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High-Performance Nanoadhesive Bonding of Space-Durable Polymer and Its Performance Under Space Environments

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The present investigation highlights fabrication of polybenzimidazole by high-performance nanoadhesive and its performance under space environments. High-performance nanoadhesive is prepared by dispersing silicate nanopowder into ultra-high-temperature-resistant epoxy adhesive. The surface of polybenzimidazole is ultrasonically cleaned by acetone and then modified by low-pressure plasma before bonding. Electron spectroscopy for chemical analysis reveals that the polymer surface becomes hydrophilic, resulting in an increase in surface energy. Thermogravimetric analysis studies show that the cohesive properties of nanoadhesive are more stable when heated up to 350°C. The adhesive joint strength of surface-modified polybenzimidazole increases considerably, and there is a further significant increase in joint strength when it is prepared by nanosilicate epoxy adhesive. When the nanoadhesive joint of polybenzimidazole is exposed to the safe-low-power critical-experiment nuclear reactor, there is a considerable increase in joint strength up to a dose of 444 kGy and then a decrease. When the joints are exposed to cryogenic (−196°C) and elevated temperatures (+300°C) for 100 h and thermal-fatigue conditions, the joint could retain 95% of the joint strength. The failure mode of the surface-modified polymer is cohesive within the adhesive.

I. Introduction

FIBER-REINFORCED polymeric composites have many attractive properties that are highly desirable for space applications due to their high strength-to-weight and stiffness-to-weight ratios. Equally well known is their typically poor stability under elevated temperatures and cryogenic environments [1]. In the course of time, polymers with much improved resistance to both high and cryogenic temperatures have been developed and are playing an important role in many advanced engineering applications, especially in space technology [2,3]. However, the use of polymeric materials in primary structures of a spacecraft is limited by the high temperatures of combustion gases, aerodynamic heating, and high-energy radiation [4,5]. Therefore, it is necessary to use such high-temperature-resistant polymeric sheets such as polybenzimidazole (PBI) sheets, which also have excellent cryogenic properties (service-temperature ranges from −260 to +500°C) as well as high stability in the radiation environments and which also show excellent fire resistance. This polymer can therefore be highly useful as a structural composite material in spacecraft [2].

These polymers are often fabricated by adhesives to form polymeric composites. The acceptance of the adhesives as a high-performance engineering material has grown steadily in the last few decades [5,6]. Adhesives contribute highly to structural integrity, ease of manufacturing, enhanced performance, improved safety, and cost and time savings.

The composite can be prepared by employing recently developed ultrahigh-temperature-resistant epoxy adhesive (Duralco 4703, service-temperature ranges from −260 to +370°C), as this adhesive retains its cohesive properties even when exposed to a cryogenic atmosphere. Presently, investigation on polymer-clay nanocomposite has become a very important research area because it is established that thermal and mechanical properties of a polymer-silicate nanocomposite are far superior to those of conventional polymer or polymeric microcomposites [7–9]. Therefore, with this principle, high-performance adhesive can be prepared by dispersing silicate nanopowder to a matrix adhesive and that could open a new horizon on the thermomechanical properties of the adhesive bonding. However, the high-performance polymer, like other polymers, is hydrophobic in nature and, in general, exhibits insufficient adhesive bond strength due to relatively low surface energy. It is therefore necessary to modify the surface of the polymer to make sure that the polymer surface becomes hydrophilic and to improve adhesive bond strength [10].

Based on these considerations, the present investigation is carried out to modify the polymer surface by low-pressure plasma under glow discharge, as it is a technique that may potentially result in better uniformity in the surface modification of the polymers [11–13]. The recent understanding of modification of polymeric composites through high-energy radiation could also be beneficial in the context of a polymeric composite [14,15]. In this regard, the surface of a polybenzimidazole sheet is modified by low-pressure plasma using a 13.56 MHz RF glow discharge for 120 s at 100 W of power [16] using nitrogen as the process gas to essentially increase the surface energy of the polymer, leading to a substantial improvement of its adhesion characteristics. The surface-modified polybenzimidazole sheets are characterized by surface energy measurements and by electron spectroscopy for chemical analysis (ESCA) and joined by the ultra-high-temperature-resistant epoxy adhesive. Other sets of experiments are carried out by dispersing silicate nanopowder into the ultra-high-temperature-resistant epoxy adhesive at a 10% weight ratio with the matrix adhesive followed by modification of the nanoadhesive after curing under high-energy radiation from 6 to 60 h in the pool of safe-low-power critical-experiment (SLOWPOKE-2) nuclear reactor with a dose rate of 37 kGy/h. Lap-shear tensile tests of all these joints are carried out to determine the joint strength. To simulate other spatial conditions, the joints are exposed to cryogenic (−196°C) and high-temperature (+300°C) environments for 100 h. Thermal-fatigue tests of the joints are carried out under 10 cycles by exposing the joint for 2 h under the

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preceding temperatures. Failure modes of the joints are investigated using the scanning electron microscope (SEM) and typical features are analyzed and correlated with the joint strength. Physichothermal characteristics of the basic adhesive and nanoadhesive are carried out by thermogravimetric analysis (TGA).

