Application Note #1519

In-Situ Fiber Push-Out Tests with the Hysitron PI 88 SEM PicoIndenter

Carbon fiber reinforced polymer composites (CFRPs) possess outstanding mechanical properties including high stiffness, high strength-to-weight ratios, and the ability to withstand dynamic loading in service without catastrophic failure. As a result, CFRPs excel as structural components in numerous applications, especially in the aerospace industry where they are quickly replacing heavier metallic components. The mechanical strength of a composite is a combination of the mechanical properties of the constituent fiber and matrix materials, as well as the strength of the interface between them. To measure the interfacial strength and sliding characteristics of individual fibers in reinforced composite materials, a fiber push-out test can be performed. In this work, the Hysitron® PI 88 SEM PicoIndenter® equipped with the 800°C heating option is used to characterize the interfacial strength between individual carbon fibers and the surrounding polyamide matrix, as a function of temperature.

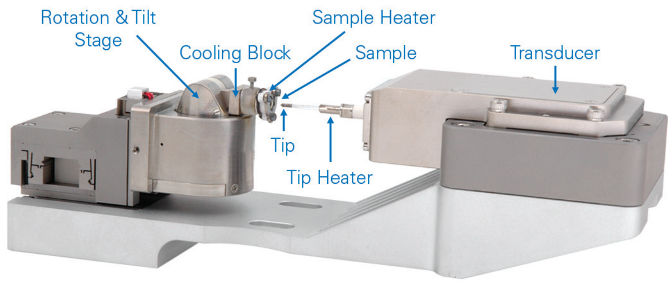

Figure 1. Hysitron PI 88 SEM PicoIndenter with 800°C module.

In-Situ Fiber Push-Out at Elevated Temperatures

Fiber push-out experiments were conducted with a Hysitron PI 88 SEM PicoIndenter equipped with a 5 µm diameter diamond flat punch on a carbon-fiber reinforced polymer suspended on a grooved steel substrate. Using displacement-controlled feedback, the fibers were loaded until complete interfacial failure was observed and the fibers began to slide through the matrix. The tests were conducted at room temperature as well as at several elevated temperatures up to 300°C. Heating was achieved through closed-loop resistive heating of both the probe and sample. The in-situ nature of the testing allowed for precise alignment of the tip on the fiber as well as direct, real-time observation of the failure progression and push-out mechanism.

Sample Preparation

A thin sample of 300 μm thickness and 5 mm x 5 mm cross-section was prepared from a bulk sample of fiber-reinforced composite. The sample and a polished substrate were clamped together and mounted on a heater that was attached to the SEM PicoIndenter, as shown in Figure 2a. The substrate was grooved in order to provide space for fiber push-out. The Hysitron PI 88 stage was used to align and position the tip above the fibers, as shown in Figure 2b.

Interfacial Failure

Two load-displacement (P-h) curves, shown in Figure 3, display different stiffness regimes during loading. The videos captured during the tests help to understand the progression of interfacial failure. The low initial stiffness of the P-h curves is a result of the thin sample bending down to meet the grooved support. Once the sample makes solid contact with the substrate, the applied load transfers entirely to the fiber and the stiffness of the curve increases. At this stage, the applied compressive load introduces shear stress at the interface with a maximum value at the surface of the sample. At a critical value of interfacial shear stress, interfacial debonding initiates and load drops. The failure load is defined as the maximum sustained load before observable debonding. Depending on the debonding mechanism and temperature, the P-h curves exhibited sudden, gradual, or stepped load drops. Sudden load drops are associated with catastrophic failure of the interface whereas gradual and multi-step load drops are related to slow propagation of crack through the interface. In some cases, surrounding interfaces also failed. An image of complete fiber push-out is shown in Figure 2c.

