

Formation of In-Situ Sequential Interpenetrating Polymer Networks Via Thermal and Radiation Curing

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Over the past years, electron-beam (e-beam) curing has been actively explored as a method to reduce the processing and fabrication cost of polymer matrix composites (PMC). In this work, a new approach towards making an interpenetrating polymer network (IPN) which is termed *in-situ sequential* IPN has been employed by developing resins from a mixture of epoxy-amine and vinyl systems. This method uses sequential steps for curing which provides control over network formation, while it eliminates the time consuming swelling step of the traditional sequential method by mixing monomers of two networks together from the beginning.

Detailed cure kinetics studies were carried out to select appropriate vinyl systems that led to IPNs with improved properties. There is side reaction that occurs between amine and vinyl groups. This is known as Michael addition reaction. The results of the kinetics experiment showed that the rate of Michael addition between primary amine and acrylate groups is about 13 times higher than that between primary amine and methacrylate groups. The kinetics study also indicated that the methacrylate group did not react with the amine at temperature below 50°C. Hence, the methacrylate group-based resin was selected with the epoxy-amine system to form an in-situ sequential IPN.

A new concept for tailoring IPNs is introduced in which the two networks are chemically bonded to each other. This required a partially methacrylated epoxy-based monomer, which was synthesized and characterized using FTIR and HPLC. The thermal cure of this resin forms the epoxy-amine network and leaves the methacrylate and / or acrylate pendant groups dispersed throughout the network. Further e-beam curing of the C-stages network creates the second network.

The esterification reaction to synthesize partially methacrylated monomer was studied under non-stoichiometric conditions and in the presence of various catalysts. The study indicated the occurrence of an etherification along with the expected esterification reaction that leads to gelled product that cannot be used. Based on this part of the work, the catalyst and processing conditions were selected to minimize the etherification side reaction and allow for the synthesis of desired monomer. Furthermore, the reaction mechanism explaining the esterification and etherification was also suggested and explained.

The performance of the developed resin modified with the methacrylated epoxy monomer was assessed in terms of the T_g and cure shrinkage. The increase in degree of covalent bonding between networks increased T_g and reduced the normalized shrinkage of the vinyl network (during e-beam curing) of the system. Further, the performance of e-beam cured systems was compared to that of thermal-cured systems, showing that thermally cured systems have lower conversion, T_g, and higher normalized shrinkage of the vinyl network than similar e-beam-cured systems.