

Dynamic Mechanical Analysis DMA 303 *Eplexor*®

Method, Technique, Applications

Dynamic Mechanical Analysis

UNLOCKING THE MECHANICAL MATERIAL PROPERTIES OF A WIDE RANGE OF MATERIALS

Dynamic Mechanical Analysis (DMA/DMTA) is an indispensable tool for determining the mechanical properties of engineering materials, particularly the viscoelastic behavior that characterizes polymers.

Changes in the viscoelastic behavior of a material can be measured by applying forces and deformations under dynamic conditions, for example, as a function of temperature, time, frequency, stress, atmosphere, or a combination of these parameters.

How Can Dynamic Mechanical Analysis Help You Fulfill Your Product Promise?

Polymers – an important material in manufacturing

Polymers have the advantage of being lightweight and easily moldable into a variety of shapes using different manufacturing processes. Especially in applications where plastic parts play an important functional role, they must perform as designed, namely in the automotive, electronics or medical industries. This requires good understanding of the material behavior from the molecular level to real-world mechanical properties.

Prediction of mechanical behavior – essential for the design of new products

Dynamic Mechanical Analysis (DMA) is a highly sensitive analysis method for evaluating material properties during design and production. A wide range of mechanical properties can be determined. These include stiffness, elasticity, damping, and viscoelastic behavior.

Information to Be Gained by Dynamic Mechanical Analysis

- Viscoelastic material properties: storage and loss modulus, loss factor, $\tan \delta$
- Stiffness and damping properties under a variety of conditions:
 - depending on temperature and frequency
 - at different levels of stress and strain
 - under defined gas atmosphere and in liquid environments
- Identification of material reactions and phase transitions
- Glass transition temperature of highly cross-linked polymers and composites
- Compatibility of polymer blends in reference to composition and structure
- Influence of filler and additive contents
- Curing and post-curing of resins
- Analysis of aging influences
- Prediction of material behavior using Time-Temperature-Superposition (TTS)
- Creep and relaxation processes



Why the DMA 303 Eplexor® Stands Out

Application of Precise Forces up to 50 N Dynamic and Static

Understand the mechanical behavior of even stiff materials with an unprecedented 50 N in a desktop DMA.

Wide Temperature Range from -170°C to 800°C

Test a wide range of materials and a material's broad thermal behavior within this unrivaled temperature range.

Frequency Range of 0.001 Hz to 150 Hz

Better understanding of a material's structure and properties is obtained when a wide range of deformation rates can be applied.

Highest Sensitivity with 1-nm Resolution

Detect minute changes in the mechanical properties of a sample to ensure exact results.

Amplitude of \pm 2.5 mm

A wide amplitude range is particularly advantageous for measurements beyond the linear-viscoelastic range.

Accessories for Multiple Measuring Modes and a Variety of Sample Holders

It is easy to adjust the measuring mode to the type of material you need to characterize. The following measuring modes are available:

- Tension
- 3-point bending
- Compression/penetration
- Cantilever
- Shear

No More Headaches when Exchanging Sample Holders

RFID* technology auto-detects sample holders

State-of-the-Art Software:

- Quick start Initialize new specimen grips directly from the start screen and automatically determine the sample length.
- Time saving Manual mode allows you to apply both static and dynamic loads to get a first impression of the specimen.
- Being prepared Launch templates for all types of measurements as well as your own pre-defined measurements or favorites directly from the home screen.

^{*} Radio Frequency Identification

Static Drive (Stepper motor)

- Responds to sample elongation
- Especially important for such investigations as tensile tests
- Travel range: 30 mm

Contactless Optical Position Sensor

- No friction
- 1-nm resolution

Custom-Designed Drive System

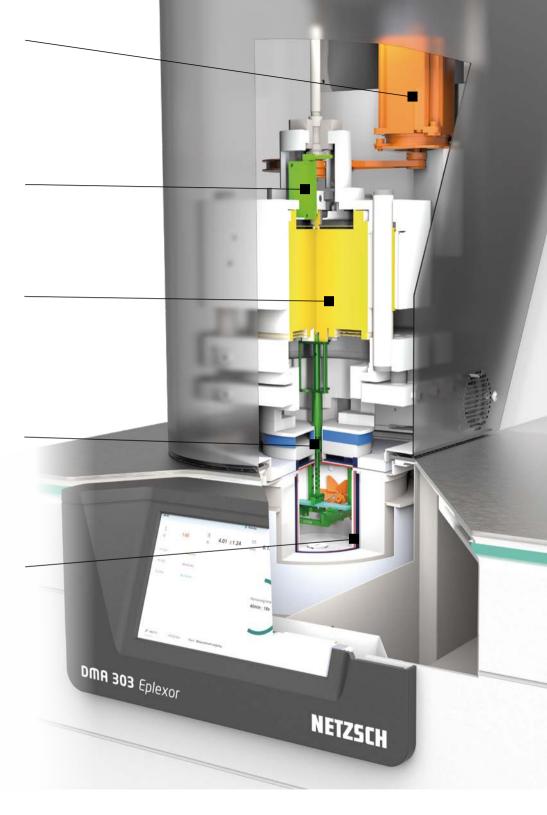
- Temperature-controlled, leight-weight electro-magnetic drive
- For precise application of forces up to 50 N

