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## Colloids and Surfaces A



# Improved adhesion between $SnO_2/SiO_2$ coating and polyimide film and its applications to atomic oxygen protection



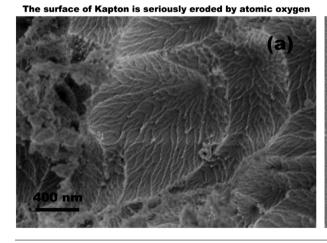
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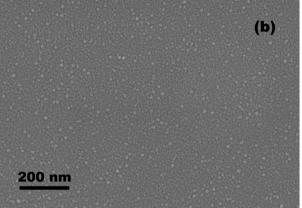
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#### G R A P H I C A L A B S T R A C T

After atomic oxygen exposure, the erosion yield of  $SnO_2/SiO_2$  coated Kapton decreased to  $0.15 \times 10^{-24}$  cm<sup>3</sup>/atom, which is only 4.1% as that of pristine Kapton.



### SnO2/SiO2 coated Kapton has good resistance to atomic oxygen



#### ARTICLE INFO

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#### ABSTRACT

Alkali solution combined with silica coupling agent were used to modify the surface of polyimide in order to improve adhesion between inorganic coating and polyimide substrate. After surface treatment by NaOH and  $\gamma$ -aminopropyltriethoxysilane (APTES), the water contact angle of polyimide substrate reduced greatly and the SnO<sub>2</sub>/SiO<sub>2</sub> coating was easily formed on it via sol-gel method. The optical transmittance of SnO<sub>2</sub>/SiO<sub>2</sub> coated samples remained steady when the number of coating layers increased from 3 layers to 7 layers. The atomic oxygen (AO) resistance of SnO<sub>2</sub>/SiO<sub>2</sub> coating was done in a ground-based AO simulation system for durability evaluation. Before and after AO irradiation, the morphology changes of the surface was analysed by means of scanning electronic microscope (SEM). The results indicated that the coating prepared by this method shows a very high quality protection to AO erosion.

#### 1. Introduction

Polyimide (PI) is one of the most widely used polymers in the space

environment because of its superior physical and chemical properties such as thermal stability and dielectric properties [1–3]. However, many environmental factors threaten the external spacecraft materials

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Received 12 March 2017; Received in revised form 10 June 2017; Accepted 11 June 2017 Available online 13 June 2017 0927-7757/ © 2017 Elsevier B.V. All rights reserved. in low earth orbit (LEO), such as atomic oxygen (AO), ultraviolet (UV) and ionizing radiation [4-6]. AO is considered to be the most destructive environmental factor to polymers, due to its strong oxidation ability and high impact velocity [7,8]. No doubt, the damaging effects of AO erosion on spacecraft materials during the periods of on orbit should be highly focused on. Many methods are used to improve the anti AO erosion properties of polymer. Recently polyhedral oligomeric silsesquioxane (POSS) is attracted more and more attention [9-11]. POSS is a new-type silicone hybrid material and used for heat-resistant and flame retardant materials and enhanced polymer materials with wider applications. POSS material also has a good anti AO erosion property, but with the increasing of addition amount of POSS, the tensile strength of POSS-polvimide (POSS-PI) is decreased and the mechanical properties will be badly affected. In addition, from the actual application perspective, there are some issues to be resolved and further studies are needed.

Recently, to protect against AO erosion of polymer, the need for functional coatings is rapidly increasing [12-15]. It is known that inorganic oxide coatings are widely used as AO resisting coatings for polymer materials, due to their high stabilities under oxidation environment, such as SiO<sub>2</sub> [16-19], Al<sub>2</sub>O<sub>3</sub> [20-22], and TiO<sub>2</sub> [23-26]. However, preparing inorganic oxide coatings, an urgent problem needed to be solved is the instability of adhesion between the coating and polymer matrix. Because the surface of polymer is hydrophobic, it is technically difficult to deposit oxide based coatings directly on it. Appropriate surface treatment is needed to obtain better wetting behavior. Numerous surface treatments and modification methods have been used to enhance the adhesive ability of coatings to polyimide. These include the uses of ion beam, plasma, and sputtering [27-33]. Most of these methods require high vacuum equipment and the productivity is low, thus there are not economically feasible. Hence, relatively simple and effective hydrophilic surface modification of polyimide become important. Polyimide has a property that it could be dissolved in the alkali solution. Fig. 1 shows the reaction of pyromellitic dianhydride-oxydianiline (PMDA-ODA) polyimide with sodium hydroxide (NaOH). Sodium hydroxide etching can introduce hydroxyl groups on polyimide surface to obtain the desired surface properties without altering the bulk properties on proper conditions [34].

