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Material Behaviour

Effect of local microstructure on the indentation induced damage of a fiber reinforced composite

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ABSTRACT

Multicycle grid nanoindentation tests, combined with high resolution Scanning Electron Microscopy (SEM) and Scanning Probe Microscopy (SPM) observations, were applied on a commercial Carbon Fiber (CF) reinforced epoxy matrix composite in order to study the induced damage mechanisms with respect to: (a) the orientation of the CFs relative to the surface and (b) the CF packing density. Normal to the surface CFs showed a multiple cracking pattern, those forming 45° showed distinct cracking, while CFs parallel to the surface did not suffer cracking. CF detachment from the epoxy matrix was observed in all cases. Pop-in type discontinuities were observed only in the samples where cracking ensued, as revealed through SEM and SPM observations. The load to induce CF cracking increased with increase of the matrix pocket area. Elastic modulus, hardness and significance of elastic deformation as an indentation energy absorbing mechanism, were reduced right after pop-in.

1. Introduction

The response of a fiber reinforced composite material to applied stress and its deformation mode are mainly governed by the properties of the interface between the matrix and the fibers [1–7]. Specifically, mechanical properties such as strength and fracture toughness are sensitively affected by the strength of the interface, since fiber/matrix debonding is an important mechanism of energy absorption during the failure of a composite material [6,8–14]. The variety of different characterization methods for the mechanical properties of the fiber/matrix interface are indicative of the attention it has gained. They fall into two main categories: (a) those applied to specially designed samples, such as embedded single-fiber tension and compression, the single fiber pull-out test, the microbond test, the fragmentation test and the push-in/push-out test [4–6,8,10,14–17] and (b) those applied to the as-produced composite, i.e. the microdebond method [17–19].

The main advantage of the methods falling into the first category is that they provide information of the active deformation micromechanisms, while the main disadvantages are the demanding sample preparation procedures and the fact that the results are not necessarily characteristic for the same materials in their bulk form [5,6,8,14,16,17]. Analogously, the main advantage of the microdebond method is the fact that the preparation procedure is simpler, the results are more indicative of their bulk counterparts, while the main disadvantage is that it cannot give exact results on the specific micro-mechanisms involved in interface debonding [18,19].

Nanoindentation has been used to characterize interface properties mainly by the push-in/push-out test, by measuring the fiber/matrix interfacial shear strength (IFSS). Based on nanoindentation's superb sensitivity on loading and displacement, a multitude of quantitative results can be obtained from the interface of a composite material [3,12,20]. Recently, nanoindentation has been used to apply grid indentation patterns across the so-called interface region [21–23], i.e. a transition region or third phase between the fiber and matrix phases which has properties that are different from those of the fiber or the matrix [3,4,7,9,11,21]. This method is appropriate to make mapping of hardness and reduced modulus over areas of several hundreds of square microns.

In this work, a commercial Carbon Fiber (CF) reinforced epoxy matrix composite was studied, utilizing grid nanoindentation testing under a multicycle loading-unloading scheme with Berkovich indenter tip geometry. The novelty of this approach stems from the fact that three different orientations of the CFs relative to the surface of bulk composite samples were studied, taking into

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account the local microstructure with a method that is usually applied to assess the debonding energy for specially designed samples (thin slices). This testing method was applied to resolve the load bearing capacity and failure behavior of the composite material from indentation-induced phenomena such as pop-in discontinuity, CF cracking and interface debonding, and shape of load-displacement curves. Experimental parameters studied were the following: (a) CF orientation with respect to the surface and (b) local microstructure, i.e. CF packing density or size of the matrix pocket. Supplementary monocycle grid nanoindentations were applied to resolve effects originating exclusively from repetitive loading during multicycle schemes.

The application of the above testing procedure utilizes the high sensitivity of nanoindentation on loading and displacement on bulk composite samples that are more easily prepared compared to the samples for the IFSS method.

2. Materials and methods

2.1. Sample preparation

The material studied was a commercial CF reinforced polymer containing T700s fibers of approximately 7 μm in diameter, with a CF volume fraction of 60% (company: Toray Carbon Fibers America Inc.). For the nanoindentation test, 1 mm thick slices were cut from the composite plate with a CNC waterjet system (Hellenic hydrocut 4020–50), in order to avoid heating. Three different types of samples were produced, differentiated by the angle between the CFs and the normal to the sample surface, namely 0°, 45° and 90°. The cut slices were mounted in epoxy forming a puck to facilitate handling during subsequent polishing. The surfaces of all samples were mechanically wet polished with SiC grinding papers of 1000, 1200, 2000 and 4000 grit, and finished with polishing Al_2O_3 pastes of 3 and 1 μm, utilizing a Struers LaboPol-2 grinding, lapping and polishing machine.

