

Erosion of POSS-polyimide films under hypervelocity impact and atomic oxygen: The role of mechanical properties at elevated temperatures

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Abstract

Low Earth orbital debris impacts on the external surfaces of satellites have increased dramatically in recent years. Polyimides are used as the outer layer of thermal control insulation blankets, covering most of the external spacecraft surfaces that are exposed to the space environment. A recently developed material, named polyhedral oligomeric silsesquioxane (POSS)-polyimide, shows significant enhancement in withstanding the space environment. In this work, the combined effect of ground-simulated hypervelocity space debris impacts and atomic oxygen (AO) on the erosion of POSS-containing polyimide films was investigated. During such hypervelocity impacts, elevated temperatures, on the order of hundreds degrees, are formed. A laser-driven flyer system was used to accelerate aluminum flyers to impact velocities of up to 3 km s^{-1} . The impacted films were exposed to an oxygen RF plasma environment, simulating the effect of AO in the low Earth orbit. Impacted polyimide films exposed to AO revealed synergistic erosion effect, while impacted POSS-containing samples showed improved erosion resistance. The increased erosion rate of the impacted polyimide film is explained by formation of residual stresses that affect the oxidation mainly by increasing the diffusivity of oxygen into the subsurface layers. Mechanical properties of the POSS-containing samples performed at $450 \text{ }^\circ\text{C}$ and fractographic examination supports the above hypothesis.

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1. Introduction

Nowadays, numerous satellites are being launched into low Earth orbit (LEO) altitudes, ranging from 200 to 800 km. A large portion of the structure of such satellites comprises high-performance polymeric materials, thanks to their lightweight and unique properties. The degrading environment for polymers in LEO includes atomic oxygen (AO), ultraviolet (UV) and ionizing radiation, ultrahigh vacuum, thermal cycles, and ultrahigh-velocity micrometeoroids and orbital debris [1,2].

The break-up of Fengyun-1C satellite on January 2007 created the most severe orbital debris cloud in history. Data analysis performed by NASA's Orbital Debris Program suggests that the total number of Fengyun-1C orbital fragments – 1 cm and larger – was at least 150,000 [3,4].

The increase in the amount of orbital debris will generate more events of ultrahigh-velocity impacts in LEO. Developing materials that can sustain such impacts and still function under the harsh conditions of the LEO environment is therefore needed.

A promising approach toward the production of LEO survivable polymers is to incorporate polyhedral oligomeric silsesquioxane (POSS) into the polymeric chains [5–7]. Previous work showed that POSS-containing polyimides have significantly lower erosion yields compared to

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polyimide. The nanometer-sized structure of POSS, with its large surface area, has led AO-irradiated samples to form a SiO₂ passivation layer, which protects the underlying polymer from further AO attack [5].

In previous studies, individual and synergistic effects of ultrahigh-velocity impacts and RF plasma (which simulates AO exposure) on polyimide and trisilanophenyl-POSS-polyimide films were studied [8,9]. A synergistic erosion effect was observed after hypervelocity impact and subsequent RF plasma exposure. Due to this effect, an impacted polyimide film eroded one order of magnitude faster than impacted 15 wt.% POSS-containing film.

The accelerated erosion of the polyimide sample is characterized mainly by the formation of RF plasma-induced new macro-holes. A model was suggested to explain this phenomenon, based on the introduction of residual tensile stresses around the impacted area. These residual stresses are formed due to the instantaneous loading and elevated temperatures that are formed at the time of ultrahigh-velocity impact. These residual stresses generate a local increase in the polymer free volume, which facilitates oxygen diffusion into the polymer, thus initiating the process of local high-rate degradation [9–11]. The low erosion rate of the impacted 15 wt.% POSS-containing film was attributed to both mechanical (i.e. no residual tensile stresses) and chemical (i.e. formation of an oxide passivation layer) mechanisms [12].

The purpose of this work is to perform a basic study of the mechanical properties of polyimide films containing various amounts of trisilanophenyl-POSS, both at room temperature and at elevated temperature. Such a study could aid in identifying the mechanism which leads to accelerated erosion of polyimide while POSS-polyimide shows only low erosion under the same conditions of ultrahigh-velocity impacts and exposure to AO.

