1004 CD
DOI: 10.1017/S1431927606064347
Microsc Microanal 12(Supp 2), 2006
Copyright 2006 Microscopy Society of America

Techniques for Polymer Composites Failure Analysis by Optical Microscopy

Luther M. Gammon, Boeing, Materials & Processes Technology
P.O. Box 3707, M.C. 73-44, Seattle, Washington 98124

This paper will discuss several composite light microscopy techniques for failure analysis. Each is designed to address challenges in composite microscopy. Most features in polymer composites are larger than 1 micron and can be resolved with the light/optical microscope. When analyzing polymers, the visible light range (400 to 700 nm) in the electromagnetic spectrum yields more data than the rest of the spectrum combined. There is a place for the electron microscope, however over 95% of failure analysis data can be obtained with the optical microscope. Bright field illumination will bring out features like porosity and fiber orientation. The more subtle features like compressive microcracks, crazes, phases, and fracture hackles can only be revealed with other selected techniques. There is a need to use the full gambit of epi-fluorescence and reflected and transmitted light illumination for the best possible material contrast.

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 dye impregnated mounting media. The initial mount is vacuum dried at 120 F^0 then coated with a dye which is absorbed by capillary action into features such as microcracks. Without the dye, the features can be so subtle and contrast very low such that features 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 at120°F or less before applying the penetrant. The mount is then back-polished to remove residual surface contamination and viewed with a fluorescent light source such as mercury or xenon. The preferred ranges of filters are the following: 390 to 440 nm excitation filters and 460 nm dichromatic mirror and a 475 nm 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 0.75gm Rhodamine-B laser dye per 100 grams of the epoxy resin mixture. Polished cross-section mounts can be examined with a variety of microscopic techniques including polarized light, brightfield, darkfield, and epi-fluorescence.

https://doi.org/10.1017/S1431927606064373 Published online by Cambridge University Press

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 hydrate totally the rhodamine. If not mixed completely, 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 and 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 (3.25 PSI).
7. Cure at room temperature, preferably at a pressure >30 PSI lb.
8. After curing at room temperature, it is recommended to post-cure the specimen at 120°F for >8hrs to fully cure. This is because the methanol will impact the mounting media's polymer molecular weight.
9. Steps 1-5 create the optimum dye / methanol / resin for high contrast mixtures useful for all polymer microscopy (@390 to 440 nanometer range).

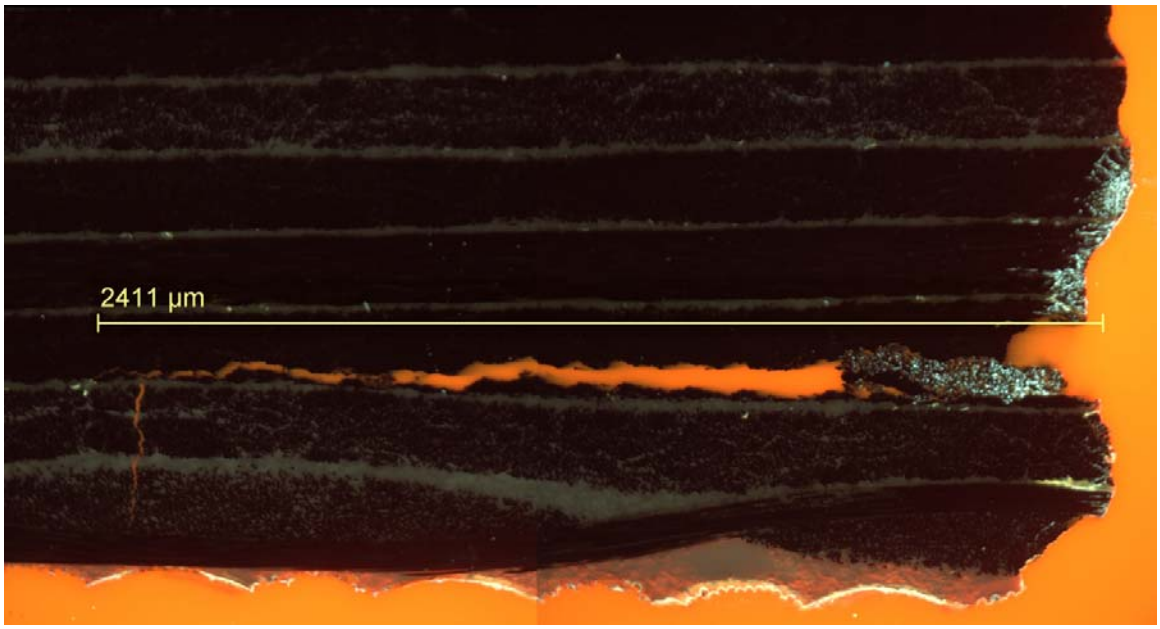

Figure 1: Carbon fiber composite with rhodamine fluorescing epoxy impregnated into the de-lamination crack caused by a diamond abrasive saw cut.

https://doi.org/10.1017/S1431927606064373 Published online by Cambridge University Press