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Composite Materials Behavior Under Hypervelocity Debris Impact

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The probability that debris will collide with a spacecraft in low Earth orbit is a rising concern of the space community. The most exposed materials are composite materials and polymers that serve as structural materials. Studying the fracture mechanisms of fiber/matrix microcomposites due to hypervelocity impact is the main purpose of this work. A laser-driven flyer-plate technique was used to simulate the debris impact, and microcomposite specimens of Spectra 1000 or Kevlar 29 fibers and epoxy resin as a matrix were prepared. In addition to studying the micromechanical response of hypervelocity impact on pristine microcomposites samples, the response of irradiated microcomposites was also examined. Pristine microcomposites and samples after exposure to gamma radiation were impacted by the aluminum laser-driven flyers at 0.9 and 1.7 km/s. The micromechanical response was correlated to the fibers and fiber/matrix interface properties.

Nomenclature

$F(x)$	=	the probability of failure up to applied stress x
x	=	applied stress
α	=	Weibull distribution scale parameter
β	=	Weibull distribution shape parameter

I. Introduction

OVER the last 50 years, many spacecraft have been launched into orbit for scientific, commercial, environmental, and national security purposes. One outcome of this activity has been the creation of a large population of debris (artificial space objects that serve no useful function) in orbit around the Earth. The space debris is an addition to the naturally occurring meteoroids. This increasing potential hazard has led the space research community to investigate the effects of hypervelocity impact of space debris (at relative speeds of 3–20 km/s) and micrometeoroids (at relative speeds of 30–70 km/s) on the properties of spacecraft structural materials.

The damage caused by debris impacts on spacecraft structural materials includes changes in mechanical properties due to the creation of the impact crater, which is often many times larger in diameter than the particle itself; [1] surface damage to optical components produced by the direct hit; internal spallation in materials resulting from the shock wave produced by the hypervelocity impact [2]; and molecular contamination of nearby surfaces generated by the vaporization of the impacting particle itself, as well as that of the material struck. Many attempts have been made to correlate the debris impact velocity and diameter to the developed crater size [3] and vice versa, to find the debris source, size, and velocity after examining the impact area [4,5]. In particular, Tennyson and Lamontagne [6] examined the effect of hypervelocity impact on polymer matrix composites and presented a correlation between the crater area and the particle impact energy.

Failure of composite materials may also be a progressive result of synergistic effects. If impact occurs, the protective coatings can be cut out, leaving the unprotected composite to degrade due to subsequent exposure to atomic oxygen [7–9] and vacuum UV radiation [10,11]. Synergistic effects might be also due to thermal cycling, which can cause microcracking in the bulk material [12–14], and ionizing radiation, which can alter the fiber/matrix interaction causing a different failure mechanism [15,16].

Upon hypervelocity impact, shock waves propagate through both the projectile and the target material. The pressures associated with these shocks typically exceed the strength of the projectile and the target material [17]. These projectile particles traveling at ultrahigh velocities generate temperatures of many thousands of degrees and pressures of hundreds of gigapascals upon collision with a surface [18,19] during the impact process. At hypervelocity impact the strain rate is as high as about $10^8\ s^{-1}$ [20].

The objective of the present research is to understand the microscopic damage mechanisms of hypervelocity impacted composite materials. Kevlar 29/epoxy and Spectra1000/epoxy microcomposites were used in this study. Both Spectra and Kevlar fibers are used in the outer surfaces of spacecraft [1,17].

This study demonstrates, for the first time, the synergistic effect of radiation and hypervelocity impact of the space environment components on the fracture behavior of microcomposites.

II. Experimental

Microcomposites were prepared in the form of thin (about 100 μm) films using epoxy resin (Araldite LY564, Ciba-Geigy mixed with hardener HY560 at a mixture ratio of 1:0.27, density of 1.2 g/cm^3, tensile strength of 50.0 MPa, and tensile modulus of 2.5–4 GPa [21]) and the following fibers:

1) Kevlar 29 (DuPont) poly(paraphenylene-terephthalamide) fibers have a nominal diameter of 12 μm, density of 1.4 g/cm^3,

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tensile strength of 2.8 GPa, tensile modulus of 65 GPa, with strain to failure of 3.5–4.0% [21].

