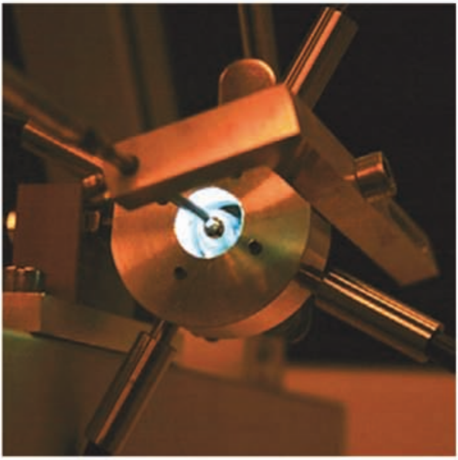

Application Lab for Volume Dilatometry

Measurements of the Specific Volume and the Thermal Volume Expansion of Materials

innomat

InnoMat GmbH

Problem definition

Volume shrinkage during the curing of resins causes problems in many applications, e.g. for moulding compounds, filling materials and adhesives.
During curing and their lifetime, reactive resins pass through various temperature cycles and thereby undergo significant changes in specific volume.

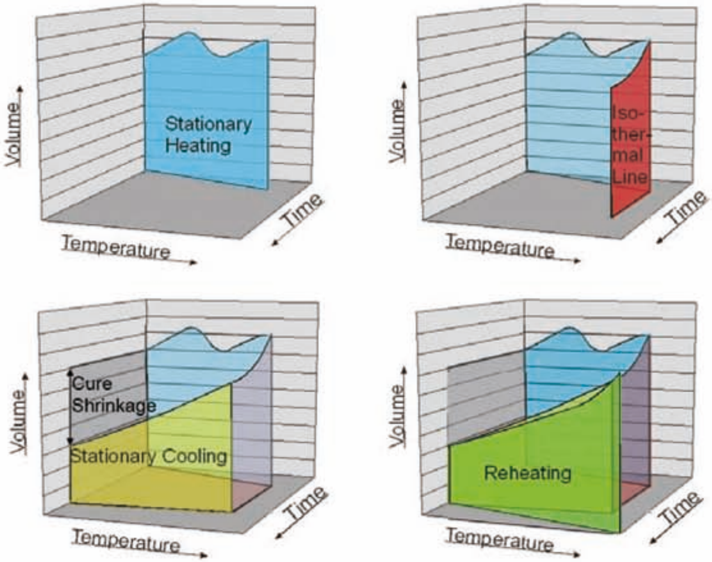

Fig. 1: *The gradient of the volume-temperature-time function in principle of a reactive resin during and after curing.*

Thus, for the development of new materials and for process control, a reliable material characterisation is very important. Nowadays, increased reliability requirements on components often demand life time predictions from simulation methods. Such computations require precise data of the volume change from resins curing and its successive temperature cycles.

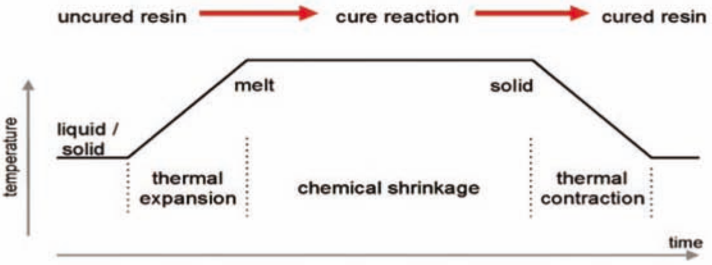

Fig. 2: *Measuring over all phases.*

Method

The InnoMat GmbH has developed a low-cost and efficient measuring device which records the specific volume of a reactive resin over all its life cycles in only one measuring process – Volume Dilatometry.

Thus, not only the volume shrinkage of the initial state of the liquid resin and the cured final state is recorded but a continuous automated recording of the volume-temperature-time function during curing is realised.

Likewise, the volume expansion of the cured resin can be determined. The measurements can be carried out under application and lifetime conditions.

With volume dilatometry, the following properties of materials can be determined:

- volume shrinkage (%) during curing,
- coefficient of thermal expansion of both the liquid and the solid (cured) resin,
- specific volume or density as a function of temperature and time,
- glass temperature.

Furthermore, volume dilatometry is ideal for monitoring reaction kinetics.

Depending on the specifications of the sample material different measurement setups are available by variation of the confining fluid (mercury, Galinstan, oil) and the curing method (thermal-induced cure up to 250°C, (UV, blue-) light-induced cure).

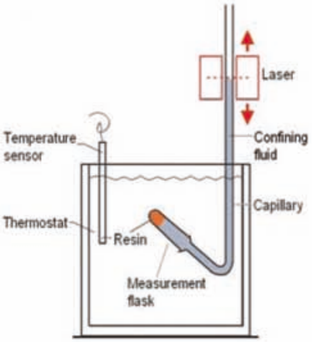

Fig. 3: Setup of a volume dilatometer for thermal curing resins.

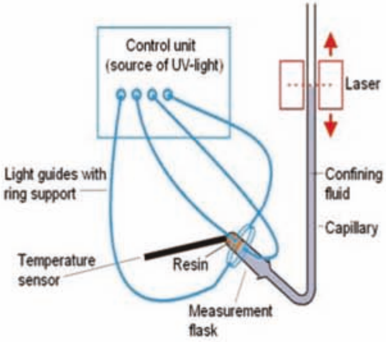

Fig. 4: Setup of a volume dilatometer for light-curing resins.