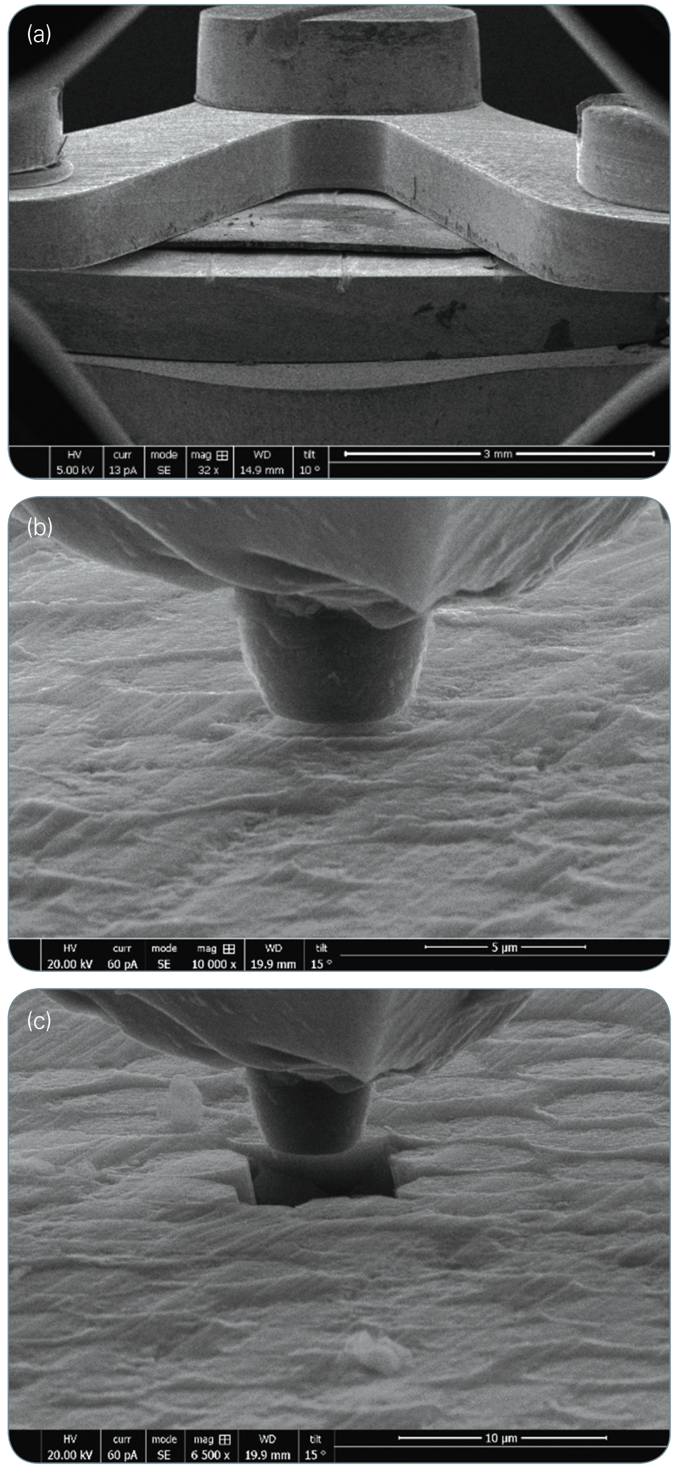

Figure 2. (a) Low-magnification SEM image of a sample clamped to a substrate; (b) pre- and (c) post-images of a fiber push-out test.

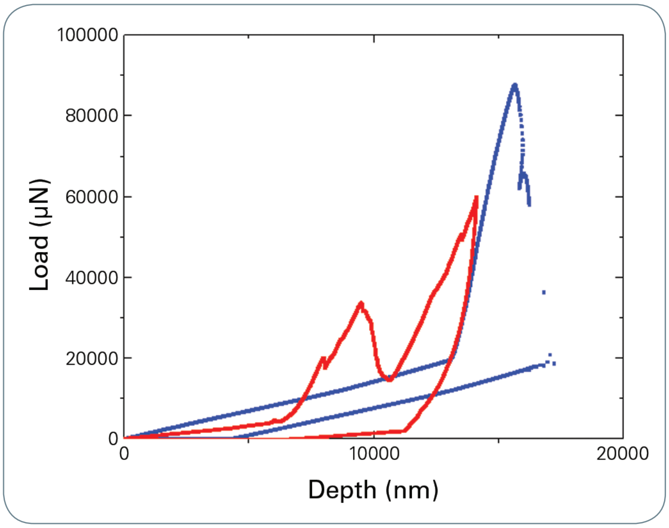

Figure 3. Load vs displacement curves of fiber push-out at room temperature (blue) and 316°C (red).