II. Experimental

A. Materials

In this investigation, PBI sheets for service temperatures ranging from −260 to +500°C, tensile strength of 160 MPa, and density of 1.3 $\mathrm{g/cm^3}$ (as reported by the manufacturer, Boedeker Plastics, Inc.) are used. The ultra-high-temperature-resistant epoxy adhesive (Duralco 4703, Cotronics Corp.) for service temperatures ranging from −260 to +370°C and a cohesive strength of the adhesive of 24 MPa is used to join the polymer sheets. The mixing ratio of resin to hardener, curing temperature, and time of this adhesive are 1:0.22, 25°C, and 24 h, respectively. Unmodified silicate nanopowder of 50 nm particle size (Glassven) is used as dispersing nanoparticles into the adhesive, and in all the cases, degassing of the adhesive is carried out in a vacuum chamber under a pressure of 100 Pa.

Two test liquids, such as deionized water and formamide, with known polar and dispersion components of surface tension are used to evaluate the polar and dispersion components of surface energies of PBI through measurement of their contact angle by the sessile drop method.

B. Low-Pressure Plasma and Surface Energy Measurement

Low-pressure plasma under RF glow discharge is used for surface modification of the PBI. The setup consists of a 220-mm-high and 225-mm-diam closed glass chamber. Through an inlet, air or other desired gases are introduced into the glass chamber. The glass chamber is fitted with a pressure gauge and a vacuum system. The electrodes are capacitively coupled to a RF power generator that operates at a fixed frequency of 13.56 MHz. The PBI sheet, ultrasonically cleaned by acetone, is kept in the chamber. The chamber is evacuated by a rotary pump up to a base pressure of 10^{-2} torr (1.31 Pa), and then nitrogen gas is introduced into the system and a steady pressure of 100 Pa is maintained. At this pressure, the RF power supply is switched on to ignite the RF glow discharge. In this investigation, the surface of the polymer is modified under 120 s of exposure under 100 W of power [16].

Contact angles of deionized water and formamide on the unexposed PBI sheets and those PBI surfaces modified by low-pressure plasma through RF glow discharge are measured by the sessile drop technique. The geometry of the sessile drop is studied at a magnification of 12.8 under an optical stereo-zoom microscope having a crosshead glass graticule fitted with an eyepiece. Vertical and horizontal reference lines of the graticule are positioned at the corner of the drop and rotated to make the vertical line tangent to the drop. The extent of the rotation angle is measured using a goniometer of ±1 deg accuracy. The surface energy is estimated by using the following equation:

$$(1+\cos\theta)\gamma_{LV} = 2(\gamma_S^D \gamma_{LV}^D)^{1/2} + 2(\gamma_S^P \gamma_{LV}^P)^{1/2} \tag{1}$$

on the unmodified PBI sheet and those PBI sheets exposed to low-pressure plasma. First, the contact angle of deionized water θ is measured on the PBI surface. Therefore, in this equation, θ is the measured contact angle of deionized water for which the surface tension of deionized water γ_{LV} and its two components, the polar γ_{LV}^P and the dispersion γ_{LV}^D, are known and the two unknowns are γ_S^P and γ_S^D for the solid PBI surface. Second, the contact angle of formamide, θ, is measured on the PBI surface; consequently, θ is the measured contact angle of formamide for which surface tension of formamide γ_{LV} and its two components such as polar γ_{LV}^P and dispersion γ_{LV}^D are known and the two unknowns are γ_S^P and γ_S^D for the solid PBI surface. Thereby solving these two equations, the unknowns γ_S^P and γ_S^D of the PBI are calculated. Finally, total surface energy γ_S is estimated by adding γ_S^P and γ_S^D as given in Eq. (2):

$$\gamma_S = \gamma_S^D + \gamma_S^P \tag{2}$$

Table 1 Ionizing particles produced by SLOWPOKE-2 at steady-state half-power operation at reactor midheight and 32 cm from the core centerline.

Ionizing particles	Percentage, %	Dose rate, kGy/h
Electrons	87	32.19
Gamma photons	9	3.33
Recoil protons	3	1.11
Fast and thermal neutrons	1	0.37
Total	100	37.00

C. SLOWPOKE-2 Nuclear Reactor

Modification of the adhesive joint of PBI to PBI is carried out through exposure to high-energy radiation for 6, 12, 24, 48, and 60 h in the pool of a SLOWPOKE-2 nuclear reactor. The dose rate at the irradiation site is 37 kGy/h and the reactor is at steady-state half-power operation of 10 kW (thermal) [15]. At this site, the radiation components contributing to this dose rate are a combination of 1% neutron, 3% proton, 9% gamma, and 87% electron radiation [15]. Table 1 listed the dose rate of each spectrum, and the following equation [17] represents the gamma spectrum within the 1.0–7.0 MeV range

$$\mathbf{N\,(E) = 8.0\ e^{-1.10\,E}\ MeV^{-1}} \tag{3}$$

with a ±15% error for the 1.0–4.5 MeV and ±40% error for energies between 4.5 and 7.0 MeV.

The sample holder used for the irradiation is an aluminum sector-shaped box designed to fit snugly against the reactor vessel. The holder could accommodate ten samples, which are all well covered by a waterproof tape to avoid all contact with water in the reactor pool. The sample holder is held in position in the reactor pool by a positioning device called the elevator, designed to position and maintain samples at a repeatable irradiation site at the midreactor plane and against the reactor vessel.

