

# Influence of sputtering times on the structural and optical properties of Al thin films for radiation-proof applications

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Al thin films for radiation-proof applications were successfully deposited by magnetron DC-sputtering with different sputtering times (15, 30 and 60 min). The effect of sputtering time on the film structures, morphology and optical properties was investigated in detail. The results show that the increase of sputtering time is in favor of being constituted in a cubic structure with a preferential orientation of Al (111), (200), (220) and (311) diffraction planes. Moreover, the morphology results implied that increasing sputtering time could vary the structure and increase the grain density and size. Finally, it is also indicated that 60 min is the best sputtering time for the optical properties in all samples.

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*Keywords:* Thin film, Al, Sputtering time, Radiation-proof, Optical properties

For several years the study of metal films on textile fabrics has been intensified in order to find new materials for radiation-proof applications [1, 2]. Among these protective films, Al film is one of the suitable candidates for the production of radiation-proof materials due to its good light reflection, heat radiation performance, high conductivity, the shielding effect of the microwave and anti-static properties [3], which make it extensive application in the military, civilian and other fields [4].

Various techniques such as DC-magnetron sputtering [5], flash evaporation [6], chemical spray [7] and electro-deposition [8] have been investigated as suitable techniques for the fabrication of Al thin films. Enormous amount of work has been already reported on structure, optical properties of Al thin films [9, 10]. However, still it requires further investigation to optimize the optical properties of Al films to use as a suitable candidate for radiation-proof applications.

It is well known that properties of Al films are strongly dependent on the preparation methods and deposition parameters [11, 12] due to obtained stoichiometry and microstructure. For this reason, it is essential to characterize their properties according to the deposition parameters.

In this paper, we report the preparation of high-quality Al nano-crystalline thin films on polyester substrates, using a DC-magnetron sputtering technique. The effect of the sputtering times on the optical performance of nano-crystalline Al films was investigated.

## 1. Experiment

### 1.1 Synthesis of Al thin films

Thin films of Al were deposited by DC-sputtering in argon gas atmosphere on polyester substrates. Substrates were cleaned ultrasonically and chemically in organic solvents. A thin Al film was deposited by an Al target of 60 mm diameter. No changes in target composition were observed with time. All films were deposited at the room temperature 25 °C, at a working gas pressure of 2.0 Pa and a sputtering power of 100 W. The samples were deposited at the different sputtering times (15, 30 and 60 min). The process parameters of Al films used in DC magnetron sputtering are shown in Table 1.

*Table 1. Sputtering parameters of Al films.*

Sample	Sputtering time / (min)	Sputtering power / (W)	Sputtering temperature / (°C)
a	15	100	25
b	30	100	25
c	60	100	25

### 1.2 Characterizations of Al films

Optical properties of the Al thin films were measured at normal incidence using a double-beam UV-VIS-NIR spec-

trophotometer (type Lambda 35 from Perkin Elmer) of optical transmittance in the photon energy range of 1.1- 6.6 eV. The resistivity calculated from the sheet resistance measured by a four-point probe. Coupled  $\theta$ - $2\theta$  X-ray diffraction (XRD) scans in the simple mode were performed in the range of  $2\theta=5^\circ$ - $90^\circ$  by using of the Cu  $K\alpha$ 1 line of the X-ray source (type Rigaku D/max2550) to investigate crystallographic properties of the films. The surface morphology of each film was examined by scanning electron microscopy (SEM-3400-N, type Hitachi).

## 2. Results and discussion

### 2.1 Influence of sputtering times on the structures of Al films

Fig. 1 shows the XRD patterns of Al films grown on polyester substrates. It can be seen there were differences between XRD patterns of Al films at the different sputtering times. The peaks at  $38.2^\circ$ ,  $44.5^\circ$ ,  $65.8^\circ$  and  $78.4^\circ$  are correspondes to Al (111), (200), (220) and (311) planes, respectively[13]. It is clear that the sputtering times had a significant effect on the preferential orientation of this cubic phase. In addition, there is some diffuse diffraction peaks indicating there were both partially amorphous organization in these samples.

Meanwhile,  $\text{Al}_2\text{O}_3$  (012) diffraction peak appeared on the position ( $2\theta = 26.5^\circ$ ) [14]. It is clear that the partial oxidation of the Al cubic phase can be detected in these Al samples while the oxidation tendency is reduced with the increase of sputtering time. It shows that the longer sputtering time (60 min) provides sufficient energy for the sputtering particles to form crystalline but not to oxidation.

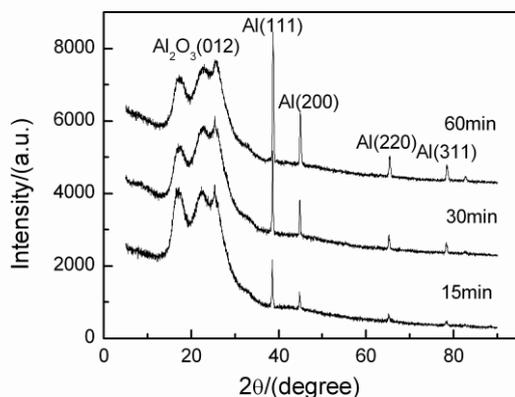


Fig. 1. XRD patterns of Al films deposited at different sputtering times.