2. Experimental

2.1. Materials and film preparation

The materials studied in this work were blends of oxydianiline-pyromellitic dianhydride (ODA-PMDA) polyimide (Pyre-M.L. RC-5019 by Industrial Summit Technology, Co.) and trisilanophenyl-POSS (Hybrid Plastics, Inc.). Fig. 1a shows a schematic presentation of the PMDA-ODA polyimide monomer, whereas Fig. 1b shows a schematic view of the trisilanophenyl-POSS molecule. Samples were produced in the form of thin films (25–30 μm thick), with different POSS contents of 0 (polyimide), 5, 10 and 15 wt.%. POSS-polyimide films were produced at a bench-scale process by casting and curing a pre-mixed solution of polyamic acid and POSS in *N*-methyl-pyrrolidone (NMP) solvent. The curing of the pre-mixed solution is based on a process developed by DuPont, Inc. After casting the pre-mixed solution into a glass mold, the samples were heated to 200 °C in air at a heating rate of 4 °C min⁻¹ and held for a period of 30 min. In a second temperature

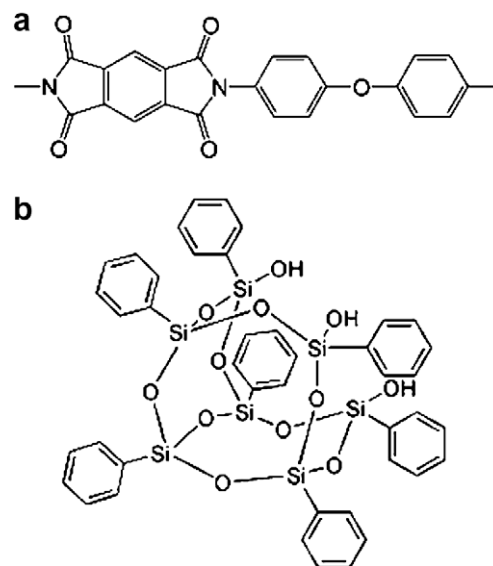


Fig. 1. Schematic presentation of PMDA-ODA polyimide monomer (a) and TriSilanolPhenyl-POSS molecule (b).

cycle, the samples were heated to 350 °C in the presence of pure nitrogen at a heating rate of 2 °C min⁻¹ and held for a period of 60 min. In order to minimize residual stresses, the final stage was slow cooling at a rate of 2 °C min⁻¹ down to room temperature [13]. At room temperature, the POSS-polyimide films were peeled off the glass mold.

2.2. RF plasma AO ground simulation system

A conventional RF plasma reactor (15 W, 13.56 MHz, Model PDC-3XG from Harrick), operating at 500 mTorr of air, was used to simulate the effect of AO in LEO. Samples were located 150 mm downstream from the reactor, in the afterglow region, where they were exposed to a mixture of atomic and molecular oxygen, excited species and vacuum UV (VUV) radiation. Nevertheless, the contribution of ions, VUV and excited species is reduced compared to the RF plasma reactor environment [14,15]. The exposure of samples in the afterglow region is referred to hereafter as AO erosion. Although LEO AO is hyperthermal, the RF plasma environment with thermal AO is considered a useful tool for assessment of materials durability in LEO. The space equivalent AO flux at the sample's position was 2.4×10^{14} O-atoms cm⁻² s⁻¹. AO equivalent fluence measurements were conducted based on 25 μm thick Kapton-HN film (DuPont, Inc.) mass loss, assuming an erosion yield of 3×10^{-24} cm³ O-atom⁻¹ [16]. The erosion yield was determined gravimetrically, using an analytical balance (Model UM3 from Mettler) with an accuracy of ±1 μg [9].

2.3. Laser-driven flyer method

A laser-driven flyer (LDF) method was used for generating simulated space hypervelocity debris [17–19]. The LDF system is based on a high-power titanium:sapphire laser (Thales Laser) with a wavelength of 810 nm, pulse energies

from 250 to 710 mJ, and a pulse length of 300 ps. The laser beam is guided into a vacuum chamber operating at a base pressure of 65 mTorr. Before entering the chamber, the beam passes through a focusing lens. The beam target is a 12 μm thick pure aluminum foil bonded to a BK7 glass using a field-assisted diffusion bonding process. The beam passes through the glass without interacting with it, until it hits the aluminum/glass interface. At the interface, a high-temperature, high-pressure plasma is formed, which then expands perpendicularly to the foil. The expanding plasma induces a series of shock and rarefaction waves, forming a spall [20]. A pressure gradient between the high-pressure plasma on one side of the spall and the vacuum on the other side of the spall causes the spalled layer to accelerate, resulting in an aluminum layer, 1 mm in diameter, flying away at an ultrahigh velocity of up to 3 km s^{-1} . The accelerated aluminum layer is composed of small flyers, 10–100 μm in size, all traveling at ultrahigh velocities [9].

2.4. Tensile tests

Tensile properties of 25–30 μm POSS-polyimide free-standing films were measured in accordance with ASTM D882-88, at a crosshead speed of 20 mm min^{-1} . The specimens used for the tensile tests were die-cut into dog-bone-shaped samples with a width of 3.5 mm in the gauge region. Tensile tests were performed using an initial distance between grips of 43 mm. Dog-bone samples having POSS contents of 0, 5, 10 and 15 wt.% were prepared. The samples were tested using a universal testing machine (Instron model 1026), equipped with a load cell of 2 kg and a temperature control chamber that allows tensile tests in a temperature range from ambient to 450 $^{\circ}\text{C}$.