2) Spectra 1000 ultrahigh-molecular-weight polyethylene (UHMWPE) fibers have a nominal diameter of 27 μm, density of 0.97 g/cm^3, tensile strength of 3.0 GPa, tensile modulus of 175 GPa, with strain to failure of 2.7% [21].

3) Surface-treated Spectra 1000 (referred to as Spectra-RF) fibers were prepared by 2-h exposure of Spectra 1000 in oxygen RF plasma. The samples were irradiated in the afterglow region (100 mm downstream) of an RF-plasma reactor (Litmas Model LB1200, power 450 W) [22].

All samples were cured for 5 h at 80°C in air. Each microcomposite sample contained seven fibers positioned in parallel to each other with an interfiber distance of 500 μm. Fiber tensile strength was measured using an Instron instrument (model 4502, strain rate of 0.1 mm/ min, and load cell f 10 N) before and after exposure to ionizing radiation. The fiber/matrix interface adhesion was characterized by a microbond test [23,24]. The results of tensile and microbond tests were averaged over 30 measurements each.

To simulate hypervelocity impact, a unique laser-driven flyer-plate system was used [2]. The laser-driven flyer-plate system is based on a high-powered titanium–sapphire laser (Thales Laser) with a wavelength of 810 nm, pulse energies from 250 to 710 mJ, and a working pulse length of 300 ps. Aluminum flyers of a maximum size of about 1 mm in diameter and 1 to 3 km/s velocity were generated as a result of the laser beam interaction with the aluminum/glass laminate (12-μm-thick Al foil tenaciously bonded to a BK7 glass). The experimental setup and velocity measurements have been described elsewhere [2,25].

Exposure to ionizing radiation was carried out using a Co^{60} source that produces γ-ray photons with energy in the range of 1.17–1.33 MeV. The total absorbed dose was 100 kGy for all fibers and microcomposites.

The fracture of the microcomposites due to hypervelocity impact was studied using an optical microscope and an environmental scanning electron microscope (ESEM) (model Quanta 200 from FEI), allowing fracture characterization without the need of a conductive coating.

III. Results

A. Ionizing-Radiation Effect on the Tensile Stress of Single Fibers

Tensile-strength measurements were carried out with pristine fibers and with fibers exposed to a 100 kGy dose. The data was then used to build a tensile-strength distribution curve. The tensile-strength distributions of the fibers were fitted to a Weibull model [21,26]:

$$F(x) = 1 - \exp\left[-\left(\frac{x}{\alpha}\right)^{\beta}\right] \tag{1}$$

where $F(x)$ is the probability of failure up to the applied stress x; β is the shape parameter, which characterizes the width of the distribution; α is the scale parameter, which is approximately the mean (or expected) failure stress. The Weibull parameters α and β are presented in Table 1.

The measured tensile strength of pristine Kevlar 29 is in reasonable agreement with previously published results [27,28]. Kevlar showed no change in tensile strength after exposure to a 100 kGy absorbed dose.

Weibull parameters for Spectra fibers are similar to previously published data ($\alpha = 3.3$ GPa and $\beta = 4.15$) [29]. Spectra tensile strength decreased drastically (about 80%) after exposure to a 100 kGy ionizing radiation. This can be explained by the fact that the polyethylene molecule undergoes scissioning when exposed to gamma radiation in the presence of oxygen [30–32]. Similar results were obtained in studies of UHMWPE [33,34].

The tensile strength of Spectra fiber treated with oxygen RF plasma was 50% lower than with pristine fibers, and after exposure to a 100 kGy ionizing radiation, their tensile strength was reduced by an additional 50%. This is most likely due to a combined effect of atomic oxygen and vacuum UV radiation present in RF plasma, which results in surface erosion, increase of surface roughness, and appearance of cracks [22].

B. Fiber/Matrix Interface Adhesion Characterization

To measure the influence of ionizing radiation on the fiber/matrix interfacial adhesion properties, microbond tests were carried out. In this test, a tiny droplet of uncured resin is spread on the fiber and cured in position. Following polymerization of the droplet, the fiber diameter and the droplet length are measured with the aid of an optical microscope to determine the embedment interface area. The specimen is then placed in a tensile testing machine so that one fiber end is gripped and the resin droplet is placed between two knife edges. The fiber is pulled against the knife edges, and the resulting shearing force is registered [23,24]. The calculated interfacial shear strength for the different microcomposites before and after exposure to ionizing radiation is shown in Table 2.

