NETZSCH

Proven Excellence.

Dynamic Mechanical Analysis – DMA 242 E *Artemis*

Method, Technique, Applications

Analyzing & Testing

Dynamic Mechanical Analysis

DMA 242 E *Artemis*

Dynamic Mechanical Analysis (DMA) is an indispensable tool for determining the visco-elastic properties of mainly polymer materials.
The new DMA 242 E *Artemis* combines ease of handling with the user-friendly *Proteus*® measurement and evaluation software. This makes it fast and easy to characterize the dynamic-mechanical properties as a function of frequency, temperature and time.
Its modular design along with a wide variety of sample holders and cooling systems allow the DMA 242 E *Artemis* to handle a broad range of applications and samples. Various add-on options make it the ideal device for any laboratory and a safe investment for the long-term.

Add-on Options

- Immersion bath for measurement of samples in a defined liquid medium
- Coupling to the dielectric analyzer DEA 288 *Ionic* for simultaneous measurement of the visco-elastic and dielectric property changes, e.g., during curing of a resin
- Coupling to a UV lamp for measuring curing on light-reactive samples
- Coupling to a humidity generator to determine the infl uence of humidity on the dynamic-mechanical properties of a material

Hang-down design
for easy accessibility, handling and changing of the different sample holders

Controlled gas flow (inert or oxidizing)
with optimal heat transfer on samples for defined measurement conditions

Various cooling options
Liquid nitrogen cooling to -170°C, Intracooler to -70°C, and air cooling down to 0°C

Controlled force range up to 24 N
for measurements of very stiff samples. Increased force resolution in the 8N measurement range.

A static travel range of 20 mm
allows for precise testing on materials which exhibit substantial changes in length during a DMA measurement. This is particularly important for the different static experiments available with the DMA 242 E *Artemis*; i.e., creep, relaxation and TMA mode

Over 30 different sample holders
for optimal adjustment of measurement conditions to material properties

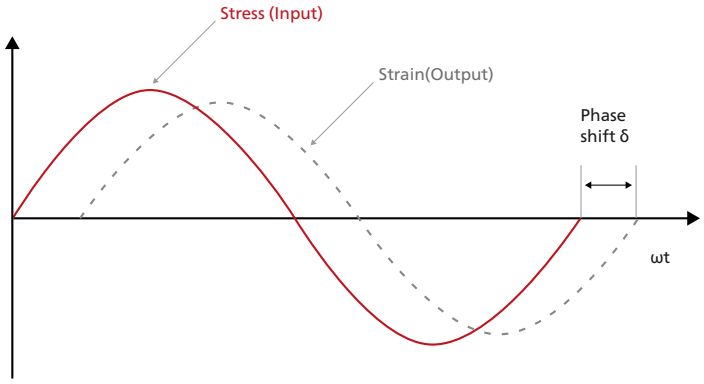
The Most Versatile DMA in the World

Dynamic Mechanical Analysis measures the visco-elastic properties of mostly polymer materials during a controlled temperature and/or frequency program. During the test, a sinusoidal force (stress σ) is applied to the sample (Input). This results in a sinusoidal deformation (strain ε) (Output).

Certain materials, such as polymers exhibit visco-elastic behavior; i.e., they show both elastic (such as an ideal spring) and viscous properties (such as an ideal dashpot).

This visco-elastic behavior causes shifting of the corresponding stress and strain curves. The deviation is the phase shift δ. The response signal (strain, ε) is split into an "in-phase" and an "out-of-phase" part by means of Fourier Transformation.

Functional Principle



DMA – Measurement principle

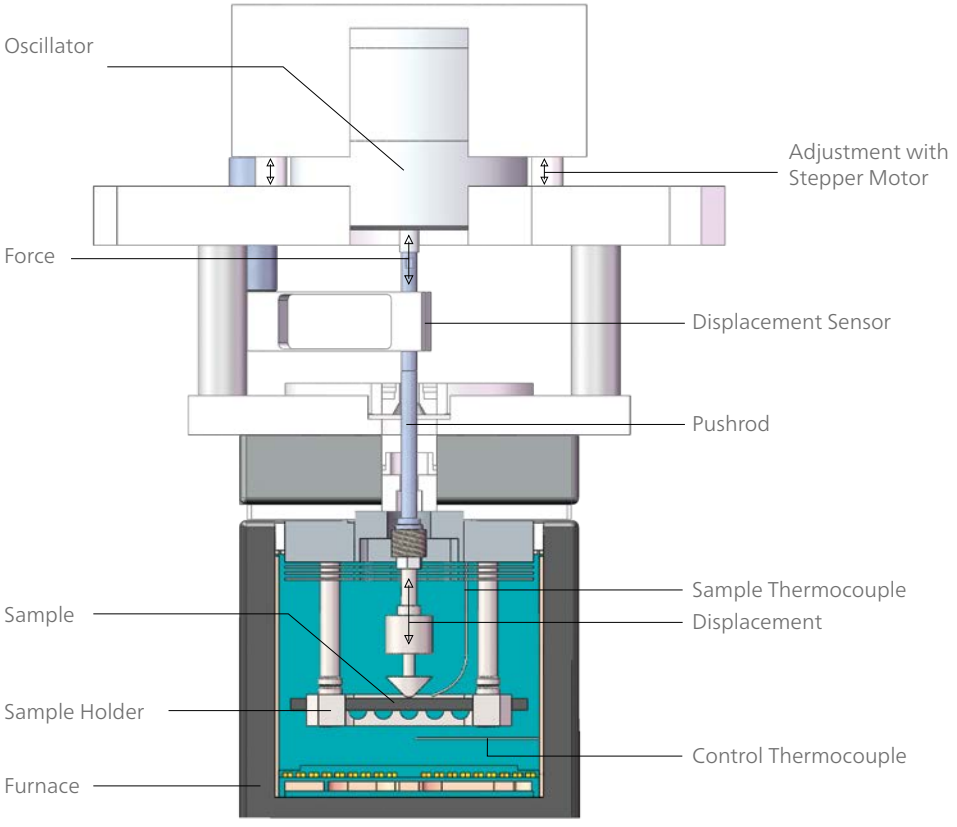
The results of this mathematical operation are the storage modulus E' (related to the reversible, "in-phase" response) and the loss modulus E'' (related to the irreversible, "out-ofphase" response).
The loss factor tanδ is the ratio between the loss modulus and the storage modulus (tanδ = E''/E').

