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Dynamic tensile strength of organic fiber-reinforced epoxy micro-composites

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ABSTRACT

Outer surfaces of spacecraft in orbit are exposed to hypervelocity impact originating from micro-meteoroids and space debris. The structural composite materials are integral parts of the spacecraft envelope. We studied the impact response of structural micro-composites containing Kevlar 29, spectra 1000 and oxygen RF (Radio Frequency) plasma surface-treated spectra 1000 fibers of 27-μm diameter, embedded in 100-μm epoxy resin films, in a series of planar impact experiments. The composites were loaded by 50-μm aluminum and polycarbonate impactors having velocities ranging from 400 to 550 m/s. The velocity of the free surface of the composite samples was continuously monitored by VISAR (Velocity Interferometer System for Any Reflector). The dynamic tensile (spall) strength of the micro-composites was calculated on the basis of the recorded free surface velocity profiles. Correlations were found between the spall strength and the separately measured: (i) fiber/matrix interfacial adhesion, (ii) tensile strengths of the fibers, of the matrix and of the micro-composites, and (iii) internal residual stresses. The spall strength of surface-treated spectra fibers micro-composites was found to be lower than that of both pristine spectra fibers micro-composites, and the pure epoxy film. The epoxy film reinforced by Kevlar fibers was found to have the highest spall strength.

1. Introduction

The continuously increasing risk of hypervelocity impact of space debris with space vehicles has led the space research community to investigate the effect of such incidents on the structural materials which make up the outer surfaces of spacecraft. Composite materials having a high strength-to-weight ratio are extensively used as space construction materials. Common impact velocities of space debris on spacecraft in low earth orbits (LEO) range 1–15 km/s, well above the velocity range of about 800–3000 m/s considered in ballistic impact [1]. Typical physical phenomena of a hypervelocity impact comprise of the generation and propagation of shock and release waves accompanied by ablation and spallation of the impacted material, which is the subject of the present study.

Spallation or spall is the process of internal failure caused by collision of two release waves originated from reflection of the compressive shock waves from free surfaces of an impacted body. As result of such collision the tensile stress is generated in the collision point. If the generated stress exceeds the tensile strength of the impacted body the spall occurs. The spall failure is accompanied by creation of two new free surfaces separating the spall plate and the spalled body. In the case of relatively low impact energy the two new surfaces are bounding an internal body cavity (incipient spall). If the impact energy is high enough the spall plate leaves the body giving way to an open spall.

Understanding of the dynamic response of composites and, in particular, of their spall failure is complicated by multiplicity of their internal parameters such as the fiber volume fraction, the fibers spatial arrangement, and the properties of the fiber–matrix interface. Along the fiber direction the response of the composite is determined by the properties of the reinforcing fibers. The response in the direction normal to the plane of the fibers is some derivative of the properties of both the matrix and the fibers and the matrix–fiber interfaces. The latter one is the most problematic of the three factors.

The role of the interface and the bonding quality between the phases was studied by Agbossou [2]. It was found that under dynamic loading conditions the off-axis response of a unidirectional fiber-reinforced composite strongly depends on the interface quality. Spall strength of glass fiber-reinforced epoxy composites were measured by Zaretsky et al. [3]. It was found that three possible deformation modes for the composite resulted in a wide variation of the spall strength. A nucleation and growth model together with a fracture model that were applied by Tokheim et al. [4], provided good estimates for corresponding experimental measurements of spall strength in a Kevlar fibers reinforced epoxy composite. Syam

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et al. [5] examined the fracture mechanism in reinforced plastics. They found that the damage zone consisted of matrix cracking, fractured fibers and debonding between the fibers and the matrix.

In the present study, the spall strengths of different micro-composites with different interface properties have been measured. The micro-composites were based on an epoxy matrix with different embedded fibers. The fibers were made of Kevlar, which is a poly-(paraphenylene terephthalamide) and of spectra, which is an ultra-high-molecular weight polyethylene (UHMWPE). Noteworthy that non-polar and inert character of polyethylene results in a poor interfacial adhesion between the UHMWPE fibers and epoxy matrix causing the increase of the anisotropy of the composite mechanical response [6]. In order to enhance the adhesion the UHMWPE fibers were exposed to oxygen RF (radio frequency) plasma treatment before being embedded into epoxy resin.

The objective of the present work was to study the impact response of micro-composites loaded normal to the fibers direction, with emphasis on the role of the interface in their dynamic strength. The response of both pure epoxy and epoxy micro-composites has been studied in a series of planar impact experiments. The velocity of the free surface of the impacted samples was continuously monitored by VISAR (Velocity Interferometer System for Any Reflector) [7]. The recorded velocity profiles were further analyzed with a special attention to the dynamic tensile failure of the impacted samples and to the role of the impact velocity and of the interface quality in the failure process.

A description of the experimental setup, the samples, and the impact parameters is given in Section 2. The results of the static measurements of composites' properties and of the dynamic measurements, in terms of the velocity profiles recorded from the free surface of the impacted micro-composite samples and their spall strength, are presented and analyzed in Section 3. Finally, the main findings are summarized in Section 4.

