Dielectric Cure Monitoring Epsilon Systems – Technical Data and Service

	DEA 230/2	DEA 230/1	DEA 230/10	DEA 231/1
Min. Sampling Time/s	2	4	4	0.055
Frequency Range/Hz	10^{-3} to 10^{5}	10^{-1} to 10^{5}	10^{-1} to 10^{5}	10 to 10^{4}
Log Conductivity Range/(S/cm)	-16 to 0	-13 to 0	-13 to 0	-11 to –4
Log Ion Viscosity Range/Ωcm	0 to 16	0 to 13	0 to 13	4 to 11
Channels	2	1	10	1

The NETZSCH Dielectric Analysis (DEA) Epsilon Systems are used in over 450 corporations, universities and research institutions worldwide. In addition to complete Dielectric Cure Monitoring Epsilon Systems, NETZSCH offers other specially designed products such as DEA 234 CurePak™ (thru-oven cure analyzer), small lab furnace and pneumatic lab press.

The various DEA systems can also be operated with other thermoanalytical instruments of the successful 200 Series such as DSC 204 *F1 Phoenix*®, TG 209 *F1 Iris*®, TMA 202 or DMA 242 C for polymers and organics or with high-temperature instruments of the 400 Series including e.g. STA 449 C *Jupiter*®, Dil 402 C or DSC 404 C *Pegasus*®.

NETZSCH offers the extensive services of our applications and accredited contract testing laboratory specializing in thermal analysis and thermophysical properties.

Technical data subject to changes

Leading Thermal Analysis.

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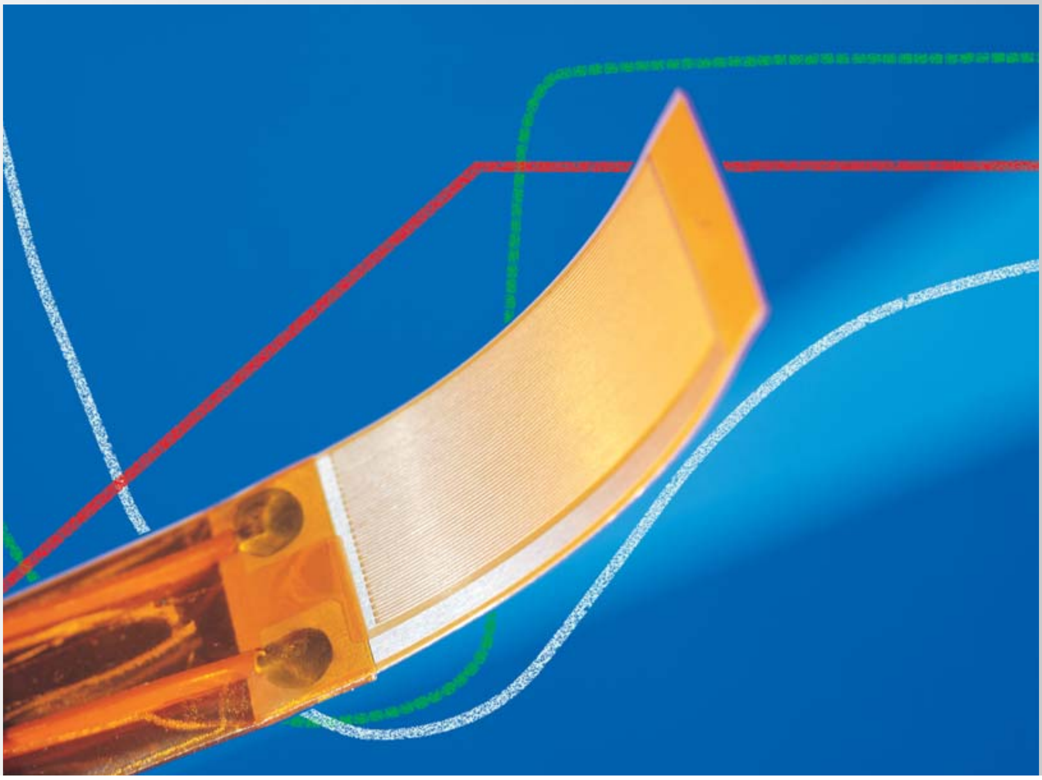


DEA

Dielectric Cure Monitoring

Epsilon

Leading Thermal Analysis.

