

NETZSCH

Proven Excellence.



Photo-DSC 204 *F1 Phoenix*®

Photocalorimetry – Method, Technique and Applications

Analyzing & Testing



Photo-DSC 204 *F1 Phoenix*®

Photo-Calorimetry Is an Extension of Classical Calorimetry for the Investigation of Light-Induced Processes.

Advantages of Photo-Calorimetry

Light- (mostly UV-) curing systems react very quickly (within a few seconds at low isothermal temperatures) and are solvent-free. This makes them very attractive for industrial applications.

Three different types exist: radical, cationic and dual-cure systems. Dual-cure systems exhibit a combination of thermal and light-curing reactions and can be found, for example, in adhesives or paints.

The ability to measure the curing kinetics and degree of cure is essential for the development of UV-curable resins and identification of optimal curing times and conditions. Photo-differential scanning calorimetry (Photo-DSC) is a powerful analytical tool for accomplishing these measurements.

Your Benefits

- Extension of the DSC technique with light radiation capability
- Analysis of photo-initiated reactions in materials
- Measurement of the light or UV-light curing of polymer resins, paints, inks, coatings and adhesives
- Study of the influence of UV stabilizers in pharmaceuticals, cosmetics and foods (aging effects)
- Optimization of temperature, atmosphere, light intensity, wave length and exposure time
- Determination of the reactivity and curing time of the polymer matrix in compounds



Made by NETZSCH

Various Commercial Lamps Can Be Used for Optimum Curing

The Photo-DSC 204 **F1 Phoenix**® can be equipped with mercury (Hg) lamps, diode laser systems or high-power LED lamps in order to achieve optimum curing results.

Defined Light Source Distance for Reproducible Measurements

Adjustable light guides provide for a constant distance between the light source and the sample and reference, respectively. Only reproducible measurement conditions ensure precise results. The light guides are mounted on the automatic cover lift for easy handling.

Flexibility and Efficiency Across a Broad Temperature Range

Photo-DSC measurements can be carried out between -100°C and 200°C. However, by simply using the additional manual furnace lid, standard DSC tests can be done – alternately or successively – in the temperature range between -180°C and 700°C with the appropriate cooling device. Even measurements with irradiation can be carried out with the automatic sample changer (ASC). The ASC can handle up to 192 samples in different crucible types.

Precise Control of the Atmosphere Around the Sample

The gas-tight DSC cell in combination with the integrated mass flow controllers allows for precise control of the composition of the atmosphere.