Generally, the storage modulus (E') refers to the material's stiffness whereas the loss modulus (E'') is a measure for the oscillation energy transformed into heat. tanδ characterizes the mechanical damping or internal friction of a visco-elastic system.

Resulting Data

Complex DMA Variable	Real Part	Imaginary Part
Complex modulus E*	Storage modulus E'	Loss modulus E''
Shear modulus G*	Storage shear modulus G'	Loss shear modulus G''
Compliance D*	D'	D''
Amplitude A*	A'	A''
Force*	F'	F''
Spring constant c*	c'	c''

General Data
Static length change dL
Offset
Static sample force F_{stat}
Loss factor tanδ



DMA 242 E *Artemis* – Functional Principle

Dynamic Mechanical Testing Supports Research and Quality Control of Polymers

R&D
The DMA method is a very sensitive tool for generating data that can help define the mechanical properties of polymers and composites in order to support product development in industries such as automotive.

Quality Control
α- and β- transitions can be used to compare production with standards and competitors' products. Our DMA experts support you by finding the right approach for specific applications and areas of interest.

DMA Measurement Information

- Design data concerning stiffness and damping properties (modulus values and damping factor under a variety of conditions)
- Data on the composition and structure of polymer blends (compatibility)
- Glass transition temperature of highly cross-linked, amorphous or semi-crystalline polymers and composites
- Curing/post-curing
- Aging
- Creep and relaxation
- Stress and strain sweeps
- Multi-frequency tests
- Prediction of the material behavior using Time-Temperature-Superposition (TTS)
- Immersion tests

DMA 242 E *Artemis*	
Temperature range	-170°C to 600°C
Heating rate	0.01 K/min to 20 K/min
Frequency range	0.01 Hz to 100 Hz
Force range with high force Force range with high resolution	24 N (max.) 8 N (max.)
Maximum controlled strain amplitude	± 240 µm
Static deformation	Up to 20 mm
Modulus range	10^{-3} to 10^{6} MPa
Damping range (tanδ)	0.005 to 100
Cooling device	▪ Liquid nitrogen: -170°C to 600°C ▪ Compressed air with vortex tube: 0°C to 600°C ▪ AIC 80 air intracooler: -70°C to 600°C; AIC 80 is activated < 300°C
Deformation modes	▪ 3-point bending ▪ Single / dual cantilever bending ▪ Shearing ▪ Tension ▪ Compression/penetration
Additional measurement modes	▪ Iso-strain ▪ TMA mode ▪ Creep / relaxation ▪ Stress / strain sweep
Sample geometries	Dependent on the deformation mode, e.g., for 3-point bending maximum sample dimensions: length: 60 mm, width: 12 mm, thickness: 5 mm
Optional accessories	▪ Immersion bath ▪ Humidity generator ▪ UV equipment ▪ Dielectric Analyzer (DEA)

Technical Specifications

Sample Holders for Different Modes

FOR ANY APPLICATION

Sample Holder	Sample Dimensions			Applications
Single/Dual Cantilever	**Free Bending Length***	**Width (max.)**	**Height (max.)**	
Standard	(2×)1 mm (2×)5 mm (2×)16 mm (2×)17 mm	12 mm 12 mm 12 mm 12 mm	5 mm 5 mm 5 mm 5 mm	Thermoplastics, elastomers
Stiff clamp	17 mm	12 mm	5 mm	For determination of the glass transition (T_g) of reinforced polymers used in the aircraft industry
Free pushrod	20 mm	12 mm	5 mm	Very stiff samples; e.g., CFRP
3-Point Bending	**Free Bending Length***	**Width (max.)**	**Height (max.)**	
Round-edged	10 mm 20 mm 40 mm 50 mm	12 mm 12 mm 12 mm 12 mm	5 mm 5 mm 5 mm 5 mm	Fiber-reinforced or highly filled thermoplastics
Knife-edged	20 mm 40 mm	12 mm 12 mm	5 mm 5 mm	Stiff fiber-reinforced or highly filled polymers, metals, ceramics
Tension	**Free Bending Length***	**Ø/Width/Thickness (max.)**		
Standard	15 mm	6.8 mm		Films, fibers, thin rubber strips
Compression/ Penetration	**Sample Ø (max.)**	**Pushrod Ø [mm]**	**Height (max.)**	
Standard	15 mm 30 mm	0.5, 1, 3, 5, 15 0.5, 1, 3, 5, 15, 30	6 mm 6 mm	Soft samples; e.g., rubber
Shearing	**Ø/Width/ Height (max.)**	**Thickness (max.)**	**Cross section (max.)**	
Flat surfaces	15 mm	6 mm	225 mm^2	Adhesives, elastomers
Grooved surfaces	15 mm	6 mm	225 mm^2	Adhesives, pastes

* The samples must be greater in length than the free bending and free tension length values listed here.

From liquids to highly-filled thermosets to metals and ceramics – all such materials can be measured with the DMA 242 E *Artemis*. Precise results require optimal adaptation of the test conditions to the materials and applications. That is why NETZSCH has developed a wide range of sample holders, accessories and measurement modes. All sample holders available are listed in the table on the left and on the following pages.