D. Electron Spectroscopy for Chemical Analysis

The ESCA of the polymer surface is carried out on the unmodified polymer as well as on modified polymer surfaces. The ESCA is carried out using a SSLSSX-100 x-ray photoelectron spectrometer at Ecole Polytechnique by using a monochromatized AlK_α x-ray source operated at a power of 200 W. The binding energy reference for the carbons of the C-C and C-H peak is set at 285.0 eV and those of C-O and C-F peaks are at 535.0 and 680.0 eV, respectively. The base pressure in the sample chamber during analysis is 2×10^{-10} Pa. Survey spectra are obtained using pass energy of 58.7 eV, and during the analysis, the electron takeoff angle is set at 45 deg and the acquisition time is 6 min.

E. Adhesive Joint Preparation

The tensile lap-shear test samples are prepared using PBI sheets with dimensions of $125 \cdot 25 \cdot 6\ \mathrm{mm^3}$ by applying the ultra-high-temperature epoxy adhesive at an overlap length of 25 mm. Any excess adhesive present at the interface is expelled by pressing the joint under a load of 10 kg. The tensile lap-shear test is performed according to the American Society for Testing and Materials D 5868–95 standard, using an Instron Universal Testing Machine under a load cell of 10 kN at a test speed of 5 mm/ min at room temperature.

The following three types of the adhesive tensile lap joints are tested:

1) The basic ultra-high-temperature-resistant epoxy adhesive lap joints of PBI to PBI are fabricated and cured at 25°C for 24 h.

2) Silicate nanopowder with a 10% weight ratio is dispersed into the preceding epoxy adhesive with a mixing through the mechanical stirrer and then the adhesive lap joints of PBI to PBI are fabricated and cured at 25°C for 24 h.

3) Silicate nanopowder with a 10% weight ratio is dispersed into the preceding epoxy adhesive with a mixing through mechanical stirrer and then the adhesive lap joints of PBI to PBI are fabricated and cured at 25°C for 24 h. After curing, the joints are irradiated for 6, 12, 24, 48, and 60 h in the SLOWPOKE-2 nuclear reactor with a dose rate of 37 kGy/h when the reactor is at steady-state half-power operation of 10 kW (thermal).

F. Durability Test

From the preceding three types of joints, durability tests of the joints are carried out on the type 3 specimens, which are exposed to the SLOWPOKE-2 nuclear reactor for 6 h. The joints are separately exposed to a cryogenic temperature (−196°C) for 100 h and to an elevated temperature (+300°C) for 100 h; further, thermal-fatigue tests of the joints are carried out under 10 cycles by exposing the joint for 2 h under the preceding temperatures.

G. Microscopic Studies of the Adhesives and Fractography

Microscopic studies of basic epoxy adhesive and silicate nanopowder dispersed epoxy adhesive are carried out under a SEM. The fractured surfaces of the adhesive joints are also examined under a SEM. Typical features of the fractured surfaces are photographed.

III. Results

A. Surface Modification of PBI by Low-Pressure Plasma and Wettability

When the PBI surface is modified by low-pressure plasma under RF glow discharge, a significant increase in polar component of surface energy is observed, as shown in Fig. 1. However, Figure shows that the dispersion component of surface energy remains almost unchanged with this exposure. Therefore, due to the increase in the polar component in surface energy, a considerable increase in total surface energy of PBI is also observed, as shown in Fig. 1.

B. ESCA Studies on the Polymer Surface

The ESCA studies of PBI are carried out for the unexposed surface and those exposed to low-pressure plasma through RF glow discharge under nitrogen atmosphere. Wide-scan spectra of the unexposed PBI shows the C1s peak, the O1s peak, and a significant concentration of the F1s peak, as shown in Fig. 2. However, the PBI surfaces exposed to low-pressure plasma show a significant decrease in the F1s peak and a small increase in the N1s peak, although the C1s peak and the O1s peak are similar, as evident from Fig. 3.

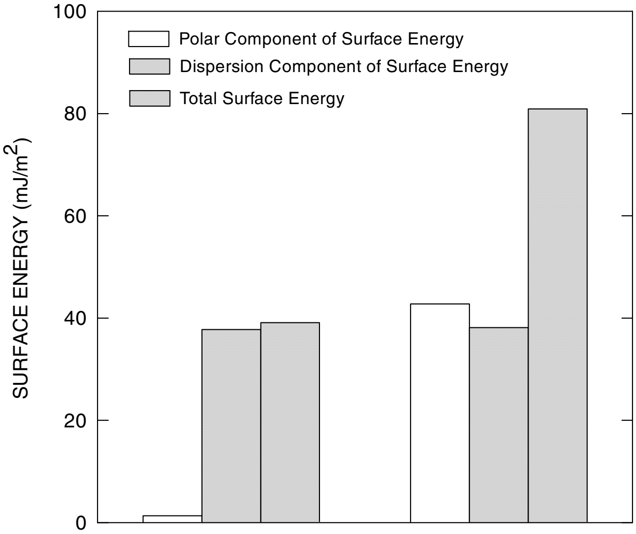

Fig. 1 Polar, dispersion, and total surface energy of polybenzimidazole when the polymer surface is modified by low-pressure plasma.