**GAS-TIGHT HEAT
FLUX CELL**

CALIBRATION WITH
OMNICURE S2000

**PRECISE CONTROL OF
ATMOSPHERE
COMPOSITION (MFC)**

SOFTWARE TRIGGER
AND CONTROL

**ADJUSTABLE LIGHT
GUIDES FOR SAMPLE
AND REFERENCE**

AUTOMATIC COVER LIFT
FOR EASY HANDLING AND
REPRODUCIBLE TEST RESULTS

**ADAPTABILITY OF
VARIOUS COMMERCIAL
LAMPS TO THE DSC**

ADJUSTABLE LIGHT GUIDES
WITH DEFINED DISTANCE
TO THE DSC CELL

**AUTOMATIC SAMPLE
CHANGER UP TO 192
SAMPLE POSITIONS**

CONTROL OF
IRRADIATION TIME

**BROAD TEMPERATURE
RANGE**

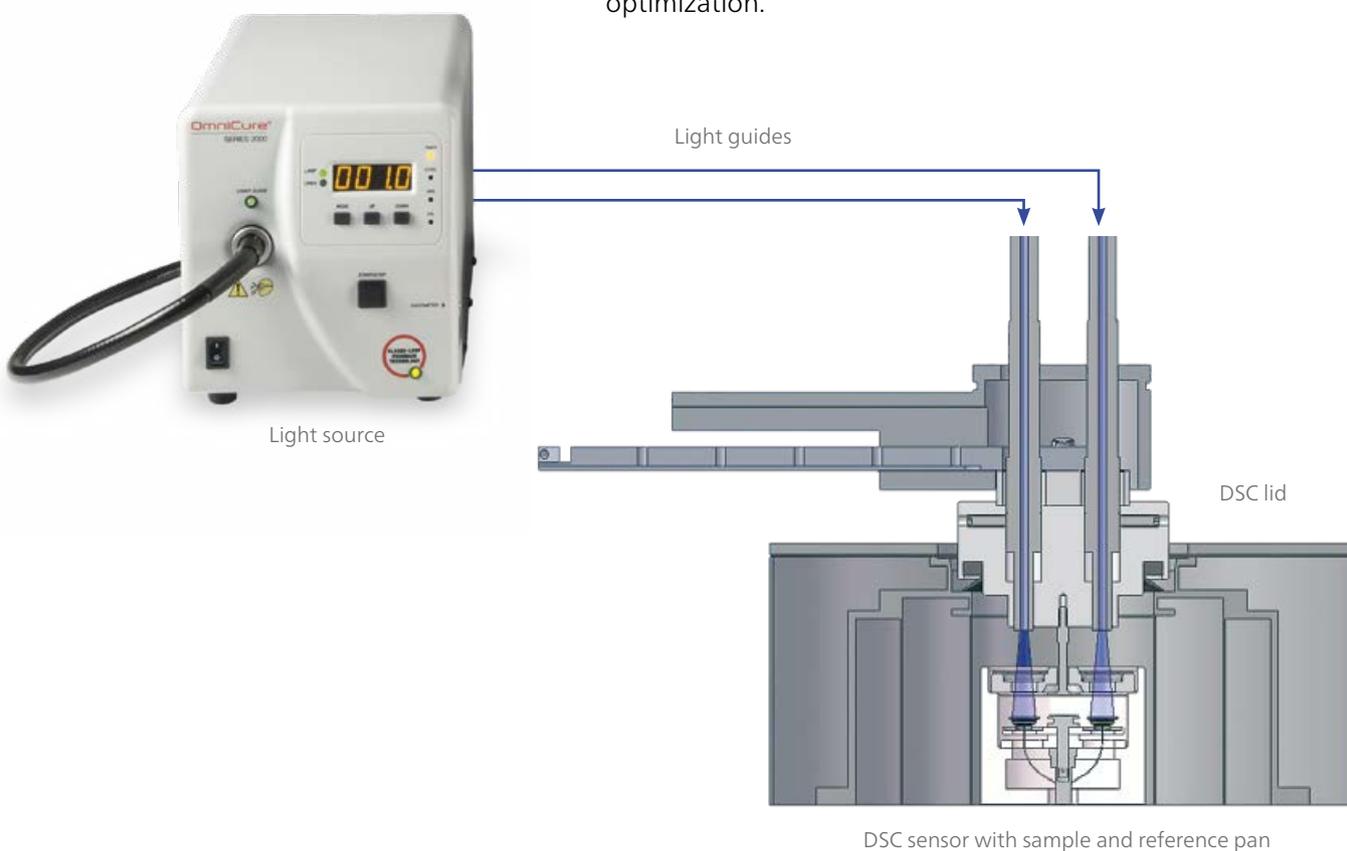
TRENDSETTING TECHNOLOGY

Photo-DSC Operation

Differential scanning calorimetry (DSC) is a thermoanalytical method in which the difference between heat flow into a sample and into a reference, when subjected to a controlled temperature program, is quantitatively determined (definition based on DIN 51 007, ISO 11357-1 or ASTM E472).

In photo-DSC measurements, both the sample and reference are located in one furnace and are irradiated simultaneously. The fiber optics are firmly installed in the lid to ensure reproducible distances between the fiber optics and the sample and reference.

The DSC measurement software communicates with the UV lamp, triggering its pulses and – for certain models (see next page) – automatically controlling the pulse length and intensity. During the course of a measurement, the signals detected are the sample temperature and heat flow difference. By integrating the heat flow peak, the heat of curing can be determined, providing meaningful data for development or process optimization.



UV Light Sources – You Have the Choice

Recommended are mercury lamps such as DELOLUX 04 or OmniCure® S 2000 which allow for the selection of wide and narrow ranges; these should have filters in the UV-A range between 280 nm and 315 nm and in the UV-B range between 315 nm and 500 nm.

However, other commercially available lamps can also be adapted. For optimized curing, a defined wavelength (e.g., 447 nm) can be used with diode laser systems or high-power LED (e.g., 365 nm, 400 nm or 460 nm) – depending on the resin to be cured.

The OmniCure® lamp is fully software controlled; selection of temperature, light intensity, wave length and exposure time can be carried out by the DSC *Proteus*® software.

The gas-tight construction of the DSC instrument allows for easy measurement of the cross-linking of paints; this process is often sensitive to oxygen, which can act as an inhibitor.

