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A TEM Investigation of the Network Structure of Electron Beam Cured Epoxy Polymers

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Glassy network epoxies have widespread applications as matrices for advanced composites. As an alternative to thermal curing, electron beam (ebeam) processing has been demonstrated as a very powerful tool for achieving fast and efficient curing [1,2]. Ebeam curing of epoxy based resins using suitable onium salts as initiators proceeds via a cationic mechanism. The final polymer network properties are highly dependent on processing conditions and curing kinetics. It was demonstrated that under certain processing conditions, heterogeneities can appear in the material, due to the agglomeration of initiator residue. Initially the initiator is miscible with the resin; however, reaction-induced phase separation occurs as the increasing molecular weight of the polymer reduces the solubility of the initiator. The formation of a heterogeneous rather than a homogeneous network depends on the polymerization kinetic, and especially on the vitrification and phase separation rate.

A TEM study was performed to better understand the curing behavior and network formation of ebeam cured epoxies. Tactix 123 resin was mixed with 3 phr of SarCat CD1012 cationic initiator and ebeam cured using an Acsion Industry 1-10/1 electron linear accelerator (Acsion Industry, Pinawa, Manitoba, Canada). The dose deposited per pass was varied from 5 to 20 kGy for a total dose of 160 kGy. The bright field micrographs in Figure 1 show submicron particles are associated with holes or embedded in the epoxy matrix. The micrograph in Figure 1b shows that the particles are an agglomerate of two particles: very small particles and much larger dark particles. Post curing this specimen for 1 hour at 175 ºC results in dissolution of many particles (Figure 2). A particle caught in an intermediate dissolution stage appears in Figure 2b. XEDS shows these particles are mostly composed of antimony. This suggests the particles are a byproduct of the initiator. Finally, the TEM micrograph in Figure 3 shows the morphology of the thermally cured epoxy. Though in general the material was featureless, there were some regions populated with submicron particles (**A**).

References

1.) B. Defoort, L. T. Drzal, SAMPE 46th International Symposium, Long Beach, CA. pp2550-2562, (2001).
2.) T. Glauser, M. Johansson, and A. Hult, “Electron-beam Curing of Thick Thermoset Composite Matrices”, Polymer, 40, 5297-5302.

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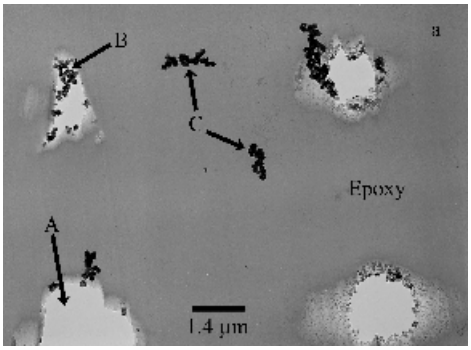

Figure 1a: Bright field TEM micrograph of 160 kGy specimen showing (**A**) holes, (**B**) particles near holes, and (**C**) particles embedded in epoxy.

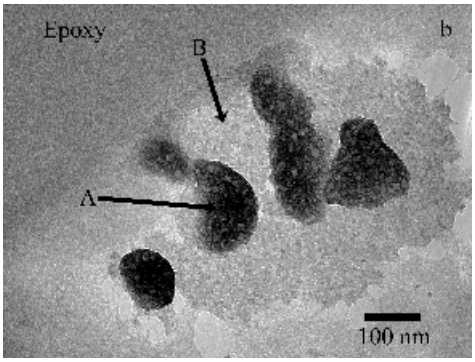

Figure 1b: Higher magnification of particle in Figure 1a, showing the particle has 2 components **A** and **B**.

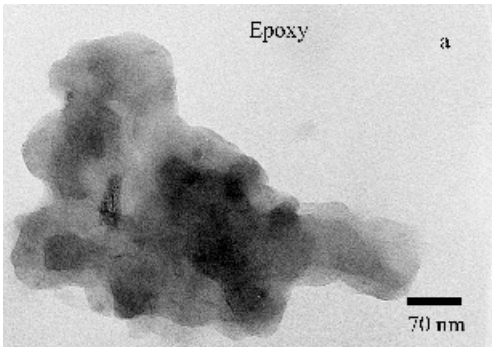

Figure 2a: Bright field TEM micrograph of a particle after ebeam (160 kGy) and post curing for 1 hour at 175 ºC.

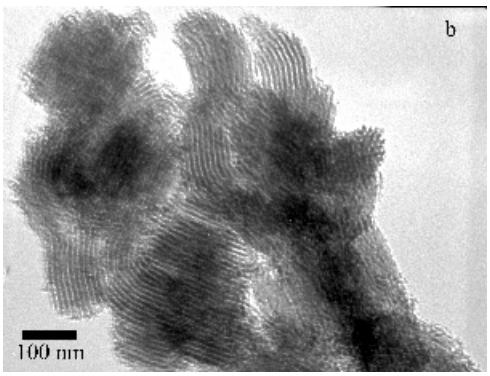

Figure 2b: Bright field TEM micrograph showing the effect of post curing on initially ebeam (160 kGy) cured material.

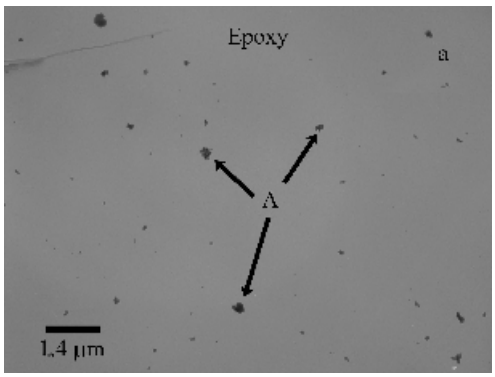

Figure 3a: Bright field TEM micrograph showing the morphology of epoxy thermally cured for 3 hours at 170 ºC.

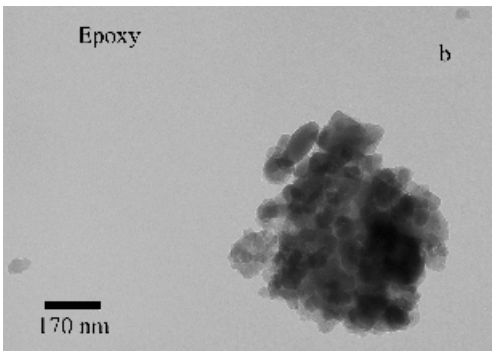

Figure 3b: A higher magnification of the particles seen in Figure3a. Note that this is an agglomeration of particles.