The Kevlar/epoxy interface is characterized by a relatively strong adhesion, compared with Spectra/epoxy, which is reflected by the high value of the interfacial shear strength of 24.6 MPa. This value is comparable with published data [35,36]. There are no significant changes in the interfacial adhesion after exposure to a 100 kGy absorbed dose. Epstein and Shishoo [37] showed that gamma radiation of aramid fibers affected the shear strength and reduced it by a factor of 2. The fact that ionizing radiation did not affect interfacial properties is surprising and has to be further studied.

In general, the adhesion of Spectra fibers to an epoxy matrix is low, because polyethylene fiber is nonpolar on its surface and is expected to have very weak compatibility with other resins unless surface treatment is applied [38]. This expectation is consistent with the obtained low value of the interfacial shear strength for Spectra/epoxy samples of 5.8 MPa. Furthermore, the exposure to ionizing radiation showed no effect on the interfacial adhesion.

The effect of the oxygen RF plasma on the Spectra fibers adhesion properties was the most prominent: the interfacial shear strength was doubled as a result of RF oxygen-plasma treatment (11.8 MPa, compared with 5.8 MPa for pristine fibers). The interfacial adhesion of Spectra-RF fibers after exposure to ionizing radiation was not measurable, because the load needed for fiber break was lower than the load needed for interfacial failure. This is consistent with a significant decrease of the Spectra-RF tensile strength after a 100 kGy absorbed dose (see Table 1).

The properties of the epoxy matrix without fibers were also characterized as a reference. The tensile strength of pristine epoxy was found to be 42 MPa. It slightly increased after exposure to a 100 kGy absorbed dose (48 ± 3 MPa).

C. Hypervelocity Impact

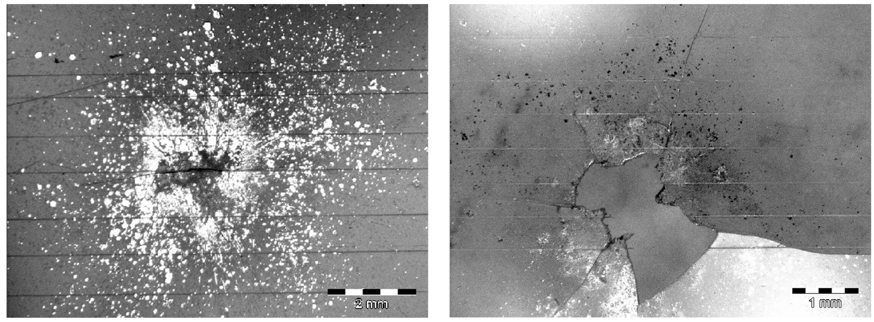
The effect of impact velocity on the extent and nature of damage developed in pristine microcomposites was studied by a comparison

Table 1 Weibull parameters [α (GPa) and β (dimensionless)] for the tested fibers before and after exposure to a 100 kGy absorbed dose

Fiber type	Kevlar 29	Spectra 1000	Spectra-RF
Pristine	$\alpha = 3.1$	$\alpha = 3.9$	$\alpha = 1.5$
	$\beta = 7.6$	$\beta = 6.3$	$\beta = 2.8$
After 100 kGy	$\alpha = 3.1$	$\alpha = 0.8$	$\alpha = 0.7$
	$\beta = 9.9$	$\beta = 4.4$	$\beta = 2.0$

Table 2 Interfacial shear strength, MPa

Composite type	Kevlar 29/epoxy	Spectra 1000/epoxy	Spectra-RF/epoxy
Pristine	24.6 ± 10.6	5.8 ± 2.9	11.8 ± 4.8
After 100 kGy	25.7 ± 11.1	5.2 ± 2.4	Fiber breaks before interface failure



a) b)

Fig. 1 Backside images of Kevlar fibers embedded in epoxy at impact velocity of a) 0.9 km/s and b) 1.7 km/s.

of low (0.9–1.2 km/s) and high (1.6–1.7 km/s) flyer velocities. Three identical impact experiments for each of the microcomposites specimens were carried out at each impact velocity to insure the reproducibility of the observed effects. Representative examples of low- and high-velocity impacts are shown in Fig. 1 for the Kevlar/epoxy microcomposite. The damage patterns observed at 0.9 and 1.7 km/s impacts are different. At a low velocity, only the matrix fails and the fibers stay intact, whereas at a higher velocity, both matrix and fibers are broken.