RFID* Tag on Probe and Frame

- Recognition of the sample holder type
- Automatic selection of the correct calibration

3D-Printed Silicon Carbide Furnace Lining

- Thin-walled but robust
- Exceptional heat transfer
- Time-saving fast heating and cooling
- Temperatures at the sample from -170°C to 800°C



Made by Material Experts for Material Expertise

Schematic setup of the DMA

Measurement Setup

- 1 Specimens of clearly defined geometry, cut from sheets, plates, or molded shapes, are mounted in a suitable sample holder.
- 2 The DMA's moving probe applies a static and/or dynamic sinusoidal force, generated by ...
- 3 ... an electromagnet, to the specimen.
- 4 A non-contact sensor detects dynamic sample deformation.
- 5 The system's additional stepper motor drive adjusts when a sample experiences a length change, such as thermal expansion.
- 6 This static displacement is monitored by a second contactless sensor.
- 7 The furnace of the DMA 303 Eplexor® features two independent temperature control loops to ensure homogeneous conditions at the sample. This is supported by a fan that provides forced convection to increase heat transfer.



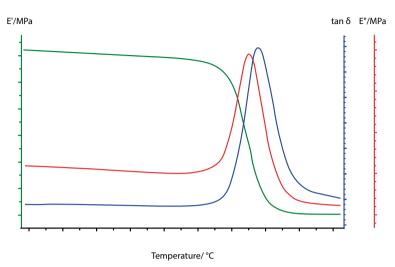
Storage Modulus E', Loss Modulus E' and tan δ

Certain materials, such as polymers, exhibit viscoelastic behavior; they have both elastic properties (like an ideal spring) and viscous ones (like an ideal damper).

The measurement curve on the right shows a typical DMA measurement result for a polymeric material that is undergoing a glass transition.

The green curve depicts the storage modulus, E', the red curve shows the dynamic loss modulus, E'', and the blue curve shows the derived phase shift and the resulting loss factor, $\tan \delta$.

DMA is extremely sensitive to glass transition as E' shows a very sharp decrease, while E' and $\tan \delta$ have distinct maxima as the temperature passes the relevant range.



Typical DMA measurement



View into the DMA furnace with fan

Operating Principle of the DMA 303 *Eplexor*®

Available Measurement Modes

- Time sweep
- Temperature sweep
- Frequency sweep
- Temperature-frequency sweep
- Static/dynamic load sweep
- Creep/relaxation
- Universal test
- Immersion for all geometries and test types

Advanced Instrument Design Offering Flexibility and Convenience

The unique suspended and moving sample holder of the DMA 303 *Eplexor*® arranged below the measuring system prevents contamination from falling sample fragments and allows easy and safe sample handling.

We Take Care of All Users

The height-adjustable measuring head accommodates for different users' heights or preferences for standing or sitting, offering optimal comfort during use.

The system design permits the use of an immersion accessory with all available sample holders to provide you with maximum flexibility and ease-of-use in your experiments.

Clear View

The illuminated sample holder area makes it easy to set up samples and geometries. This, in combination with height-adjustable sample holders, offers unparalleled user-friendliness over other instruments on the market.



Suspended design with illuminated sample holder area



Immersion bath compatible with all geometries



Height-adjustable working position with suspended sample holder design

Difference between closed and fully open instrument: 80 cm.



IMPROVED STATUS INFORMATION – EVEN FROM AFAR

Get complete insights into your measurements with the DMA 303 *Eplexor**. Our advanced instrument offers a comprehensive overview of your current measurement on the built-in display.

The innovative LED status bar allows you to check the general instrument status of the instrument even from afar.