C. TGA and SEM Studies on Basic Adhesive and Nanoadhesive

The results from the TGA on physicothermal characteristics of the epoxy adhesive and the silicate nanoepoxy adhesive are shown in Figs. 4 and 5, respectively. The peak of the TGA (especially the second peak) represents the phase changing temperature followed by severe weight loss of the adhesive, corresponding to each stage in the whole process. It is found that the degradation of the basic epoxy adhesive is a two-stage process: a relatively short stage with a very small percent of weight loss beginning at about 125°C, followed by about 8% of weight loss up to 350°C, attributed to a degradation of the adhesive over the ranges of temperatures up to 350°C; thereafter, there is a massive degradation of the adhesive. However, for the silicate-epoxy nanoadhesive, degradation of the adhesive occurs in one stage as steady state continues up to 350°C, and there is no sign of degradation of the adhesive up to 350°C. In this case, the percent of weight loss up to 350°C is almost negligible, as clearly evident from Fig. 5. However, thereafter, as in the previous case, there is a massive degradation of the adhesive. A scanning electron micrograph of basic

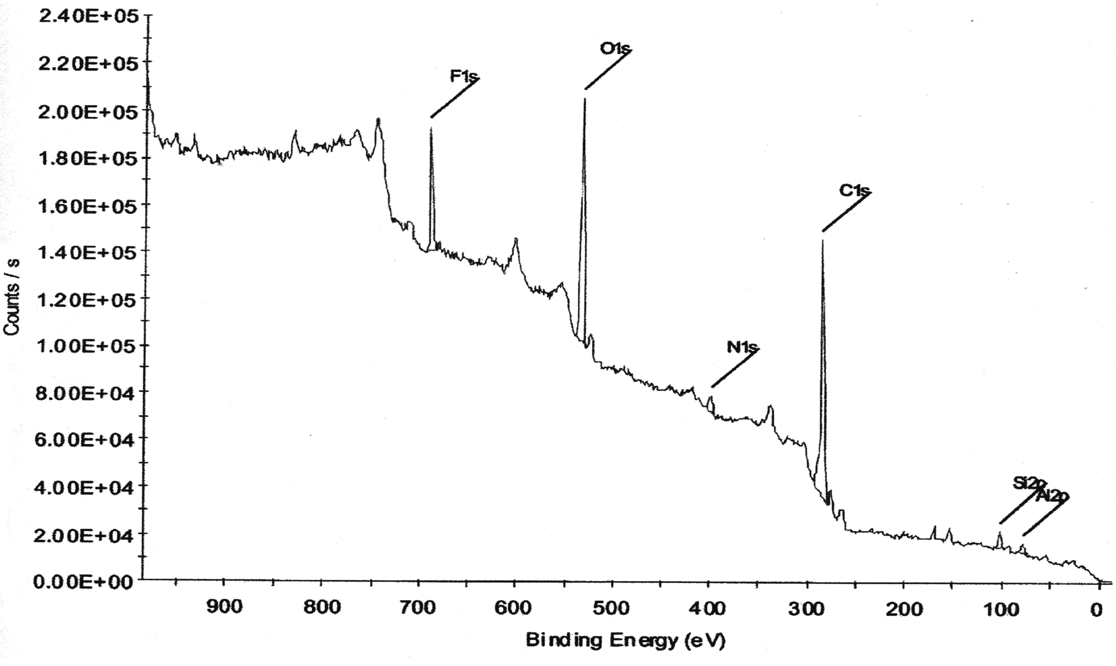

Fig. 2 ESCA wide-scan spectra of the unexposed polybenzimidazole surface.

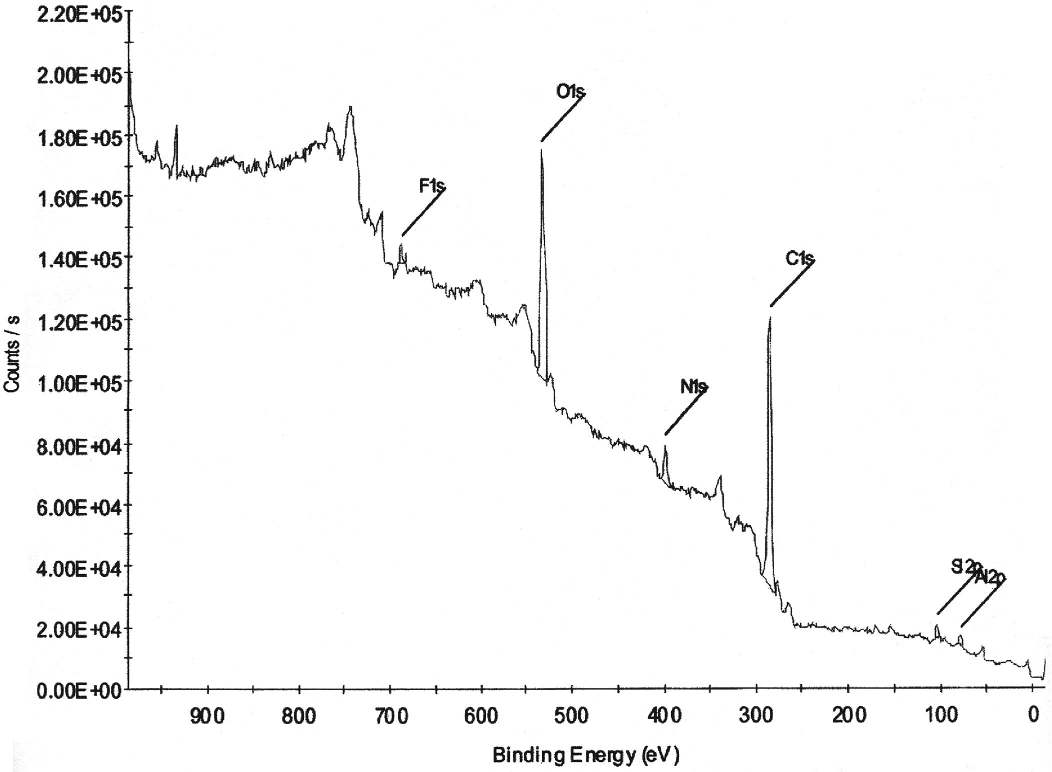

Fig. 3 ESCA wide-scan spectra of the polybenzimidazole surface modified by low-pressure plasma.

