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Protection of Kapton from Atomic-oxygen Erosion Using Alumina Film Deposited by Magnetron Sputtering

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Abstract. Ultrathin alumina film was deposited on Kapton substrate by direct current (DC) magnetron sputtering technique and their atomic oxygen (AO) erosion resistance was tested in a ground-based AO simulator. The AO fluence and the erosion yield of the alumina coated Kapton was calculated. The surface thickness and morphology of alumina film was investigated by scanning electronic microscope. The results indicate that as the depositing time increases, alumina film tend to become thick. Under AO environment, the AO erosion resistance of alumina coated Kapton is improved and enhances as the depositing time increases. It is noted that the alumina coated Kapton deposited in 60 min shows the best AO resistance and its erosion yield is two orders of magnitude less than that of pristine Katpon.

1. Introduction

Atomic oxygen (AO), the most prevalent of the atmospheric species in low earth orbit (LEO), can erode most space materials, especially polymers, resulting in the structural failure and performance degradation of the space crafts [1]. Since this erosion phenomenon was recognized, much effort for the development of atomic oxygen-resistant material has been carried out for more than two decades [2-3]. Numerous schemes are available to mitigate the AO damage to polymers and polymer matrix composites in the LEO environment. Researchers found that organic coatings have excellent AO resistance. However, it is very different to make a defect-free AO protective layer in a big surface, and the oxidation erosion of the underlying polymer can occur at pinhole and scratch defects through undercutting erosion. In contrast, the inorganic metallic oxide coatings, such as silica and alumina are proposed as potential protective coatings due to their unique combination of the merits of organic and inorganic coatings. Metallic oxide films can be prepared by physical or chemical deposition methods including filtered arc, laser ablation, direct ion beam, sputtering and plasma assisted chemical vapor deposition techniques. Magnetron sputtering is an extremely versatile technique that has been widely used for the

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preparation of both alloy and metallic oxide films. The superiority of this technique lies in its easier adaptability to industrial applications, as homogenous films on large-area substrates can be grown with high deposition rates. Comparatively, the magnetron sputtering method is a simple, inexpensive and low-temperature technique for preparing films, and especially suitable for application on complex shapes. In recent years, some data have been reported about using magnetron sputtering method to fabricate anti-AO films. In this paper, the alumina film was prepared on Kapton substrate by magnetron sputtering method and their AO erosion resistance was also investigated.

2. Experimental Procedure

Alumina thin film was deposited on Capton polyimide substrate using direct current (DC) magnetron sputtering technique at room temperature. The target is a circular disk with 76mm diameter and 0.5mm thickness and is fitted with a moveable shutter. The base pressure of the chamber was about 3.0×10^{-5} Pa for all deposition processes. Pure argon gas is introduced to the chamber as sputtering gas via a mass flow controller. The argon flow was changed to give chamber pressures between 0.35 and 1.5 Pa with a fixed throttle position on the vacuum pump. Stable plasma was established with an applied DC power of 90W to the magnetron gun. The alumina target with purity 99.99% and diameter of 60 mm was water-cooled. After pre-sputtering of the alumina target for 5 min, the shutter was removed and the deposition started. The substrates were not heated during the deposition and there was no substrate bias. The target to substrate distance was 60 mm. All samples were deposited for 30min without intentionally heating the substrates. The AO erosion resistance of silica-modified Kapton was tested in a ground-based AO simulator, where the AO beam with 5eV energy was produced using a laser detonation technology. The mass loss of all samples was measured at different time intervals using a microbalance with a precision of 10^{-5}g. For obtaining the accurate mass loss measurements, the samples was dehydrated and outgassed in vacuum for less than 24 h before AO exposure. The exposure of the samples to air during weighing was kept to the minimum to prevent rehydration. From the mass loss of the pristine Kapton, AO fluence was calculated using the following formula:

$$F=\triangle M/(A \bullet \rho \bullet E).$$

where $\triangle M$ is the sample mass loss (g), A is the sample surface area (cm^2), ρ is the sample density (for Kapton, $\rho = 1.42$ g/cm^3), and E is the sample erosion yield (for Kapton, $E = 3*10^{-24}$ cm^3/atom).

For surface microstructure analysis, a Scanning Electron Microscope (SEM) (Hitachi S-4200) was used at the accelerating voltage of 15 kV.