Dielectric Analysis (DEA) for Cure Monitoring and More...

Dielectric Cure Monitoring and Dielectric Analysis (DEA) are techniques for investigating the processing behavior and physical and chemical structure of thermosetting resins and other polymers by measuring the changes in the dielectric properties of the material.

In a typical test, the sample is placed in contact with two electrodes (the dielectric sensor) and a sinusoidal voltage (the excitation) is applied to one electrode. The resulting sinusoidal current (the response) is measured at the second electrode. The response signal is attenuated in amplitude and shifted in phase in relation to the mobility of the ions and alignment of dipoles. Dipoles in the material will attempt to align with the electric field and charged ions (present as e.g. impurities) will move toward the electrode of opposite polarity. The dielectric properties of permittivity ε' and loss ε" factor are then calculated from this measured amplitude and phase change.

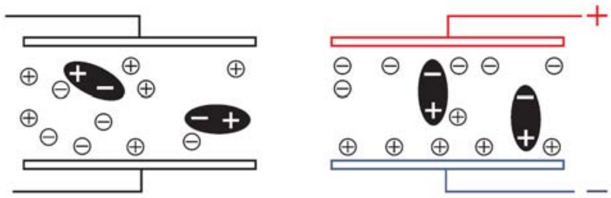
Studying the changes in the dielectric properties of the sample under processing conditions or as a function of time or temperature (as detailed in ASTM E 2038 or E 2039) provides information about the polymer:

- **Viscosity**
- **Cure Rate**
- **Cure State**
- **Cure Time**
- **Degree of Cure**
- **Glass Transition and other Polymer Transitions**
- **Diffusion Properties Aging & Decomposition Effects**

Application areas include:
- **Research**
- **Process Development**
- **Process Monitoring**
- **Process Control**
- **Quality Control**
- **Quality Control**
- **Quality Assurance**

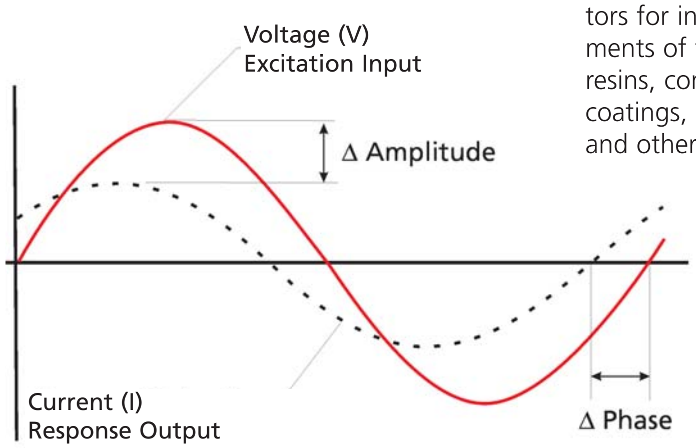
The DEA 230 Epsilon can be connected to the Dynamic-mechanical analyzer DMA 242 C to perform **Simultaneous DMA-DEA** experiments. Combining these complementary techniques can provide additional information to fully characterize the processing behavior of polymers.

Remote dielectric sensors allow testing to be performed in actual or simulated processing conditions as well as in the laboratory. This versatility helps bridge the gap between the laboratory, process development, production and quality control since the same sensors and instruments can be used across these areas. Dielectric sensors are routinely located in ovens, presses, molds, autoclaves, and batch reactors for in-process measurements of thermosetting resins, composites, paints, coatings, oils, lubricants, and other polymers.



Dipolar and Ionic Behavior

The NETZSCH Dielectric Cure Monitoring Systems are based on technology and research work originated at the Massachusetts Institute of Technology (MIT) and further developed by Micromet Instruments. We have over 25 years of experience in supplying dielectric cure monitoring systems to the polymer processing industries.