Sample holder for 3-point bending

Sample holder for single/dual cantilever

Sample holder for tension

A variety of sizes for frame and pushrods allow for optimal adaptation of the compression/penetration sample holder to the test parameters.

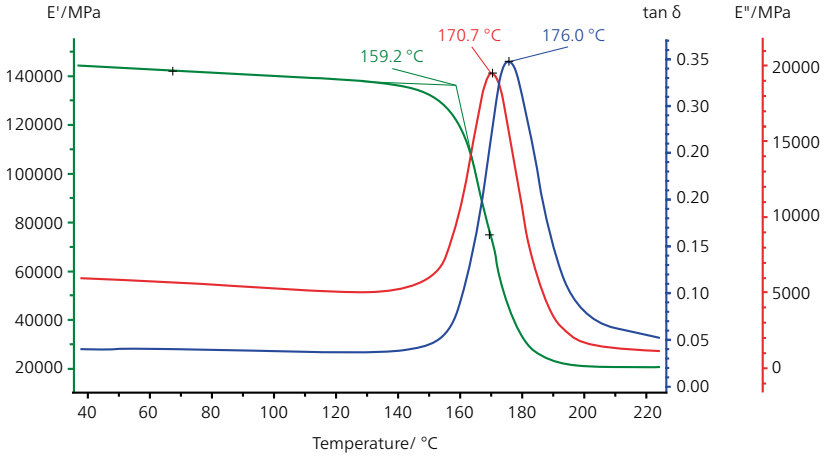
Sample holder for shearing

WIDE CHOICE OF SPECIAL SAMPLE HOLDERS

Sample holder for measurements on pasty samples in compression with insert

Sample holder for single cantilever bending with free pushrod, especially used for stiff materials

Special Sample Holder	Sample Dimensions			Applications
Compression/ Penetration	**Sample Ø (max.)**	**Pushrod Ø [mm]**	**Height (max.)**	
Pushrod made of fused silica and free alumina disk	15 mm	5 mm	6 mm	Insulation foams, expansion measurement in TMA mode
Sample insert	7 mm	3 mm	2.5 mm	Curing of pasty samples with higher viscosity
Ball-shaped pushrod	Container: Ø 19 mm, height 15 mm Pushrod ball: Ø 8 mm			Curing of viscous samples
Fused silica window for UV light	15 mm	15 mm	6 mm	Curing of UV-sensitive materials
Simultaneous DMA-DEA measurements	15 mm	15 mm	6 mm	Curing of reactive resins



DMA measurement on a very stiff, carbon fiber-reinforced epoxy resin
Sample holder: single cantilever bending; 20-mm with free pushrod
Measurement parameters: heating rate 3 K/min, frequency: 10 Hz, amplitude: ± 40 µm

Young's Modulus of a CFRP

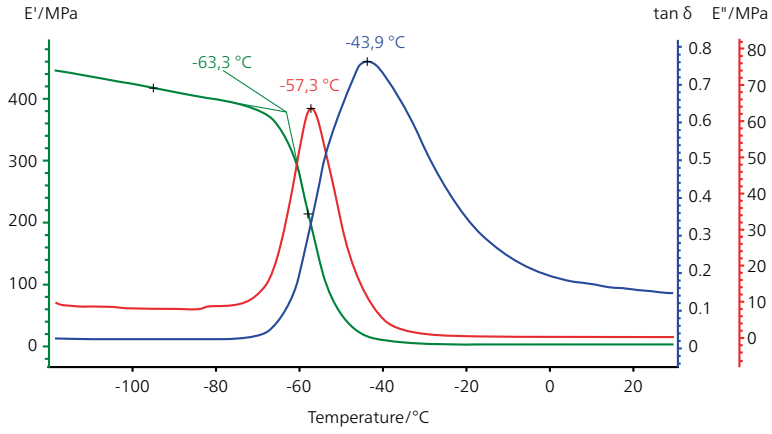
The single cantilever sample holder with free pushrod was specially developed to accurately measure very stiff materials. The sample is tightly fixed at one end and a free pushrod oscillates at the other end.

The results of a DMA test on a carbon fiber-reinforced epoxy resin are presented in the plot to the left. The high storage modulus at 50°C (approx. 145,000 MPa) indicates that this material is even stiff er than metallic titanium. The drop in the curve at 159°C (onset temperature), related to the maxima in the loss modulus and loss factor curves at 171°C and 176°C, marks the glass transition of the epoxy matrix.

Sample Holder for Insulation Foams

Because of the very low heat conductivity of foams and insulation materials, heat can be lost if a standard metallic pushrod is used. It is thus advisable to work with the fused silica pushrod with free disk made of alumina especially developed for measurements in the compression mode. This pushrod is also recommended for measuring expansion in TMA mode.

Pushrod made of fused
silica with free alumina disk



Compression measurement on an insulation foam (height 5 mm)
Sample holder: compression with fused silica pushrod / alumina disk
Measurement parameters: -120°C to 30°C at 2 K/min, frequency: 10 Hz,
amplitude: ±30 µm

Visco-elastic Properties of a Foam

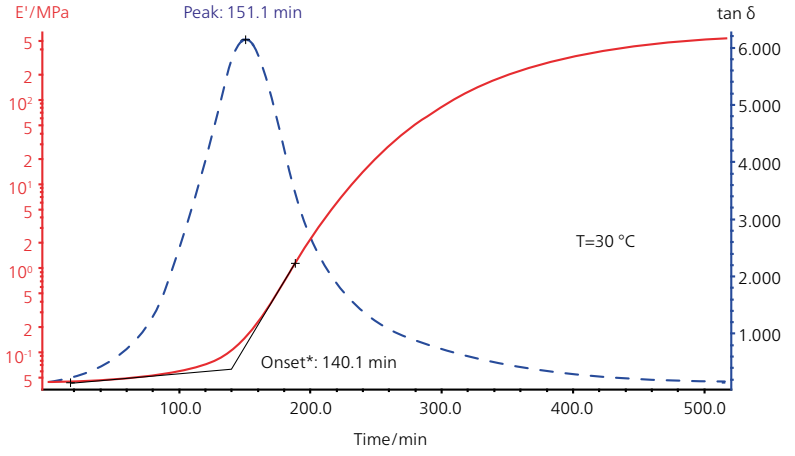
Insulation foams are increasingly important in the building industry for both new construction and building renovation. They help to lower energy consumption by preventing heat loss through the walls.

