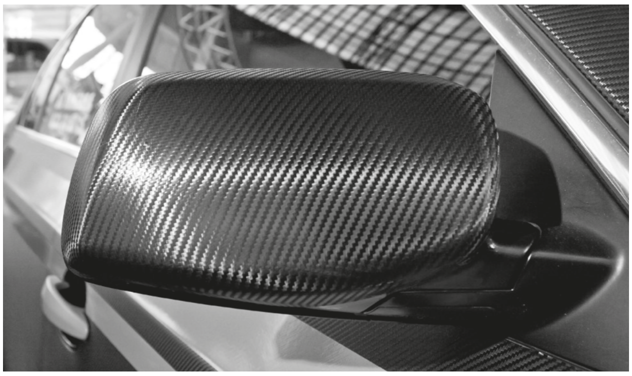
WHITE PAPER

Curing of Thermosetting Resins for Fiber-Reinforced Parts



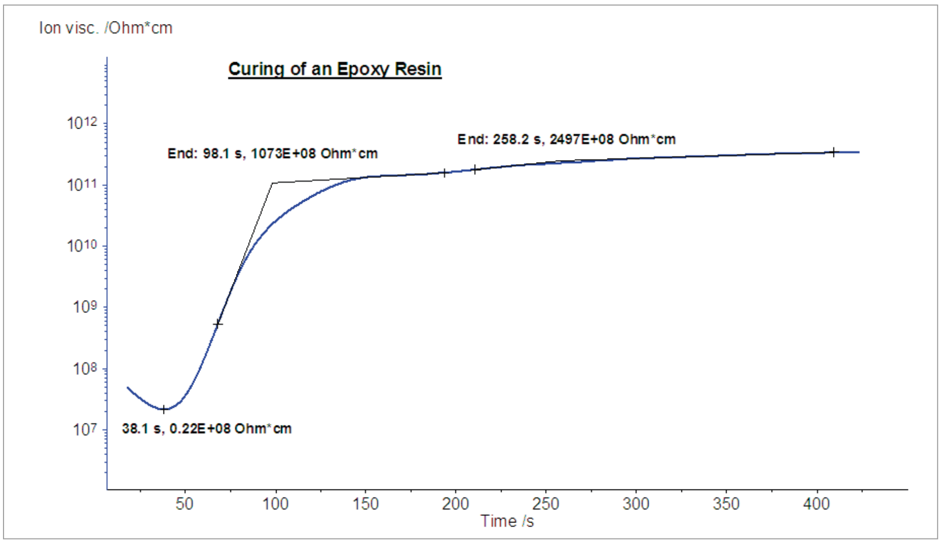
1 Use of carbon-fiber reinforced plastics in automotive industry

A variety of questions may arise during the curing process for thermosetting resins. At which temperature, or after how much time, does the resin begin curing? How high is the reactivity? When is curing complete? Is the curing profile identical for every position of the mold? How can the curing cycle be optimized in order to save energy and costs? Is there any potential for post-curing?

The answers to questions such as these often serve as catalysts for new developments in a variety of industries. For example, epoxy, polyester and polyurethane resins are increasingly being used in conjunction with fiber materials for reinforced parts such as spoilers or panels in the automotive industry (fig. 1). The primary objective is to find substitutes for traditional steel – and nowadays, even for light metals such as aluminum and magnesium – to create lighter cars without compromising the necessary stiffness across a broad temperature range. Such "high-modulus composites" have already proven their performance in the aircraft and aerospace industries. However, cycle times for the production of car parts are much faster and the thermosetting resin must therefore cure in only a few minutes, not a few hours.

Cure Monitoring by Dielectric Analysis (DEA) – Method and Instrumentation

Dielectric Analysis (DEA), according to ASTM E 2038 and E 2039, allows for the measurement of changes in the dielectric properties of a thermosetting resin during curing. The liquid or pasty resin must be placed in direct contact with two electrodes comprising the dielectric sensor. A sinusoidal voltage (excitation) is applied and the resulting current (response) is measured, along with the phase shift between voltage and current. These values are then used to determine the ion mobility (ion conductivity) and the alignment of dipoles. In turn, the dielectric properties of permittivity ε' and loss factor ε" are calculated from these effects. Of primary interest with regard to curing is the ion viscosity. This is the reciprocal value of the ion conductivity, which is proportional to the loss factor.
Use of the DEA technique is not limited to the lab environment; it can also be applied to in-situ curing in the mold under processing conditions. For production monitoring and process control, a specific ion viscosity value can be programmed to trigger de-molding when the part being produced is sufficiently cured. This reduces cycle times and increases throughput, thereby lowering costs and potentially allowing lower prices to be charged for the finished part.



2 DEA measurement result for the curing of an epoxy resin in a mold

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The plot in figure 2 depicts the curing of a liquid epoxy resin at a mold temperature of 80°C, measured at a frequency of 10 Hz. Initially, the ion viscosity drops due to the increase in temperature; the minimum value exhibited represents the lowest viscosity and therefore the best flow behavior. Curing begins immediately thereafter, with an increase in ion viscosity of more than four orders of magnitude. The slope of this high increase correlates with the high reactivity of the resin (curing speed). After 258 s, a small second curing step is detected. After approx. 300 s, the ion viscosity curve levels out, signaling that curing is practically completed and that the cured epoxy part can be safely de-molded at this time.

DEA 288 *Epsilon* – Cure Monitoring of Thermosetting Resins in Composites – Even In-Situ

3 The versatile DEA 288 *Epsilon* (lab version) for cure monitoring

4 Slim version of the DEA 288 *Epsilon* with up to 2 channels

5 Industrial version of the DEA 288 *Epsilon* with up to 8 channels

DEA 288 *Epsilon* – One Modular Concept to Meet Your Needs

The modular concept of the brand-new DEA 288 *Epsilon* allows for the study of the curing behavior of thermosetting resins, adhesives, paints and coatings in nearly any application. The lab version (fig. 3), with up to 8 channels can be used in conjunction with a newly designed furnace for research & development which reaches temperatures up to 400°C. The industrial versions are intended for production monitoring and process control, and are designed with up to 16 channels (figures 4 and 5 depict models with up to two and up to eight channels, respectively). The 8- and 16-channel versions can also be implemented in a 19” rack. The industrial devices are connected via rugged extension cables and connectors to various sensor types which are located in molds, presses, ovens or autoclaves.
Reusable tool mount sensors (TMS) in a variety of geometries and temperature/pressure ranges can be applied to molding processes such as RTM (Resin Transfer Molding) or SMC (Sheet Molding Compound). For composites with carbon fibers, the sensor must be protected with a glass-fiber cloth; this allows the liquid resin to penetrate while still preventing contact with the electrically conductive carbon fiber, since such contact would create a short circuit and therefore impede curing results. For paints, adhesives and thin coatings, various implantable (disposable) comb sensors are available, tailored to different application needs.

DEA 288 *Epsilon* – At a Glance

- Modular concept for the laboratory (R&D) and/or for production processes
- Simultaneous multi-channel cure monitoring (up to 16 channels)
- Multi-frequency scans (from 1 mHz to 1 MHz)
- Extremely fast data acquisition time (<5 ms)
- A variety of reusable in-process TMS sensors
- A variety of disposable comb sensors
- New *Proteus*® software for controlling a press, an oven or a UV lamp

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