3. Results and Discussion

3.1AO Exposure

The thickness of alumina film as a function of depositing time is shown in Fig. 1. It can be seen that the film thickness increases linearly with the depositing time. The thickness of alumina film is below 100 nm when the depositing time is 10 min. The thickness of alumina film increased to about 700 nm when the depositing time rose to 90 min. The mass loss versus AO fluence for pristine and alumina coated Kapton with various deposited time is shown in Fig. 2. It can be observed that the mass loss for all samples increases linearly with AO fluence, but the gradient of these lines decreases with the deposited time. This indicates that the erosion yield of alumina-modified Kapton decreases as the depositing time increases. After AO exposure to a fluence of 2.5×10^{20} atoms/cm^2, the mass loss of pristine Kapton is 9.2 mg/cm^2, while those of coated samples deposited in 10, 30, 60 min are 4.3, 2.1 and 1.2 mg/cm^2 respectively. However, as the depositing time sequentially increases, the mass loss of coated samples presents increase sharply. The coated sample deposited in 90 min shows a mass loss of about 6 mg/cm^2 after AO exposure of the same fluence. Comparatively, the coated Kapton deposited in 60 min displays the least mass loss, demonstrating that this sample has the best AO resistance. These results suggest that there is an optimum thickness about 500 nm of the alumina film to protect Kapton substrate from AO erosion.

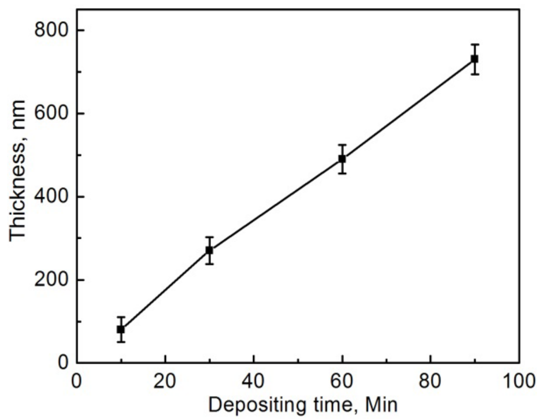

Fig. 1 Thickness of alumina film in relation to depositing time

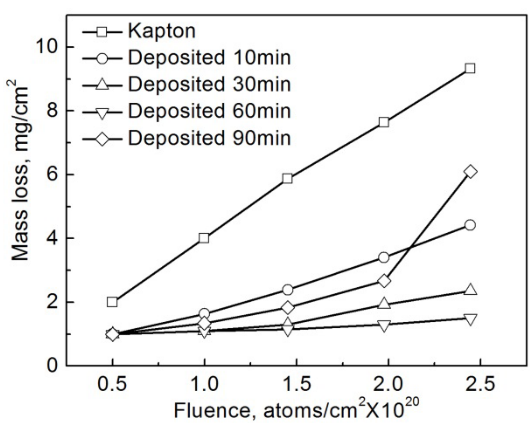

Fig.2 Mass loss versus AO fluence for pristine and alumina-coated Kapton with various depositing time

3.2Surface Analysis

SEM photograph of pristine and alumina coated Kapton are shown in Fig. 3. The pristine and all alumina coated Kapton in various depositing time except 90 min have a smooth surface. The EDX results make clear that alumna film generated in the surface of Kapton. After AO exposure, the pristine Kapton suffers serious erosion and presents a

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carpet-like appearance (Fig. 4(a)). This is consistent with the results in the literatures [4]. For the coated samples with various depositing time, they all exhibit a smooth, uniform and no-crack surface before AO exposure (same as Fig. 3). However, as seen in Fig. 4(b)–(d), the coated samples suffer different degrees of damage after AO exposure. The coated Kapton deposited in 10 min shows scratch-like erosion, with many erosion micro-holes evenly distributed on the surface. A few erosion micro-holes can be still observed on the sample surface deposited in 30 min. The erosion morphology includes two types: erosion micro-holes and tiny cracks. During AO exposure, the as-received pinholes in alumina film become the initiation sites to erode the underlying substrate, resulting in expanding into micro-holes. Some pinholes cause serious undercutting erosion of Kapton substrate, so that the alumina film is peeled off or cracked when alumina film is too thin. For the depositing time arrived 60 min, no erosion micro-holes appear on the surface, implying that the alumina film completely covers the substrate. However, as the depositing time sequentially increases, many cracks already formed on the alumina coated surface before AO exposure as shown in Fig. 5. The result may due to the thickening-induced embrittlement effect. Accordingly, the formed cracks in alumina film become a path for AO penetrating into the substrate. Therefore, there are two factors to determine the AO erosion resistance of alumina film: the film coverage and the thickening-induced embrittlement effect. When depositing for less than 30 min, the incomplete coverage of alumina film is the main reason of causing AO erosion. As more than 90 min, the thickening-induced embrittlement effect leads to the crack formation in alumina film, resulting in invalid protection effect. The alumina film deposited in 60 min completely covers the substrate and has the fewest cracks, thus providing the best AO erosion resistance.