This plot shows a measurement on an insulation foam between -120°C and 30°C at a frequency of 10 Hz.

The decrease in the storage modulus curve beginning at -63°C is related to the peaks at -57°C (loss modulus) and at -44°C (tanδ). It corresponds to the glass transition of this insulation material, thereby limiting the application range.

Curing of a Liquid Epoxy Adhesive

The results of DMA measurements on a liquid epoxy adhesive are shown in the plot below. The sample holder with container and ball-shaped pushrod, which was developed for the curing of liquids, was used for the testing. The increase in storage modulus after 140 minutes (time onset) results from the curing reaction. It is related to a peak at 151 minutes in the tanδ curve. The further increase of the storage modulus value after approximately 500 minutes indicates that the curing has not been finished.



DMA measurement using the sample holder with ball-shaped pushrod
Sample: epoxy adhesive
Sample holder: compression sample holder with container and ball-shaped pushrod
Measurement parameters: isothermal 30°C, frequency: 1 Hz, amplitude: ±20 µm

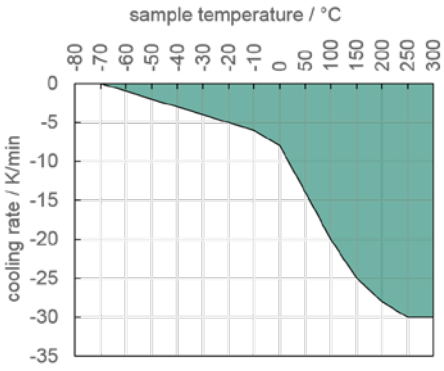
Special sample holder with ball-shaped pushrod for curing of high-viscous liquids

WIDE SELECTION OF ACCESSORIES

Air Intracooler –
An Economic Solution for Typical DMA Applications

Many applications in the field of, e.g., polymers with lower stiffness, require a measurement start below room temperature. The AIC 80 cooling system is a compact air intracooler that works entirely without liquid nitrogen. It is a compact chiller based on a heat exchanger system with a long insulated connection line, which allows the air intracooler to be placed under the table or on the side – as it is most convenient in your laboratory.

The valve is software-controlled and can be operated in an on/off mode in each measurement segment. An inlet for compressed air allows for the connection of an air dryer (outlet dewpoint -70°C).



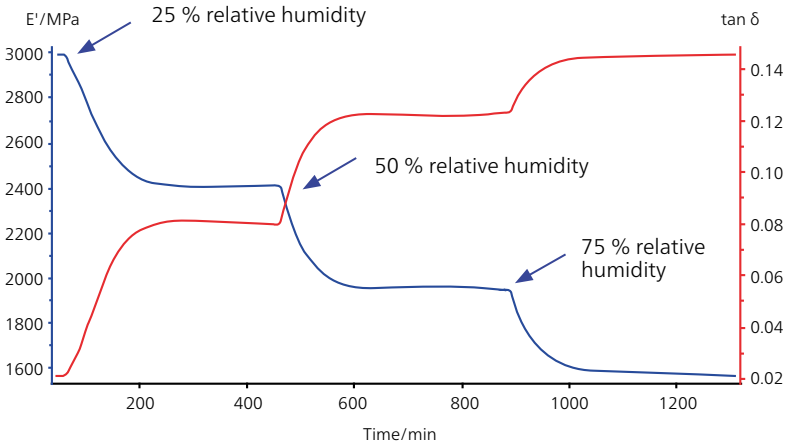
Cooling performance of the AIC 80

Technical Specification of the AIC 80 Air Intracooler

Temperature range	-70°C to 600°C, AIC is activated < 300 °C
Max. input pressure	2 bar
Air throughput	Max. 50 slm (standard liters/min)
Dimensions (w x d x h)	0.38 m x 0.55 m x 0.8 m
Length of probe	3 m
Weight	65 kg
Outlet tube length	3 m
Max. ambient temperature	5°C to 35°C (specifications refer to 25°C)

Influence of Humidity on the Mechanical Properties of a Polyamide Film*

For this example, a polyamide film was dried and measured in tension mode by using the DMA with the humidity generator. First, the humidity generator was switched off and the storage modulus E′ was constant at at ≈ 3000 MPa. As soon as humidity was introduced into the furnace, E′ of the polymer decreased sharply; it reached a plateau at approx. 2400 MPa.Increasing the humidity content to 50% and 75% (after 7 h and 14 h) led to further decreases in the storage modulus. These results show that the humidity content has a great influence on the storage modulus of polyamide because water acts as a plasticizer on polymers.



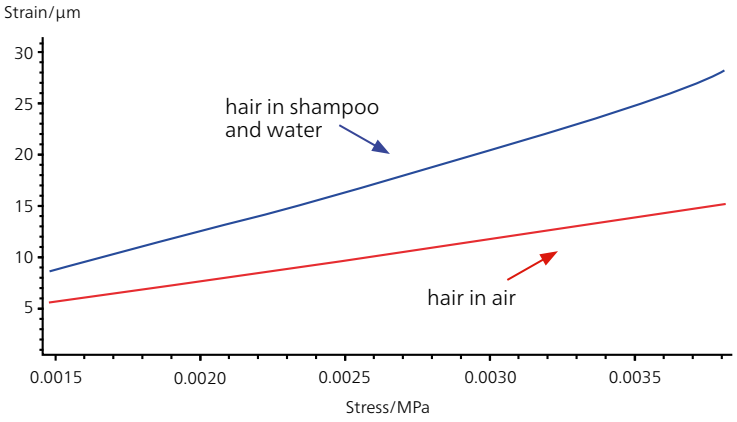
Container for immersion tests

Easy Measurement in Liquids: Immersion Bath

An immersion bath (inserted into the furnace) can be used in combination with any sample holder to check the influence of a given liquid on the visco-elastic properties of a material. The temperature can be varied at will during the measurement.