Tensile tests were performed on five specimens of each POSS-polyimide composition. The highest reading at peak load of each tensile test was reported as the tensile strength. The elongation values at the breaking point were used to obtain the percentage elongation. The resilience of each specimen was calculated by measuring the area under each stress–elongation curve until yielding. Due to the absence of a definite yield point, the yield stress was calculated from the intersect of the tangent of the curve from the origin and the tangent of the curve within the yield region [21], as shown in Fig. 6a. The toughness of each specimen was calculated by measuring the area under each stress–elongation curve up to the point of fracture [22].

2.5. Characterization techniques

The morphology of fracture surfaces (resulting from hypervelocity impacts, AO erosion or tensile tests) was studied using an environmental scanning electron microscope (ESEM; Model Quanta 200 from FEI). This microscope allows characterization of degassing and non-conductive samples, such as polyimide and POSS-polyimide samples. Thermo-gravimetric analysis (TGA) was carried out with a

TA TGA Q500 thermo-gravimetric analyzer, scanning from 25 to 650 $^{\circ}\text{C}$ under a nitrogen environment, at a scanning rate of 20 $^{\circ}\text{C min}^{-1}$.

3. Results and discussion

3.1. The synergistic effect of ultrahigh-velocity impacts and AO exposure

Fig. 2 shows AO erosion of polyimide films containing 5, 10 and 15 wt.% POSS, relative to that of pure polyimide, when subjected to an AO equivalent fluence of 1.7×10^{20} O-atoms cm^{-2} . POSS-containing films reveal increased AO durability as the POSS content is increased. The 15 wt.% POSS sample reached an erosion rate of 74% of the erosion rate of the pure polyimide sample. Fig. 3 demonstrates the synergistic effect of ultrahigh-velocity impacts and AO erosion on the pure polyimide and 15 wt.% POSS samples. The fractures of the pure polyimide (Fig. 3a) and 15 wt.% POSS (Fig. 3c) samples were created using flyers with velocities of 2.4 and 2.2 km s^{-1} , respectively. During such ultrahigh-velocity impacts, elevated temperatures, on the order of hundreds of degrees Celsius and above, are established and are accompanied by shock pressures in the range of tens of GPa [23].

After being impacted by the ultrahigh-velocity flyers, the samples were exposed to an AO equivalent fluence of 2.6×10^{20} O-atoms cm^{-2} . Significant erosion of the pure polyimide film (Fig. 3b) is evident after such exposure. This significant erosion is mainly by formation of new holes, which means total erosion of the polyimide film at certain areas of the sample.

The thickness of the pure polyimide sample is 24 μm . A total erosion of pristine 24 μm thick polyimide requires an AO equivalent fluence of 8.0×10^{20} O-atoms cm^{-2} . Therefore, the total erosion of the polyimide film that occurred

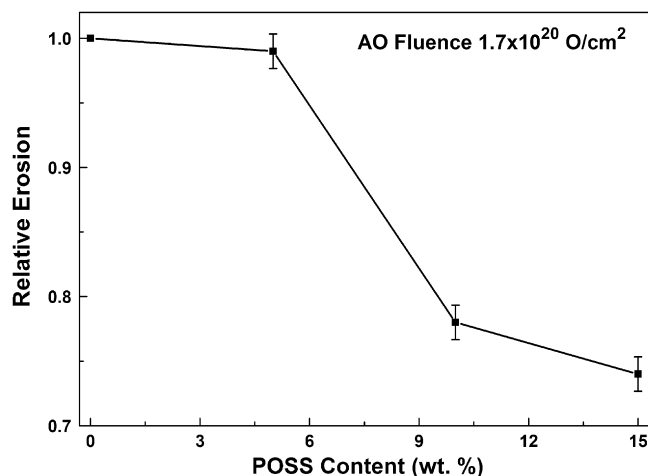


Fig. 2. AO erosion of films containing 5, 10 and 15 wt.% POSS, relative to pure polyimide (0 wt.% POSS) erosion. Samples were subjected to AO equivalent fluence of 1.7×10^{20} O-atoms cm^{-2} .