2. Materials and methods

Thin films (of about 100-μm thickness) of epoxy resin (Araldite LY564, Ciba-Geigy mixed with hardener HY560) and of different micro-composites based on this epoxy resin were prepared (5 h, 80 °C curing in air). The fibers that were embedded in the epoxy were: 12-μm diameter Kevlar 29 [poly-(paraphenylene terephthalamide)] fibers from Du Pont, 27-μm diameter UHMWPE spectra 1000 fibers (spectra in follows), and surface-treated spectra 1000 fibers (spectra-RF in follows). The spectra fibers were exposed to an oxygen RF plasma (Litmas Model LB1200, 450 W power) for 2 h.

Each micro-composite sample contained 20 parallel fibers. The distance between the aligned fibers is about 100-μm and the fibers are centrally embedded in the matrix. The fibers tensile strength was measured using Instron Model 4502 testing machine (strain rate 0.1 mm/min, load cell 10 N). Epoxy matrix and micro-composite tensile strengths were measured using tensile testing Mini-Mat machine (Rheometric Scientific Model Minimat 2000). The fiber/matrix interfacial adhesion was characterized by a microbond test [8,9]. The results of the fibers tensile strength and the microbond tests were averaged over 30 measurements each.

The micro-composite stress–strain characteristics were analyzed using micro-Raman spectroscope (Renishaw, with 633 nm laser light). The spectrum of the Raman scattering is sensitive to the sample internal stress state: departure of the internal sample stress from zero causes a shift of the Raman peak. High, about 1 μm, spatial resolution of the Raman technique makes it possible to study stress distribution along the composite fibers and, thus, to derive the variation of interfacial shear stress along the fiber–matrix interface [10,11].

Dynamic response of the composites was studied using a 59-mm bore diameter, 4 m long gas gun of the laboratory of dynamic behavior of materials at BGU [12]. Producing the spall (dynamic tensile) failure in a sample of some 100-μm thickness requires the use of impactors of ~50-μm thickness with free rear surface. In order to satisfy this condition the two-stage scheme of the impact-loading of the samples by impactors made of 60-μm thick aluminum foil or of 50-μm thick polycarbonate film was chosen. The hollow projectile equipped with plane-parallel primary impactor of 2-mm thickness and 55-mm diameter struck the 2-mm thickness plane-parallel buffer of 20-mm diameter made of the same, as the primary impactor, material. The secondary (and the actual) impactor (aluminum foil or polycarbonate film) was rolled over the rear surface of the buffer. In order to provide the acoustic contact between the buffer and the secondary impactor the Panametrics silicone grease was used. In the case of aluminum secondary impactor the primary impactor was made of copper. In the experiments with polycarbonate secondary impactor the aluminum primary impactors were employed. The studied samples were fixed (with use of the plane-parallel glass spacers) at the distance of about 0.15 mm from the rear surface of the secondary impactor. Such scheme of the secondary impactor acceleration utilizes the impedance mismatch between the buffer and the secondary impactor materials. The acoustic impedance of copper is equal to $Z_{Cu} = \rho_{Cu}C_{Cu} \approx 36 \times 10^6$ kg m^{-2} s^{-1} while the impedances of aluminum or plastic are equal to $Z_{Al} \approx 16 \times 10^6$ kg m^{-2} s^{-1} and $Z_{Pl} = 3 \times 10^6$ kg m^{-2} s^{-1}, respectively. When the shock wave generated at the buffer by the impact of the primary impactor arrives at the interface between the buffer and the secondary impactor the "light" secondary impactor acquires velocity almost twice as high as the velocity of the primary impactor and separates from the rear buffer surface. Further motion of the secondary impactor is the "free flight" along 0.15-mm path towards the front sample surface. With the aid of such technique the secondary impactors were accelerated up to velocities ranged from 400 to 550 m/s. The impact direction was normal to the plane of the composite sample. Electrically charged pins were used for measuring the velocity of the primary impactor and for controlling its tilt (the impactor–buffer misalignment), which did not exceed 0.5 mrad. The tilts estimated for collision of the secondary impactor with the sample did not exceed 1 mrad. The velocity of the free surface of the impacted samples $w(t)$ was measured by VISAR. Since the reflectivity of the studied materials is insufficient for producing measurable VISAR signal a 0.5-μm gold layers were vapor deposited on the free surfaces of all studied samples. The dynamic response of free-of-fibers epoxy samples was studied in two separate impact experiments.

The spall strength σ_{spall} of the impacted samples was determined on the basis of the velocity pull-back signals Δw of the recorded velocity profiles $w(t)$ using formula [13]

$$\sigma_{spall} = \frac{1}{2}\rho_0 C_b \Delta w, \tag{1}$$

where ρ_0 is the initial sample density, and C_b is the bulk speed of sound in the sample material.

3. Results

The micro-mechanical properties of the fiber, the matrix and the micro-composites were measured as described below (Sections 3.1–3.3).

3.1. Fiber strength

The tensile strength of the fibers was measured (30 measurements each) and for each of the fiber type a tensile strength distribution curve was fitted to a Weibull distribution [14,15]: