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Evolution of surface roughness and mechanical properties of Sputtered Aluminum thin films

F.M. Mwema ^{1*}, E.T. Akinlabi ¹, O.P. Oladijo ^{1,2}

¹Department of Mechanical Engineering Science, University of Johannesburg, South Africa.

² Department of Chemical, Materials & Metallurgy Engineering, Botswana International University of Science & Technology, Palapye, Botswana

*Email: fredrick.mwema@dkut.ac.ke

Abstract-

In this work, aluminum thin films were sputtered on steel substrates at a varying substrate temperature ranging between 40 and 100 degrees Celsius. The films were characterized for microstructure by field emission scanning electron microscope, topography by atomic force microscope, mechanical properties by nanoindentation and the results related to the wear behavior of the films under very high sliding load of 30 N. The mechanism of failure of such films were observed and the relationship between the substrate temperature and sliding failure discussed. The study is important in understanding the failure mechanisms and improvement of the surface properties of sputtered aluminum thin films.

Keywords: Aluminum, sputtering, temperature, thin films, wear

1. Introduction

Aluminum thin films find applications in microelectronics, optics and as coatings for protection of surfaces [1]–[3]. These films can be deposited on metallic, polymers, ceramics and glass substrates depending on the required application. When deposited on metals, aluminum films can act as sacrificial agents for corrosion protection and barrier metals for semiconductors [1], [4]. The films can be prepared by either chemical or physical deposition methods and published literature has shown that physically deposited thin films especially through sputtering exhibit attractive properties for different applications [1].

It has been extensively reported that the sputtering parameters influence the microstructure and performance of Al thin films [1], [5], [6]. The most important parameters during sputtering of aluminum thin films are temperature, substrate type, power, bias voltage, argon flow rate, etc. [1]. In this work, we illustrate the effect of substrate temperature on microstructure, topography, mechanical and wear properties of Al thin films sputtered on a mild steel substrate.

2. Methodology

The aluminum thin films were prepared using radiofrequency (RF) magnetron sputtering system described elsewhere [7] and some of the processing parameters and conditions are summarized in Table 1. Prior to the sputtering, the mild steel substrates were ground using SiC papers for up to #1200 grain and then finely polished with a diamond paste of 1 µm. They were then cleaned in a vibrating bath with distilled water, acetone and dried in high-pressure and hot air.



Both the target and substrates were pre-sputtered for 10 minutes to remove any oxides and other contaminants on their surfaces.

The details of the sputtering process were discussed in our previous articles [7], [8]. After deposition, the samples were left to cool in vacuum for at least 6 hours to reduce oxidation and formation of other defects. The samples were then removed from the chamber and sliced into sizes of 10 mm by 10 mm for analysis. The microstructure of the films was obtained via field emission scanning electron microscope (FESEM) as detailed elsewhere [7], topography through atomic force microscopy (AFM) as described in references [9]–[11] nanoindentation and wear [12].

Table 1: Sputtering parameters and materials

Parameter	Conditions
Substrates	Mild steel [11]
Targets	99.9% aluminum; diameter 75 mm and 3 mm thick [7]
Temperature	40, 60, 80, 100 degrees Celsius (°C)
Target-substrate separation	130 mm
RF Power; time	200 W; 2 hours
Argon flowrate	12.0 sccm

3. Result and discussions

The evolving surface microstructural features of the Al thin films with a substrate temperature between 40 and 100 °C are shown by the representative FESEM micrographs in figure 1. At 40°C, the microstructure appears amorphous with fine structures of aluminum structures. There are also occasionally larger and long structures appearing as bright features (Fig. 1). At 60 °C, the microstructure is highly porous and containing interconnected amorphous aluminum. It is very difficult to observe Al grains at 60 °C. As the temperature increases to 100 °C, there is the formation of larger and well-defined Al grains with fewer porous structures. These observations are attributed to an increase in adatom energy at a higher temperature and therefore enhancing grain growth as earlier described in our previous studies [7], [11].

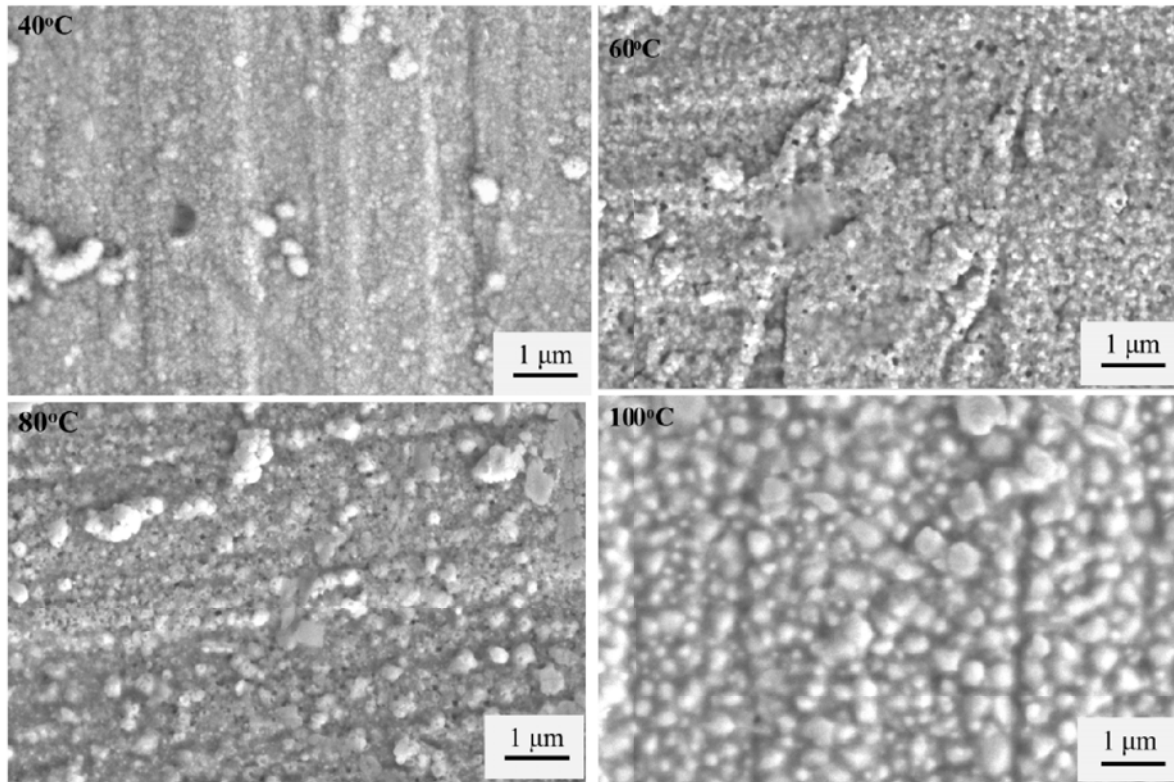


Figure 1: FESEM micrographs of aluminum thin films sputtered on mild steel substrates at different substrate temperatures. The images were obtained at a magnification of 30,000X.

The topographical properties of the films prepared at different substrate temperatures are represented in figure 2. For each surface, ten AFM images were taken from which the root mean square surface roughness values were averaged as; 13.85 ± 5.42 nm, 16.78 ± 0.96 nm, 13.29 ± 1.38 nm and 19.15 ± 0.08 nm for films deposited at 40, 60, 80 and 100 °C respectively. These values show that high roughness is an indication of either very high porosity (60 °C) or very large grain sizes (100 °C) as observed in figure 1. The large error observed at 40 °C roughness value is an indication of the non-uniform distribution of surface structures-the microstructure, in this case, consists of fine and large structures as well as small pits on the surface [6]. The error is insignificant at 100 °C, indicating uniform distribution and growth of Al grains at higher sputtering temperature.