In general, an increase of impact velocity results in an increase of strain rate, which leads to a higher energy-transfer rate between the flyer and the sample. This reduces the energy absorption by composite strain, whereas other failure modes (e.g., fracture or delamination) absorb more energy [39]. The material does not have sufficient time to absorb the shock wave energy as strain, and a large fraction of this energy is absorbed by new-surface-creation mechanisms such as delamination, debonding, pullout, matrix failure, and fiber break. In all of the specimens, it was noticed that at an impact velocity of about 1 km/s, only the matrix is deformed but the fibers stay intact. However, at a higher impact velocity, the different composites (Kevlar/epoxy, Spectra/epoxy, and Spectra-RF/epoxy) showed different responses to the impact, as will be discussed subsequently.

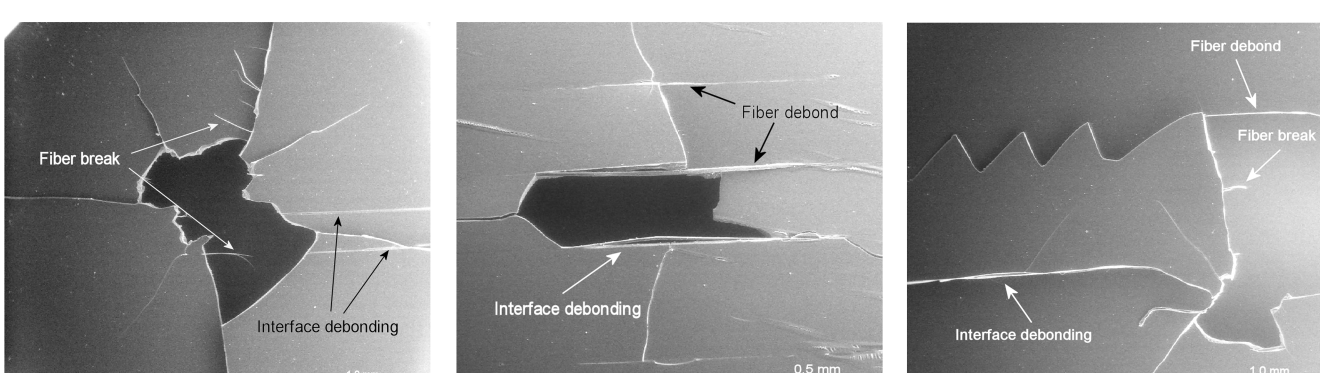
D. Effect of Fiber Nature on the Fracture Mechanism

At low-velocity impacts, similar qualitative results were achieved for all types of the fibers/matrix composites, whereas higher-velocity impacts revealed different fracture patterns for Kevlar/epoxy, Spectra/epoxy, and Spectra-RF/epoxy composites, as shown in Fig. 2.

A scanning electron microscope (SEM) image of the impacted Kevlar/epoxy sample (Fig. 2a) demonstrates that the matrix fails in a brittle manner, as can be seen by the SEM image of the crack surface, and all embedded fibers are broken. Examination of the fiber buckling in ESEM images of impacted samples allows the estimate of the strain ($\varepsilon = \Delta l / l_0$) of the fiber in the debonding region. It was concluded that the fiber was under higher strain (about 10%) than it can stand according to literature data (4%) [21]. There is a primary damaged area, which is reflected in an isotropically spalled matrix, and secondary cracks. Most of the cracks in Kevlar/epoxy are radial cracks; however, some cracks propagate in the direction of the fiber/matrix interface (Fig. 2a). Using image analysis [40], the sheared damaged area (or spalled area) and perimeter were found to be 3 mm^2 and 7.0 mm, respectively (average over all samples).

