

## Healing Carbon Fiber/Polymer Composites by Resistive Heating

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Interface is the key region which determines, to a great extent, the set of properties of all heterogeneous systems, including composite materials. We reported interface healing of carbon fiber reinforced thermoplastic composite material via resistive heating. The carbon fiber, T700 carbon fiber, with a resistivity of  $1.66 \cdot 10^{-3} \Omega \cdot \text{cm}$  was used as the heating element while the matrix is polyarylether sulfone with cardo. Micro-droplet experiment was used to study the interface strength before and after heating to determine the healing efficiency. The measurement shows (experimental results show) that resistive heating is an efficient way to heal cracks near interface.

**Keywords:** Interface Healing, Resistive Heating, Thermoplastic, Carbon Fiber.

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### 1. INTRODUCTION

Composite materials always suffer from delaminating caused by long term usage or external impact. However, damages are difficult to detect since most of them are buried deeply within the structures. Once damages developed, the integrity of the structure would be greatly compromised. Therefore, it is desirable to repair the composites to elongate the lifetimes and to lower the maintenance cost.

Healing composites can be carried out by several methods. Dry [1] and White [2] used microcapsules filled with healing agents to heal microcracks. Kausch and co-workers [3] showed that single cracks in PMMA could be healed by rejoining and welding above the glass transition temperature. Kwon et al [4, 5] reported an electrical resistive heating method via carbon fiber network. Park et al [6-18] reported electrical resistive heating to heal a thermally mendable polymer.

In this paper, we studied the healing efficiency of resistive heating induced interface healing by micro-droplet experiments. A polyarylether sulfone with cardo (PESC) microdroplet is repeatedly pulled of and rehealed, and the interface shear stress is measured for the rehealed microdroplets.

### 2. EXPERIMENTS

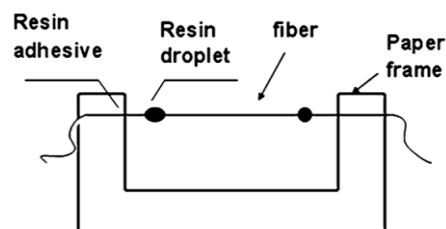
#### 2.1 Materials

Commercially available carbon fiber T700SC-12000-50C (Toray, Japan) with a diameter of about  $7 \mu\text{m}$ , was used. A thermoplastic resin, polyarylether sulfone with cardo (PESC) was provided by Wuxi Resin Factory (China). The N-Methyl pyrrolidone (NMP) and acetone were provided by J&K Chemical Ltd.

#### 2.2 Preparation of micro-droplet specimens

We first detached a single filament of carbon fiber from the fabric and then fastened it to a thin paper holder ( $20 \times 70 \text{ mm}$ ) with double sided adhesive tape.

The free fiber length was approximately 30mm. The micro-droplet specimen was made by applying PESC resin/ NMP at a weight ratio of 20:80. Then the specimen was adhered on a single filament with an embedded length of  $40\text{--}80 \mu\text{m}$  using a fine-point applicator (Fig.1). Then the specimens were cured in an air dry oven (101A-1, Shanghai Laboratory Instrument Works Co., Ltd.) under the normal pressure at  $350^\circ\text{C}$  for 3 h.



**Fig. 1** - Schematic drawing of micro-droplet composite specimens

#### 2.3 Mechanical tests

Interfacial shear strength was measured by the micro-droplet test to determine the interfacial adhesion ability of fibers and matrix. Specimens were tested at room temperature on the interfacial strength machine (Tohei Sanyon Corporation of Japan) at a cross-head speed of  $0.5 \mu\text{m}/\text{min}$ . Two knives were brought very close to each other, as shown in Fig. 2, and eventually came in contact with the solid resin droplet. The fiber was pulled by an actuator while the force required to de-bond the droplet from the fiber was recorded. The force to pull the fiber out of the epoxy composite is measured and used to calculate the interfacial shear strength  $\tau$ , which is expressed as

$$\tau = \frac{F_{\max}}{\pi d l} \quad (2.1)$$

where  $F_{\max}$  is the peak pullout force,  $d$  is the diameter of carbon fiber, and  $l$  is the embedded length of a micro-droplet.

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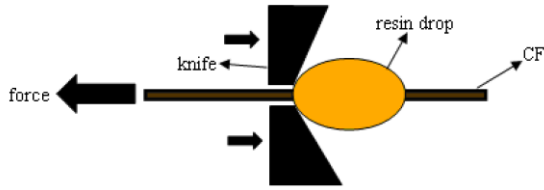


Fig. 2 - The process of micro-droplet test

The SEM images of the debonded microdroplet are shown in Fig. 3. It is found that the surface of the droplet gets rough and some microcracks appears, indicating plastic deformations during the pulling process. A meniscus shaped resin was left on the fiber showing the breaking position.

