Experimental Investigation on Viscosity-Temperature Relationship of CNFs Filled Epoxy

Robert L. Clark III, Nathan Brock and Kuang-Ting Hsiao*

Department of Mechanical Engineering, University of South Alabama, Mobile, Alabama 36688, USA

* Email: kthsiao@jaguar1.usouthal.edu; Phone: 1-251-460-7889.

Introduction

Viscosity of resin has always been important to the manufacturing process of composites [1]. A resin with a high viscosity has a greater chance of creating voids or dry spots during the infusion process. On the contrary, a lower viscosity causes less resistance to resin flow, which creates a better chance of trapped bubbles being pushed out of the exit port during the composite infusion process. Voids in composite materials are undesired and cause a weakening of the integrity of the composite [2]. Epoxy infused with carbon nanofibers (CNFs) is thought to have a higher viscosity than pure epoxy at the same temperature. An experimental investigation on the viscosity-temperature relationship of CNFs filled epoxy resin is presented here.

Experimental

Materials and Equipments

The purpose of this experiment is to examine the change in viscosity of the CNFs filled liquid epoxy resin. The materials for this experiment are EPON 862 epoxy (provided by Miller-Stephenson) and PR24-LHT CNF (provided by Pyrograf). The equipment used were a digitally controlled hot plate, vacuum oven, thermocouples, a microscope (Nikon Eclipse LV150), multimeters (Agilent 34405A), and a Brookfield Dv-E Viscometer.

Procedure

A pure EPON862 epoxy is used as the control sample for comparison purposes. CNF-filled EPON 862 samples with CNF weight fraction of 0.5wt%, 1.0wt%, and 1.5wt% were carefully prepared with mechanical mixing and sonication for approximately one hour each. To characterize the viscosity-temperature relationship, the resin was first heated on a hot plate to a desired temperature and then allowed to stabilize at that temperature for approximately a half hour. This temperature was verified by a thermocouple inserted into the resin. After assuring the stabilization of the resin temperature, the viscosity was measured. Because the hot plate could not reach the desired maximum heating temperature for the 1.0wt% and 1.5wt% nano-resins (maybe due to the high thermal conductivity), they were alternatively heated in the vacuum oven without a vacuum being applied. The temperatures of the resin and vacuum oven were measured by inserting 2 K-type thermocouples inside the vacuum oven and the nano-resin. CNF-filled EPON 862 was a non-Newtonian fluid [1, 3]; its viscosity changed with different shear rates; therefore, a fixed spindle speed at 100 rpm of the viscometer and a spindle size of 2 was chosen for all viscosity measurements.

Results and Discussion

The temperature and viscosity relationship of pure EPON 862 epoxy is shown in Figure 1. The major changes in viscosity began to decrease around 60^0C; therefore, the appropriate operation temperature for resin flow can be any temperature above 60^0C. For this reason, the nano-resin study was conducted within the temperature range from 60^0C to 130^0C.

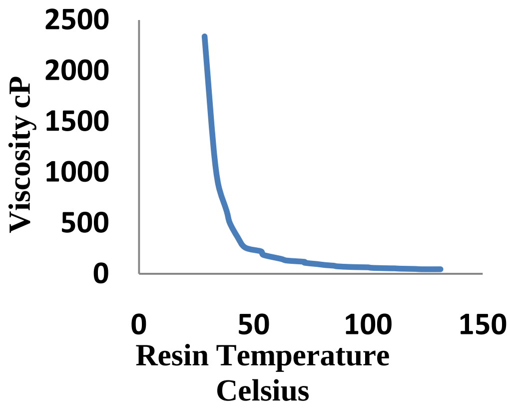

Figure 1 Pure Epoxy Viscosity

At 28, 85, and 120^{0}C, the viscosity was 2339, 80, and 48 centipoise respectively. Figure 2 shows the viscosity-temperature relationships for the 0.5wt%, 1.0wt%, and 1.5wt% CNFs-filled EPON862 resins. The results of the pure epoxy are also included in Figure 2 for comparison.

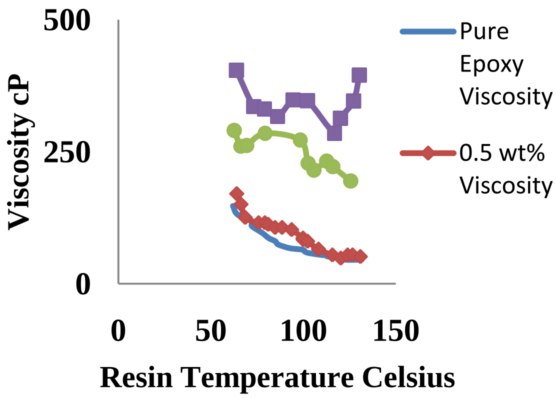

Figure 2 Combined Viscosity Plot

At 102^{0}C, the viscosity was 80, 227, and 346 cP for the 0.5wt%, 1.0wt%, and 1.5wt% respectively.

At a given temperature, the addition of CNFs generally increases the viscosity of the resin system. However, the viscosity increase of 0.5wt% CNFs-filled resin is not as significant as the increases observed in the 1.0wt% and 1.5wt% CNFs-filled resins. In terms of temperature dependence, the viscosities of the control sample and the 0.5wt% CNFs-filled resin significantly reduced as the temperature increased. Contrarily, the high CNF loading cases such as the 1.0wt% and 1.5wt% CNFs-filled resins present much weaker viscosity-temperature dependence. The results suggest the direct contact among CNFs becomes more significant when the epoxy is filled with more than 1.0wt% CNFs.

Conclusions

Generally, CNF fillers always increase the EPON862 epoxy viscosity. For diluted CNFs-filled resin, the viscosity difference from the pure resin is moderate. However, the high concentration CNFs-filled resin behaves very different from the pure resin for its significantly higher viscosity value and the much weaker viscosity-temperature dependence. This may be due to the CNFs contact in the high concentration nano-resin.

Acknowledgements

The authors acknowledge the financial supports from National Aeronautics and Space Administration (Award# NNM07AA09A-01), National Science Foundation (NSF-EPSCoR Cooperative Agreement 0814103 and Subagreement No. 39-21530-081), and the NASA ASGC Fellowship (Award# SUB2005-152-A11-04)

References

[1] Advani, Suresh G., and E. Murat Sozer. *Process Modeling in Composites Manufacturing*. 1st. New York: Marcel Dekker Inc., 2003.

[2] Clark III, Robert L., Michael Skinner, Basil Farah, Justin Farris, Dr. Kuang-Ting Hsiao, Dr. Martin R. Parker. "Thermal and Electrical Conductivities Characterization of CNF-Modified Glass Fiber/Polyester Composite." *Society of Advanced Materials and Process Engineering* (2008): Print.

[3]Owczarek, Jerry A. *Introduction to Fluid Mechanics*. 1st. Scranton, PA: International Textbook Company, 1968.