Equipment capabilities

- Temperature range: 25°C – 250°C.
- Individual temperature-measurement profiles according to the customers' needs / application process.
- Measurement at normal pressure.
- Small sample quantity (0.5 - 1g).

Field of application

- Determination of the volume shrinkage of filled and unfilled as well as light-curing resins.
- Development of low-shrink resins.
- Obtaining input data for FE simulation.
- Optimisation of technological (curing) processes.
- Observing reaction kinetics.

Measurement examples

In Figure 5, the specific volume for the entire curing cycle of two different hot curing cyanate ester resins is represented.

The marked sections of graph given below are:

1 Heat phase
2 Melting of crystalline monomer
3 Isothermal curing
4 Cooling phase

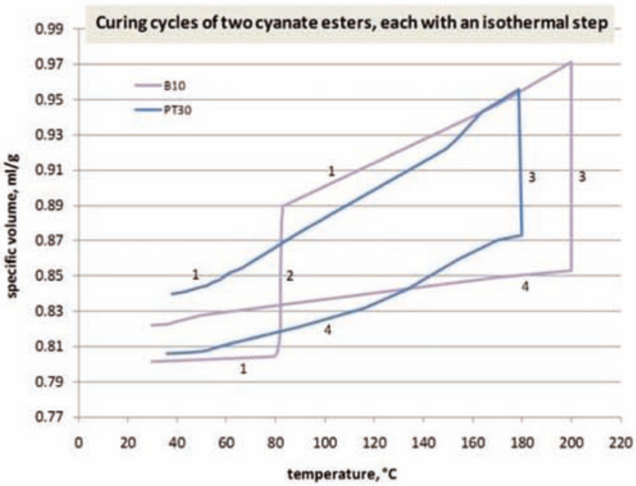

Fig. 5: Curing cycles of two hot curing cyanate ester resins, each with one isothermal step.

Measurement examples

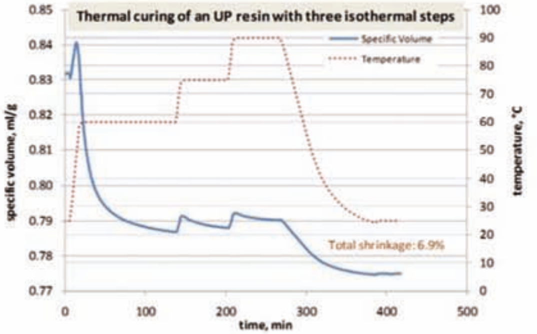

***Fig. 6:** Thermal curing of an UP resin with three isothermal steps.*
Figure 6 shows the gradient of the specific volume for a curing cycle of a hot curing UP resin. Shortly after reaching 60 °C the resin starts to shrink. The shrinkage is not finished after the first isothermal step. Thus, in every isothermal step a continuing shrinkage is detectable.

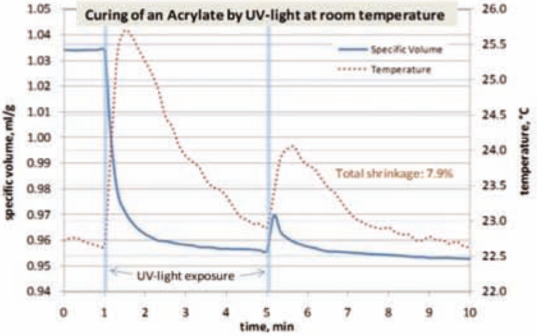

***Fig. 7:** Curing of an acrylate by UV light at room temperature.*
Figure 7 displays an example of a light induced shrinkage measurement. At the first UV light exposure of the material, a considerable shrinkage occurs. To insure that all reactive species have reacted, a second UV light exposure is performed. The exothermal nature of polymerisation is clearly detectable.

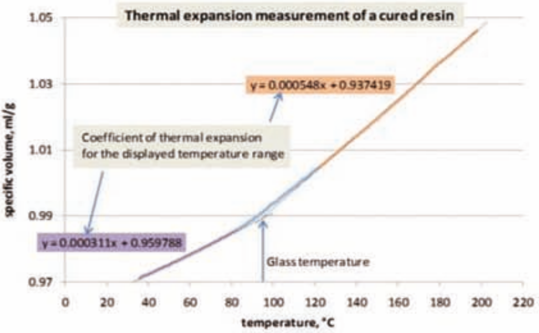

***Fig. 8:** Volume dilatometric measurement of the thermal expansion.*
Figure 8 gives an example for a measurement of the volumetric thermal expansion coefficient. These measurements are usually performed on cured resins with a heating of 1 K/min. The graph displays different volumetric coefficients of thermal expansion below and above the glass transition temperature.

Contact

InnoMat GmbH is a service provider in the field of research and development of innovative materials. Among others, innovative materials are materials with carefully balanced, often opposed properties, materials for new technologies and materials for system solutions. However, our main focus lies in polymeric materials.

InnoMat GmbH offers a wide range of services in the field of innovative materials:

- research and development, e.g. 3D-structured fibre-reinforced plastics, nano-particle-filled polymers, new core materials
- prototyping and small series production, e.g. prepregs, laminates, sandwiches
- material characterisation, e.g. volume dilatometry
- material studies and expertise on innovative scientific-technical questions and developments
- material assessment including simulation of material data and lifetime forecasts
- apparatus and prototype construction
- seminars and conferences, e.g. organisation of the “Thermosets 2009” conference
- project arrangement and support
- support of postgraduates and PhD students

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