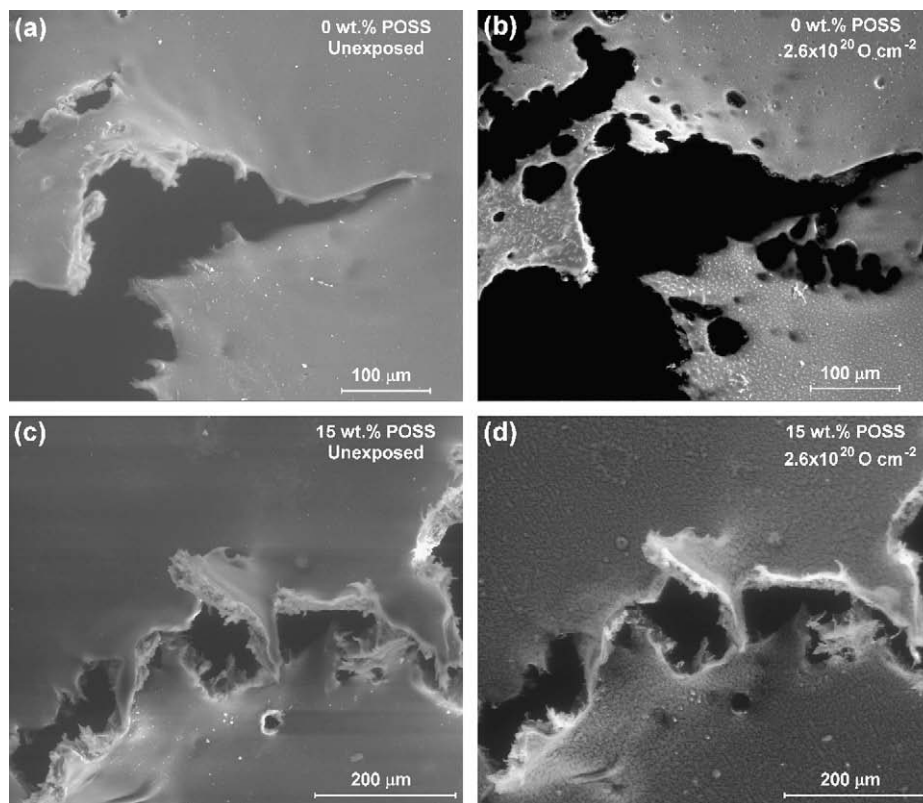


Fig. 3. ESEM images of pure polyimide and 15 wt.% POSS-polyimide films impacted by flyers at velocities of 2.4 (a), and 2.2 km s⁻¹ (c), respectively, and subsequently exposed to AO equivalent fluence of 2.6×10^{20} O-atoms cm⁻² (images (b) and (d), respectively) [12].

at certain areas cannot be explained by AO erosion only after exposure to 2.6×10^{20} O-atoms cm⁻². As stated earlier, the combination of residual stresses and elevated temperatures is a key factor in the local immense erosion of the impacted polyimide [9,10].

The 15 wt.% POSS-containing film, on the other hand, shows better AO erosion resistance (Fig. 3d). No expansion of existing holes or formation of new holes was observed after AO exposure. If ultrahigh-velocity impact-induced residual tensile stress was relevant in the case of the 15 wt.% POSS sample, local erosion of the film at certain areas of the sample was expected, since the addition of 15 wt.% POSS to the polyimide matrix retards the AO erosion rate only by 26%.

The large difference in erosion rate between the two samples is therefore attributed to the impact-induced residual tensile stresses formed in the pure polyimide, while in the case of the 15 wt.% POSS-containing sample a lower level of residual stresses is established under the same conditions of loads and elevated temperatures. Results of impacted 5 and 10 wt.% POSS samples exposed to AO (not shown) support the above impact-induced residual tensile stress erosion mechanism. In the case of the 5 wt.% POSS sample, a smaller number of new holes were formed after erosion compared to the polyimide sample. The 10 wt.% POSS sample revealed behavior similar to that of the 15 wt.% POSS sample, with the formation of only a few new holes due to AO erosion. The impacted POSS-con-

taining samples' durability to AO may be attributed to a basic difference in the mechanical properties of the POSS-containing samples at elevated temperatures, as discussed below.

3.2. Fractographic examination

Prior to examining the durability of pure polyimide and POSS-polyimide samples to elevated temperatures, TGA was performed, as shown in Fig. 4. Both the pure

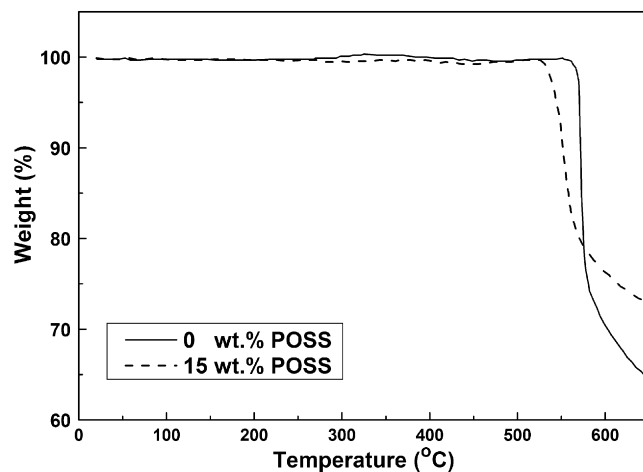


Fig. 4. TGA curves of POSS-polyimide films containing 0 and 15 wt.% POSS.

polyimide and the 15 wt.% POSS-polyimide samples showed no mass loss up to a temperature of 540 °C, which is above the temperature at which the elevated temperature mechanical tests were conducted.

