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Quantifying methods for the evaluation of carbon-based composite surfaces for subsequent adhesive bonding

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The production of fibre-reinforced composite parts has revolutionised the aviation industry due to properties such as high specific stiffness and resistance to environmental deterioration, but composite parts can potentially be damaged both during fabrication and during use, and composite repair is still an expensive and delicate process.

If a defect is found after cure and during assembly, or if a defect is introduced through wear and tear, the defect must be cut out, replaced with prepreg and cured and bonded in place. In order for a repair to be effective, the prepreg must form a strong bond with the precured composite surface. This type of composite repair is both labour-intensive and expensive and directly impacts manufacturing and maintenance costs.

Fibre-reinforced composites absorb water into the resin phase, mostly during use or in storage under a humid atmosphere. Before a strong bond can be formed, the composite must be thoroughly dried. This can take hours of heating and vacuum pumping the composite before the bond is formed with new prepreg. There is a need for a simple nondestructive test that can be used to identify whether the composite is wet or dry, both on the shop floor and in the field.

Water dissolved in plastic produces a unique and reproducible signature in the near Infrared (IR) spectrum. Transmission near IR spectroscopy can be used to look into and analyze very thick samples of plastic. However, carbon fibres absorb near infrared energy very efficiently and therefore transmission near IR cannot be used to analyze carbon fibre-reinforced composite samples. It was thought that a relatively new near IR detector based on diffuse reflectance technology, the integrating sphere (marketed by Thermo Electron) could overcome this problem by analyzing near IR radiation that reflects off the surface of the composite without passing through the composite. Near IR spectrometers can be custom built as small portable units that can fit on a cart. If successful, there will be reason to redesign the integrating sphere as a hand-held unit attached to the spectrometer with fibre optic cables. The detector could then be developed to touch the part and be used to label it as dry or wet prior to repair.

A near IR integrating sphere module was built for the Nexus research grade IR spectrometer. Sample preparation consists of placing the sample on top of the module, as shown in Figure 1. A three-step strategy was undertaken to test the validity of this technique for the identification of water in a composite part.

In the first two steps, researchers attempted to determine if it is possible to obtain meaningful near IR spectra using the integrating sphere module with mid- and high-performance epoxy resin and if it is possible to quantify the effect by constructing calibration curves of normalized peak area versus measured water content in the resin.

In the first case, sheets of an in-house synthesized medium performance epoxy resin based on DGEBA and phenylene diamine were submerged in water at high temperatures to accelerate penetration of water into the resin. In the second case, sheets of a high performance epoxy resin, similar to “934” resin and containing MY 720 epoxy and diaminodiphenyl sulfone curative, were submerged in water at high temperatures. In both cases reproducible near IR calibration curves could be constructed for both absorption and desorption of water from the resin.

The third study focused on obtaining meaningful IR spectra using the integrating sphere module with a high performance epoxy resin (carbon fibre prepreg) and if the effect could be quantified. In this study composite plaques were fabricated using carbon fibre-reinforced 934 resin woven prepreg. Samples were vacuum-bagged but not pressurized to produce a composite containing a high resin content and to speed equilibration of water through the sample. Both the bag and tool face of the composite were examined by near IR spectroscopy using the integrating sphere module.

Based on the results of these and other experiments on composite samples, it was concluded that the third technique had well-defined and reproducible strengths and limitations when applied to composite materials. Researchers are consistently able to produce good spectra when examining the bag (or peel ply) face of the composite. In contrast, reproducible spectra cannot be obtained from the tool face of the composite. Both composite faces consist of resin over the first layers of composite. Upon examination of composite plaques in cross section by optical microscopy and SEM, it was discovered that the layer of surface resin is consistently thicker over the bag or (peel ply) face than over the tool face.

The research has shown that diffuse reflectance near infrared spectroscopy, as measured using integrating sphere technology, is a nondestructive technique for the measurement of water in composite systems.

To complete this preliminary phase of the project, composite plaques will be conditioned to zero (the control), a low water content and a high water content, as measured by gravimetry. The near IR spectra of these composites will be measured. The composites will then be bonded into simple test pieces for mechanical testing to determine if it is possible to identify samples likely to form strong and weak adhesive bonds as a function of water content in the composite.

In parallel, near IR instrument makers will be lobbied to design and build a portable version of integrating sphere technology that can be brought to the part. The availability of this equipment will allow for further testing on this project and determine if this method is the most accurate for measuring water in composite systems.

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