and nanosilicate epoxy adhesive is shown in Figs. 6 and 7 at a magnification of 5000. Figure clearly demonstrates a different feature, with the inclusion of silicate nanopowder.

D. Lap-Shear Tensile Properties of the Polymer: Polymer Adhesive Joint

Figure 8 shows the lap-shear tensile properties of the adhesive joints of PBI to PBI when the PBI surface is modified by low-pressure plasma. Figure reveals that the adhesive joint strength of the unmodified PBI to PBI is 1 MPa and increases to 13 MPa when the PBI surface is modified by low-pressure plasma. It is observed that there is a considerable increase in joint strength up to 22 MPa, when the surface-modified PBI joint is prepared by nanosilicate epoxy adhesive and further modification of the adhesive joint under high-energy radiation at the SLOWPOKE-2 nuclear reactor for 6 h results in a significant increase in joint strength up to 30 MPa, as shown in Fig. 9. It is observed that joint strength increases up to 32 MPa with the increasing dose of high-energy radiation up to 480 kGy, as shown in Fig. 10. However,

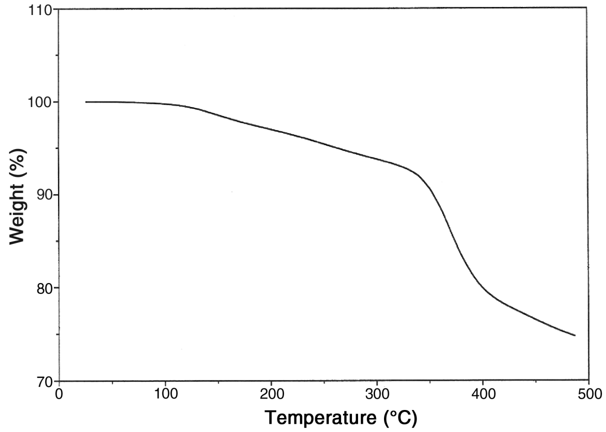

Fig. 4 TGA plot of the basic high-temperature-resistance epoxy adhesive.

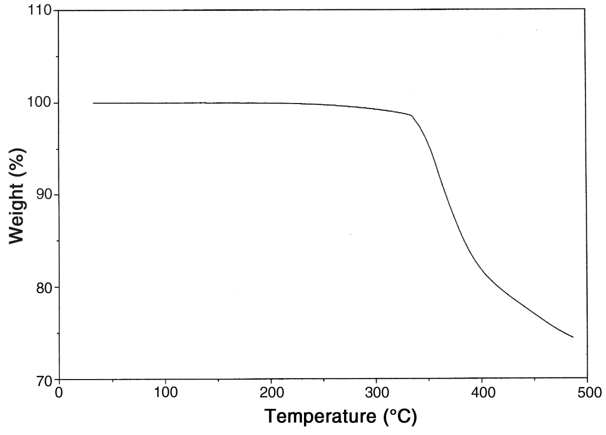

Fig. 5 TGA plot of the high-temperature-resistant epoxy adhesive when silicate nanopowder is dispersed into the adhesive in 10% weight ratio.

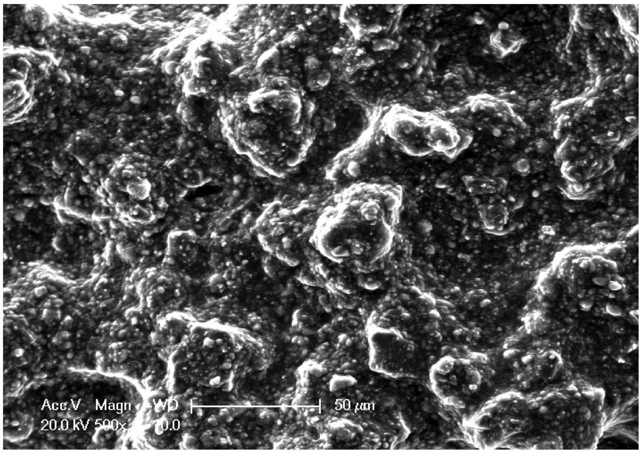

Fig. 6 Scanning electron micrograph showing the basic epoxy adhesive.

