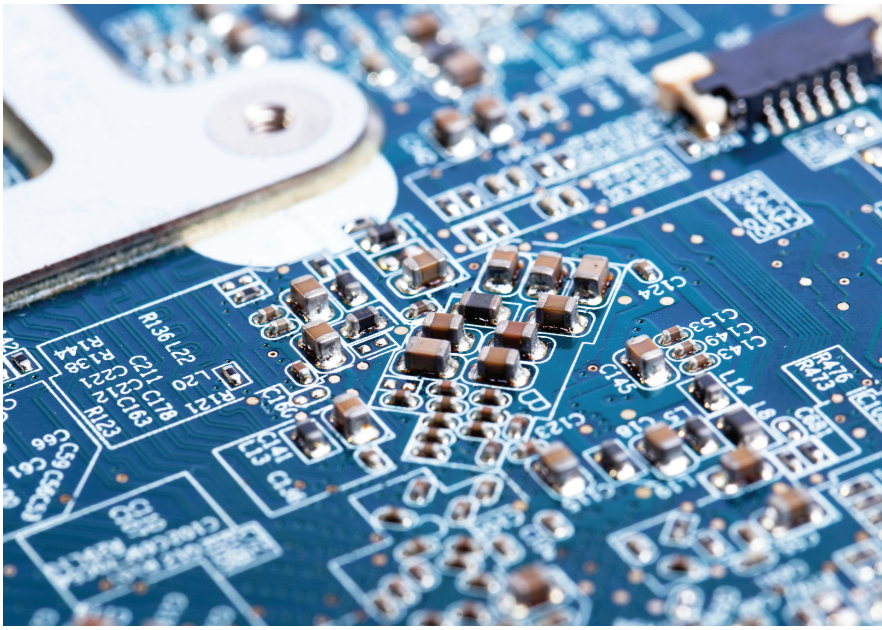

APPLICATION NOTE

Differential Scanning Calorimetry

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Curing of an Optical Adhesive by UV Irradiation in the DSC 8000

Introduction

Optical adhesives are used in many industries where solvents are undesirable. Semiconductors and chip manufacturers for example cannot afford solvents depositing on components. Photo-DSC allows fast analysis of the curing profile and measurement of the energy of the curing reactions. Because photo-initiated reactions are fast and energetic, good temperature control and responsiveness are needed to get good data. Power compensated instruments are the best choice for these applications

Experimental

A PerkinElmer DSC 8000 with UV irradiation system was used. A specialized DSC pan with a quartz cover can be used although an open pan often is acceptable. The sample is heated or cooled to the isothermal temperature and allowed to equilibrate. Pyris™ software allows triggering the shutter of the light source to open and close for irradiation of the sample. Data can be collected at various intensities and times to develop the best cure cycle for the material.

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Figure 1. **DSC 8000.** The DSC's high sensitivity and excellent temperature control makes it ideally suited for demanding applications like photo curing studies.

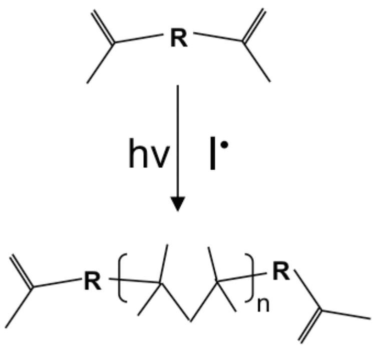

Figure 2. UV curing materials are commonly used as adhesives. UV light generates a free radical which causes the cure.

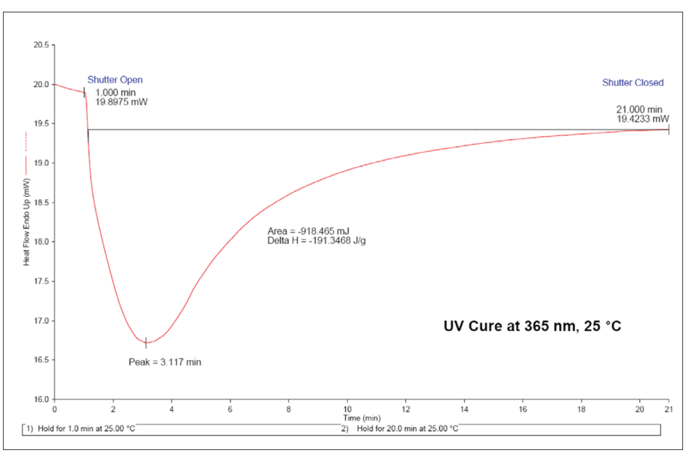

Figure 3. DSC results from the UV driven cure of an adhesive. Light turned on at 3 minutes (red line marked shutter open) for 30 seconds.

Advantages of Power Compensation Design

The DSC 8000 (Figure 1) is the ideal choice for these studies because the power compensated design allows the instrument to quickly detect and respond to changes in the optical material. The initial transient is very short, typically around 12 seconds, which means that rapid reactions can be accurately measured without significant energy loss during the transient. Power compensation design also ensures true isothermal operation, power is adjusted to maintain the desired isothermal temperature and it is this power compensation that is read out on the screen, so even rapid increases in temperature often associated with fast reactions are compensated for. In fact, the extremely rapid energy response, fast data collection rate of up to 100 data points a second, and extremely wide dynamic energy range of power compensation systems are essential to control and measure rapid energetic reactions. All of the recorded energy therefore comes from the reaction shown in Figure 2. The totally stable design also means that curve subtraction procedures often used as part of the measurement technique are totally quantitative. Single furnace design (heat flux) systems have significantly longer transient times, cannot control rapid energetic reactions and only make measurements when the temperature deviates from isothermal so are incapable of true isothermal operation.

Results

When the UV source is triggered, the sample is exposed to the UV light. A curing exotherm develops as the material undergoes polymerization. The energy in this peak can be used to calculate the kinetics of the curing process. Figure 3 shows an example of this. When the light is applied at 3 minutes for 30 seconds, a exothermic reaction occurs as the material crosslinks and forms a solid. Since the material is its own solvent, no volatiles are lost and contamination of the circuit board is minimized. Looking at the thermogram, one see the maximium of the exotherm occurs very quickly after applying the light, emphasizing the need for fast response. The energy of the reaction is calculated from the area under the peak, which is why the ability to compensate for the heat coming from the UV lamp is important.

Depending on the material, the exposure can be varied in terms of time as well as the number of exposures. Light frequency and intensity can be varied using filters on the UV source. As UV sources generate heat, the DSC's temperature control must be able to compensate for the energy from the lamp.

Conclusion

Photo-DSC using power compensation techniques is a powerful tool for studying and quantitatively characterizing optically curing materials. For more information, visit http://www.perkinelmer.com

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