrow marked R indicates the radius of the equivalent circle of the bright feature of the FFT process.

Fig. 9 shows the load (P) against displacement (h) plots obtained from the nanoindentation tests of the surface of Al thin films deposited at different substrate temperatures. As shown, all the films experienced elastic-plastic behaviour during load and unloading with the minimum deformation reported at 100°C . The Youngs moduli were determined as 54.94 ± 23.13 , 26.79 ± 19.26 , 55.15 ± 23.41 and 63.87 ± 44.40 GPa for films deposited at 40°C , 60°C , 80°C and 100°C respectively. The nano-hardness values were obtained as 1.02 ± 0.6040 , 0.5617 ± 0.5069 , 1.08 ± 0.4627 and 1.38 ± 1.20 GPa for 40°C , 60°C , 80°C and 100°C respectively. The mechanical properties confirm the FESEM and AFM observations on dense and continuous Al thin films deposited at 100°C as compared to those produced at lower temperatures.

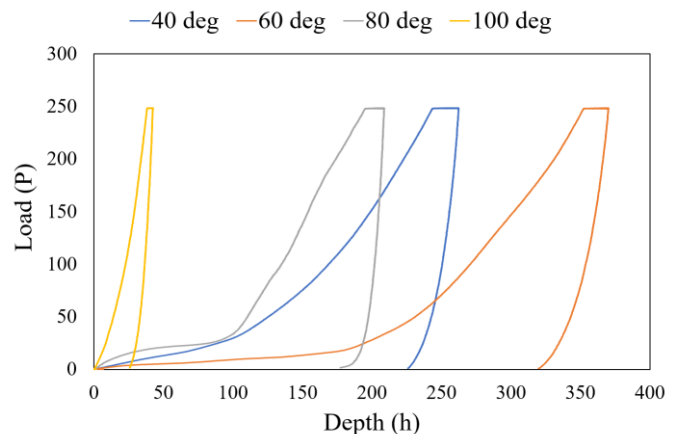


Figure 9. Nanoindentation curves for the Al thin films deposited at various substrate temperatures.

III. Conclusions

The FESEM and AFM analyses on the evolution of surface properties of Al thin films sputtered on mild steel substrates at different temperatures in this work has led to the derivation of the following significant concepts:

- ❖ As the sputtering temperature of the substrate increases, the formation of the clusters of aluminum structures evolves to grains with well-defined grain boundaries.
- ❖ There is a reduction in porosity and other surface defects (observed as pits in AFM) as the substrate temperature increases between 40°C and 100°C.
- ❖ As indicated by the power spectra of the 2D-FFT algorithm of the FESEM images and the line profiles of the AFM images, there is lateral growth of the surface structures with the increase in the substrate temperature. The lateral growth is characterized by fine, smooth and well-defined grains.
- ❖ The Al thin films exhibiting higher lateral development and less porosity (at 100°C) exhibit the highest elastic modulus and nano-hardness values as obtained via the nanoindentation tests.

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