Influence of Shampoo on Human Hair

Stress-sweep tests were carried out on a human hair in an air atmosphere and in a mixture of water and shampoo (same hair for both tests) The force was varied from 0.1 N to 1 N and the strain measured. The plot represents the stress-strain plot for both measurements. The stress-strain plot for both tests shows the difference in the slope of the curves: the hair has a higher strain – i.e., is softer – when in contact with the water-shampoo-mixture than with air.

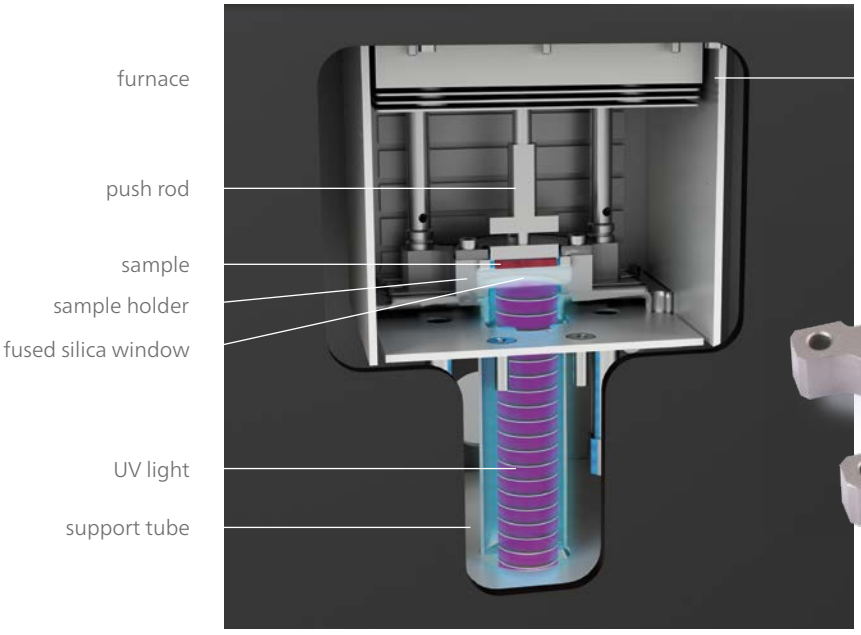


Softness of human hair (thickness 70 µm and 80 µm) measured in tension mode at 25°C and a frequency of 1 Hz;. force varied between 0.1 and 1 N.

* Our thanks go to Prof. Dr. T. Rödel and M. Wendt from the University of Applied Sciences in Merseburg for the measurement and discussions.

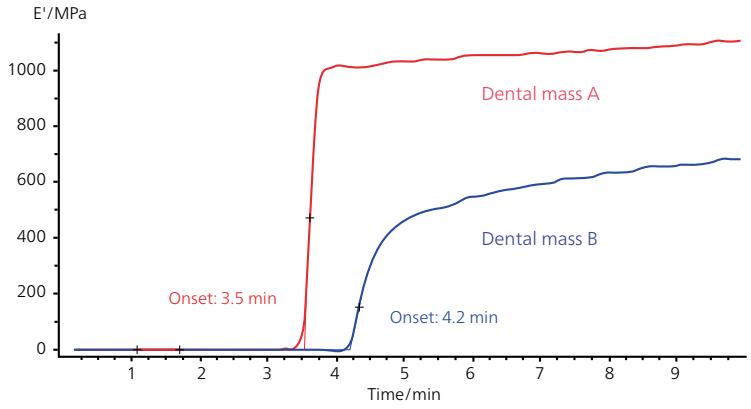
Light-Curing: UV Add-On

The furnace of the DMA 242 E *Artemis* can be connected to a light source in order to measure the curing of UV-reactive materials. A special compression sample holder allows the light to pass through a fused silica window.



Instrument set-up for a DMA 242 E *Artemis* connected to a light source

Special sample holder with fused silica window for DMA measurements under the influence of UV light



Comparison of the curing behavior of two dental masses
Measurements parameter: compression mode, temperature: 30°C, frequency: 10 Hz, amplitude: ±15 µm

Light Curing of Two Dental Masses

The curing behavior of two dental masses under light were compared. The storage modulus of dental mass A red) increased sharply after 3.5 minutes, which can be attributed to curing of the material. The reaction of dental mass B (blue) began nearly one minute later and ran more slowly, as can be seen by comparing the slopes of the two materials. The difference in the final storage moduli (1100 MPa for dental mass A and 700 MPa for dental mass B) is due to differences in the mechanical properties of the cured products.