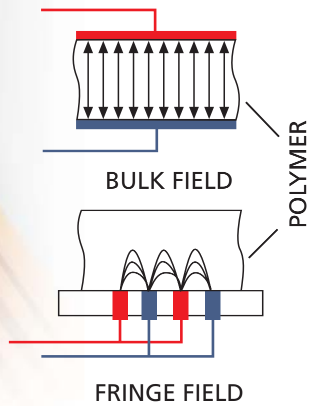


Sensitive and Versatile Sensors for Every Application - Including In-situ Curing

Implantable (disposable) Sensors:

- Low Conductivity Integrated Circuit (IC) Chip Sensor (up to 250°C)
- High Conductivity Sensor (up to 300°C)
- Interdigitated Electrode (IDEX) Mid Conductivity Sensors (up to 375°C)
- Filtered IDEX Sensors (up to 375°C)
- Micron Sensors (MS, up to 375°C)

The dielectric sensors are available in a wide variety of configurations to meet the requirements of a wide range of polymer processing applications. Sensor types consist of either Implantable or In-process varieties. Implantable sensors are single use sensors that can be placed at the desired location in a part or simply coated with material. In-Process Sensors (TMS™) are permanently mounted in a mold, tool, or batch reactor and have virtually unlimited lifetimes. Sensors are available that can withstand high temperatures (up to 400°C) and high pressures of over 350 bar (35 MPa).
Most sensors are comprised of two interdigitated comb electrodes on an inert substrate. The resulting field of measurement is a fringing pattern which makes a localized measurement of the dielectric properties near the sensor/sample interface. Sensors are also available that use a parallel plate electrode configuration and measure the bulk properties of the sample.



Rugged cabling (up to 30 meters in length) is available to make reliable connections between the sensors and measurement system. Vacuum-tight feedthrough glands are available for installing cabling into autoclaves and other pressurized vessels.

In-Process (reusable) Sensors:

- Tool Mount Sensors (TMS™) for Molding Processes
- Monotrode Sensors for Molding Processes
- Dielectric Fluid State (DFS) Sensors for Liquid Processes
- Autoclave Feedthrough Gland for Vacuum-Tight Connections

Wide Product Range to Meet Your Needs

The NETZSCH Dielectric Cure Monitoring Epsilon Systems encompass the wide range of customer needs and applications.

Systems are available that sweep a wide range of measurement frequencies (0.001 Hz to 100 kHz) in order to accurately measure the large change in dielectric properties that can occur during the cure of a thermosetting resin. Other systems are designed to make high-speed measurements (up to 55 ms) to measure fast curing resins. Systems are available that can measure up to 10 dielectric sensors during an experiment. All systems are equipped to measure one or more thermocouples so that the temperature data can be collected along with the dielectric data. Some systems can measure other analog inputs from pressure sensors, LVDTs, or other sensors.

The NETZSCH-DEA product line provides systems for every application:

DEA 230/2 Epsilon
High-performance dielectric measurement system designed for both laboratory dielectric analysis of polymers and in-process cure monitoring of thermosets. The DEA 230/2 Epsilon is the only system that can utilize the **Low Conductivity Integrated Circuit Sensor.** This sensor provides highly accurate measurements even at lowest frequency of both loss factor and permittivity and for measuring the lowest conductivity values. The DEA230/2 Epsilon can measure the curing of most thermosetting resins, composites, paints and coatings with **2 dielectric sensors** (2 channels) in one experiment.

DEA 230/1 Epsilon
Single channel standard dielectric measurement system designed for cure monitoring of most thermosetting resins.