Software Features

- Light source can be triggered and controlled by DSC measurement software (time control and intensity control)
- Radiation impulse is selectable by segment, by means of activation/deactivation through a check box in the temperature program
- Selectable delay time for irradiation
- Evaluation of effective heat of reaction by means of curve subtraction and time offset capability
- Determination of relative or absolute integral (degree of conversion)

Technical Specifications

Photo-DSC 204 F1 Phoenix®

| | | |
|----------------------------------|---|-------------------------|
| Temperature range | <ul style="list-style-type: none"> ▪ -100°C to 200°C with irradiation module ▪ Standard DSC measurements: -180°C to 700°C | |
| Crucibles | Open aluminum | |
| Automatic Sample Changer (ASC) | up to 204 sample pans (option) | |
| Recommended Hg-lamp types | DELOLUX 04 | OmniCure® S 2000 |
| Max. power | 9.9 W/cm ² | >10 W/cm ² |
| Wavelength range | 315 nm to 500 nm* | 320 nm to 500 nm* |
| Irradiation time | 0.1 s to 1000 s | 0.2 s to 1000 s |
| Orifice diameter | 8 mm, 4 mm, 2 mm | 8 mm, 4 mm, 2 mm |
| Life time (lamp) | 1500 h | 2000 h |

* Variable and extendable with various filters

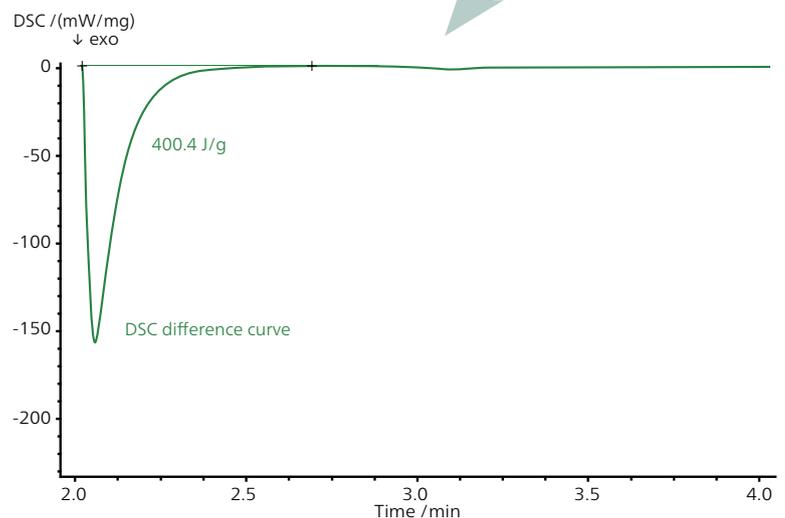
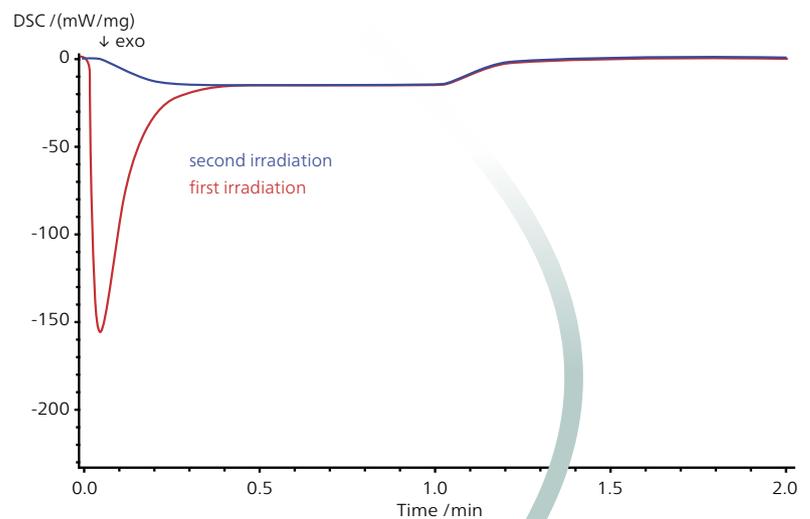


APPLICATIONS

- Paints/Coatings
- Adhesives/Inks
- Dental Composites
- Pharma/Cosmetics
- Food/Chemicals

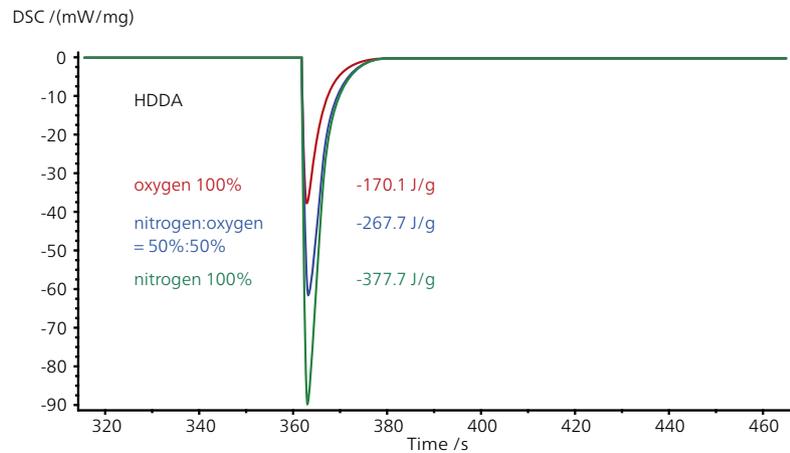
Photo-DSC Measurement and Evaluation

The sample and reference are irradiated with UV light at a constant temperature until the sample is cured. Afterward, the cured sample and the reference are irradiated for a second time for the same duration and at the same temperature. Finally, the difference between the first and second irradiation is calculated to determine the pure heat of reaction (curve subtraction).



