

## Fracture Analysis of Composites

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This paper will discuss composite fracture analysis using the light microscope. Because of the translucent nature of polymers there is a wide range of techniques available for viewing polymeric composite materials. If brightfield illumination is the only method used, much of the data will be overlooked.

Often, enhanced contrast is required to study defects having ultrahigh aspect ratios (length but virtually no width, such as a microcrack). Polymer composites with translucent fibers, like glass, Kevlar, polyester and nylon can be examined using colored dyes. The mount is vacuum dried at 120 degrees Fahrenheit, then coated with a dye that is absorbed by capillary action into features such as microcracks. Without the dye, the features can be so subtle they can go undetected. When viewed with darkfield, polarized light or epi-fluorescence, the dyed features will be easily observed.

Carbon fiber reinforced composites require a different set of sample preparation parameters due to the fact that carbon fibers are not translucent. With thermoset polymers most of the features like microcracks and porosity will show up with brightfield illumination. However, with thermoplastic matrix composites the porosity may be visible but microcracks will remain hidden. A fluorescent penetrant dye, like Magnaflux Zyglo (ZKL-H), is used. The cross-section mount is vacuum dried at 120°F. or less before applying the penetrant. Then the mount is back-polished and viewed with a fluorescent light source such as mercury or xenon. The preferred ranges of filters are the following: 390 to 440nm excitation filter and 460nm dichromatic mirror and a 475nm barrier filter.

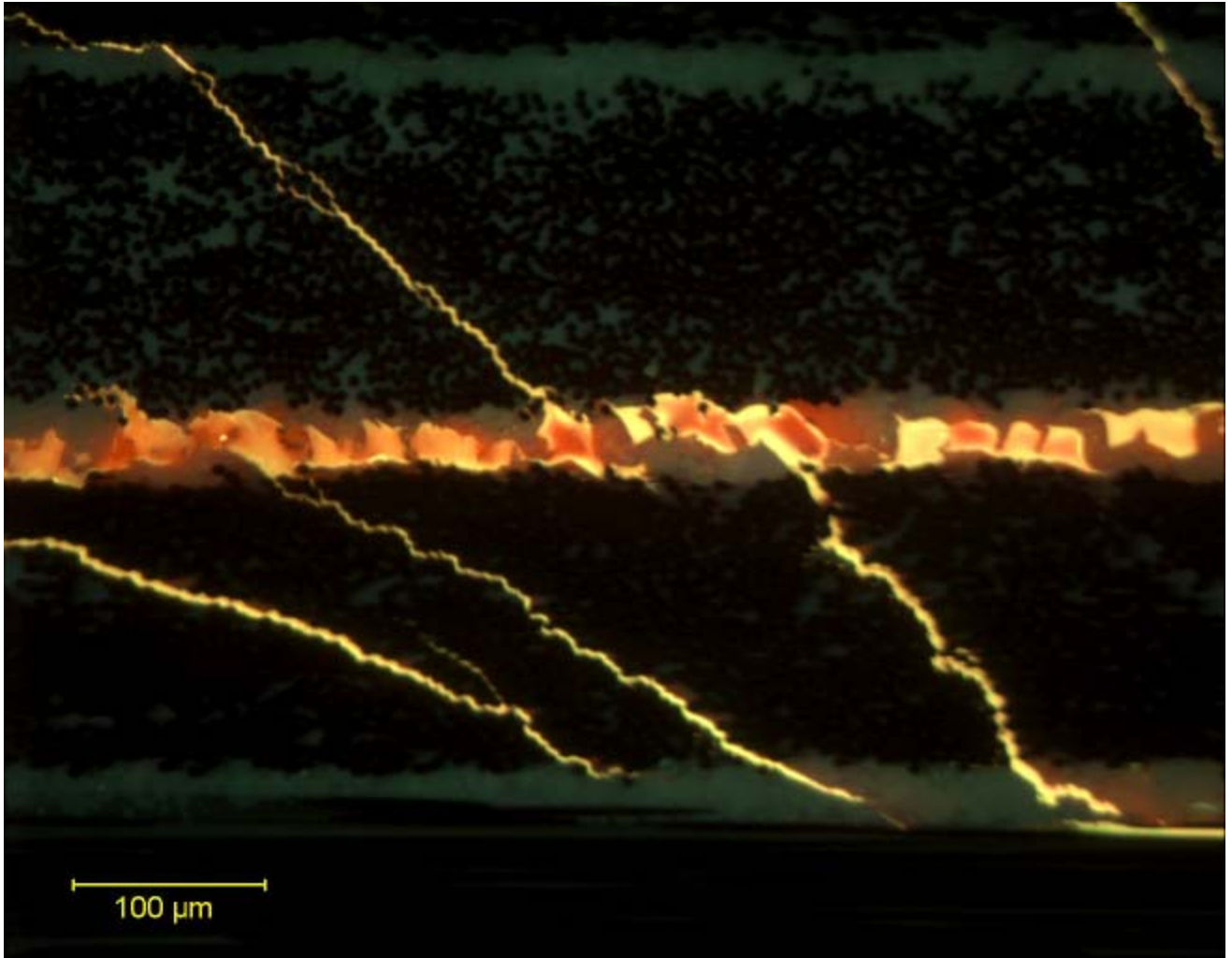
With some analyses there is a need to distinguish the specimen polymer from the mounting polymer. This is best accomplished by using a mounting resin with a red dye such as Rhodamine-B. It is important to hydrate the Rhodamine-B laser dye in the epoxy mixture so that the dye will fluoresce after the specimen mount is prepared. Hydration of the dye is accomplished by adding 7 ml of methanol to 100 grams of the epoxy resin mixture. The methanol and dye are mixed with the resin component of the epoxy prior to mixing the two components together. Polished cross-section mounts can be examined with a variety of microscopic techniques including polarized light, brightfield, darkfield, and epi-fluorescence.

### *RHODAMINE / FLUORESCING EPOXY IMPREGNATION STEPS*

1. Use a glass beaker and a non wooden stirring stick.
2. Mix 0.75g Rhodamine-B laser dye into 7.5 ml methanol. Mix thoroughly to totally hydrate the rhodamine. If not mixed totally it can leave un-hydrated rhodamine that can bleed out from the mount.

3. Mix 100g Buehler Epoxicure Resin (#20-8130-128) into the Rhodamine-B methanol mixture
4. Mix thoroughly and do not vacuum off the excess methanol
5. Mix 20g of Buehler Epoxicure Hardener (#20-8132-32) into the resin Rhodamine-B methanol mixture
6. Mix thoroughly and vacuum impregnate at 225mbar
7. Cure at room temperature, preferably at a pressure >30 PSI lb
8. After curing at room temperature, because the methanol will lower the molecular weight it is recommended to postcure the specimen at 120°F for >8hrs to bring up the molecular weight.

Steps 1-5 create the optimum dye / methanol / resin mixture (@390 to 440 nanometer range) for all polymers



*Figure 1: Carbon fiber composite with rhodamine fluorescing epoxy impregnated into fractures from a compression after impact (CAI) test sample.*