Differential Scanning Calorimeter

Thermal analysis of Polymers: Photo-DSC as a tool for UV-curing

Chip-DSC 10

Introduction

Thermal analysis is very useful tool for the analysis of various compounds. Differential Scanning Calorimetry (DSC) gives information about phase changes and chemical reactions.

As the Chip-DSC comes with some unique hardware options, there is the possibility to access analysis techniques and combinations of techniques that are not available with other systems on the market. One example is the availablity of optical interfaces that can be directed at the sample during the measruement, such as a UV lamp.

Methods

During the last decades, ultraviolet (UV)-curable resin systems have been gaining more and more market share in the coating, lacquer and other related industries due to their unique advantages including fast curing, low energy consumption and low VOC emission levels. Therefore, the UV-curable resins market size was estimated to be worth over USD 3.5 billion in 2015 and is expected to exceed USD 8.6 billion by 2023, which means an annual growth of more than 9.2%.[1]

Latest trend which is believed to be contributing to this growth, is the additive manufacturing industry. 3D printing processes such as the DLP (digital light processing) or the CLIP (continuous liquid interface production) method, which promise up to 100 times faster production times compared to other 3D printing processes, are on the verge of overturning parts of the industrial landscape.[2] Here, LED-based light sources are used for precise manufacturing where in the whole industry a change to LED-curing due to longer service life and lower energy consumption is visible.

In cooperation with the University of Bayreuth, the Chip DSC was proven as a perfect model to measure adhesives while irradiating with a new UV lamp. These measurements are important for entrance tests and development business like 3D printing as well.

"The Linseis Chip DSC in combination with an UV/LED source is a powerful tool for the characterization of fast photocurable resin systems" [L. Gonzales, University of Bayreuth]

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Table 1. Experimental Conditions

Instrument	Chip-DSC 10
Heating rate	200K/minute
Sample Mass	approx. 10 mg
Sample Pan	open aluminum pans without lid
Gas	air

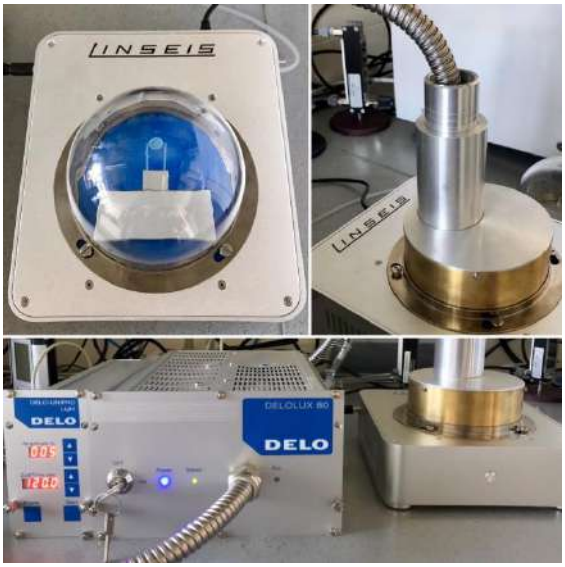

Fig 1: Linseis Chip DSC 10 with LED Lamp; RT to 600°C (+/- 0.2°C) Heating Rate 0.001 – 300 K/min

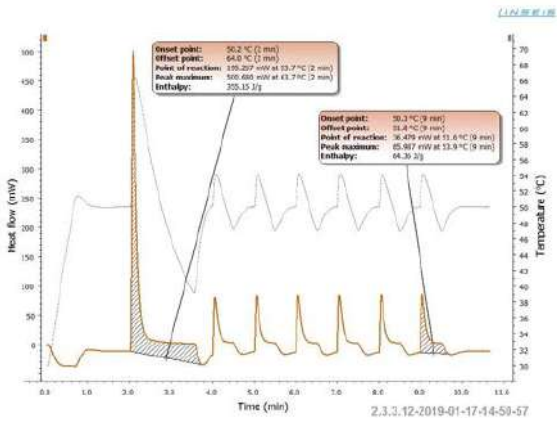

Fig. 2: Determination of UV curing enthalpy by light pulses

Experimental

The new Linseis ChipDSC10 was connected with a 400nm-LED-lamp (DELO Industrie Klebstoffe, Windach, Germany), where selection of temperature, atmosphere and irradiation time is easily adjustable (see Figure 1).

Results

In this specific case, a mixture of a photocurable acrylate and thermally curable epoxy was used and around 10 mg of material was placed in an open crucible and irradiated with several UV light pulses until no change in peak area was detected.

The difference between the first irradiation peak and the last one, (when the area under the peak reaches a plateau, thus it is assumed that no reaction is taking place), is calculated to determine the heat of reaction for the UV-curing part, and to derive a conversion curve.

Conclusion

The Linseis ChipDSC10 in combination with an UV/LED source is a powerful tool for the characterization of fast photo-curable resin systems. Deep insight into curing kinetics as well as into the quality of the raw material is possible. The technique allows experimental flexibility and the software, that accompanies the device, facilitates the evaluation of the collected data.

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