The influence of RF power and gas pressure on the surface characteristics of aluminium oxide deposited by RF magnetron sputtering plasma

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Abstract. Aluminium oxide thin films are deposited in the transition mode by RF magnetron sputtering of aluminium target at different applied RF power and different pressures of argon and oxygen gases. The films exhibit grainy surface microstructure with varying size and density. Nanostructured alumina film can be grown at lower gas pressure and optimized power. The chance for grain formation is lowered by the increase of power. Investigation reveals that it is possible to grow crystalline alumina film at low substrate temperature. Deposition conditions are optimized for protective alumina film coating on bell metal with good adhesion and corrosion resistance properties.

1. Introduction:
The Aluminium oxide thin films are widely used in different industrial applications from microelectronic and optical applications to wear resistant coatings [1-5]. All these properties of alumina film depend on different system parameters such as sputtering rate, target to substrate distance, pressures of buffer and reactive gases, substrate temperature and also the nature of the substrate material. Low temperature synthesis of crystalline alumina is of great technological interest as high substrate temperature requirement limits the selection of substrate. Recently, results have been published on crystalline alumina deposition at low temperature in poisoned mode of ac magnetron sputtering [6]. It has been studied that significant amount of negative oxygen ions is formed at the target surface during poisoned mode of sputtering. These negative ions are accelerated to high energy (a few hundred eV) through the sheath potential and bombard the substrate during film growth, which can account for crystalline alumina formation [7-8].

2. Experimental
The experiment is carried out in a stainless steel chamber of 40 cm in diameter and 45 cm in length placed vertically as described in ref. 9. The substrate is placed on a stainless steel substrate holder at a fixed distance of 6 cm from the target and kept grounded. The film deposited at larger target to substrate distance than 6 cm leads to poor deposition rate, while at smaller target to substrate distance the deposited film is damaged by the high electron current. The square size (8×8 mm²) bell metal substrates are initially polished with the help of a polishing machine using sand paper of different roughness rating. The final polishing of the samples is done by diamond paste of range 8 to ¼ microns. After polishing, the sample is cleaned ultrasonically for about 30 minutes with propan-2-ol
solution in an ultrasonic bath. This helps in removing any foreign materials or contaminants from the sample surface. Then the sample is dried and placed on the substrate holder.

The deposited films are characterized using X-ray diffractometry, Scanning Electron Microscope, Energy-dispersive X-ray analysis and Atomic force microscopy. The corrosion resistance of the films is investigated using copper accelerated acetic acid salt spray (CASS) test. Scotch tape test is done to see the adhesion quality of the films.

3. Result and Discussion:

It is observed that the deposited films show poor adhesion quality when substrate temperature increases above 150 °C. This is due to the stress developed due to the difference of the coefficient of thermal expansion and contraction between the film and the bell metal surface films. Therefore additional substrate heating is avoided in the present experiment.

It has been shown in previous studies that the deposition of aluminium oxide films in the transition region leads to highest rate of formation of aluminium oxide. Therefore in the present investigation, films are deposited in the transition region as described in following table 1. It is seen that the film B has higher deposition rate than film A. This is due to reduced gas phase scattering at lower pressure, which enables for effective sputtering and subsequent deposition on the substrate. Film C and Film D shows the increase of deposition rate. With the increase of power, the poisoning level of the target decreases which results into higher sputtering rate and also higher aluminium oxide formation rate.

| Film name | Pressure (torr) x 10^-3 | Argon Flow Rate (sccm) | Oxygen Flow rate (sccm) | Applied Power (Watt) | DC Self-bias (V) | Deposition Rate (nm/min) | Substrate temperature (°C) |
|-----------|-------------------------|------------------------|------------------------|---------------------|------------------|--------------------------|-----------------------------|
| A         | 1.3                     | 12                     | 6                      | 100                 | -178             | 8.37                     | 100                         |
| B         | 82                      | 10                     | 6                      | 100                 | -152             | 10.02                    | 86                          |
| C         | 82                      | 10                     | 6                      | 125                 | -184             | 11.35                    | 99                          |
| D         | 82                      | 10                     | 6                      | 150                 | -213             | 12.24                    | 120                         |

![Figure 1. XRD pattern of film D.](image)

The X-ray diffraction studies of the films A, B and C do not show any significant peaks, which indicates the formation of amorphous alumina. The film D shows two peaks corresponding to θ and γ alumina as shown in Fig. 1. This implies that in the poisoned mode of sputtering and controlled deposition condition there can be formation of crystalline alumina. This finding is attributed to the higher substrate temperature as well as the bombardment of the substrate by the high energetic negative oxygen ions that generally formed above the oxidized target.

Fig.2 shows the surface microstructure of the films A, B, C and D. The films are found to show agglomerated grainy surface structure. Interestingly it is found that film C and D contains nano-sized
grains (50-100 nm). Film D has comparatively less number of grains, which indicates that the increase of power decreases the chance for grain formation.

![Figure 2. SEM micrograph of films A, B, C and D.](image)

Fig. 3 shows the AFM image of the film B. It can be seen that the films are very much continuous and uniform with maximum roughness about 50 nm. The cracks observed in the film are due to the difference of coefficient of thermal expansion and contraction.

![Figure 3. AFM image of film B.](image)

The Copper Acetic Acid Salt Spray (CASS) test is done to determine the anti-corrosive property of the aluminium oxide thin films. The test solution is prepared using 5% NaCl solution and 0.026% CuCl2 solution, the PH of which is found to be close to 7. Required amount of glacial acetic acid is used to get the standardized test solution having PH value of 3. All the samples have been visually examined over a period of time to determine their capability to protect the bell metal alloy from corrosive attack. The cracks observed on the surface of the film after the test can be attributed due to the permeation of the salt solution. The corrosion test of the films reveals that the film A is
slightly more corrosion resistant than film B. However films C and D are found to have almost identical corrosion resistance properties with film B. This indicates that the corrosion resistance is almost independent of power.

The adhesion test with scotch tape shows that no portion of the films A, B and C peels off in the test indicating excellent adhesion quality of the film. However a portion of the film D is found to be peel off in the test. The bad adhesion of the film D on the bell metal substrate may be due to increase of the substrate temperature with the increase of applied power and the difference of coefficient of thermal expansion and contraction of the film and the substrate, which results into poor adhesion of the film.

4. Conclusion:
The characteristics of aluminium oxide coatings on bell metal substrate by RF magnetron sputtering technique have been investigated at low substrate temperature. The films are generally amorphous in nature. However, sputtering in the poisoned mode with optimised power can result in the formation of crystalline alumina. The surface of the films exhibits agglomerated grainy structure with varying size and density. The nanostructured alumina films can be deposited at lower argon flow rate. At higher substrate temperature (120 °C), the films are found to have poor adhesion quality.

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