In addition, avoiding or minimizing charging is an important consideration in the design of spacecraft. Energetic charged particles can penetrate a near surface polymer layer, thus depositing a charge onto insulating materials. This can result in electrostatic charge (ESC) buildup, development of large electric fields, and eventually a discharge [35,36]. However, the widely used SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> protective coatings physically block the interaction with AO but can not prevent ESC buildup due to their insulating properties [37]. Stannic oxide is a standard conductive material, combining it with SiO<sub>2</sub> to fabricate a composite coating not only can resist AO attack but also prevent the polymer from surface charge accumulation effectively, and thus eliminate or reduce the electrostatic effect.

In this paper, technological conditions of polyimide substrate modification process by alkali treatment combined with silane coupling agent was investigated. First, polyimide was treated by NaOH solution to remove the surface layer, then  $\gamma$ -aminopropyl-triethoxysilane (APTES) was used as modifier to improve the interfacial adhesion between coating and polyimide matrix. Finally the SnO<sub>2</sub>/SiO<sub>2</sub> sol prepared by sol-gel method can be applied on the surface of polyimide to form a coating. [38–40]. The AO resistant property of the prepared samples were tested in a ground-based simulation system, and the mass loss, morphology change of the samples during AO irradiation were determined.

#### 2. Experimental

#### 2.1. Materials

The polyimide (Kapton HN) substrates used in this study were purchased from Dupont Company with a thickness of 50  $\mu$ m.  $\gamma$ -amino-propyltriethoxysilane (APTES),  $\gamma$ -(Methacryloyloxy) propyltrimethoxysilane (MPS),  $\gamma$ -mercaptopropyltriethoxysilane (MPTES), dimethoxydimethylsilane (DMDMS), methyltrimethoxysilane (MTMS), tetraethylorthosilicate (TEOS), tin(II) chloride dihydrate (SnCl<sub>2</sub>·2H<sub>2</sub>O), ethanol (EtOH), hydrochloric acid (HCl) and sodium hydroxide (NaOH) were purchased from qualified chemical suppliers, and were used as received without further purification.

#### 2.2. Surface treatment

Kapton substrate used in this work was sequentially sonicated in water and ethanol for 10 min each [16,49]. Next, the substrate was treated with different concentration of NaOH solution and then taken out and rinsed with deionized water for at least 3 times. Afterwards, the substrate was placed in a beaker containing silane coupling reagent and EtOH (20:80 v/v) at room temperature for 1 h. Then the substrate was lifted out tardily and cleaned with enough amounts of EtOH, and dried at room temperature.

#### 2.3. Preparation of silica-based sol

Silica-based sol was prepared according to the reported method in the literature [49]: (1) 5 mL TEOS was added to 50 mL EtOH, and the mixture was stirred sufficiently at room temperature. (2) 0.04 mL HCl and 2 mL H<sub>2</sub>O were added to another 50 mL EtOH. (3) The mixed solution was added into the TEOS/EtOH solution by dripping slowly, with the stirring speed of 300 rpm at 30 °C for 1 h. (4) The obtained mixture was aging at 70 °C for 6 h and then stored under room temperature for 7 days. At the same time, stannic oxide sol was prepared as following: A solution of SnCl<sub>2</sub> in the EtOH was prepared by dissolving 0.52 g SnCl<sub>2</sub>:2H<sub>2</sub>O in 50 mL EtOH under vigorous stirring at 80 °C for 2.5 h. Finally, the silica-based sol was mixed with stannic oxide sol at room temperature for 30 min until the solution became transparent [41,42].

Using the same method, the intermediate layer sols were prepared as above, just the TEOS was simply replaced by the 80:20 ratio of TEOS and DMDMS (v/v) as the first layer and the 80:20 ratio of TEOS and MTMS (v/v) as the second layer.

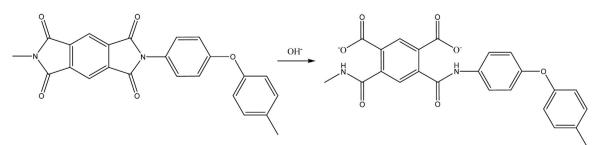


Fig. 1. The reaction of Kapton during NaOH treatment.

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