### 2.2 Influence of sputtering times on the morphology of Al films

Fig. 2 shows the Low multiples ( $\times 1k$ ) of SEM images of the Al films at the different sputtering times. It can be seen from Fig. 2 that the surface of film at 60 min was rougher and the number of grain boundaries was higher than that of 15 and 30 min sputtering time. It shows that the microstructures and surface morphologies of the films can be improved by the increase of sputtering time. This could be attributed to the thorough diffusion and better growth of the Al grains on the increase of sputtering particles at the longer times [15, 16].

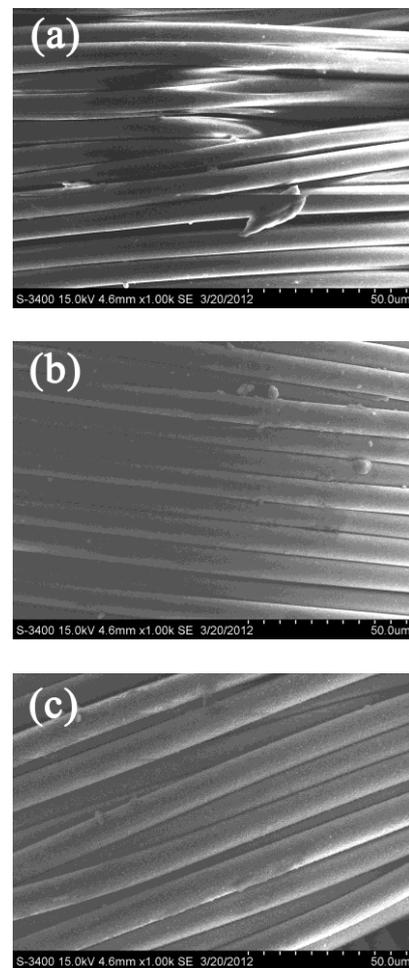


Fig. 2. Low multiples ( $\times 1k$ ) of SEM images of Al films deposited at different sputtering times: (a) 15 min, (b) 30 min and (c) 60 min.

Fig. 3 is the high multiples ( $\times 30k$ ) of SEM images of the Al films at the different sputtering times. It shows that the film deposited at 15 min has a few isolated grains (Al grains) with uniform size and well-defined boundaries on the polyester substrates at low sputtering rate. When the sputtering time of 30 min deposited, the grain boundaries

become irregular and some of the grains are connected due to the increase of the solute concentration or variation of the applied potentials [17]. However, the shapes of grains were changed from the circular to the trapezoidal when the sputtering time increased from 30 to 60 min. Also, the particle sizes and intensities are increased as the increase of the sputtering times can be seen in Fig.3. This result may be due to the improvement of the surface diffusion energy of deposition atoms on substrates at the longer time, which will promote atoms to escape from the surface [18], thus the holes among the sputtering particles were reduced.

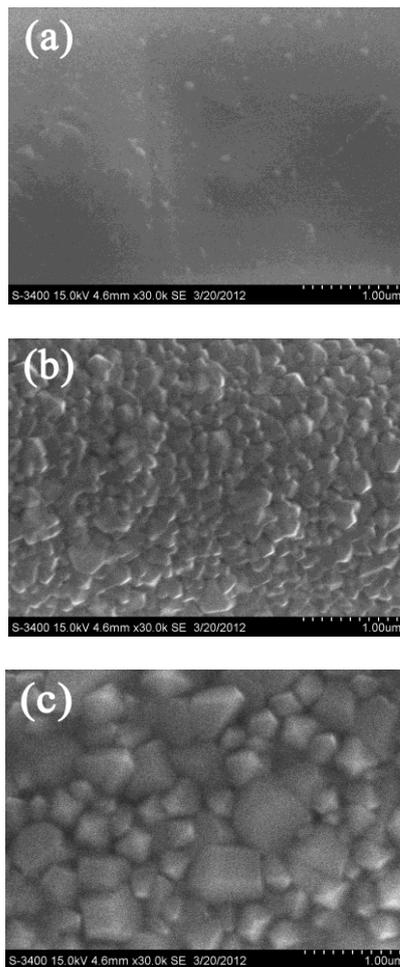


Fig. 3. High multiples ( $\times 30$  k) of SEM images of Al films deposited at different sputtering times: (a) 15 min, (b) 30 min and (c) 60 min.

### 2.3 Influence of sputtering times on the optical properties of Al films

Fig. 4 is the transmittance spectra curves of Al films deposited at different sputtering times. It can be seen in Fig. 4 that the optical transmittance of Al films (15 and 30 min) both exhibited good optical transmission compared with the Al film (60 min). This indicates that radiation-proof properties of Al films were enhanced on the increase of the

sputtering times. The reason is due to that the bigger particle size and density of Al cubic phases are more helpful to improve the reflectivity. In addition, a certain number of grain boundaries can reduce the optical transmittance up to a certain extent, which also increased the radiation-proof properties of Al films.