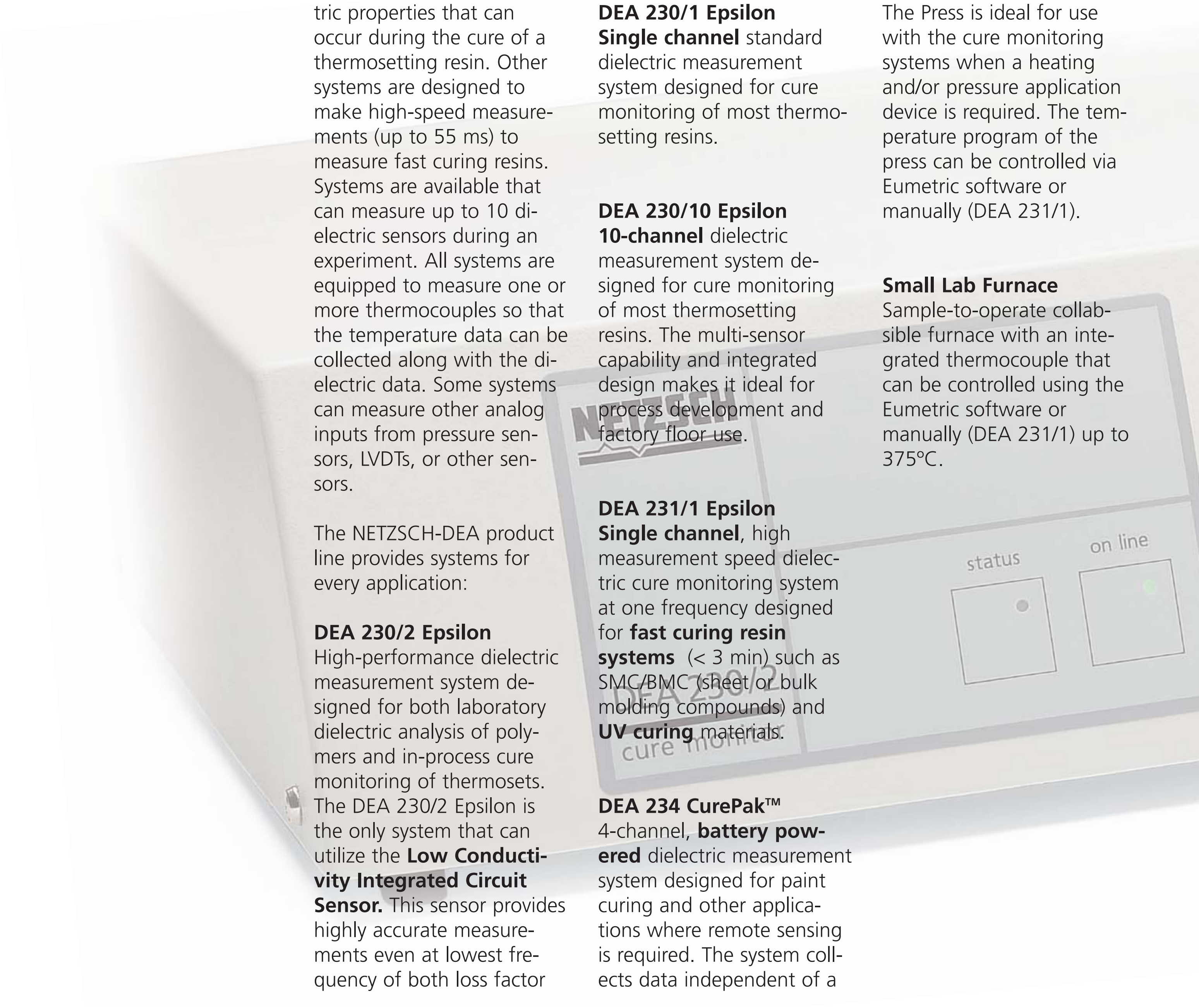
DEA 230/10 Epsilon
10-channel dielectric measurement system designed for cure monitoring of most thermosetting resins. The multi-sensor capability and integrated design makes it ideal for process development and factory floor use.

DEA 231/1 Epsilon
Single channel, high measurement speed dielectric cure monitoring system at one frequency designed for **fast curing resin systems** (< 3 min) such as SMC/BMC (sheet or bulk molding compounds) and **UV curing** materials.

DEA 234 CurePak™
4-channel, **battery powered** dielectric measurement system designed for paint curing and other applications where remote sensing is required. The system collects data independent of a controlling computer. Packed in an insulation box it travels through a conveyor oven along with the sensors.

Pneumatic Lab Press
Bench-top compression press designed for applying heat (up to 300°C) and force (up to 10 kN at a pressure of 5 bar) to the sample. The Press is ideal for use with the cure monitoring systems when a heating and/or pressure application device is required. The temperature program of the press can be controlled via Eumetric software or manually (DEA 231/1).

