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Evolution of surface chemistry and morphology of hyperbranched polysiloxane polyimides in simulated atomic oxygen environment

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ABSTRACT

Hyperbranched polysiloxane (HBPSi) polyimide membranes were fabricated by copolymerizing amine-functionalized HBPSi and imide monomers. The atomic oxygen (AO) resistance of the resulting polyimides were investigated in simulated AO environment, based on their evolution of surface chemistry and morphology. Results indicated that a silica passivating layer finally formed on the membrane surfaces and, there was a percolation threshold of HBPSi addition to achieve the most desirable AO resistance. This is explained by the formation of a much denser and more connected silica passivating layer in shorter time on the membrane surface at high HBPSi loading upon AO exposure.

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1. Introduction

In the 1950s, the first man-made satellite was successfully launched into space, witnessing human stepping into a new age using spacecraft to explore the outer space. To speak of, most of the spacecrafts function in low earth orbit (LEO). LEO is an orbit that extends from the earth's surface at sea level to an altitude of about 200–700 km [1,2], and most of it lies within the earth's atmosphere, where plenty of space environmental hazards, such as atomic oxygen (AO), thermal cycle, space radiation, high vacuum, ultraviolet (UV) and vacuum-ultraviolet (VUV) radiation, micro meteor, and space debris are present [1–5]. As a consequence, most organic materials onboard spacecrafts and artificial satellites may be rapidly eroded away due to the damage created by those aggressive environmental factors in LEO, especially the AO [5,6]. In LEO, AO may collide with spacecraft surfaces at a high relative velocity of approximately 7–8 km/s, which imparts the equivalent of 4.5–6 eV of translational energy (TE) to AO, thereby causing erosion called AO attack [7–9].

Polyimides, in particular aromatic polyimides like Kapton[®] H, are extensively used onboard spacecrafts mainly as surface protective materials and lightweight flexible substrates in solar cells, owing to its inherent strong mechanical strength, high-temperature stability and desirable dielectric properties

[3–5,9–15]. However, the presence of AO in LEO makes a considerable threat to polyimides, which can cause significant reduction in physical, mechanical and optical properties of polyimides due to its ultra-strong oxidability [3–5]. As is reported, polyimide coated with TiO₂ or SiO₂ has exhibited superior AO resistance than unprotected one [3,16]. However, inherent and hypervelocity debris-induced defects within the coating will permit AO attack of the underlying polymer, although the SiO₂ coating itself is highly resistant to AO [17]. Therefore, rational design focusing on the molecular modification of polymeric materials, other than coating technology, is the key to manufacture new polyimides with desirable AO resistance.

One effective strategy is to incorporate siloxane units into polyimide molecular chains via copolymerization to fabricate AO-resistant polyimides [4,18,19], for silicon–oxygen bonding energy is about 8 eV, a little higher than AO translational energy, and this bond is intact and impervious upon AO exposure [4,9]. Among a few examples of siloxane-containing polyimides, those fabricated with polyhedral oligomeric silsesquioxane (POSS) have drawn great attention in the past several years, owing to the flexible tailorability of POSS monomers [4,12,20–22]. When exposed to AO, POSS polyimides tend to form a silica passivating layer on the surface, thus preventing the underlying polymer from additional erosion [4,12,20]. As is reported by Minton's group [4,12], POSS polyimides are highly resistant to atomic-oxygen attack, with erosion yields that may be as little as 1% those of the standard Kapton[®] H, making them an attractive AO-resistant material in aerospace.

While these recent advancements lead to improved space survivability of POSS polyimides over pure polyimides, relatively high

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development costs limit their wide applications. For this reason, we have previously fabricated hyperbranched polysiloxane (HBPSi) polyimides, which employ less expensive monomers and remarkably simplify the experimental operations. AO exposure results indicate that HBPSi polyimides exhibit desirable thermal stability and superior AO resistance (though not as high as POSS polyimides) than HBPSi-free polyimide [18]. It is also found that there is a percolation threshold of HBPSi addition to achieve the most desirable AO resistance [18]. Unfortunately, the underlying mechanism for the threshold has not been revealed in that work, which is vital to provide some insights into the erosion mechanism and degradation behavior of HBPSi polyimides in AO environment.

In this work, the mechanism for the threshold is revealed by investigating the evolution of surface chemistry and morphology of the resultant HBPSi polyimides in simulated AO environment and, the relationship between AO resistance and surface morphology is also provided.

2. Experimental

2.1. Materials

Tetraethoxysilane (TEOS), γ -aminopropyltriethoxysilane (APS) and diphenyldimethoxysilane (DPDMS) were purchased from Nanjing Chengong Silicon Co., Ltd. (Nanjing, China) and used as received. 4,4'-Diaminodiphenyl ether (ODA) and pyromellitic dianhydride (PMDA) were provided by Sinopharm Chemical Reagent Co. Ltd. (Beijing, China) and purified by vacuum sublimation prior to use. *N,N*-dimethylacetamide (DMAc) was purchased from Tianjin Fuyu Fine Chemicals Co. Ltd. (Tianjin, China) and purified by distillation under reduced pressure over phosphorus pentoxide and stored over 4 Å molecular sieves prior to use.