Simultaneous DMA-DEA: Two Measurements in One

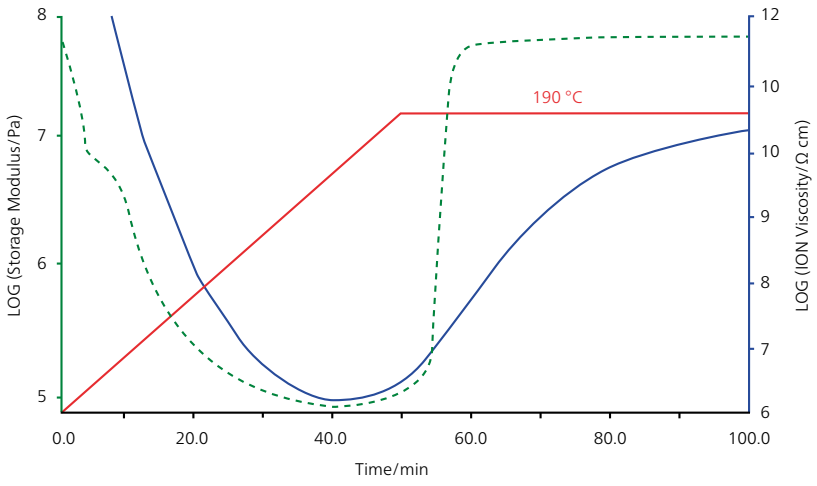
DEA (Dielectric Analysis) is a method for determining the curing behavior of reactive resins by monitoring the ion viscosity. In the DMA-DEA coupling test, the DEA sensor is set on a special compression sample holder, and both DMA and DEA measurements run simultaneously during the same temperature program.

Sample holder for
simultaneous DMA-DEA

DMA-DEA Measurement on an Epoxy Resin

In this example, an uncured epoxy resin was heated to 190°C and the temperature was kept constant. The initial decrease in the storage modulus and ion viscosity during heating is due to softening of the sample. The increase in the storage modulus is related to the beginning of curing. The subsequent sharp increase in storage modulus demonstrates the sensitivity of DMA at the beginning of the curing reaction.

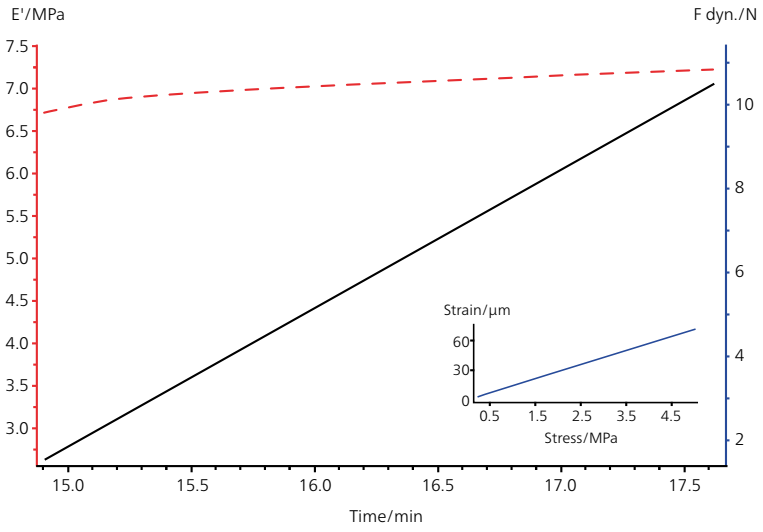
During the isothermal hold at 190°C, the storage modulus stabilizes in compression mode. However, the ion viscosity continues to increase; the more sensitive DEA method makes it possible to determine that curing has still not completely finished after 100 minutes.



Curing of an epoxy resin
Sample holder: special compression sample holder for DMA-DEA
Measurement parameters: room temperature to 190°C at 3 K/min and isothermal at 190°C, frequency: 10 Hz

DIFFERENT MEASUREMENT MODES

Higher Forces for More Information



Stress-sweep test of a natural rubber with a thickness of 2.01 mm
Sample holder: compression, 15 mm diameter
Measurement parameters: room temperature, frequency: 10 Hz

Sample holder for measurements in compression

The DMA 242 E *Artemis* works with a force range up to 24 N. Thanks to this broad range, very thick and stiff samples can be investigated, especially in the compression and tension modes. Here, a natural rubber was measured in the compression mode. The maximum static force was set to 12 N. The dynamic force was varied between 0.5 N and 10.5 N, and the resulting strain was measured (stress-sweep test). The dynamic force which was applied and the resulting storage modulus are presented in the plot. Additionally, the strain curve is depicted as a function of the applied stress (inset) to check that the test was carried out in the Hooke's region (linearity of the curve).

Static Modes : Creep, Relaxation, TMA

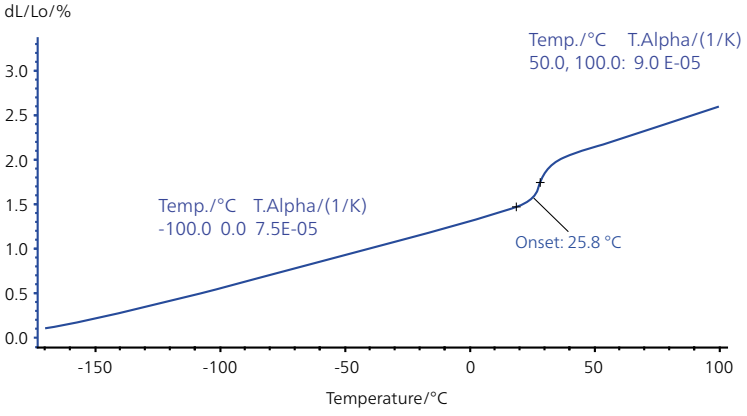
Along with dynamic measurements, the DMA 242 E *Artemis* also allows for tests in the three static modes creep, relaxation and TMA.

In the creep mode, a constant static force is applied to the sample and the resulting deformation is measured. The relaxation test determines the static force required to attain a defined constant deformation.
In the TMA mode, the thermal expansion of materials is determined. For this, a small static force is applied to the sample and the resulting length change is measured as a function of the increasing temperature.

TMA Mode:
Thermal Expansion of PTFE

In this example, the length change of PTFE was measured from -170°C to 100°C with the NETZSCH DMA 242 E *Artemis* in the TMA mode.

