Simultaneous Rheological-Dielectrical Characterisation Using the HAAKE MARS Rheometer Platform

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The result of a rheological test tells us how a substance behaves under a given stress or strain. What it does not tell us is the reason why, rheology is a macroscopic method and regards the sample as a homogeneous amount of substance. To be able to explain the rheological behaviour, we need additional information about the sample's microstructure, supplied by an additional "microscopic" method. Microscopy, FT(IR) spectroscopy, Raman spectroscopy or dielectrical analysis (DEA) are well established microscopic methods.

Using the traditional approach, running two independent test methods on two different samples, always bears the risk that due to the different ways of sample preparation or sample history in general, the tests happen under different conditions and the test results are not then really comparable. This risk can be avoided by running both test methods on the same sample simultaneously using a combination of two analytical methods in one setup. Subsequently, the two resulting data sets can be correlated without any doubt since they have been collected at the same time on the same sample. As an additional benefit, this approach saves a significant amount of time since only one sample has to be prepared and both tests run at the same time.

The Thermo Scientific™ HAAKE™ MARS™ rheometer can be equipped with parallel plate geometries with an integrated DEA sensor for simultaneous investigations on rheological and DEA behaviour. This method combination can be used in temperature range up to 220 °C using a temperature chamber (Fig 1).

Typical applications, which can be investigated using this method combination, are the curing behaviour of reactive thermosets, composites, glues, inks, coatings and dental materials.

Depending on the viscosity range, plates with different diameters are available. The sample is loaded into a plate/plate geometry, like for classical rheological tests covering the DEA comb sensor on the lower plate. During the test, a sinusoidal voltage (excitation) is applied and the resulting current (response) is measured together with the phase shift between voltage and current. Both current and phase shift are used to determine the loss factor, based on which

Fig.1: Parallel plates geometry with integrated DEA comb sensor in a temperature chamber for the HAAKE MARS rheometer.

the ion mobility (ion conductivity) is calculated. For curing reactions, the inverse ion conductivity is used, the so-called ion viscosity.

It is essential for simultaneous measurements and their evaluation to present both results in one data file. For this purpose the rheological results can be automatically exported into an ASCII file with the Thermo Scientific™ HAAKE™ RheoWin™ Software at the end of a measurement. This format allows the import into the *Proteus*® Software from NETZSCH. By this means both results can be displayed in one graph, as for example shown in Fig. 2.

Whilst with a rheometer the start and the progress of a curing reaction can be measured very well, the DEA delivers valuable additional information, e.g. if the curing reaction is completed or if there still is potential for post-curing. Based on the ion viscosity the progress of the reaction can be read from the data and the degree of curing can be determined (conversion curve). On top of that, the DEA is capable of expanding the frequency range from mHz to MHz.

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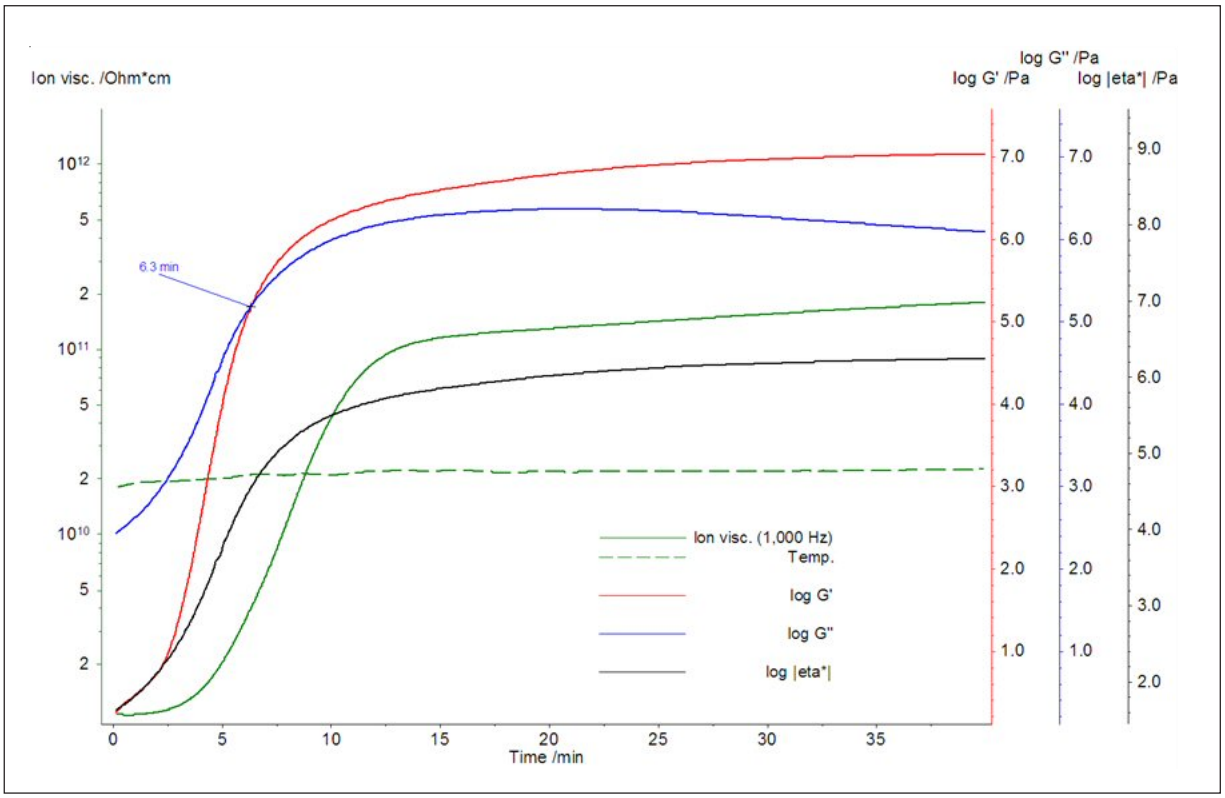

Fig. 2: Time-dependent curing reaction of a two component epoxy resin described with the rheological moduli (red and blue) and the dynamic viscosity (black) as well as the simultaneously measured ion viscosity (green) by DEA at room temperature and a measuring frequency of 1 Hz.

Order information

Triangular adapter plate for HAAKE MARS including lower holder with plate and integrated DEA sensor as well as thermocouple.
With different plate diameters available:
603-1200 20 mm*
603-1201 25 mm*
603-1230 35 mm*
*upper plate not included.

Necessary configuration

HAAKE MARS with controlled test chamber CTC,
NETZSCH DEA 288 *Epsilon*

Material Characterization

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