Fig. 5 shows low-magnification ESEM images of the fracture surfaces of 0, 5 and 15 wt.% POSS-polyimide films. The samples were tensile tested at either room temperature (images 5a, 5b and 5c, respectively) or the high temperature of 450 °C (images 5d, 5e and 5f, respectively). A temperature of 450 °C is above the glass transition temperature of the polyimide, which falls within the range of 360–410 °C [24]. The tensile tests were performed at such an elevated temperature in order to simulate the conditions during ultrahigh-velocity impacts. Fig. 5g shows the dog-bone sample and the orientation of the ESEM images. The images shown in Fig. 5a–f are representative images out of the three fractographic images that were acquired for each type of sample (i.e. for each POSS content).

The fractographic images of the samples tested at room temperature (Fig. 5a–c) do not show any significant differences. In all images, the fracture surface appears brittle. This brittle behavior is the consequence of a main crack propagating perpendicular to the direction of loading under unidirectional tension.

At an elevated temperature of 450 °C, the fractographic characteristics of all three samples are different. The fracture surface of the 15 wt.% POSS sample (Fig. 5f) is brittle. The fracture surface of the pure polyimide sample (Fig. 5d) is deformed and rippled. The fracture surface of the 5 wt.% POSS sample (Fig. 5e) is also rippled, but to a lesser degree compared to the pure polyimide sample. The ripples that are characteristic

of the fracture surfaces of the 0- and 5 wt.%-POSS samples are the result of a bidirectional tension field. During the tensile test, the primary tensile stresses are formed in the direction of loading. At the same time, secondary stresses are formed perpendicular to the direction of loading. During fracture, a main crack propagates perpendicular to the direction of loading. After fracture, the two newly created free films spontaneously form non-trivial three-dimensional ripple structures in order to minimize their elastic energy [25]. The secondary residual stresses cause the fracture surface to ripple, as is apparent in Fig. 5d and e. The ripples are indicative of a process of release of residual stresses that were formed during the high-temperature tensile test and the cooling-down process of the samples back to room temperature. In the case of the 15 wt.% POSS sample, no such release process is apparent. The fractography of the 10 wt.% POSS-containing sample (not shown) shows the same characteristics as for the 15 wt.% POSS-containing sample. In the following section, basic differences in mechanical properties of these samples, affecting the above-observed phenomenon, will be discussed.

3.3. Mechanical properties

Typical tensile stress–elongation curves for the investigated POSS-polyimide nanocomposite systems and for the parent polyimide are shown in Fig. 6. As mentioned earlier, tensile tests were performed at either room temperature (Fig. 6a) or 450 °C (Fig. 6b). At room temperature, the polyimide sample (Fig. 6a, curve A) exhibits a gradual transition from elastic to plastic behavior without a well-