At the beginning of the test, the sample length increased linearly. The step in the sample expansion at 26°C is related to the transition from the well-ordered phase of PTFE to its disordered phase.



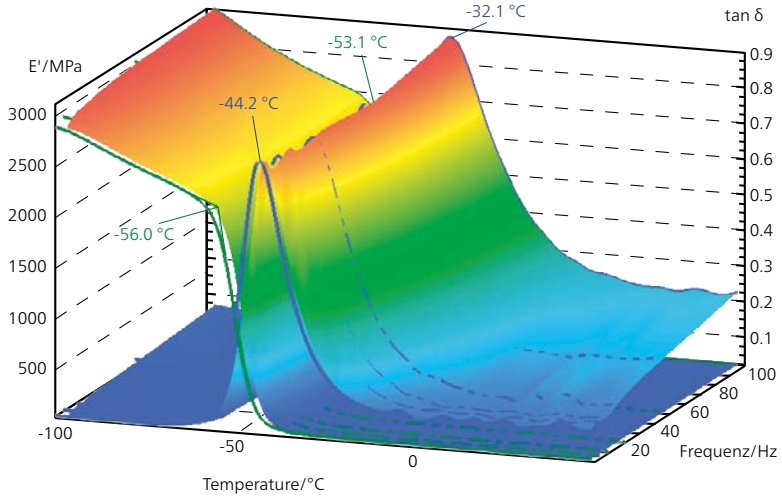
TMA measurement of a PTFE
Sample holder: compression in the TMA mode
Measurement parameters: -170°C to 100°C at 2 K/min, static force: 0.1 N

3D-Plot, Multifrequency

Sample holder for
dual cantilever bending

Multi-Frequency Measurement on an Elastomer

In addition to the ability to carry out multi-frequency measurements, the user also has the possibility to present results in a three-dimensional plot: the visco-elastic properties of the tested material can be viewed as a function of both temperature and frequency at one glance.



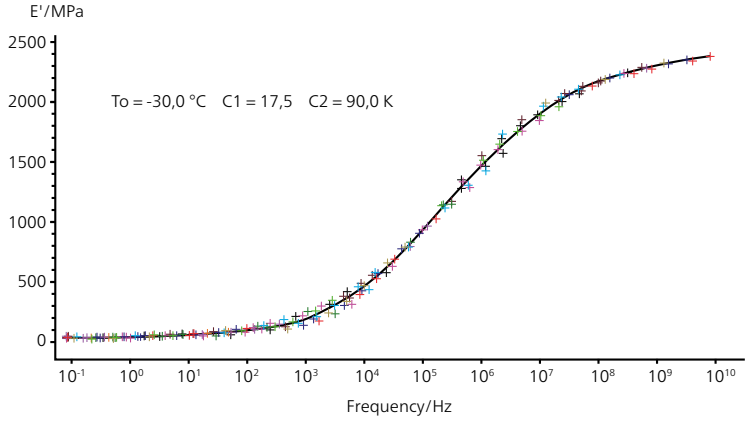
3D-plot of the visco-elatic properties of an elastomer
(height: 2.66 mm, width: 7.77 mm)
Sample holder: dual cantilever 2×16 mm
Measurement parameters: heating from -100°C to 50°C at
2 K/min, frequencies: 1, 5, 10, 20, 50 and
100 Hz, amplitude: ±40 µm

In this example, an elastomer was heated from -100°C to 50°C and its visco-elastic properties were determined for frequencies from 1 to 100 Hz.

The plot depicts the curves of the storage modulus and loss factor as a function of temperature and frequency. For each frequency, the decrease in the E´curve is associated with a peak in the tanδ curve. This effect is due to the glass transition of the sample. As expected, the glass transition is shifted to significantly higher temperatures with increasing frequency. The values given on the graph are the onset temperatures of the storage modulus curve and the peak temperatures of the loss factor curve for 1 Hz and 100 Hz.

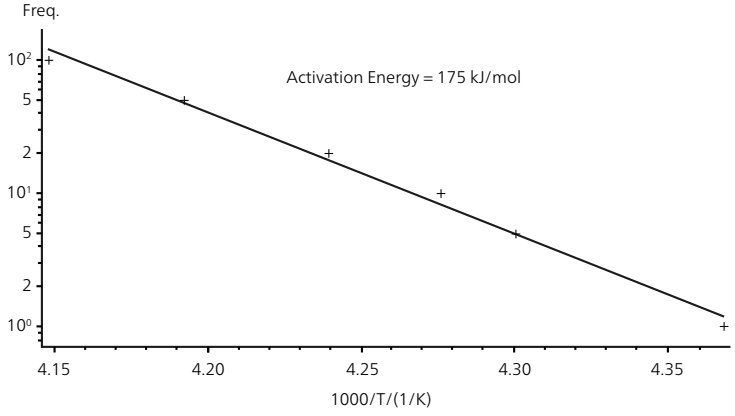
Master Curve and Arrhenius Plot of an Elastomer

The visco-elastic behavior of a polymer as a function of frequency can easily and quickly be determined using the master curve calculated from a single multi-frequency measurement. To do this, the time-temperature superposition is used: the dependency relationship of E', E'' and tanδ on frequency can be extrapolated to frequencies exceeding the measuring range of the device. With the WLF (Williams-Landel-Ferry) equation, the shift factor can be calculated and a master curve can be established at a given reference temperature.



Master curve of an elastomer at a reference temperature of -30°C

In the example, the master curve of the storage modulus was calculated at a reference temperature (T0) of -30°C. The DMA software evaluated the coefficients C1 and C2 of the shift factor according to the WLF equation. The measure of E´over the extrapolated frequency range up to 10^{10} Hz can be demonstrated.