Meanwhile, it can be found in Fig.4 that the radiation-proof properties of Al films can be affected by the oxidation of Al grains. It can be seen in SEM images that the grains seem to be rather compact and density of the holes decreased in the Al film (60 min). Obviously, the reduction of defects in the films can also reduce the optical transmittance of visible lights. In this result, the radiation-proof properties of Al films can be improved in the less oxidation film at 60 min.

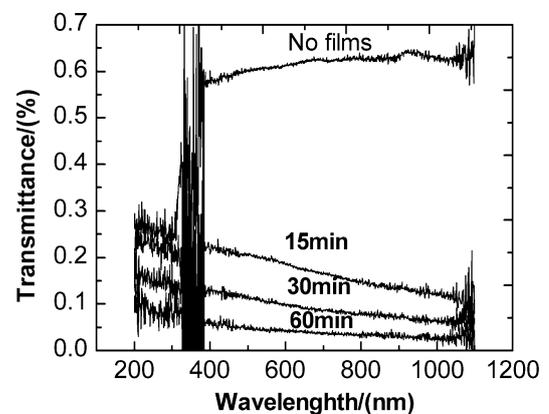


Fig. 4. Transmittance spectra curves of Al films deposited at different sputtering times.

### 3. Conclusions

Al films for radiation-proof applications were successfully deposited on polyester substrates by magnetron sputtering at different sputtering times. It is found that the sputtering time had a significant effect on film structure, surface morphologies and optical properties in the sputtering process. Moreover, the grain growth and crystalline improvement of Al cubic phases are more helpful to improve radiation-proof properties of Al films. Furthermore, the reduction of defects in the oxide layer at 60 min can also reduce the optical transmittance of visible light.

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## References

- [1] X. Ji, Y. M. Mi, Z. Yan, C.M.Zhang, *Optoelectron. Adv. Mater. – Rapid Commun.* **5**(9), 977 (2011)
- [2] M. A. Popescu, *J. Non. – Cryst. Solids.* **169**(1-2), 155 (1994).
- [3] X. Ji, Z. Yan, Y. M. Mi, C. M. Zhang, *Optoelectron. Adv. Mater. – Rapid Commun.* **6**(1-2), 300 (2012).
- [4] M. A. Popescu, *J. Non-Cryst. Solids.* **35-36**, 549 (1980).
- [5] N. Djedid, B. Hadoudja, B. Chouial, S. Yousfi, A. Chibani, *Optoelectron. Adv. Mater.–Rapid Commun.* **5**(8), 827 (2011).
- [6] X. Ji, Z. Yan, Y. M. Mi, C. M. Zhang, *Optoelectron. Adv. Mater. – Rapid Commun.* **6**(3-4), 483 (2012).
- [7] P. S. Menon, B. Bais, A.A.M. Jhi, S. Shaari, *Optoelectron. Adv. Mater. – Rapid Commun.* **6**(5-6), 535 (2012).
- [8] A. Cheniti, O. Ponta, L. Tirle, T. Radu, S. Simon, *Optoelectron. Adv. Mater. – Rapid Commun.* **6**(5-6), 560 (2012).
- [9] Z. Rizwan, B. Z. Azmi, M. G. M. Sabri, *Optoelectron. Adv. Mater. – Rapid Commun.* **5**(4), 393 (2011).
- [10] N. Touka, B. Boudine, O. Halimi, M. Sebais, *Optoelectron. Adv. Mater. – Rapid Commun.* **6**(5-6), 583 (2012).
- [11] D. E. Baciú, J. Simitzis, *Optoelectron. Adv. Mater. – Rapid Commun.* **6**(5-6), 648 (2012).
- [12] V. Senthamilselvi, K. Saranakumar, R. Anandhi, A. T. Ravichandran, K. Ravichandran., *Optoelectron. Adv. Mater. –Rapid Commun.* **5**(10), 1072 (2011).
- [13] F. H. Lu, H. D. Tsai, Y. C. Thin Solid Films. 516,1871 (2008).
- [14] Y. Yu, A. Ito, T. Rong, T. Goto, *Appl. Surf. Sci.* **256**, 3906 (2010).
- [15] B. S. Li, K. Akimoto, A. Shen, *J. Cryst. Growth.* **311**, 1102 (2009)
- [16] K. Y. Chan, B. S. Teo, *Microelectron. J.* **38**, 60(2007).
- [17] L. Zhang, F. D. Jiang, J. Y. Feng. *Solar Energy Mater. Sol. Cells.* **80**, 483 (2003).
- [18] Y.Y. Jiang, Z.Q. Lai, Z. D. Wang, Q. H. Huang, *Journal of Nanchang University (Natural Science)*, **31**, 545 (2007) (in Chinese).

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