As seen in figure 2 (through inspection), the pits (dark regions) generally decreased from 60 °C to 100 °C. These pits represent porosity in the FESEM micrographs (figure 1). The values of skewness and coefficient of kurtosis were obtained as 0.345 ± 0.238 and 0.218 ± 0.221 , -0.211 ± 0.015 and -0.513 ± 0.053 , 0.442 ± 0.251 and 1.006 ± 0.114 , -0.053 ± 0.021 and -0.380 ± 0.018 for 40, 60, 80 and 100 °C respectively. These results indicate that all the surfaces are bumpy with the negative skewness values at 60 °C and 100 °C indicating porosity and large grain sizes respectively [7]. These results indicate a nonlinear relationship between roughness and substrate temperature [8] although the highest roughness value is obtained at 100 °C.

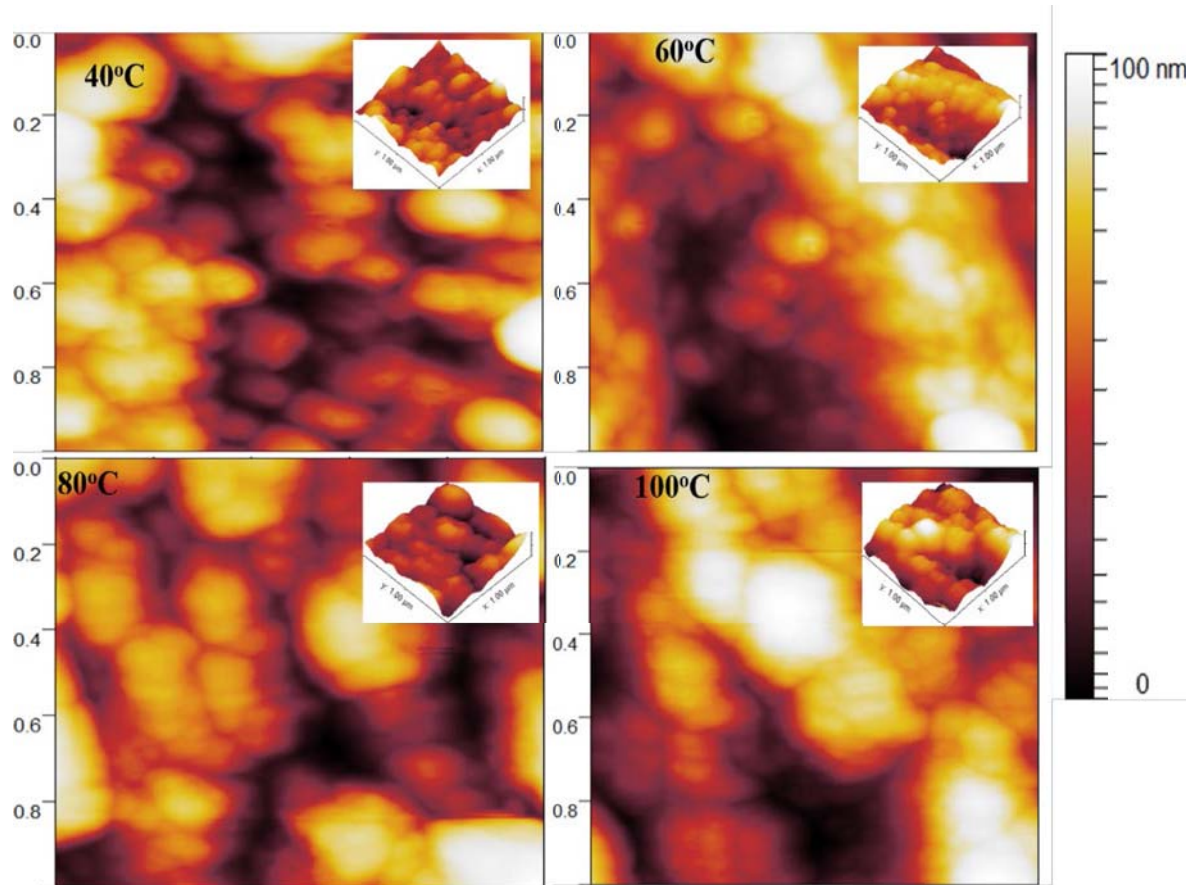


Figure 2: Representative 2D AFM images of aluminum thin films sputtered on mild steel substrates at different temperatures. The AFM scan area was $1 \mu\text{m}^2$. The scale bar of the images is shown to the right with a maximum height of 100 nm.

The results of mechanical properties of the Al thin films characterized through nanoindentation and wear techniques are represented in figures 3-5. The mechanical properties in figure 3 were obtained from the nanoindentation test via the established theory [13], [14]. The Young's modulus and hardness are shown to vary across the different films; with the higher values obtained at 100 °C and 40 °C while the lower obtained at 60 °C and 80 °C. There is no linear relationship between temperature and mechanical properties as reported in other studies [15]; however, the highest modulus and hardness are obtained at well-defined and porosity-free microstructure (at 100 °C). The high hardness and modulus at 40 °C can be attributed to fine structures observed on the FESEM images. The lower values at 60 °C can be attributed to the very porous structure and pits observed via FESEM and AFM measurements respectively. The low values of hardness and modulus at 80 °C can be attributed to the presence of large aluminum structures with unclearly defined microstructure.