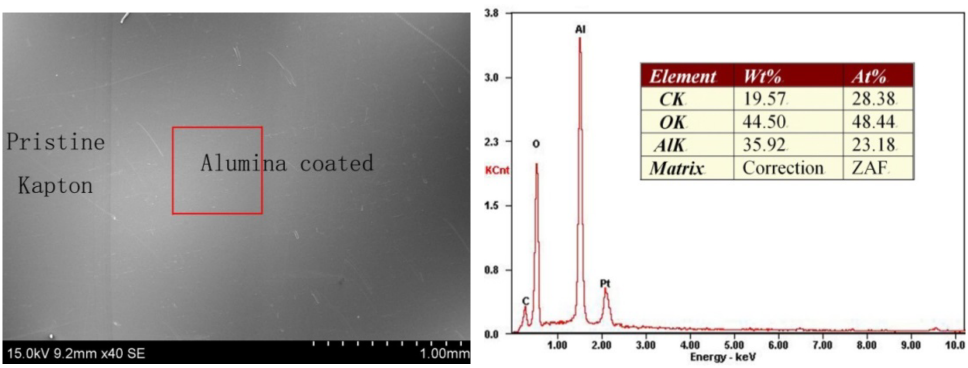
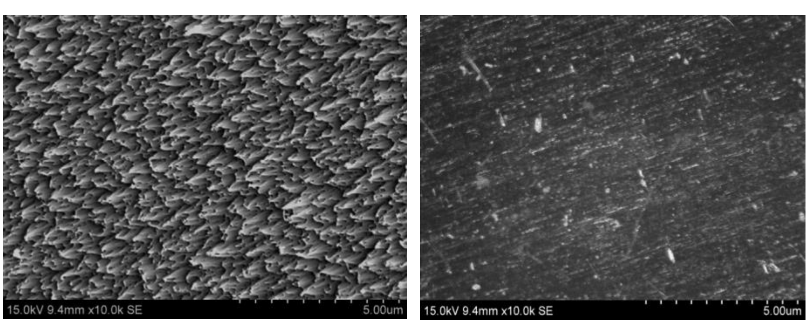
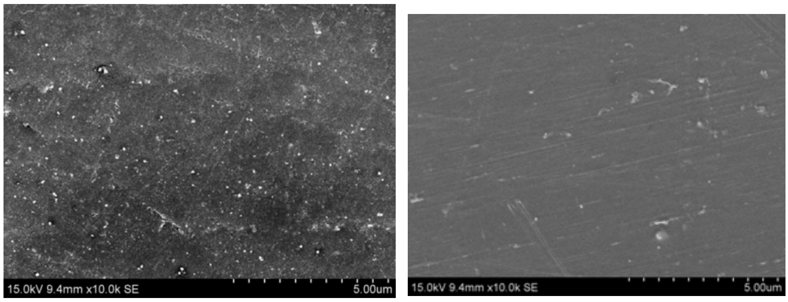


Fig. 3 SEM photograph of pristine and alumina coated Kapton with EDX analysis



(a) (b)



(c) (d)

Fig.4 SEM photographs of pristine and alumina coated Kapton deposited in different time after AO exposure. (a) Kapton, (b) 10 min, (c) 30 min, (d) 60 min

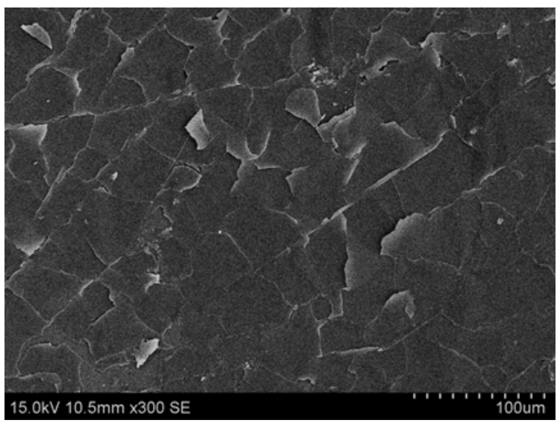


Fig. 5 SEM photograph of alumina coated Kapton deposited in 90 min

The thermal expansion coefficient of alumina is $6.6*10^{-6}$ K^{-1}, while that of Kapton is $5.0*10^{-5}$ K^{-1}. Owing to the larger difference of thermal expansion coefficient between the film and Kapton substrate, the larger stress concentration would be formed in the alumina film when the film is too thick. Thus the cracks are easy formed in the film. Comparatively, if the alumina film in thin enough, the stress concentration is not evident. Therefore, no cracks are detected in the alumina coated Kapton deposited below 60 min. The alumina film has some micro-pores when the deposing time is below 30 min. With the continual depositing process the interspaces of the alumina particles could be filled. In appropriate time a stable interface is formed between the alumina and Kapton substrate. As a result, the as deposited alumina film shows the best toughness and densification, thus they have the best AO erosion resistance.

4. Conclusions

Ultrathin alumina film was deposited on Kapton using magnetron sputtering technique. After depositing, the AO erosion resistance is improved. As depositing time less than 30 min, incomplete film on Kapton causes the AO erosion, as exceed 90 min, the thickening-induced embrittlement effect leads to early break of film. The coated Kapton deposited in 60 min shows the best AO resistance: its erosion yield decreases by two orders of magnitude compared with pristine Kapton.

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