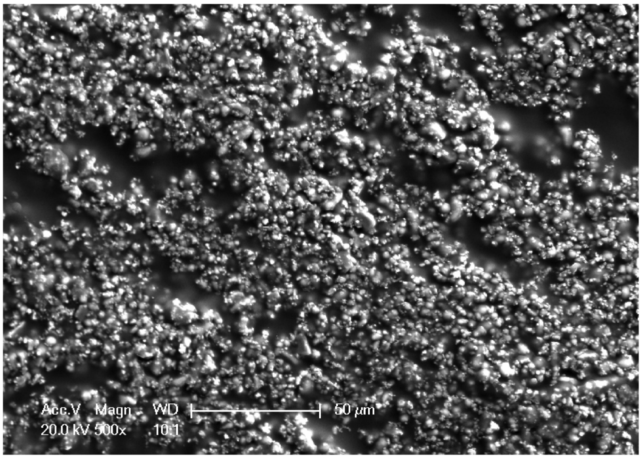

Fig. 7 Scanning electron micrograph showing epoxy adhesive when silicate nanopowder is dispersed into the matrix adhesive at a 10% weight ratio.

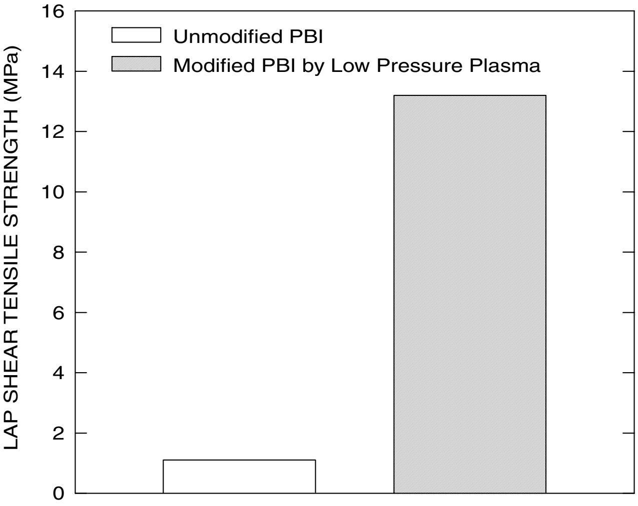

Fig. 8 Lap-shear tensile strength of the adhesive joint of surface-modified polybenzimidazole.

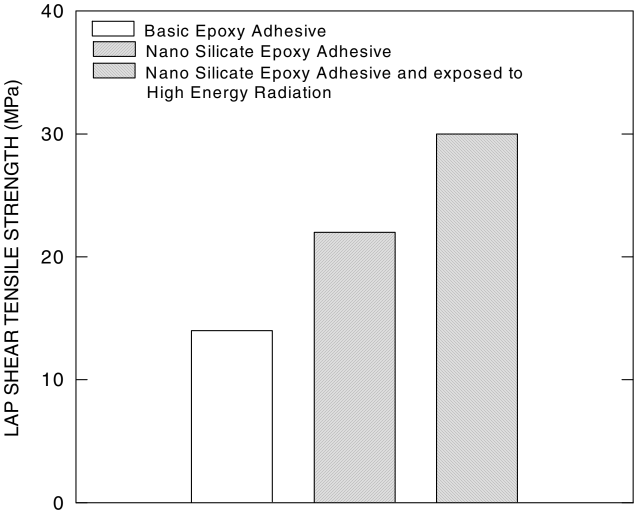

Fig. 9 Lap-shear tensile strength of the joint prepared with basic epoxy adhesive, nanosilicate epoxy adhesive, and nanosilicate epoxy adhesive joint exposed to the SLOWPOKE-2 nuclear reactor for 6 h.

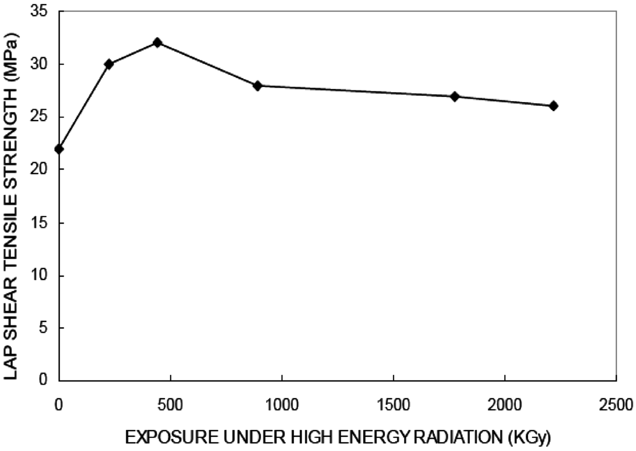

Fig. 10 Lap-shear tensile strength of the joint prepared with nanosilicate epoxy adhesive and the joint exposed to high-energy radiation at the SLOWPOKE-2 nuclear reactor.

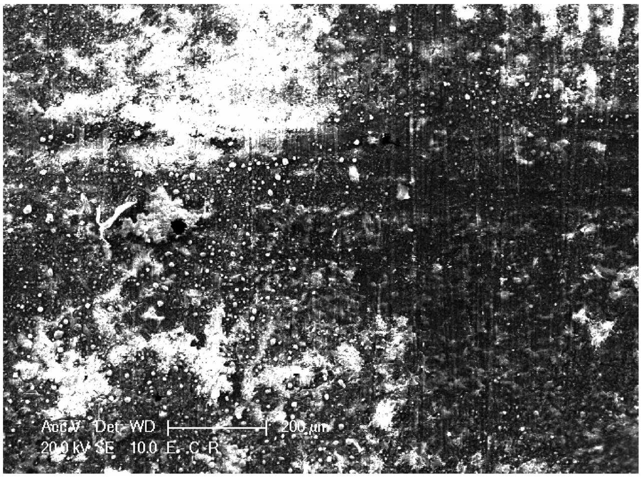

Fig. 11 Scanning electron micrograph showing the fractured surface of the adhesive joint of unmodified polybenzimidazole.

with the higher dose of high-energy radiation, the joint strength decreases, as shown in Fig. 10. The joint with the unmodified PBI failed at the interface between the adhesive and the PBI, as shown in Fig. 11. But in the other cases, the joint fails cohesively within the adhesive, and therefore the features are different, as evident from the SEM images shown in Figs. 12 and 13.