Small Lab Furnace
Sample-to-operate collabsible furnace with an integrated thermocouple that can be controlled using the Eumetric software or manually (DEA 231/1) up to 375°C.



Software - Powerful - Easy to Use

The NETZSCH Dielectric Cure Monitoring Epsilon Systems feature easy-to-use MS® Windows™ software packages.

The **Eumetric Software** is the standard software package for all DEA 230 Epsilon systems. This software features easy experiment set up, data collection and data analysis routines while maintaining maximum flexibility.

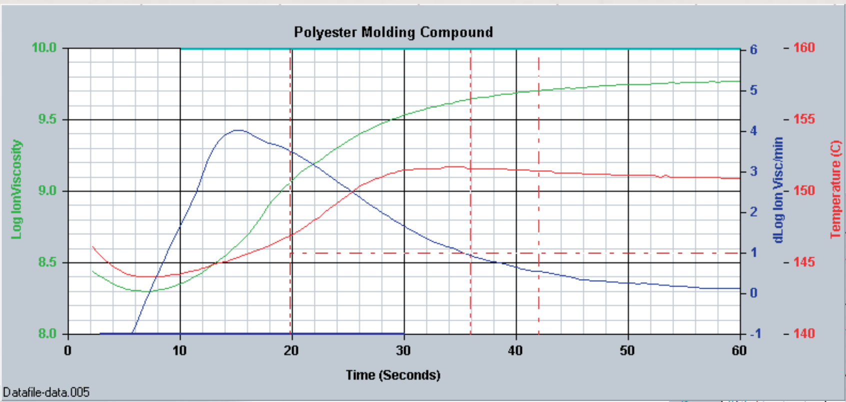
- Data Acquisition & Data Analysis Modules
- Frequency Sweeping
- Multi-Sensor Experiments
- Conductivity and Ion Viscosity (Resistivity) Calculation
- Virtually unlimited data file size
- Auto-scaling of axes & modification of the data collection parameters during an experiment
- Simultaneous data collection, data analysis and the operation of other Windows programs
- Exporting of data to other programs
- Cole-Cole Plots
- Cure Index data analysis routine
- Diffusion Coefficient analysis routine

The **CPC (Critical Point Control) Software** is the standard software package for the DEA 231 Epsilon systems. The CPC software can also be used with the DEA 230 Epsilon systems. The CPC software features the ability to identify 4 "Critical Points" on the dielectric cure curve to provide quantitative values for use in R&D, QA/QC, or event based process control.

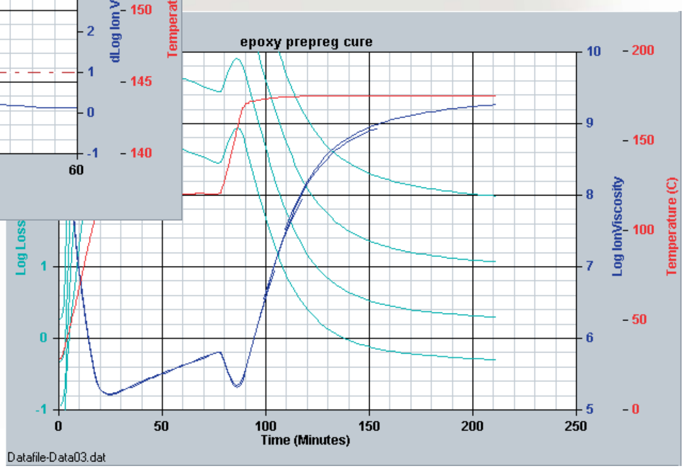
These Critical Points, along with other values associated with the dielectric and temperature data (and pressure and LVDT, if present) can also be stored in a database file for statistical analysis. Features include:

- Critical Point Identification & Storage
- Trigger issued at Critical Point (e.g. opening of a mold)
- Remote Start of Data Collection
- Specialized SQC (Statistical Quality Control) and Production Modules
- Database Analysis Software Module
- Filtering and Averaging of Data
- Data Export

All DEA results can be imported into our comprehensive Proteus® software for e.g. direct curve comparsion with DMA and/or DSC.