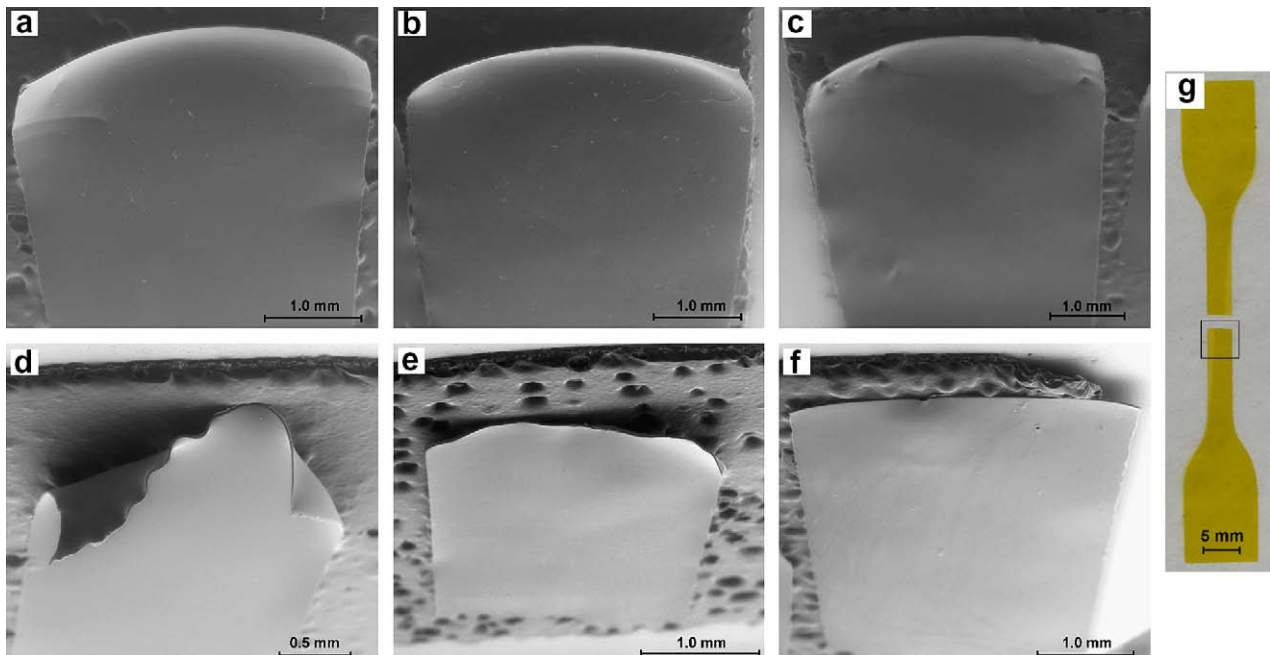


Fig. 5. Low-magnification ESEM images of the fracture surfaces of 0, 5 and 15 wt.% POSS-polyimide films tensile tested at room temperature (a, b and c, respectively), and at 450 °C (d, e and f, respectively). The dog-bone sample used in these tests and the orientation of the ESEM images are also shown (g).

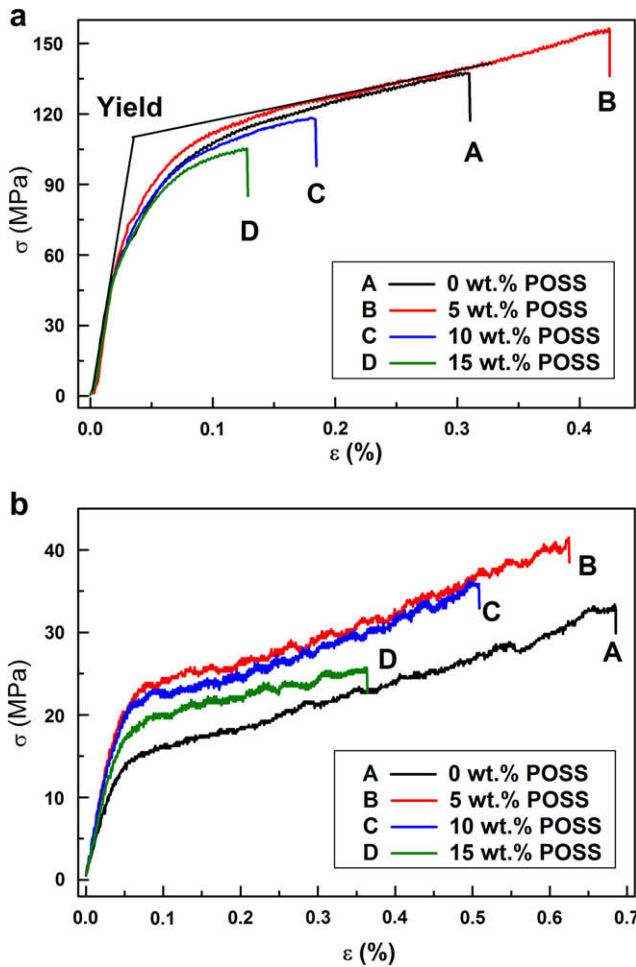


Fig. 6. Typical stress-elongation curves of POSS-polyimide films containing 0, 5, 10 and 15 wt.% POSS, performed at ambient conditions (a) and at an elevated temperature of 450 °C (b).

defined yielding point. A similar behavior is observed at room temperature for the POSS-polyimide samples (Fig. 6a, curves B, C and D). At 450 °C (Fig. 6b, curves

A–D), the transition from elastic to plastic behavior is better defined. Besides yield strength, the tensile test provides data on other mechanical properties, such as tensile strength, elongation, toughness and resilience, as described below.

Fig. 7 shows the tensile strength of the POSS-polyimide samples at ambient conditions and at 450 °C, as a function of POSS content. At both room temperature and 450 °C, the tensile strength of the POSS-polyimide samples as a function of POSS wt.% exhibits a similar trend. The tensile strength of the 5 wt.% POSS-polyimide sample is the highest: 154 MPa at room temperature and 41 MPa at 450 °C. The tensile strength of the polyimide sample is lower than that of the 5 wt.% POSS-polyimide sample, 138 MPa at room temperature and 30 MPa at 450 °C. The tensile strength of the 5, 10 and 15 wt.% POSS-polyimide samples is found to decrease monotonically with increasing POSS content.

Fig. 8 shows the elongation at break of the POSS-polyimide samples at ambient conditions and at an elevated temperature of 450 °C, as a function of POSS content. At room temperature, the elongation at break pattern vs. POSS content follows the same qualitative pattern as the tensile strength. The elongation at break of the 5 wt.% POSS-polyimide sample is the highest, at 42%. The elongation at break of the polyimide sample is lower than that of the 5 wt.% POSS-polyimide sample at 31%. The elongation at break of the 5, 10 and 15 wt.% POSS-polyimide samples is found to decrease monotonically with increasing POSS content. At 450 °C, however, the elongation at break decreases monotonically from pure polyimide to 15 wt.% POSS, with no optimum point at 5 wt.% POSS.

In general, in terms of tensile strength and elongation at break, the addition of 5 wt.% POSS to the polyimide matrix enhances its mechanical properties. The further addition of POSS up to 15 wt.% causes a reduction in its mechanical properties compared to pure polyimide.