Arrhenius curve of an elastomer

Additionally, the *Proteus*® software allows for calculation of the activation energy for the glass transition. To do this, the logarithmic frequency dependence of the loss factor (tanδ) is plotted over the inverse absolute temperature. The activation energy is given as the slope of the linear fit through the data points. An activation energy of 175 kJ/mol was found for the glass transition of the elastomer.

Proteus® Software

for the DMA 242 E *Artemis*

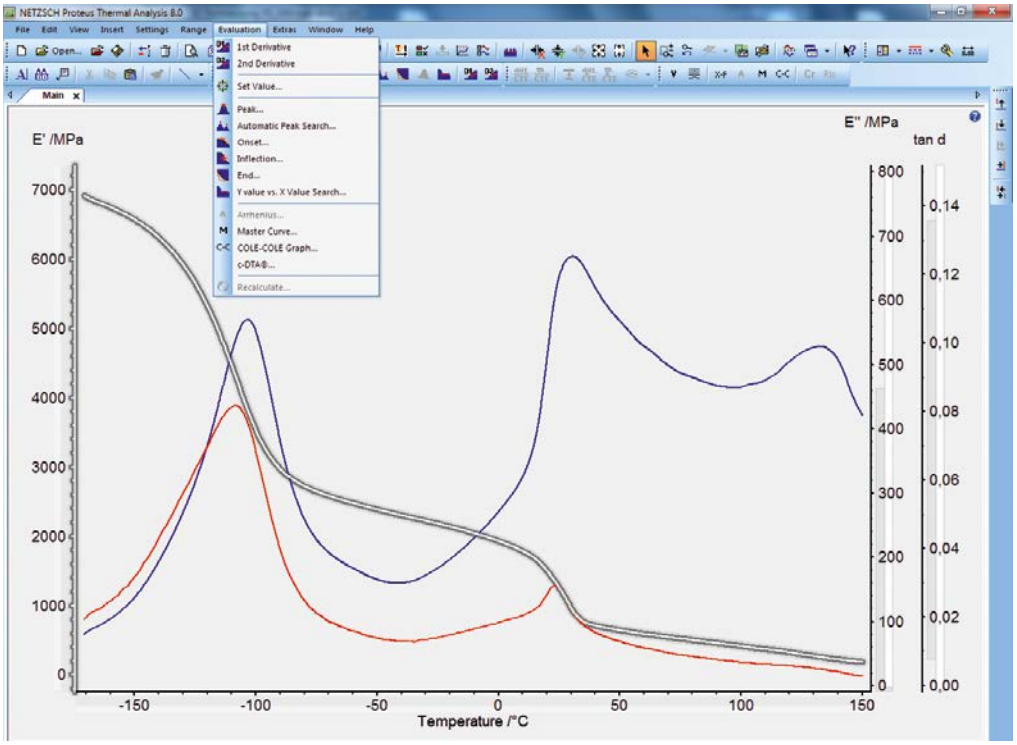
The DMA 242 E *Artemis* runs under a 32- and 64-bit Windows® operating system and includes everything you need to carry out a measurement and evaluate the resulting data. Userfriendly menus combined with automated routines make *Proteus*® very easy to use while still providing sophisticated analysis.

Key Features of the General Software

- For Windows XP Professional®, Vista® (Enterprise, Business), Windows 7 (Professional®, Enterprise®, Ultimate®) operating systems
- Simultaneous measurement and evaluation
- Combined analysis: comparison and/or evaluation of DSC, TGA, STA, DIL, TMA, DMA and DEA measurements in a single plot with up to 64 curves/temperature segments from the same or different measurements
- Storage of the analysis results and status with all analysis windows and preview-graphic in a file for later restoration and continuation with analysis
- Printout possible in 9 different languages
- Export graphics with evaluation results to clipboard or to common formats such as EMF, PNG, BMP, JPG, TIF or PDF
- ASCII-file export
- E-mail support: status messages or measurement files can be sent automatically following the measurement or in case of error
- Online evaluation of the measurement in progress (snapshot)

Key Features of the Measurement Software

- Multiple programmable temperature segments (isothermal, dynamic) and temperature ramps with single or multiple frequencies; free selection of force values, deformation amplitudes and frequencies for each segment
- Online graphics with up to eight separate freely selectable axes, with online zoom, time- or temperature-scaled, single-segment or full-curve view
- Calibration routines: Dynamic mass, empty system, system stiffness, rotation tuning, temperature
- Oscillation control: Easy choice of stress control, strain control and special mixed mode (strain control with additional force limit) for materials with visco-elastic properties exhibiting considerable change



Typical DMA measurement with graphical presentation of E', E'' and tanδ.

Integrated Special Measurement Modes

- Creep mode ··Relaxation mode with deformation range up to 20 mm (depending on the sample size and chosen sample holder geometry)
- Stress-sweep mode
- Strain-sweep mode
- Iso-strain
- TMA mode
- Force modes: Force range with higher force (24 N), force range with higher resolution (8 N)

Key Features of the Analysis Software

- Determination of storage modulus E', loss modulus E'' and loss factor tanδ
- 1st and 2nd derivative
- Superposition of the frequency-scaled curves (master curves)
- 3D plot functionality for multifrequency DMA data (for e.g., visualization of the frequency-dependent shift of the glass transition temperature)
- Determination of the activation energy (Arrhenius plot)
- Determination of Cole-Cole plot (graphical presentation of log(E'') or log(tanδ) as a function of log(E'))
- Graphical presentation of the static length change, both in absolute units (dL in µm) for all types of sample holders, and in relative units (dL/L_0, dL/L_0 in %) for all sample holders of the 'compression' or 'tension' type
- TMA Mode: Graphical presentation of the static length change, 'dL' (TMA mode), with the possibility for calibration correction and calculation of expansion coefficients (CTE) in dynamic segments
- Graphical presentation of creep and relaxation behavior
- Graphical presentation of stress- and strain-sweep behavior, stress-strain graph

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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.

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