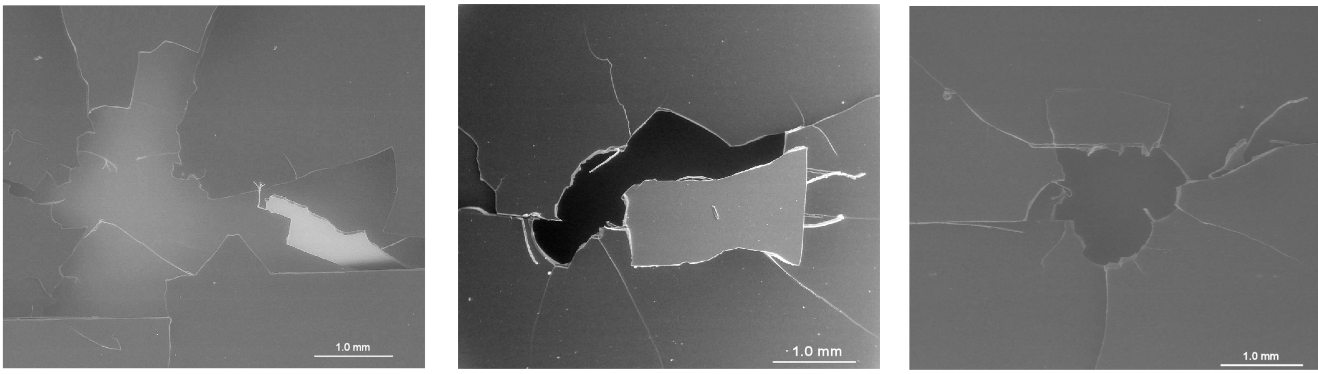
In the case of Spectra/epoxy (Fig. 2b), the damaged area is bordered by the strained *but intact* Spectra fibers. This most important difference between the behavior of Kevlar and Spectra fibers may be explained by the poor adhesion properties of the Spectra/epoxy interface, compared with Kevlar/epoxy, as shown in Sec. III.B (Table 2). The interfacial shear strength of Spectra/epoxy was found to be 4 times lower than the interfacial adhesion of Kevlar/epoxy. As a result of the weak interface, the load does not transfer properly to the fibers, and debonding between the fibers and matrix occurs, causing the fibers to slip out without breaking. Figure 2b clearly shows the fiber detachment and the fiber/matrix interface failure. The strain estimated from ESEM images is about 1.5%, which is low in comparison with the strain imposed on the sample under hypervelocity impact (see the subsequent discussion). The fibers split from the matrix and there are cracks propagating along the interface. In this case, the average damaged area was significantly lower than with Kevlar/epoxy (1.2 mm^2 compared with 3 mm^2), and the perimeter was 4.1 mm, out of which 3.1 mm was the fiber/matrix detachment length.

Figure 2c demonstrates the fracture of the Spectra-RF fibers. In this case, both fibers and matrix experienced severe damage: fibers were broken under impact and the spalled area was found to be 8.1 mm^2, which is the highest of all composites. After RF-plasma treatment, fiber/matrix interface adhesion was improved by 50% compared with the pristine Spectra fibers (Table 2). This improvement caused a better load transfer between the matrix and the fiber, causing the fibers to break, similar to the case of Kevlar/epoxy. A



a) Kevlar/epoxy b) Spectra/epoxy c) Spectra-RF/epoxy

Fig. 2 ESEM images of the backside of a) Kevlar/epoxy, b) Spectra/epoxy, and c) Spectra-RF/epoxy composite specimens at impact velocity of 1.6 km/s.



a) Kevlar/epoxy b) Spectra/epoxy c) Spectra-RF/epoxy

Fig. 3 ESEM images of the backside of a) Kevlar/epoxy, b) Spectra/epoxy, and c) Spectra-RF/epoxy composites exposed to a 100 kGy absorbed dose and further subjected to 1.6 km/s hypervelocity impacts.

crack is observed running in a zigzag pattern between the two fibers: an indication of better adhesion. However, in the case of Spectra-RF, interface failure is also observed and it may be noticed that the damaged area is bordered by the fibers. The perimeter was also measured and was found to be 11.4 mm, out of which 3.3 mm is the fiber/matrix detachment length.

E. Ionizing-Radiation Effect on the Fracture Mechanism

The effect of ionizing radiation on the damage produced by hypervelocity impact at 1.6 km/s is shown in Fig. 3 for Kevlar/epoxy, Spectra/epoxy, and Spectra-RF/epoxy composites.

