Establishing process requirements to fabricate pre-impregnated glass fabric using resin chemistry based on polybenzoxazine

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Introduction

Prepreg technology is a material class used in formulating aerospace quality composites, which are low in void content and high in fiber volume fraction. Prepreg materials are typically formed by mixing a resin with a fiber reinforcement under heat and pressure (Federal Aviation Administration, 2012). Composites can be used to create military grade equipment, such as armor for soldiers and vehicles. Epoxy is a type of resin that most commercial applications use for structural performance because it has a historical precedent. Polybenzoxazine (PBO) is a type of resin with minimal completed characterization, but discovered characteristics of the resin have proven to be better than most resins. Unlike some other resins, PBO does not require an acid catalyst, it releases no byproducts during curing, it has near zero change in volume when cured, and has a long shelf life (Ghosh, Kiskan, & Yagci 2010).

Epoxy is well-studied, so it has a manufacturing process for forming composites. The project demonstrates the processing and manufacturing to form composite panels from PBO and glass fabric. Comparing SC-15 epoxy (Applied Poleramic, Benicia, CA) and CTD 870 PBO (Composite Technology Development, Lafayette, CO) chemical and mechanical characteristics will determine relevancy of the materials for military applications of interest. CTD 870 could help improve the strength of military grade equipment if manufactured properly.

Materials and Methods

In order to proceed with manufacturing efforts, baseline chemical reactivity and resin flow data for CTD 870 was required. The SC-15 epoxy was also analyzed as a direct comparison.

Utilizing a Differential Scanning Calorimetry (DSC) machine, samples of resin were analyzed to find parameters such as reaction rate, fractional conversion, pre-exponential factor, heat of reaction, and reaction order. The glass transition temperature (T_g), or the temperature at which the material softens and flows, of both resins were found in order to determine processing temperature and cure rate of the composites. The Dynamic Mechanical Analysis (DMA) machine was utilized to find the T_g. The single cantilever test on the DMA oscillates the sample as the temperature changes to determine the T_g.

The determined basic chemical behavior of the resins helped design processing conditions that produced appropriate forms of CTD 870 and SC-15 for manufacturing. Figure 1 shows the equipment and flow diagram for producing the intermediate material class known as prepreg. CTD 870 must be heated up in an oven until it becomes a liquid then quickly poured into the heated resin bath and coated onto the fabric to make the prepreg. SC-15 is a two-part chemistry that is time and temperature sensitive and must be run through the process quickly before the reaction is completed (Federal Aviation Administration, 2012). The reinforcement matrix of choice for this study was e-glass fiber. The matrix went through the resin bath and into the oven, as shown in Figure 2, and the newly formed prepreg came out the other end. Then the prepreg was rolled up, with paper between the layers, as shown in Figure 3, so the composite did not stick to itself. The material roll was stored in a freezer until it was cut into coupons for testing.

Materials and Methods (cont.)

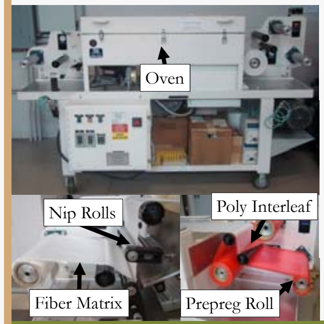
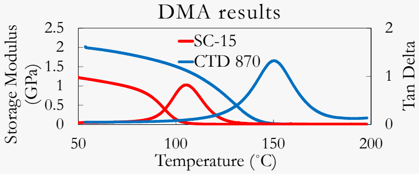


Figure 1 (top): Lab prepreg machine. Figure 2 (bottom left): Prepreg feed side. Figure 3 (bottom right): Prepreg product side.

CTD 870 and SC-15 were made into a composite samples to observe the mechanical behavior by stacking layers of prepreg and curing in an oven under vacuum and pressure. The in-plane shear test and the three-point bend test were conducted in order to find flexural and tensile strength. SC-15 and CTD 870 composites were compared by mechanical and chemical characteristics to validate the resin's performance benefits in composite application.

Results

The results of this research effort were three-fold: a feasibility demonstration for new resin material, a demonstration of processing technology, and a quantitative analysis of composite performance. Tan Delta measures the energy dissipation of a material and determines how good a material is at absorbing energy. The ratio of the storage modulus to the loss modulus of a DMA run is the Tan Delta of a sample.



Graph 1: Tan Delta and Storage Modulus are shown. The Tan Delta peak is the glass transition temperature.

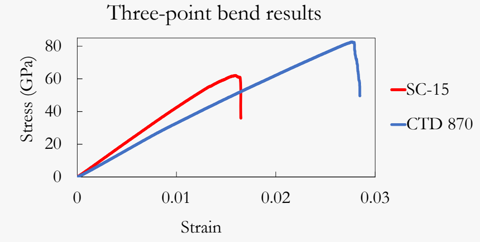
To demonstrate the ability to process CTD 870 into a product from that could be converted into component level testing, material characterizations were completed. A characterization is the DMA mechanical spectrum as shown in Graph 1. The material is stressed using a standard protocol that enables direct comparison of the materials properties for the cured composite system.

Specifically, the T_g was evaluated. In comparison to the baseline SC-15 epoxy material, the CTD 870 had a 45 °C higher T_g. This leads to a greater field level stability and environmental performance.

To demonstrate the component level product, an intermediate product, called the prepreg, is generated. The benefit of this technology is to enable high quality component level products to be manufactured from a wide range of material sources, allowing direct comparison between materials that would not be able to be formed under other process methods.

Results (cont.)

The final success in the project was the ability to manufacture coupon level composites and test the mechanical response of the materials.



Graph 2: The average calculated stress and strain from the three-point bend test of 4 samples of each composite displays the flexural strength.

A *t*-test showed that there was a significant difference in yield stress between CTD 870 (M = 82 GPa, SD =.73 GPa) and SC-15 (M = 62 GPa, SD = 1.5 GPa), $t(4) = -23.98$, $p < 0.0001$ (Graph 2). Also, there was a significant difference in maximum strain between CTD 870 (M = 0.028, SD = 0.0002) and SC-15 (M = 0.016, SD = 0.0008), $t(4) = -30.42$, $p < 0.0001$, which suggests that CTD 870 is mechanically stronger than SC-15. Tensile tests showed similar trends, meaning that CTD 870 is mechanically stronger than SC-15.

Conclusion

All of the chemical and mechanical characterizations show that CTD 870 is a better resin to utilize in manufacturing military grade composites than the traditional epoxy. The direct comparison of the resins in multiple tests shows that CTD 870 performed better overall.

The data collected is a partial characterization of both materials, so there still could be situations where SC-15 is a better resin than CTD 870. The CTD 870 had very little previous data published, but with these positive results, CTD 870 will be researched even further. The next step is conducting a full characterization of CTD 870 for the possibility of replacing SC-15 in some military grade composites in the future.

References

Federal Aviation Administration. (2012). *Aviation maintenance technician handbook-airframe* (FAA-H-8083-31). Washington, DC: U.S. Government Printing Office.

Ghosh, N. N., Kiskan, B., & Yagci, Y. (28 October 2010). Polybenzoxazine-based composites as high-performance materials. *Wiley Online Library, 60,* 167-177. doi: 10.1002/pi.2961