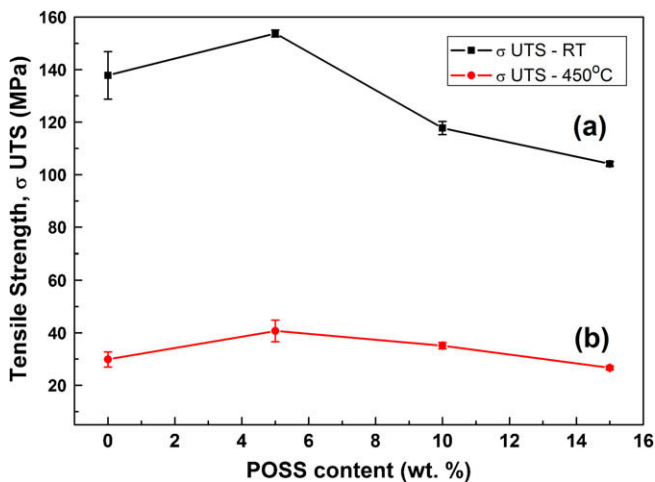


Fig. 7. Tensile strength of the POSS-polyimide samples at ambient conditions (a) and at an elevated temperature of 450 °C (b) as a function of POSS content.

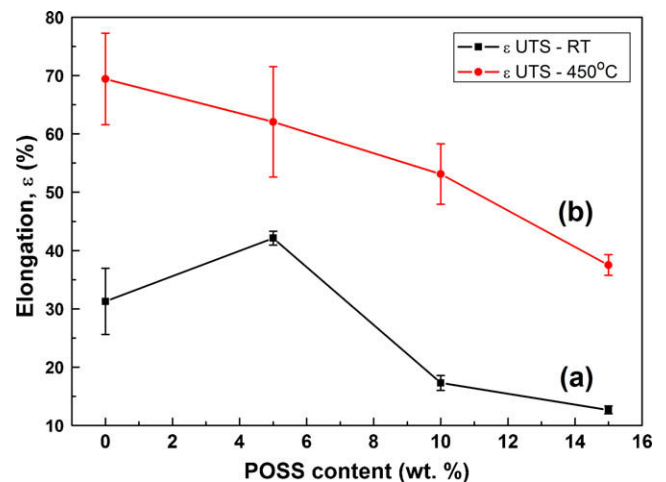


Fig. 8. Elongation at break of the POSS-polyimide samples at ambient conditions (a) and at an elevated temperature of 450 °C (b) as a function of POSS content.

Fig. 9 shows the resilience of the POSS-polyimide samples at ambient conditions and at an elevated temperature of 450 °C, as a function of POSS content. The dependence of the resilience on the POSS content is different for the room temperature and elevated temperature tests. At room temperature, the 5 wt.% POSS-polyimide sample shows the highest resilience, 1.4 MJ m⁻³. In general, at room temperature the addition of 5, 10 and 15 wt.% POSS causes the resilience to decrease monotonically. For the 15 wt.% POSS-polyimide sample, the resilience is 1.1 MJ m⁻³, as compared to 1.3 MJ m⁻³ for polyimide. At 450 °C, however, the opposite trend occurs – addition of POSS causes the resilience to increase in comparison with the pure polyimide. The resilience of the pure polyimide sample is 0.3 MJ m⁻³, while the resilience of the 5, 10 and 15 wt.% POSS increases to 0.4 MJ m⁻³, an increase of 33%.

Fig. 10 shows the toughness of the POSS-polyimide samples at ambient conditions and at an elevated temperature of 450 °C as a function of POSS content. At both room temperature and 450 °C, the 5 wt.% POSS-polyimide sample shows the highest toughness – 50.4 and 18.1 MJ m⁻³, respectively. At both temperatures, the toughness of the polyimide sample is lower – 34.6 and 14.3 MJ m⁻³, respectively. At 450 °C, addition of 10 and 15 wt.% POSS causes the toughness to decrease monotonically, down to 7.9 MJ m⁻³ for the 15 wt.% POSS-polyimide sample. At room temperature, addition of 10 and 15 wt.% causes a dramatic decrease in the sample toughness – down to 15.9 and 9.9 MJ m⁻³, respectively. These values of toughness are almost the same as those obtained at 450 °C.