2.2. General characterization

The molecular structure parameters of HBPSi were determined on a DAWN EOS size exclusion chromatography/multiangle laser light scattering (SEC/MALLS) instrument equipped with a viscometer (Wyatt Technology). The viscometer provided Mark–Houwink–Sakurada (MHS) relationships. HPLC grade tetrahydrofuran (THF) was used as the eluent with a flow rate of 0.5 mL/min at 25 °C. The molecular weight and its distribution were determined by a SEC/DAWN EOS/Optilab rEX/QELS model. Amino content of HBPSi was acquired by elemental analysis (Heraeus VarioEL-III, Germany, in CHN mode) and acid–base back titration in THF solution. Fourier transform infrared (FT-IR) spectra were determined with a FT-IR spectrometer (Bruker Tensor 27). Thermal gravimetric analyses (TGA) were performed on a TGA/DSC 1 synchronous thermal analyser on membrane samples at a heating rate of 10 °C min⁻¹ under air and nitrogen atmosphere from room temperature to 1000 °C. Dynamic mechanical thermal analysis (DMA) were evaluated by utilizing a Mettler Toledo DMA/SDTA861e instrument. All samples were analyzed using a 5 °C min⁻¹ temperature ramp and a frequency of 1 Hz from room temperature to 450 °C in a tensile mode. The glass transition temperatures (T_g s) of resulting polyimide films are regarded as the peak temperature in the $\tan \delta$ curves. Membrane thickness was measured with a QUJ thickness gauge (Tianjin Yongda, China) with an accuracy of $\pm 1 \mu\text{m}$. Membrane densities were obtained by liquid suspension method with a mixture of toluene and tetrachloromethane at 25 °C. Inherent viscosities (η_{inh}) were obtained on 0.5% (w/v) polyamic acid (PAA) solutions in freshly distilled *N,N*-dimethylacetamide (DMAc) at 25 °C with an Ubbelohde viscometer. Presented η_{inh} values are the average of at least nine individual tests of each sample.

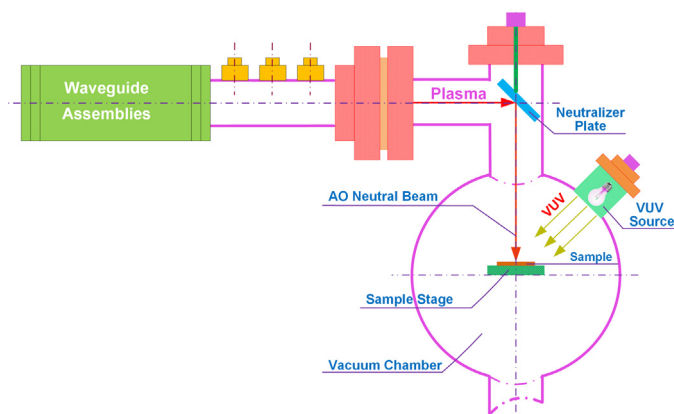


Fig. 1. Cross-section view of low-energy neutral beam facility configured for atomic oxygen (AO) irradiation measurements.

2.3. AO exposure testing

Ground-based AO exposure measurements of HBPSi polyimides were performed on a combined space effects testing facility equipped with neutral AO beam and vacuum ultraviolet ray (VUV) sources, as shown in Fig. 1. The specific operation procedures and details of the system were described in our previous study [18,23]. The AO flux at the sample's position was finally calculated to be approximately $4.89 \times 10^{15} \text{ atom cm}^{-2} \text{ s}^{-1}$ from mass loss of Kapton[®] H. During AO exposure, all samples were handled in vacuum chamber and irradiated to various AO fluences ranging from 0.88 to $3.87 \times 10^{20} \text{ O atoms cm}^{-2}$ or larger for some select samples. Three or more individual tests were carried out for each sample under the same exposure conditions.

2.4. Surface characterization

Surface morphologies of polyimide membranes before and after AO exposure were observed with a Scanning Electron Microscope (SEM, JEOL JSM-6700F). TappingMode[™] atomic force microscope (AFM) images and root-mean-square surface roughness (RMS) of the polyimide membranes before and after AO exposure were obtained using an SPI3800-SPA-400 (Japan, NSK Ltd.) scanning force microscope. The elemental compositions and variations of the electron state of surface elements before and after AO exposure were analyzed on an AXIS Ultra DLD X-ray Photoelectron Spectroscopy (XPS) instrument (Kratos Co., UK). The shifts of binding energy of XPS curves were corrected by assuming that the lowest C 1s component was 284.6 eV for the unexposed polyimide (0 wt% HBPSi) sample. The high resolution XPS spectra of C 1s, Si 2p and O 1s were curve-fitted according to XPS standard spectra databases and theoretical compositions.

2.5. Electrochemical impedance spectroscopy (EIS) tests

The Electrochemical impedance spectroscopy (EIS) of polyimide samples before and after AO exposure were tested in a sandwich structure [platinum (Pt)/polyimide/platinum (Pt)] at room temperature on a TH2817A model LCR meter (Changzhou Tonghui, China) with frequencies ranging from 50 Hz to 100 kHz and measuring voltage of 1 V. The Pt electrode, with an area of 0.5 mm \times 0.5 mm, was sputtering on both sides of polyimide films in a vacuum at 4×10^{-6} Pa. Then, these films were dried at 120 °C under vacuum for 24 h. The thicknesses of polyimide films are 30–34 μm . The impedance spectra were fitted using electrical equivalent circuits with Ec-Lab impedance software to give the equivalent circuits

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