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Thermal Analysis

PETAN-47

Characterization of (Epoxy) Adhesives Using Multiple Thermal Analysis Techniques

Introduction

Adhesives are widely used today to bond two surfaces together. Epoxy film adhesives represent a large group of materials consisting of resin/hardener mixtures that are cast into a film and partially staged (partially cured). Thermal analysis techniques are used to determine if an epoxy adhesive has advanced beyond a useful state of cure (i.e., shelf-life studies), and in determining the glass transition and the degree of cure. Thermal analysis techniques are also used to find the amounts of moisture and volatiles present in a material, as well as measuring mechanical properties such as expansion, contraction and modulus.

Each of the various thermal analysis techniques supplies information about materials used in adhesive technology. Together, the combined results answer many questions relating to the formulation and development of these materials.

The DSC (Differential Scanning Calorimeter) is used for analyzing a material's glass transition, degree of cure and cure state. Residual cure in the material is determined by calculating the ΔH of the exotherm. The TGA (Thermogravimetric Analyzer) is used for analyzing the composition of an adhesive by quantifying the amount of moisture that is present and the amount of volatiles associated with the reaction. The TMA (Thermomechanical Analyzer) is valuable in the determination of the glass transition and expansion of the material. DMA provides modulus information related to the cure state of the epoxy. All of this information is then used to determine the compatibility of a material with a specific application.

Purpose

To completely characterize an epoxy adhesive using several thermal analysis techniques.

Experimental

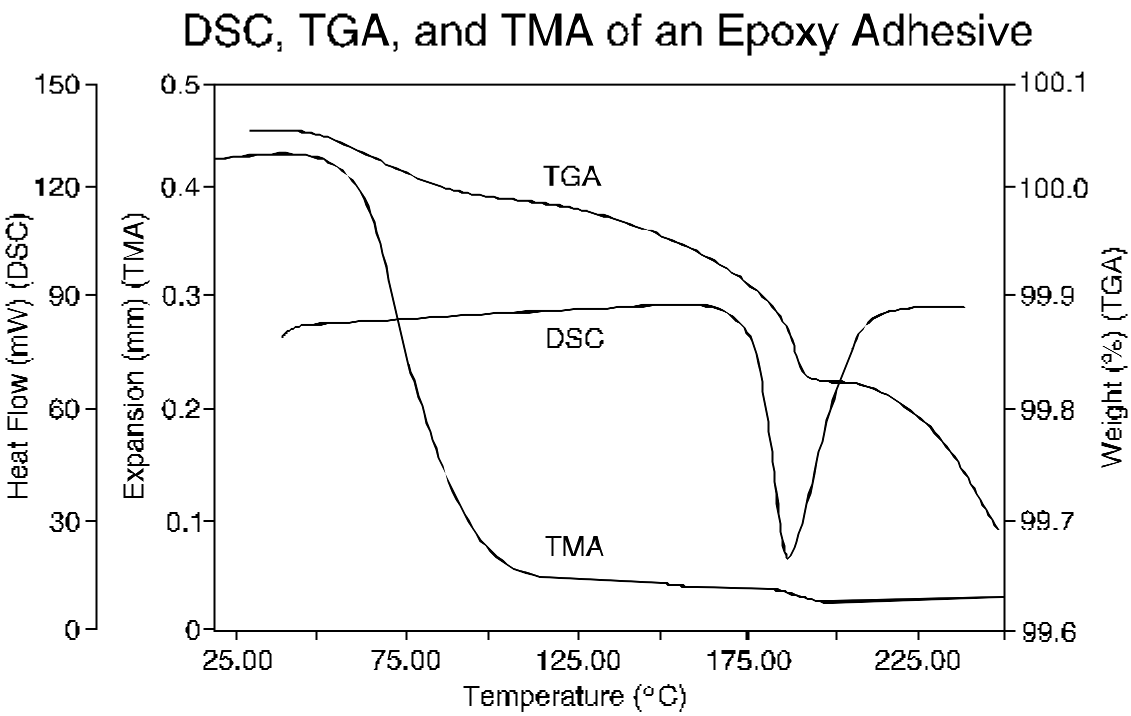
From the adhesive film, a paper punch was used to prepare samples for all of the analyzers. The DSC sample was weighed on a PerkinElmer AD-6 Ultramicrobalance and encapsulated in a standard aluminum DSC pan. The instrument was purged with nitrogen at 20cc/min and the sample scanned from 50°C to 250°C at 20°C/min. The TMA was zeroed with an empty DSC aluminum pan and cover. Two disks of sample were placed in the pan, then covered with the lid and positioned under the probe of the analyzer. This sample was scanned from 0°C to 250°C at 10°C/min in a helium environment. The sample for the TGA was placed directly into the TGA sample pan and scanned at 20°C/min in air up to 250°C. The PerkinElmer DSC, TGA and TMA were used in the evaluation of this high-temperature epoxy film adhesive.

Results

A small weight loss (.05%) was found in the TGA between 50°C and 100°C. This weight loss is associated with the loss of volatile material from the sample. In the same temperature region, the TMA trace shows a significant compression of the sample related to the softening point or onset of the Glass Transition of the film.

As the temperature is increased, the crosslinking (curing) reaction proceeds. This is shown by the large exothermic reaction in the DSC curve beginning at approximately 165°C. During the crosslinking (170°C-225°C), volatilization continues to occur and is seen as a weight loss of 0.15% in the TGA. At the same temperature, the TMA data shows another compression, presumably due to continued softening prior to the onset of gelation.

As the reaction proceeds to completion, the TMA exhibits expansion in the sample while the TGA shows the beginning of another weight loss.



Conclusion

Each thermal analysis discipline by itself supplies necessary information about epoxy materials, but none will supply a complete characterization. It is the ability to analyze the material with instrumentation which has high sensitivity and accuracy, along with the ability to simultaneously study data from multiple instrument disciplines, which gives the thermal analyst insight into the entire curing process.

References

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