Epoxy Cure Analysis

Application Note 140

Relaxation measurements can be very useful to monitor cross linking reactions. Conventional methods such as gel timers or rheological measurements apply shear to monitor the cross-linking reaction. Shearing the sample during crosslinking interferes with the formation of polymer chains. Shear also provides mixing that would not occur during actual use of a polymer used as an adhesive or encapsulant where diffusion of reactive species to the forming polymer may control the final stages of cure. Lastly, relaxation measurements have minimal cleanup. Resin is added to a disposable sample tube and then the cured resin filled tube is discarded.

In this example, a commercial 2-part quick setting epoxy resin (Clearweld, JBWeld) was injected into a disposable open ended sample tube after mixing in the static mixer included with the dual syringe epoxy. A short video illustrating the sample loading process is available here (https://youtu.be/0Tbj5_9FR8g).

T2 relaxation measurements using a CPMG pulse sequence were performed at room temperature periodically for 220 minutes. Figure 1 shows representative results from the first experiment, with a relaxation time of 2.2ms, followed by results obtained after 16 minutes following mixing. Initially we observe the presence of a second shorter relaxation time indicated by the divergence of the experimental data (red x) from the single relaxation fit. This is likely due to the presence of unreacted epoxy within silica filler domains in the epoxy. In the data acquired at 16 minutes, extensive crosslinking has occurred, and we only observe a single relaxation time.

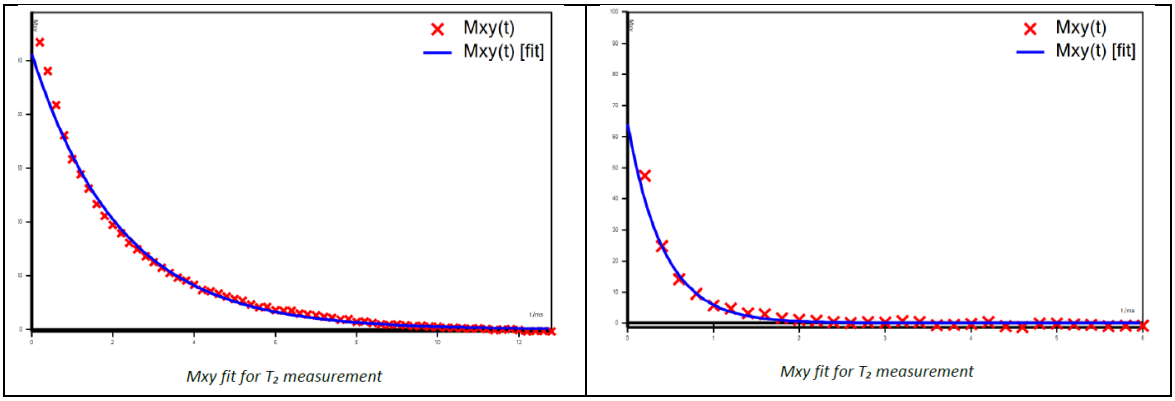

Figure 1 Representative Relaxation Results

The results in figure 2 are the compilation of relaxation time and Mxy(0) as a function of cure time. Time is shown logarithmically to better understand the dynamics of the rapidly curing epoxy at short and long times. Crosslinked solid polymer has a relaxation time too short to measure accurately with this approach. Mxy(0) is the intercept of the relaxation experiment, and is proportional to the

quantity of unreacted polymer. We observe that both the relaxation time T2 and the relaxation intercept Mxy(0) decrease with time.

It is useful to be able to capture the reaction dynamics without complicating the data interpretation by shear, as you would observe in a gel timer or rheological test. We do see an inflection point after about 8 minutes of cure which is likely related to the ability of reactive components to diffuse to a reactive site on the polymer chain. The Area has the ability to control temperature at elevated conditions. The interplay between filler concentration, particle size, surface area, epoxy reaction rates should be optimized to provide the right combination of flow and mechanical properties for the best product performance.

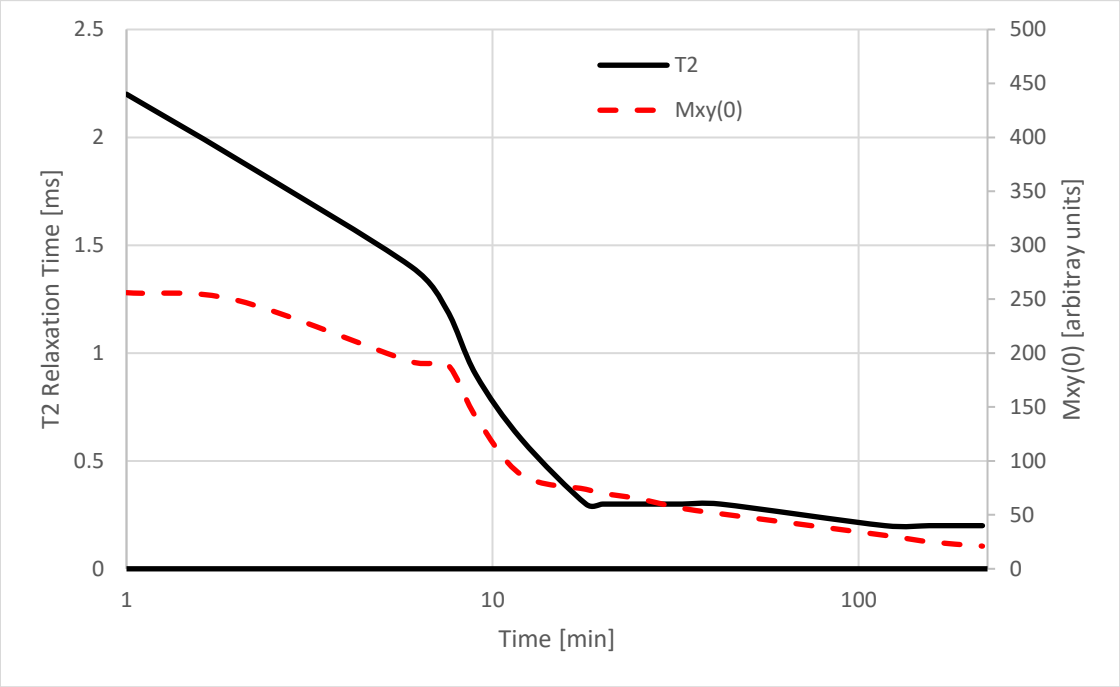

Figure 2 Relaxation time & Mxy(0) as function of cure time