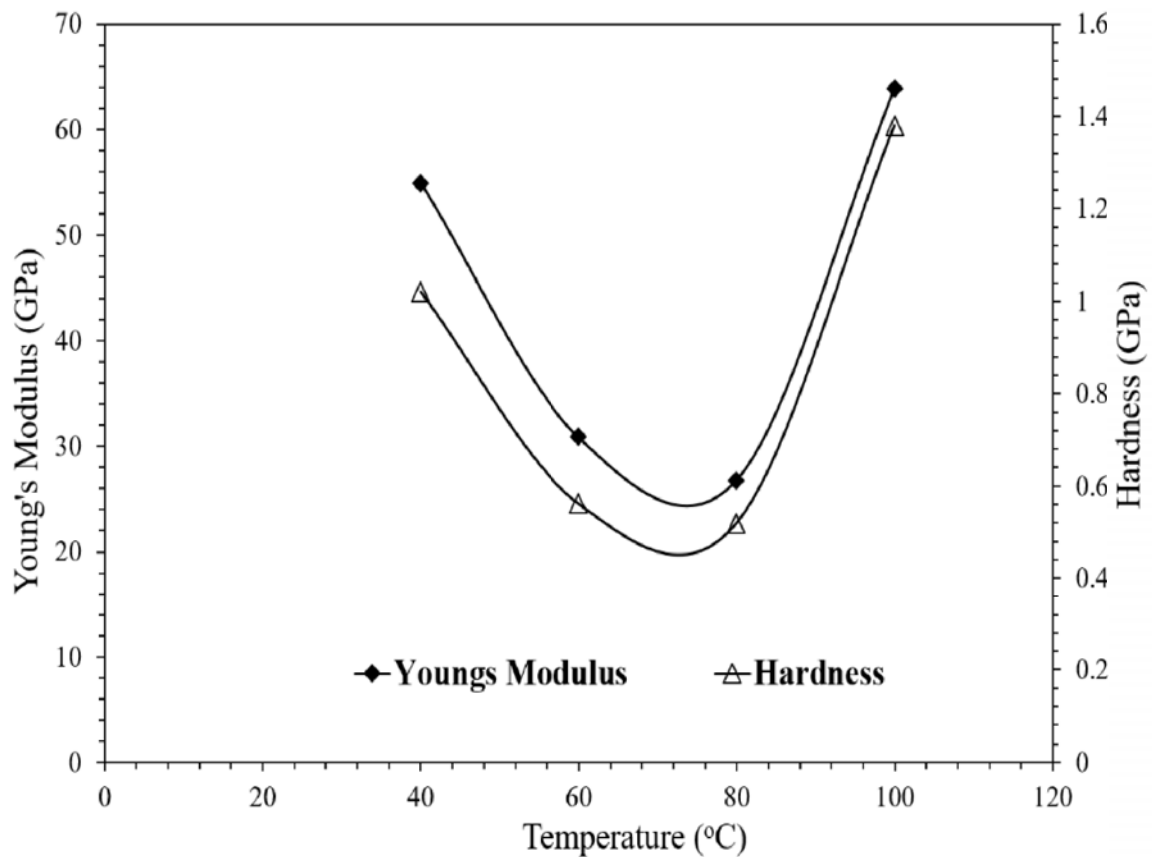


Figure 3: Mechanical properties of aluminum thin films deposited on mild steel substrates at different temperatures.

A plot of coefficient of friction (μ) against the time (in seconds) of sliding for all the films is presented in figure 4. Generally, within the first second (1 s) of the wear test, the coefficient of friction increases steadily and then a nearly sinusoidal variation is experienced. Similar results were reported for aluminum films deposited on stainless substrates [12]. The mean constant coefficients of friction are computed as 0.1271, 0.0982, 0.1071 and 0.1683 for 40, 60, 80 and 100 °C respectively. High values of coefficient of friction indicate better resistance of the film to failure due to sliding load [12], [16]. The results indicate that the films deposited at 100 °C and exhibiting well-defined structural properties have a higher resistance to wear failure. Aluminum films consisting of porous structures exhibit the lowest coefficient of friction indicating that porosity enhances failure in such films. Figure 5 shows the optical images of the surfaces of the wear tracks for all the samples. In such extreme loading, the wear mechanism can be understood by inspecting the edges of the track. At 60 °C, the edges are discontinuous which can be attributed to compaction of the porous structure as earlier explained in our previous article [12]. The small and discontinuity observed at the central regions of the wear track at 100 °C are indications of the film's resistance to sliding loading.