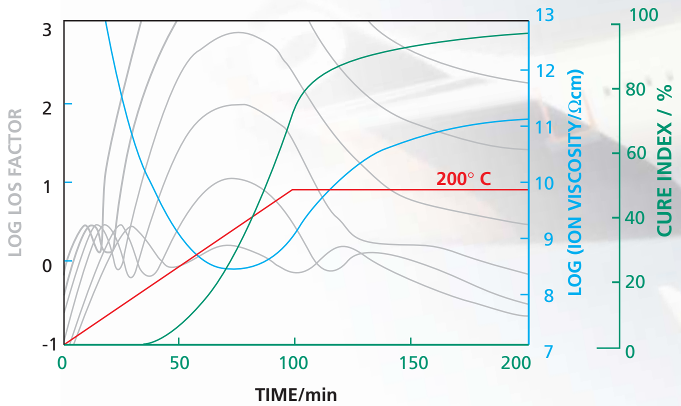
CPC Software



Eumetric Software

Various Applications

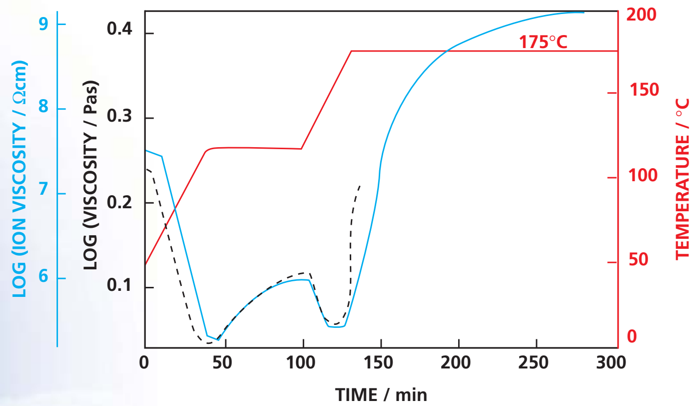
Epoxy Resin during Melting and Curing



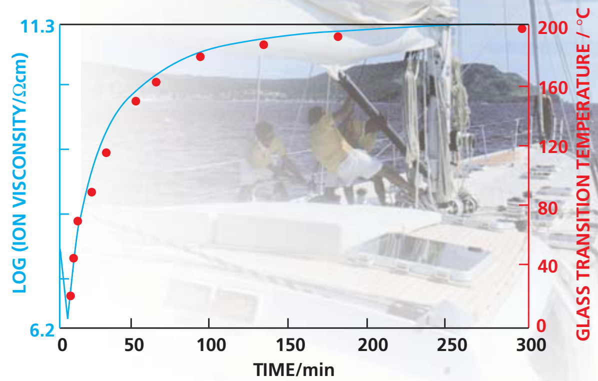
As temperature is ramped, the multi-frequency loss factor ε″ shows a series of dipole relaxation peaks as the epoxy resin passes through its glass transition temperature. The loss factor then rises rapidly as the epoxy melts, reflecting the dramatically increasing ionic mobility in the resin. The ion viscosity curve is derived from the ionic mobility component of the loss factor and is a frequency independent parameter related to the viscosity of the polymer before gelation and to rigidity after gelation. The ion viscosity initially decreases reflecting the effect of increasing temperature on the dynamic viscosity, η of the resin. The initiation of reaction, however, competes with the temperature effect by restricting mobility and results in a clearly defined viscosity minimum. After the minimum the ion viscosity rises, reflecting the increasing viscosity and cure state of the material. To illustrate the degree of cure, the dielectric cure index may be utilized.

Correlation with Viscosity Measurements

A comparison of DEA and rheometer data during the cure of an epoxy-graphite composite system shows that for the first 150 min the mechanical viscosity and ion viscosity curves nearly superimpose, demonstrating the clear correlation between the two properties. Early in the 175°C final hold, the epoxy resin goes through gelation and the mechanical viscosity can no longer be measured. The ion viscosity signal continues to follow the entire reaction, even as the material cures into a rigid glass.