2.2. Morphological characterization

Microstructure and surface morphology, before and after indentation tests, were studied by optical microscopy (OM), utilizing a Zeiss AXIO Imager. A2m metallographic microscope under incident illumination, and Scanning Electron Microscopy (SEM), utilizing a Nova NanoSem 230 instrument (FEI Company) with tungsten filament, operating at 25 kV and SPM (see Nanoindentation part).

The varying parameters of the grid nanoindentation were the orientation of the CFs with respect to the surface vector (Fig. 1a) and the type of microstructure, which is characterized by the local volume fraction of the CFs or the area of the matrix pocket. Two types of microstructures were qualitatively recognized (Fig. 1b): regions with densely packed CFs (type A) and regions with sparsely packed CFs (type B). Fig. 1c depicts an optical micrograph under low magnification of the 0° sample, where the two types of microstructures are indicated.

2.3. Nanoindentation

A Hysitron Tribolab® Instrument, equipped with a standard three-sided pyramidal Berkovich tip, was used for the nanomechanical tests, at a load range from 1 μN to 35 mN with simultaneous recording of the displacement. Load resolution (1 nN) and displacement resolution (0.04 nm) in the vertical direction were high enough to allow detailed characterization of nanomechanical properties. The test instrument used in this work supports the application of Scanning Probe Microscopy (SPM), in which a sharp probe monitors the sample surface with a three-axis piezo system and produces three dimensional representations of the sample surface. Nanomechanical testing was applied in a clean area environment with 45% humidity and 23 °C ambient temperature. A standard fused silica sample was used to calibrate the area function of the Berkovich indenter tip before the measurements.

Nanoindentations were mainly performed using a multicycle load-unload scheme (Fig. 2) under force control. This scheme was

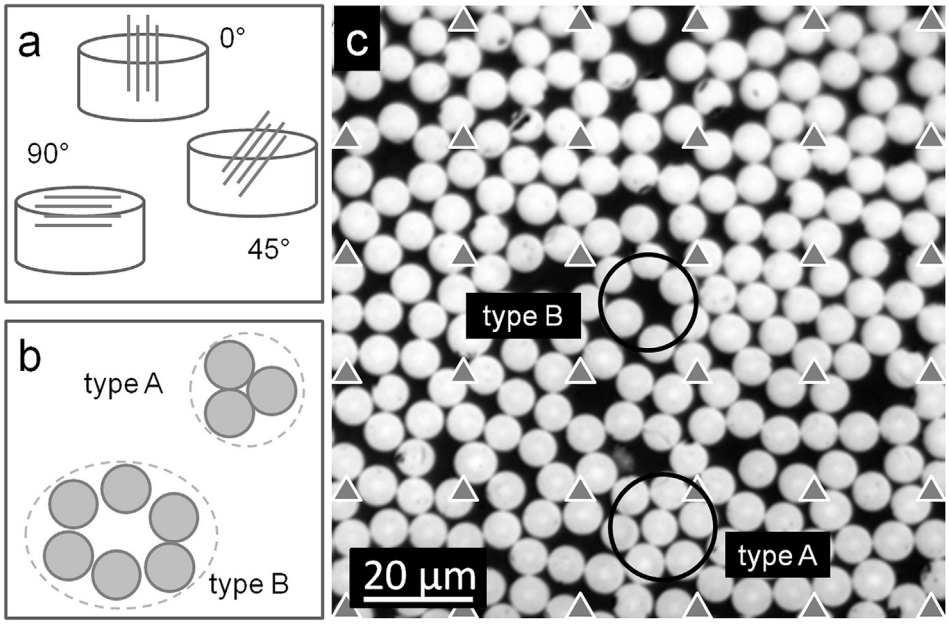

Fig. 1. Schematic diagrams and respective sample nomenclature of (a) the orientation of the fibers with respect to the surface and (b) the type of microstructure, with respect to the density of fibers. (c) Optical micrograph of the surface of the 0° sample, where the two types of microstructure are indicated with black circles.