3.4. Residual stress effect

The impacted and AO-exposed pure polyimide revealed accelerated erosion compared to the 15 wt.% POSS-polyimide sample (Fig. 3). This effect is attributed to formation

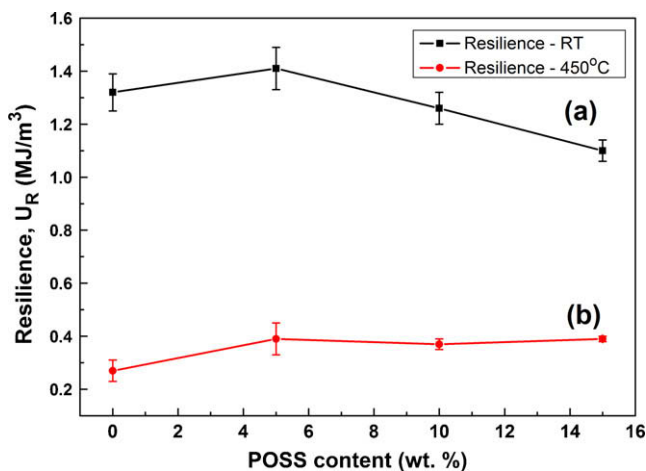


Fig. 9. Resilience of the POSS-polyimide samples at ambient conditions (a) and at an elevated temperature of 450 °C (b) as a function of POSS content.

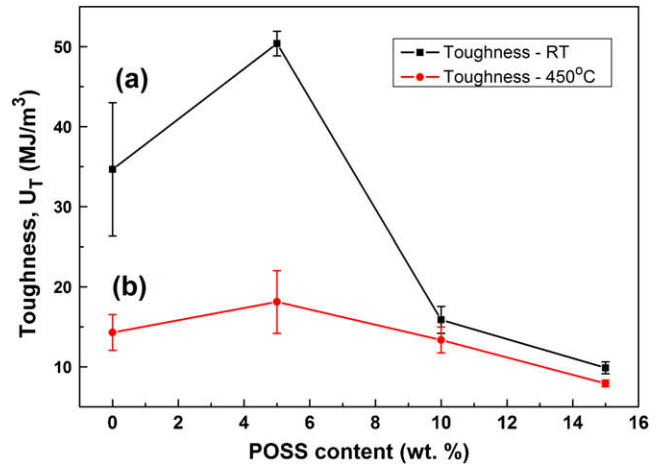


Fig. 10. Toughness of the POSS-polyimide samples at ambient conditions (a) and at an elevated temperature of 450 °C (b) as a function of POSS content.

of residual stresses at elevated temperatures, developed upon ultrahigh-velocity impact. Fractography of the pure polyimide and 15 wt.% POSS-polyimide samples (Fig. 5) indicates the formation of residual stresses only in the pure polyimide sample fractured at elevated temperature. The residual stresses effect is evident by the appearance of ripples at the fracture surface. Mechanical properties reveal the susceptibility of the pure polyimide to the formation of residual stresses. At 450 °C, while the toughness of the 15 wt.% POSS-polyimide is lower than that of the pure polyimide, its resilience is higher. This difference, evident only at an elevated temperature, reflects the ability of the 15 wt.% POSS-polyimide to absorb less energy before reaching its breaking point (lower toughness compared to polyimide) while releasing a higher percentage of this energy in the form of elastic energy (higher resilience compared to polyimide). The pure polyimide, on the other hand, absorbs more energy during tensile tests and releases less energy after breaking, resulting in a larger amount of stored residual stress. Residual stresses generate a local increase in the polymer free volume, which facilitates oxygen diffusion into the polymer, thus initiating the process of local high-rate degradation [11]. The absence of residual stresses in the POSS-containing samples improves their durability to AO attack, in agreement with the results shown in Section 3.2. The lower the residual stresses, the lower the AO erosion.

4. Conclusions

A synergistic effect of ultrahigh-velocity impacts and AO erosion on the degradation of POSS-polyimide samples was demonstrated. After ultrahigh-velocity impact, the samples were exposed to AO. Polyimide film revealed significant erosion, including formation of new through holes. The 15 wt.% POSS-polyimide sample showed better resistance to AO erosion without the formation of any new holes. Residual stres-

ses formed due to elevated temperatures are a key factor in the impacted polyimide local immense erosion. Fractographic examination of fracture surfaces of pure polyimide and 15 wt.% POSS-polyimide samples, tensile tested at room temperature and at 450 °C, supports the above hypothesis. At 450 °C, the fracture surface of the 15 wt.% POSS-polyimide sample is smooth and brittle, while the fracture surface of the pure polyimide sample is rippled. The ripples indicate on residual stresses formed during the elevated temperature tensile test. No residual stress effects are evident in the case of the 15 wt.% POSS-polyimide. Tensile tests showed that addition of 15 wt.% POSS to the polyimide matrix led to a reduction in some of its mechanical properties compared to pure polyimide, both at room temperature and at 450 °C. However, at 450 °C the resilience of the 15 wt.% POSS-polyimide sample is 33% higher compared to polyimide, while its toughness is lower by 45%. This behavior reflects the ability of the 15 wt.% POSS-polyimide sample to absorb less energy (lower toughness) while releasing higher percentage of its absorbed energy after fracture (higher resilience), making it less likely to accumulate residual stresses. Residual stresses generate a local increase in the polymer free volume [11], facilitating oxygen diffusion into the polymer, thus initiating the process of local high-rate degradation.

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