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Radiation Effects on Epoxy/Carbon-Fiber Composite

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INTRODUCTION

Piping in the Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS) must withstand the stresses involved during an unlikely but potential deflagration event. One method proposed for protection and reinforcement of piping during such an event is the use of a carbon fiber-reinforced epoxy composite (Diamond-Wrap®). In the DWPF, this reinforcement composite product would be required to maintain its safety function for a 20-year service life. This product has been ASME-approved (nuclear code case 589) for post-construction maintenance and is DOT-compliant per 49CFR 192 and 195. However, its radiation resistance properties have not been evaluated. This report documents initial radiation resistance testing of the product and microstructural effects. Additional testing is recommended to evaluate radiation effects on specific properties such as burst strength, chemical resistance/weeping and for service life prediction in critical applications.

EXPERIMENTAL

Tensile bars of Diamond-Wrap® composite of approximate dimension 8 x 1 x 1/16" were provided by Citadel Technologies. Wrapped pipe samples were also provided. Several samples were irradiated to dose of 50, 100, and 200 Mrad in the SRNL Gamma Irradiation Facility. Two bars were subjected to each radiation dose for duplication. Irradiated samples were tensile tested, viewed under scanning electron microscope, and analyzed for changes in the glass transition temperature via dynamic scanning calorimeter on select samples.

RESULTS

Six irradiated composite bars and three baseline bars were tensile tested in a universal testing machine (Sintech 1125). Sample bars were loaded in a swivel upper grip with a 4" grip separation. A load cell of 20 kip was used with a crosshead speed of 0.05 in/min. Each bar was tested until failure by fracture. The strain at failure and moduli values calculated from the stress/strain curves are given in Table 3.

The tensile bars before exposure to radiation are shades of gray in color with some lighter patches most likely due to the presence of excess cured resin. The as-received tensile samples fractured approximately perpendicular to the length of the tensile bar with protruding fibers visible. Minimal evidence of delamination between the resin and carbon fibers is visible. The visible damage due to fracture is primarily contained to the fracture area.

After exposure to a gamma radiation dose of 50 Mrad, the resin undergoes a color change from clear/transparent to light yellow. Discoloration of polymers due to radiation exposure is typical.

After a 100 Mrad dose, the resin shows an increased color change with a visible darkened yellow color. The tensile bars fractured with visible damage primarily contained to the fracture area. Similar to the baseline and 50 Mrad samples, protruding fibers are visible at the fracture site. Visible delamination exists at the fracture site, with minimal damage extending beyond the fracture area.

At the higher dose of 200 Mrad, the resin undergoes a significant color change with a visible dark yellow color. Damage extends along the length of the tensile bar and is not contained to the fracture site. In addition to color changes, pockets of resin are missing at the interface between carbon fiber woven points. This damage extends several inches from the fracture site down the length of the tensile sample. Noticeable volume of resin is also absent from the interior of the tensile bars. Weight measurements were taken for a bar from each radiation dose. While baseline, 50 Mrad, and 100 Mrad samples weighed between 7.7 and 7.9 g, the 200 Mrad weighed 7.5 g, which is a more significant weight loss.

Scanning electron micrographs were taken of the fracture surfaces of the tensile bars. With no radiation, the composite exhibits significant amounts of interfacial failure typical for composites in tension. Fiber pullout found in the baseline samples contained minimal polymer debris, suggesting failure at the interface of the fibers and the epoxy. At sites of epoxy failure, signs of ductile fracture are evident from the ductile dimples elongated by shear.

After 50 Mrad of gamma radiation, minimal debris on fibers suggest interfacial failure as the primary failure mechanism, however, the fracture mode of the epoxy has undergone a transformation to a more brittle fracture, evident from sharp cleavage fracture.

At radiation doses of 100 Mrad significant amounts of polymer debris are present on the carbon fibers. Limited evidence of interfacial failure exists. The primary failure mechanism is through the epoxy rather than along the interface of the carbon fibers and epoxy. The fracture surface of the epoxy is increasingly brittle in nature.

At radiation doses of 200 Mrad (a typical nuclear qualification dose), the integrity of the composite is diminished. Significant epoxy debris remains on carbon fibers and numerous cracks run through the epoxy core.

Several budges covered in cracks have formed on the irradiated sample. These are most likely due to the release of gases from the epoxy upon radiation decomposition. Offgassing of polymers during radiation exposure is expected. Heavy microcracking is present throughout the resin and the macroscopic disconnect between sections of epoxy on the carbon fiber weave, see Figure 1. Laden with cracks, the epoxy appears to have lost much of its reinforcing strength. Might want to say that although significant changes are observed in the composite/interfacial regions, the impact upon specific properties is unknown and requires further investigation. Depending on service conditions, the degradation observed may be acceptable. Alternative epoxy resins with superior radiation resistance could also be incorporated, if other properties are suitable.

Also should note that the composite should be irradiated at varying dose rates to determine the potential for dose rate effects and for life prediction purposes. High dose rate irradiation does not accurately predict service life, particularly if operating at significantly different dose rates.

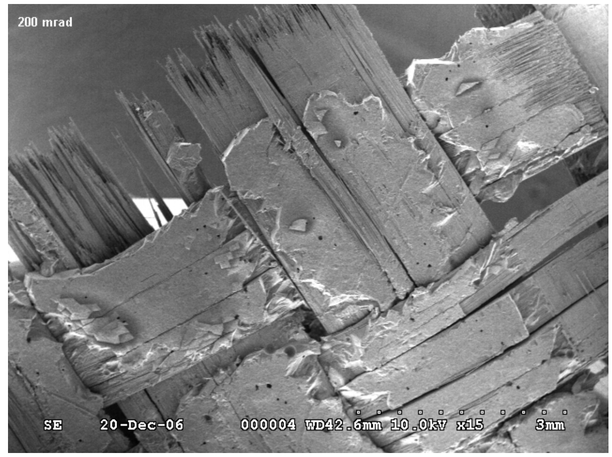

Fig. 1: Epoxy surface on tensile bar subjected to 200 Mrad gamma radiation. Macrocracks form at the interface of carbon fiber bundles as well as along the length of the carbon fiber bundles. The crack formation suggests the epoxy has lost significant reinforcing properties.