In the case of Kevlar/epoxy, after exposure to a 100 kGy absorbed dose, the matrix becomes very brittle. As a consequence, for the same impact velocity, the spalled damaged area (7.2 mm^2) is higher than that created in a nonradiated sample (3 mm^2), as shown in Figs. 2a and 3a, respectively. The damaged area is also bordered by a fiber/matrix interface. Using imaging software [40], the total damage perimeter was found to be 18 mm, out of which 2 mm is due to fiber/matrix splitting.

Figure 3b demonstrates the effect of ionizing radiation on the fracture pattern of the Spectra/epoxy composite. It can be seen that due to the ionizing radiation, the fiber becomes more brittle and it breaks. In addition, after exposure to ionizing radiation, there is less delamination in the fiber/matrix interface, indicating better fiber/matrix adhesion. The effect of ionizing radiation on the adhesion will be discussed subsequently. As in the case of Kevlar/epoxy, the crater is much bigger than that observed for pristine samples, due to the fact that the matrix becomes more brittle upon irradiation; the spalled damaged area is 4.8 mm^2, compared with 1.2 mm^2 for pristine fibers.

Figure 3c shows the effect of gamma radiation on the fracture pattern of the Spectra-RF/epoxy composite. It can be observed that the fiber/matrix interface adhesion has increased, because the cracks propagate in the matrix area rather than in the fiber/matrix interface zone. Exposure to a 100 kGy dose resulted in the matrix becoming very brittle for the Kevlar/epoxy, Spectra/epoxy, and Spectra-RF/epoxy. At the same time, the interface adhesion increased, as can be noticed in Table 2, and the fiber tensile strength was reduced by about 50%, to the same level as the Spectra fibers. Consequently, the spalled damaged area is reduced to 3.6 mm^2, in comparison with 8.1 mm^2 found for pristine samples at the same impact velocity.

IV. Discussion

The combined effects of hypervelocity impact and ionizing radiation on composite materials were studied. To understand the combined effects, the samples were divided into two groups; the first set was kept untreated, and the second was exposed to a 100 kGy dose. Following that, all samples were impacted by the aluminum laser-driven flyer at velocities of 0.9–1.6 km/s, simulating hypervelocity debris impacts in space. The analysis and the conclusions are based on postimpact observations of specimen damage. The fracture pattern of different microcomposites was studied and was correlated to the fibers' tensile strength and fiber/matrix interfacial adhesion strength, which were also characterized in this research.

The fracture observed under hypervelocity impact was analyzed according to the effect of the simulated space environments on the strength of 1) fibers, 2) matrix, and 3) interface. The main interest for us is the nature of the fiber/matrix interaction that defines the interface strength. The adhesion between the fiber and the matrix is controlled by many factors, including 1) interlocking of polymer chains, 2) chemical bond formation, 3) polar–polar interaction, and 4) mechanical interference, including frictional force or grabbing force generated by differential thermal shrinkage of the matrix polymer and the fiber during the curing process [41].

A. Kevlar/Epoxy

The tensile strength of pristine Kevlar was found to be within a reasonable agreement with published data (Table 1). Ionizing radiation did not affect the fiber strength. Wu et al. [35] considered the adhesion between Kevlar fibers and most matrices as poor, due to the high crystallinity of Kevlar leading to a chemically inactive surface and to the relatively smooth surface of the fiber. Wu et al. used laminates to measure an interlaminar shear strength of 20.6 MPa, which is comparable with the value obtained with micro-composites in this work (24.6 MPa). As in the case of the tensile strength, no change in interfacial shear strength was observed after ionizing radiation.

The strength measurement results are supported by the fractographic evidence (Figs. 2 and 3). The hypervelocity impact broke the fibers and the matrix (Fig. 2a) altogether, indicating a high interfacial shear strength. Ionizing radiation resulted in formation of a large hole upon impact, which is attributed to radiation embrittlement of the epoxy matrix.