Role of Temperature

The tests were conducted at a temperature range from room temperature to 316°C. A reduction of the failure load was observed at elevated temperatures as the interface becomes weaker and the interfacial shear strength reduces, as shown in Figure 4. Moreover, as the matrix softened at elevated temperatures, it was more easily pulled away from the surrounding fibers as the loaded fiber was pushed-out. After the tests, the sample was removed from the Hysitron PI 88, to observe the back-side of the sample, push-out fibers, and surrounding matrix. Figure 5 shows an image of the push-out fibers.

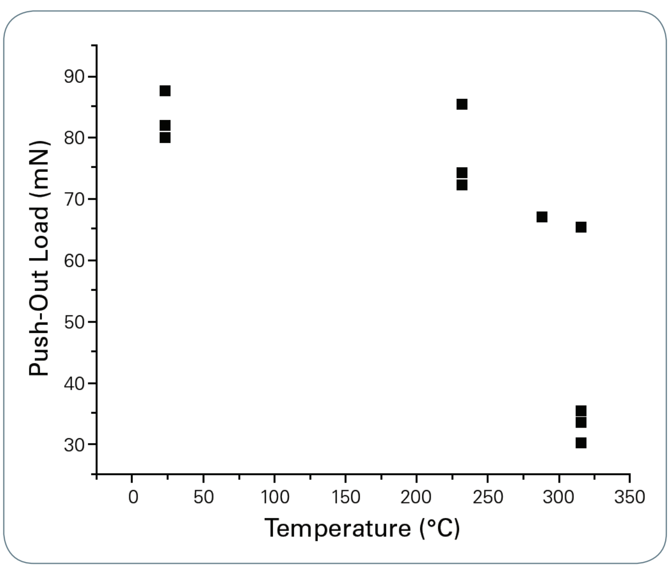

Figure 4. Push-out force vs. temperature. A sharp reduction in push-out force was observed as the temperature approached 300°C.

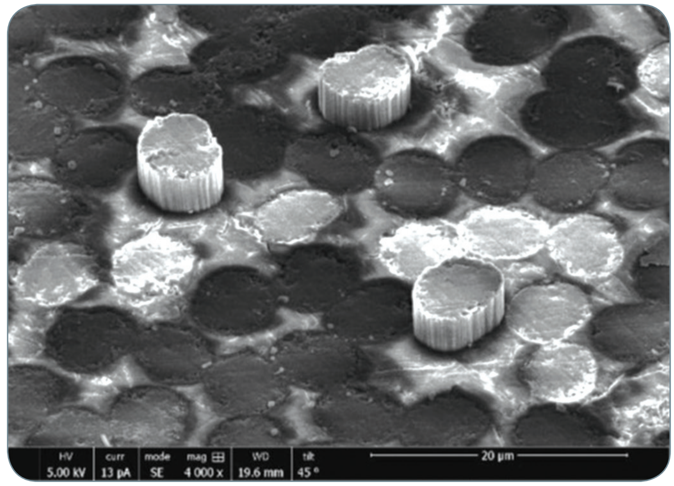

Figure 5. The back-side of the sample showing complete push-out of the tested fibers.

Conclusions

The fiber push-out study demonstrates a technique to measure debonding strength of single fibers within fiber-reinforced composites. The in-situ nature of the tests also provides valuable insight into the progression of failure as well as the impact of temperature on the deformation process—observations that would go unnoticed without performing the tests inside an SEM.

Acknowledgements

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