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Thermal stability of high temperature epoxy adhesives as measured by accelerated TGA, DMA, and adhesive strength

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High temperature adhesives able to operate continuously at high service temperatures are an important class of adhesives and have use in electronics, automotive, and aerospace. It is crucial in these high temperature applications to have an accurate prediction of the adhesive life in order to predict failure in applications that tend to push adhesives to their thermal limit.

Measurement of adhesive life has heavily relied on thermal gravimetric analysis (TGA) to determine material degradation kinetics under accelerated thermal aging. [1-4] Thermal degradation studies performed through TGA measurement relate the rate of mass loss to the rate of chemical bond scission. In TGA experiments, the rate of mass loss is measured and is assumed to be proportional to the rate of bond scission. As chemical bonds are broken, volatile species are released leading to a loss of mass. For the small sample size employed in TGA experiments, the mass loss of volatiles is assumed to be much faster than bond scission and therefore rate limited by the scission of chemical bonds.

A criticism of TGA measured degradation is that it measures degradation at temperatures far above the anticipated operating environment of the adhesive and relies on large temperature extrapolations to lower service temperatures under the assumption of a consistent degradation mechanism. [5] In addition, TGA measures degradation in terms of weight loss. In many applications, other properties such as loss of adhesive or bulk mechanical strength is the more important variable leading to ambiguity in how TGA weight loss measurements relate to adhesive or mechanical degradation at the anticipated service temperature.

In this paper, the extent of degradation of two epoxy adhesives known for their thermal stability is measured in terms of loss of adhesive strength and reduction of glass transition temperature (T_g) and compared to high temperature accelerated TGA weight loss measurements. The loss of adhesive strength is measured by napkin ring torsional adhesion measurements and the glass transition is measured by dynamic mechanical analysis (DMA). Measurements are directly compared to TGA weight loss measurements. We are interested in characterization of adhesive degradation at temperatures closer to potential continuous operating temperatures of high temperature resistant adhesives in order to compare the thermal sensitivity of adhesion degradation and T_g reduction performed at lower temperatures with high temperature TGA weight loss measurements and test the Arrhenius relation of epoxy thermal degradation over a wide temperature range through independent measurements of thermal degradation. In theory, the loss of adhesion and reduction of T_g are governed by bond scission of the polymer network in a manner similar to TGA weight loss measurements and will have a similar sensitivity to temperature. The loss of adhesive strength and reduction in T_g are shown to be well described by

an Arrhenius expression with an activation energy that agrees well with the same parameter determined from high temperature TGA measurements. The comparison allows information to be gained about the relationship between specific material properties and thermal degradation.

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