E. Durability of the Polymer Joint Under Space Environments

It is observed that when the best joints (i.e., the type 3 joints) are exposed to a low temperature (−196°C) and an elevated temperature (+300°C) for 100 h and to thermal-fatigue conditions with these temperature variations for under 10 cycles, by exposing the joint for 2 h at each temperature, the joints could retain almost 95% of the joint strength, as shown in Fig. 14.

IV. Discussion

This study examined the performance of the space-durable polymer polybenzimidazole fabricated by high-performance nanoadhesive in the context of its applications to space. However, like any other polymers, the space-durable polymer is also hydrophobic in nature and therefore results in weak adhesive bond strength [2]. Therefore, to ease fabrication, it is necessary to modify the surface of this polymer to become hydrophilic, leading to an increase in surface energy, resulting in high adhesive joint strength when fabricated for structural applications. Exposure to low-pressure plasma results in the formation of polar groups on the polymer surface, leading to an increase of the surface energy of the polymer [1,3,12,13]. The wide-scan spectra of ESCA shows a relatively high nitrogen peak and

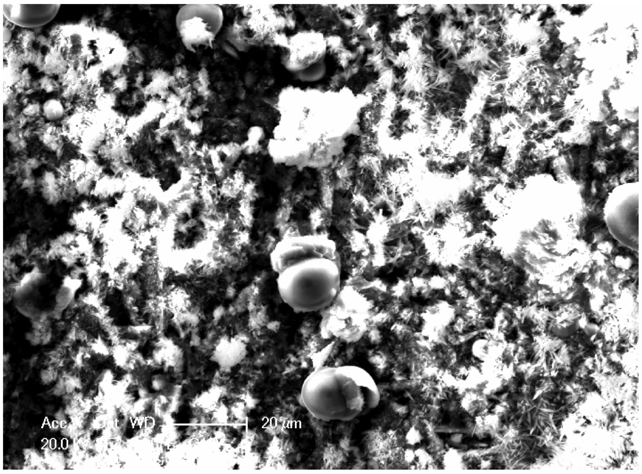

Fig. 12 Scanning electron micrograph showing the fractured surface of the adhesive joint of low-pressure-plasma-exposed polybenzimidazole.

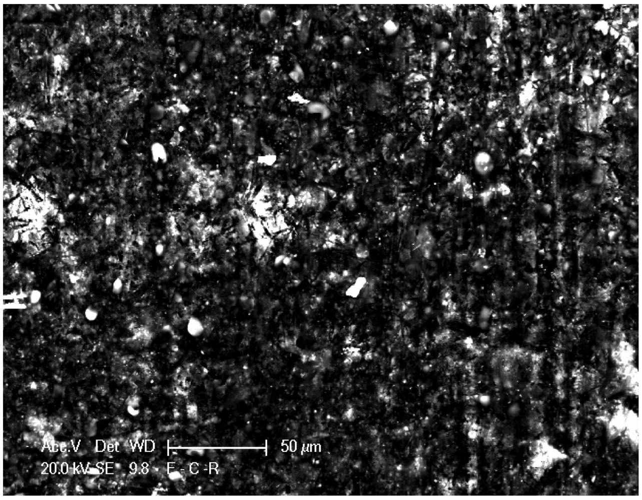

Fig. 13 Scanning electron micrograph showing the fractured surface of nanosilicate adhesive joint of polybenzimidazole when modified by low-pressure plasma and then irradiated in the SLOWPOKE-2 nuclear reactor for 6 h.

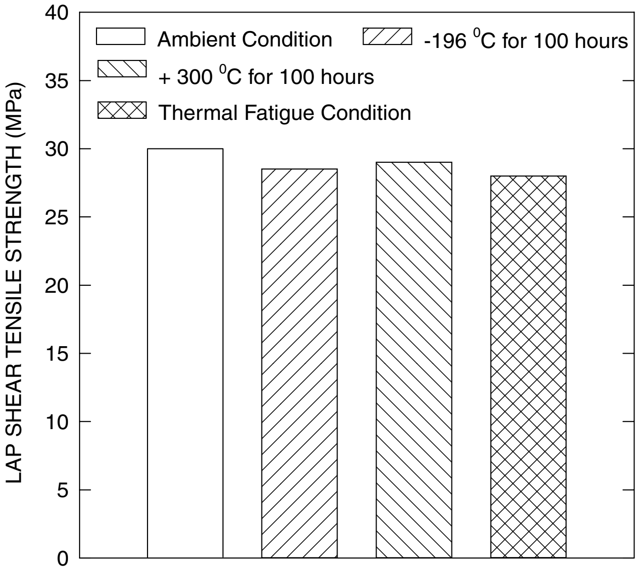

Fig. 14 Durability of the best adhesive joint of polybenzimidazole under ambient, cryogenic, elevated temperature, and thermal-fatigue conditions.

significantly low fluorine peak when the PBI is exposed to low-pressure plasma through RF glow discharge at 100 W for 120 s under nitrogen atmosphere, as evident from Figs. 2 and 3. Therefore, the exposure of the polymer to low-pressure plasma has essentially made the polymer significantly hydrophilic; consequently, there is a significant increase in surface energy. The increase in surface energy of the polymer results in a significant improvement of the adhesion characteristics of the polymer, as evident from Fig. 8.