B. Spectra/Epoxy

Significant ionizing-radiation effects were observed for the Spectra/epoxy system. The tensile strength was reduced by a factor of about 5 (Table 1), and the Weibull scale parameter α was reduced from 3.9 to 0.8 GPa. The interfacial shear strength, however, was not affected by the ionizing radiation: 5.8 MPa for pristine specimens, compared with 5.2 MPa for irradiated ones (Table 2). Similar results were reported by Epstein and Shishoo [37], who found no effect of 60 kGy exposure on interlaminar shear strength. This is an indication that the main radiation effect is most likely on the fiber properties and less on the matrix.

Ionizing radiation of Spectra fibers could result in scissioning and/or cross-linking, depending on the environment [31]. For polymeric materials, the primary mechanism of damage occurs via free-radical formation, which is the main result of ionizing treatment. Radical formation in a polymer exposed to radiation in air could lead to oxidation, which could be the main reason for the observed degradation. Spectra fibers being highly stressed, due to the structure of nanofibrils, are susceptible to oxidation, as was shown by Tyler [42]. The oxidation process can be slowed down by either cross-linking or recombination. According to the decreased radical recombination hypothesis, the role of stress (such as residual stress that exists in Spectra nanofibrils) is to increase the separation of the radical fragments produced by photolysis. An increased separation leads to slower radical–radical recombination, which increases the probability of trapping oxygen and thus of degradation of the fibers.

The fractography of the Spectra/epoxy (Fig. 2b) supports the tensile and interfacial strength results. High fiber tensile strength accompanied by relatively low interfacial shear strength caused an impact effect of shearing off only the matrix, with no damage to the fibers. Ionizing radiation, which has reduced the fibers' strength, caused the fibers to break along with the matrix.

C. Spectra-RF/Epoxy

One of the common methods for improving the adhesion between the fiber and the matrix is by treating the fiber surface in RF plasma. The plasma reaction could result in a chemically or physically modified surface that will enhance the interfacial strength. In this study, the Spectra fibers were exposed to oxygen plasma for 2 h, resulting in an oxidized and roughened surface [34]. The effect on the Spectra fiber strength was remarkable and its tensile strength was reduced from 3.9 to 1.5 GPa. The reduction in strength is attributed to the etching of the amorphous domains between the crystalline fibers and the formation of surface microcracks. This is mainly a surface effect, which was less dramatic than the ionizing-radiation effect. As was shown earlier for the Spectra/epoxy composites, ionizing radiation causes severe damage to the fiber, and for the Spectra-RF/epoxy, the strength was reduced to 0.7 GPa. The interfacial shear strength (Table 2), on the other hand, was increased from 5.8 to 11.8 MPa, mainly due to the formation of new surface features, enabling better mechanical and chemical adhesion. Ionizing radiation was shown to reduce the Spectra-RF fiber strength intensely, thus preventing microbond measurements. However, fractography shows (Fig. 2c) that the interface was probably not affected by ionizing radiation, and all the fibers in the impacted area were broken. Similar results were shown by Epstein and Shishoo [37], who reported that corona (plasma) and gamma-radiation-treated Dyneema UHMWPE fibers had clearly better adhesion than untreated Dyneema (Dyneema fibers are similar to Spectra fibers).

V. Conclusions

The fracture modes of Kevlar/epoxy and Spectra/epoxy microcomposites under hypervelocity impact were studied. The study included also the effect of ionizing radiation before impact. The fracture mechanisms were characterized using measurements of fiber/matrix tensile strength and interfacial shear strength, as well as optical and electron microscopy morphology analysis of the fractures.

For all systems, it was found that impacts at velocities of 0.9–1.2 km/s resulted in matrix breakage only, with no damage to the fibers. Higher impact velocities (about 1.6 km/s) were required to damage the fibers. The following conclusions refer to the higher-impact-velocity effects.

1) Kevlar/epoxy microcomposites revealed strong interfacial strength, thus causing damage to the matrix as well as the fiber. Ionizing radiation did not affect this behavior.

2) Spectra/epoxy revealed relatively low interfacial strength with high fiber strength, thus resulting in matrix damage confined by the fibers. Ionizing radiation weakened the fibers, causing them to break along with the matrix. Strengthening of the interface was achieved for the Spectra/epoxy system by oxygen-plasma pretreatment of the fiber surfaces. Plasma-treated Spectra fibers were broken under impact. Ionizing-radiation exposure of plasma-treated Spectra fibers composites before impact did not affect this result.

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