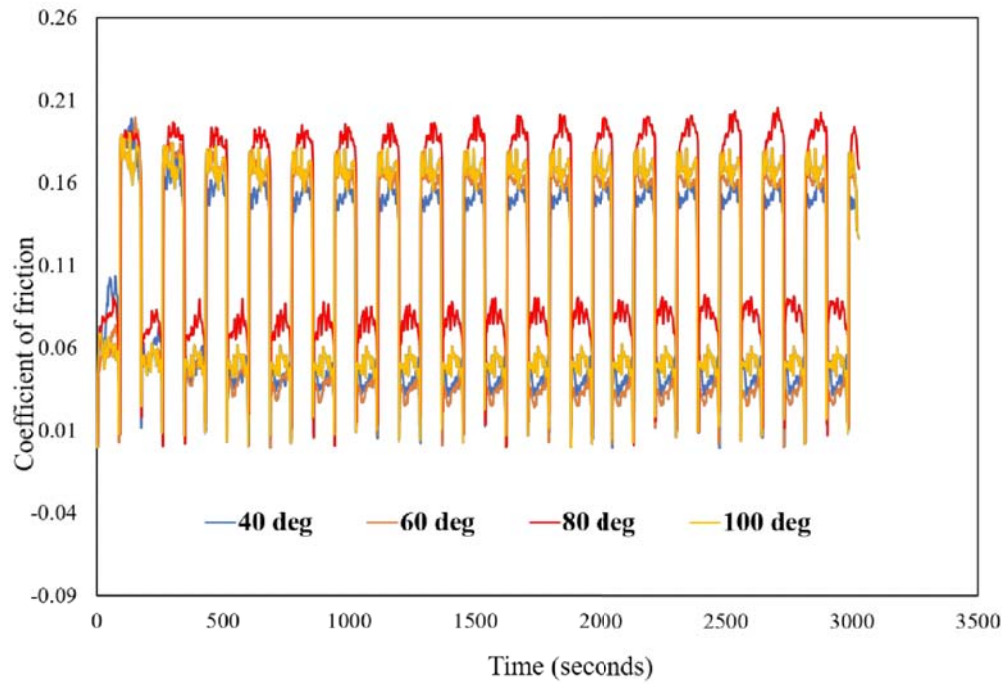


Figure 4: The coefficient of friction against the time of sliding for films sputtered at different temperatures

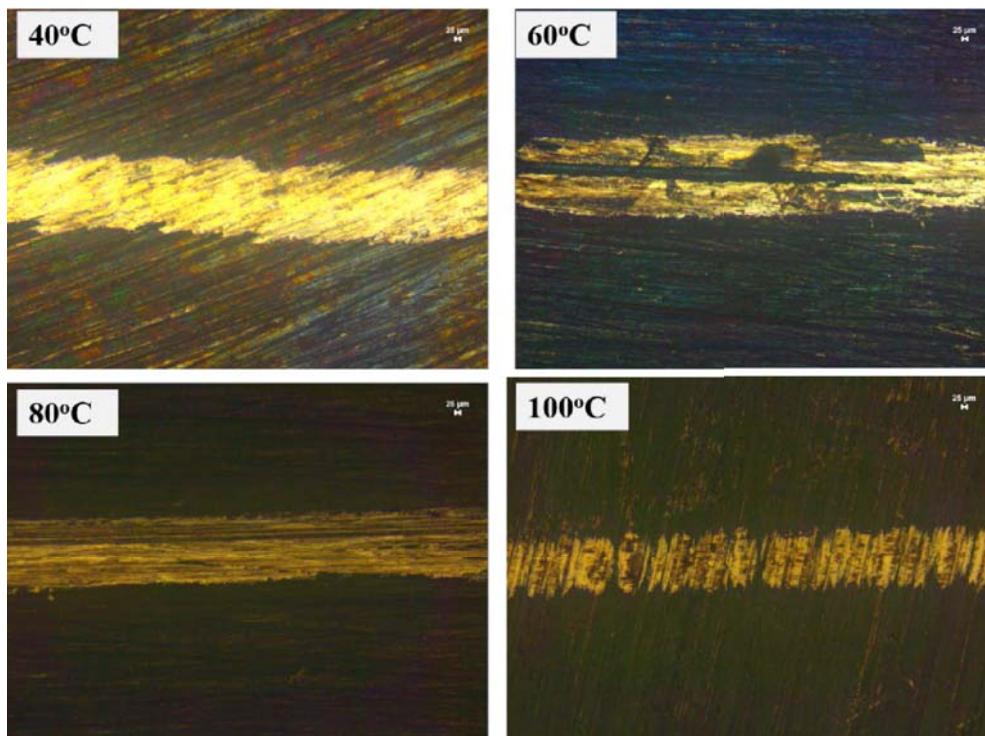


Figure 5: The optical images of the wear tracks on the Al thin films. A scale bar of 25 μm is shown in each image.

4. Conclusion

Aluminum thin films deposited on mild steel substrate at varying substrate temperature have been investigated for topography, structure and mechanical characteristics and the following conclusions can be drawn:

- The microstructure consists of large, well-defined and evolved grains at 100 °C whereas at 60 °C a highly porous microstructure is observed.
- The higher values of roughness obtained at both 60 and 100 °C indicate porosity and evolved grains respectively. Topography imaging reveals more pits on samples prepared at 60 °C than the rest of the samples.
- The highest hardness and Young's modulus values are obtained at 100 °C and those films show better wear properties at an extreme sliding load of 30 N.

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