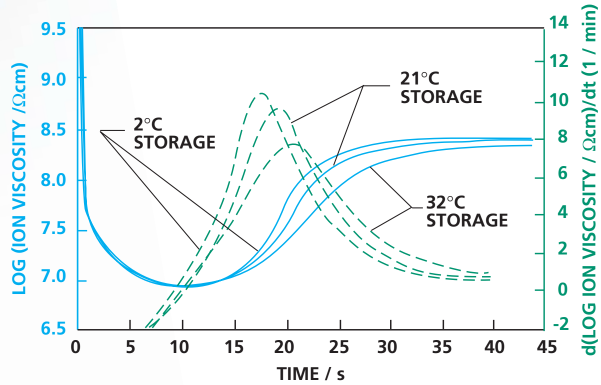
Correlation with Glass Transition Temperature



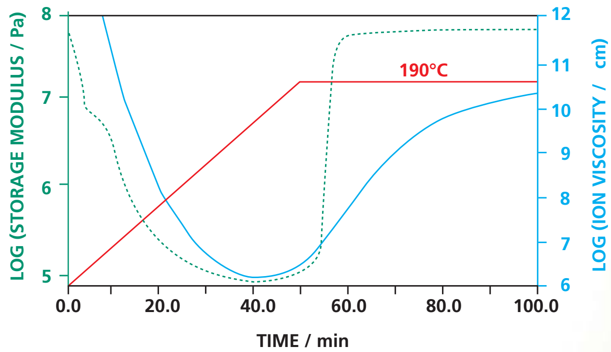
During the isothermal cure of an epoxy resin, the increase in log ion viscosity correlates well with the increasing glass transition temperature of the resin. This demonstrates that the ion viscosity can be used to continually monitor the increase in the cure state of a resin during processing. Since the dielectric measurements can be made in environments such as ovens, presses and autoclaves where most laboratory measurement instrumentation cannot, dielectric cure monitoring can provide previously unattainable information about the curing behavior of the material.

Dielectric cure monitoring provides a convenient method for R&D and QA/QC screening of different products, batches, or formulations of materials. Three samples of a fast curing polyester bulk molding compound (BMC) were stored for six weeks at temperatures of 2, 21, and 32°C. The logarithm of the ion viscosity (solid lines) and its 1st derivative (dashed lines) clearly identify differences in curing reaction rates.

R&D and QA/QC of Polyester Molding Compounds (SMC/BMC)

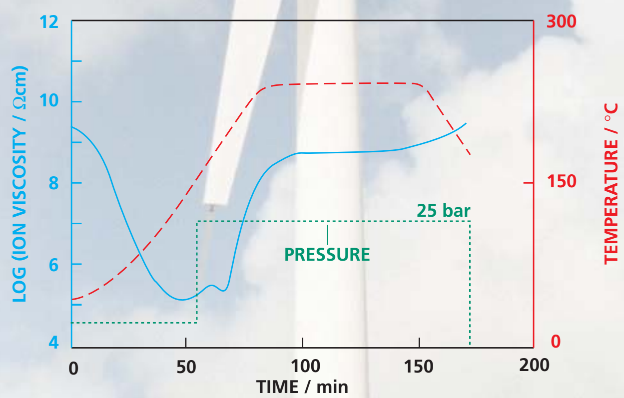


Simultaneous Dynamic-mechanical and Dielectric Analysis (DMA-DEA)



Combining the DEA 230 Epsilon with the DMA 242 C, the simultaneous measurement of a polymer's dielectric and dynamic-mechanical properties can be investigated in a single experiment. In this configuration, the compression sample holder of the DMA is used as parallel plate electrodes for the DEA. The techniques are complementary: DMA can clearly identify gelation and vitrification of the resin, while DEA is more sensitive in the viscosity minimum region and to the end of the curing reaction.

Production Monitoring and Control



The molding of a polyimide/graphite prepreg in a production press was automated using the ion viscosity to initiate processing steps. Pressure was applied when the ion viscosity detected the viscosity minimum of the material, the critical time when the resin would flow properly but the excess volatiles would not be trapped. An additional critical point was employed to trigger de-mold when the part was sufficiently cured, thus reducing cycle times and increase throughput. This is reflected in a much lower price for the molding.