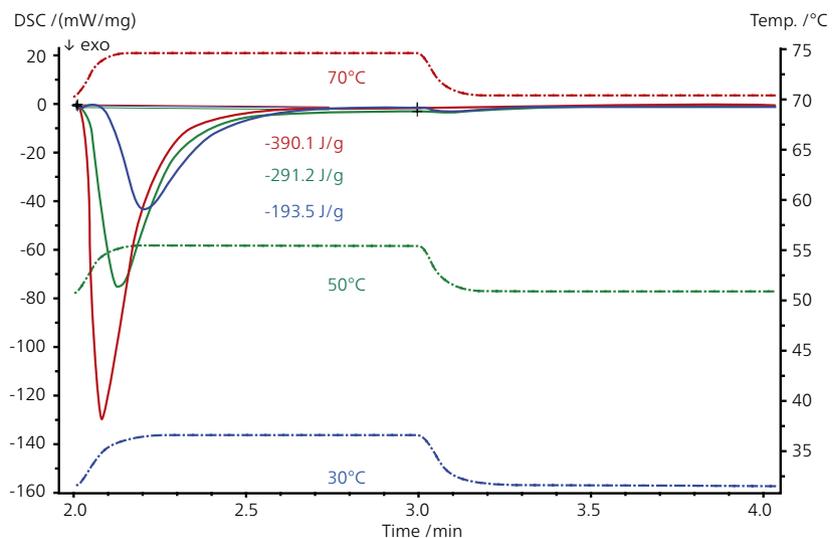
Paints under Different Atmospheres

The irradiation for 1s of a sample of hexanediol diacrylate (HDDA) was investigated using three different atmospheres. The heat of crosslinking was at its highest under an inert atmosphere of 100% nitrogen (green curve) with 378 J/g. A mixture of 50% nitrogen and 50% oxygen yielded 268 J/g (blue curve); a pure oxygen atmosphere, only 170 J/g (red curve). There is obviously a competitive reaction due to the influence of oxygen.



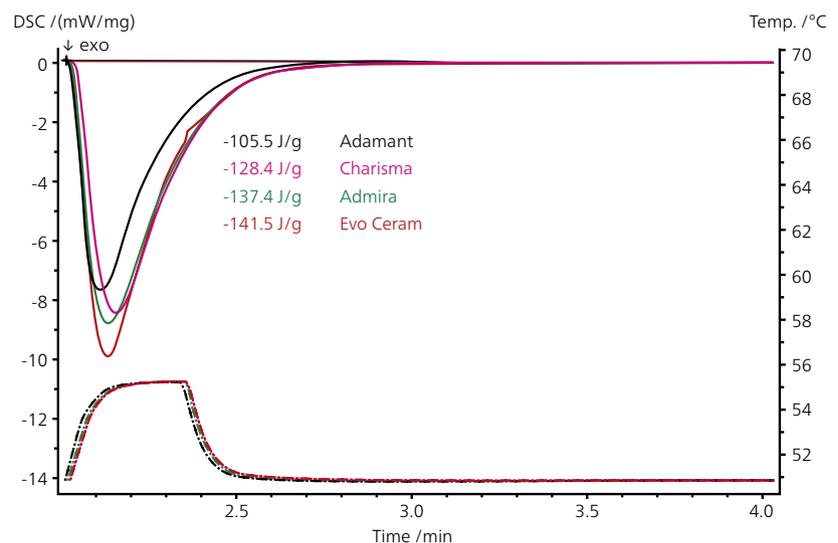
Adhesives at Different Temperatures

This fluorescent one-component modified epoxy resin is activated with visible light of 400 nm to 500 nm. A cationic curing mechanism allows the adhesive to cure after having assembled the different components. A higher temperature accelerates this reaction. The adhesive is used for bonding metal, glass, or plastics and especially for stress-equalizing bonding or sealing products.



Different Dental Composites

In dental applications, light-curing dental composites are used as restoratives (fillings) or veneering materials. The materials are generally composed of methacrylate systems such as bis-glycol-dimethacrylate (bis-GMA) or urethane dimethacrylate (UDMA). Additional monomers are used as diluent or to guarantee the cross-linking abilities of the resin. Inorganic fillers up to 80 weight percent improve the mechanical properties and reduce shrinkage during cross-linking.





The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

Proven Excellence.■

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