The ESCA studies related to the surface modification of polymer show that when the polymer surface is exposed to low-pressure plasma under glow discharge, there is a considerable incorporation of polar functional groups on the polymer surfaces [18]. The ESCA spectra of exposed polyolefin [1] clearly reveals the generation of new peaks resulting from plasma oxidation: namely, C2 at 286.5 eV due to C-O groups (hydroxyl, ether, or epoxide), C3 at 288.0 eV due to $C=O$ or O-C-O (carbonyl or double ether), and C3 at 289.4 eV due to $O=C$-OH or $O=C$-O-C. Similarly, the present investigation clearly indicates that the adhesive joint strength of unexposed PBI to PBI is significantly low, resulting in interracial failure from the polymer to adhesive, and increases drastically up to the order of 13 under the surface modification of PBI through low-pressure plasma, resulting in cohesive failure from the adhesive.

It is well documented by various researchers that thermophysical and thermomechanical properties such as melting, crystallization, thermal conductivity, coefficient of thermal expansion, and tensile and breaking strength of epoxy and polyolefins improves considerably due to incorporation of silicate nanopowder into the matrix polymer [7–9]. The present investigation of the nanoadhesive bonding of PBI by dispersing silicate nanopowder to ultra-high-temperature-resistant epoxy adhesive also shows a similar trend, as there is a considerable improvement of thermophysical and thermomechanical properties. This is because, in respect to any adhesive, the thermomechanical properties of silicate are significantly high and therefore essentially influence the overall performance of the adhesive. However, dispersion of silicate nanopowder with different weight ratios (2–20%) into the adhesive could be useful to investigate the more detailed influence of silicate nanopowder on the properties of the adhesive joints.

An earlier investigation carried out on high-energy radiation on a polymeric composite revealed that there is a considerable influence on the mechanical properties on the polymeric composite when exposed to high-energy radiation [19], because the exposure under high-energy radiation promotes cross-linking, essentially affecting the overall behavior and mechanical properties of the polymer. However, with the longer exposure at high-energy radiation, mechanical properties of the adhesive joint degrade because of chain scissions within the adhesive [15]. A similar observation has been reported by Xu et al. [20] and Berejka and Eberle [21] on electron beam irradiation on polyolefins and polymeric composites. They state that with the lower dose rate, the mechanical properties of the polymer increase; however, with the higher dose rate of the electron beam, there is a chain scission of polymer, resulting in the deterioration of mechanical properties of the polymer. Therefore, in this investigation, the joints are essentially exposed to high-energy radiation for those polymer surfaces modified by low-pressure plasma to promote cross-linking within the silicate nanopowder dispersed adhesive, and as surface-modified polymer essentially fails cohesively within the adhesive, it is a clear justification to increase the joint strength even 20 times with respect to unmodified PBI. However, when these polymeric joints are exposed to climatic conditions such as the elevated and cryogenic temperatures related to space conditions, the joints could retain the joint strength of about 95% of the strength of the joint tested under ambient conditions. This could be due to the generation of residual stress along the interface leading to a minor decrease in the joint strength within a reasonable limit. Strauss [2] has also revealed a similar observation and stated that when polybenzimidazole laminates are exposed to elevated temperature there is little degradation in joint strength. Therefore, the adhesive bonding of PBI by high-performance nanosilicate adhesive could be highly useful for space applications as a high-performance polymeric composite.

V. Conclusions

The present investigation has led to the following conclusions:

1) When the PBI sheet is modified by low-pressure plasma under RF glow discharge at 100 W for 120 s using nitrogen as the process gas, the polar component of surface energy and total surface energy of the polymer increases significantly.

2) Studies under ESCA confirm a sharp decrease in fluorine peak and a small increase in nitrogen peak, resulting in an increase in hydrophilic properties of the polymer.

3) The adhesive joint strength of the PBI increases about 13 times when the polymer surface is modified by low-pressure plasma. Failure of tensile lap-shear joints of unexposed PBI joined by adhesive is essentially through the interface of the polymer to adhesive. For the surface-modified polymer, the failure of the joint is essentially cohesive within the adhesive.

4) There is a significant increase in joint strength up to 22 MPa when the low-pressure plasma exposed PBI joint is prepared by nanosilicate epoxy adhesive.

5) When the polymer surface is modified by low-pressure plasma and the nanosilicate epoxy adhesive joints are irradiated for 6 h in the pool of the SLOWPOKE-2 nuclear reactor, the joint strength increases up to 30 MPa. There is a further increase of joint strength up to 32 MPa with the increased exposure of 12 h and then it deteriorates with the exposure from 24 to 60 h.

6) When the joints are exposed to cryogenic and elevated temperature for 100 h, the joints could retain about 95% of their strength in comparison with the joint tested under ambient conditions.

Acknowledgments

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