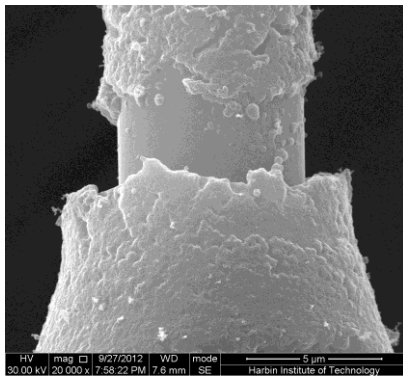


Fig. 3 - SEM images of the debonded microdroplet

### 2.4 Healing experiment

After pulling off the resin micro-droplet, electrical current was applied to the specimen to heal the interface. After healing, the specimen was cooled down to room temperature and the whole process is monitored by an optical micrograph. Fig. 4 shows a healed microdroplet, and by zooming in, it is found that the resin attaches to the carbon fiber again. However, the surface of the droplet is not as smooth as the original one and the contact angle becomes larger.

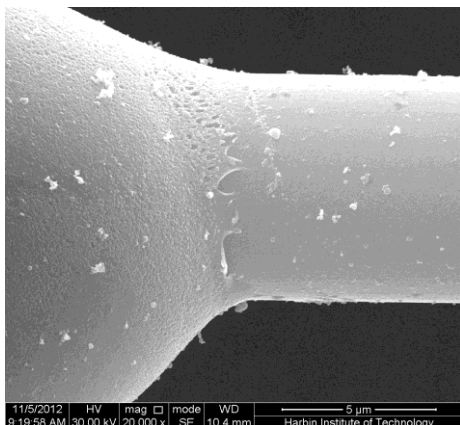


Fig. 4 - SEM images of the debonded microdroplet after resistive heating

To determine the healing efficiency accurately, pulling off and resistive heating was repeatedly applied to a single microdroplet, and the interface adhesion was recorded for each cycle. SEM study shows that

after each heating cycle, the microdroplet tends to vary its shape and dimensions a little bit, while the interface resumes attachment. This corresponds to some mass loss for each pulling off – resistive heating cycles, since after each cycle there will be a meniscus left on the carbon fibers. This mass loss applied a limitation on the heating times in the case of microdroplet test, however for the case of bulk carbon fiber reinforced composites, this is not an issue, since the lost mass will always be recovered for next heating cycle.

### 3. RESULTS AND DISCUSSIONS

The shape of the microdroplet varies after resistive heating, as well as the interface structures. This indicates a potential change of the interface shear strength. So we studied the healing efficiency as a function of the heating electrical current and the heating times.

The healing efficiency was defined to be the strain energy ratio of healed sample and virgin sample up to the maximum strength of the virgin sample.

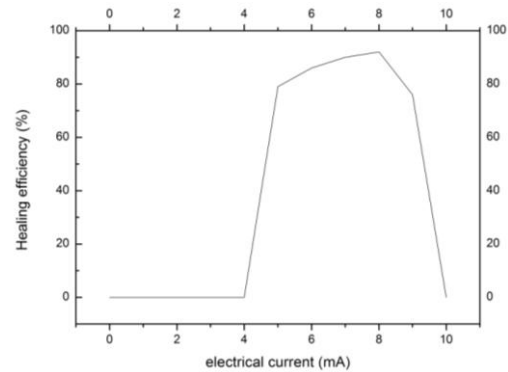


Fig. 5 - Healing efficiency dependence of electrical current

Fig.5 shows healing efficiency as a function of the electrical current. For an electrical current smaller than 5mA, the interface can not be healed, indicating that the temperature is lower than the glass transition temperature of PES. As the current increased to 7mA, healing takes place and the interracial shear strength recovers.

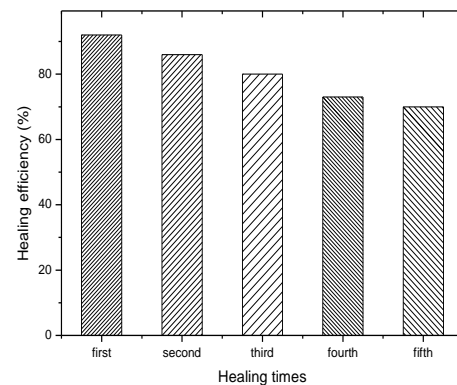


Fig. 6 - Healing efficiency as a function of healing times.

However, increasing the current further tends to cause the healing efficiency to decline. This can be attributed to the thermal decomposition of the matrix material due to the extra energy input. This indicates

that controlling the heating current is critical for healing carbon fiber reinforced composite materials, since the heating tends to result in multi-effect.

The effect of healing on actual interfacial bonding behavior was evaluated using micro-droplet tests. After the resistive heating process, the microdroplet recovered its interfacial shear strength. Fig.6 shows the healing efficiency of the same specimen as a function of the healing times with an electrical current of 7 mA. The healing efficiency ranges from 91.1 % to 87.9 %, and shows little degradation after multi healing cycle. From the high healing efficiency of the specimens, multiple healing of PESC composites with carbon fibers using electrical resistive heating was confirmed.

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## 4. CONCLUSIONS

Interface damages in carbon fiber reinforced thermoplastic composite were healed using resistive heating. Micro-droplet tests were used to discern the possibility of healing from the healed specimen showed high interfacial shear strength. The best healing current was found to be around 7mA. The healing times has little effect to the healing efficiency, the maximum and minimize of healing efficiency are 91.1% and 87.9%. The self-healing ability of the carbon fiber/PESC composite was confirmed by electrical resistive heating.