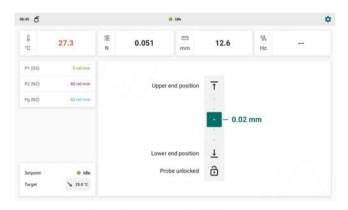
The integrated color display provides real-time updates on important information, such as:

- Measurement progress
- Temperature
- Force
- Frequency

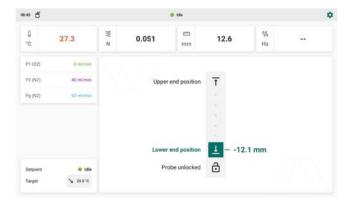
No more time-consuming logging into your PC – the information is right there.

The DMA 303 *Eplexor**'s display also offers visual support when changing sample holders, to simplify the process.

Thus, the DMA 303 *Eplexor*® offers complete information and easy operation to take your research to the next level.



Visual support when changing sample holders



EFFECTIVE CHOICE OF COOLING SYSTEMS

Smart and Economical Cooling Solution for Your Low-Temperature Experiments

Many applications, such as low-stiffness polymers, require a measurement start below room temperature.

Air Chiller System

The AIC 80 cooling system is a compact air intracooler that operates entirely without liquid nitrogen. It is a compact cooling unit based on a heat exchanger system with a long insulated connection line, allowing the air intracooler to be placed under the table or on the side, whichever is most convenient in your laboratory.

The valve is software-controlled and will be automatically switched on or off depending on whether cooling power is required. An inlet for compressed air allows for the connection of an air dryer (outlet dewpoint -70°C).

Liquid Nitrogen Cooling

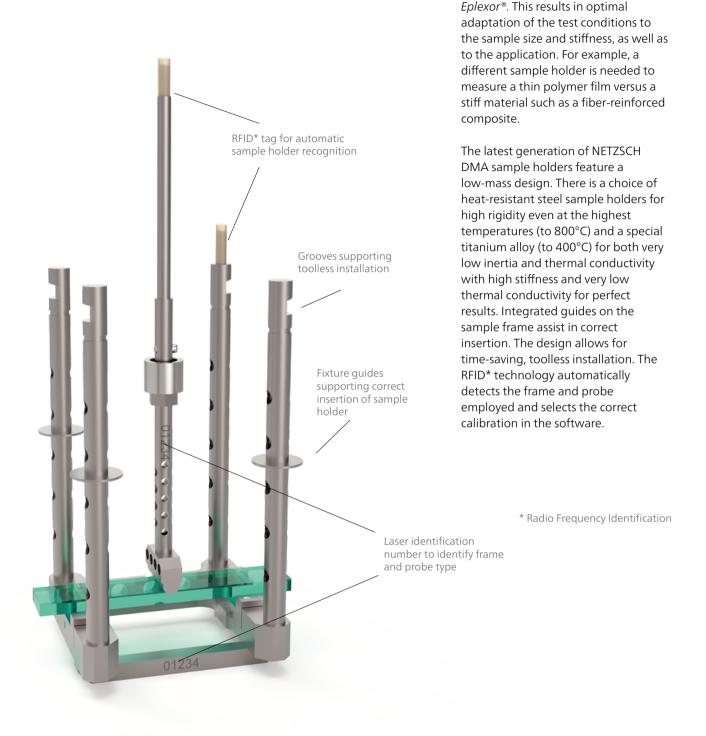
To reach temperatures as low as -170°C, a liquid nitrogen cooling system is available as a reliable and cost-efficient solution. This allows for controlled cooling rates over the entire temperature range of the DMA.



Temperature Range According to Cooling Option

Liquid Nitrogen -170°C to 800°C

AIC 80 air intracooler -70°C to 800°C



NETZSCH offers a wide variety of sample holders for its DMA 303

Excellence in Sample Handling





Clamping Systems to Suit Your Every Need

3-point bending

This sample holder is perfect for solid materials. The sample doesn't suffer from clamping effects and results are therefore ideal. The three-point bending tool is equipped with self-adjusting outer sample supports. This allows for good results to be achieved even on slightly warped samples.

Tension sample holder

Tensile clamps are commonly used for films and fibers. With a force up to 50 N, even strips of moderately stiff materials can be measured. Specimens are fixed with only one screw, ensuring evenly distributed clamping pressure. Laser-etched, non-slip contact surfaces and an additional grub screw ensure perfect grip on the sample.



Liquid immersion accessory suitable for all available sample holders



Sample holder for simultaneous DMA-DEA







Compression/penetration sample holder

Soft to moderately stiff specimens such as foams or elastomers can be studied in compression.

Probes are available in a variety of diameters to achieve the desired penetration. A self-aligning version is also available for specimens with slightly non-parallel surfaces.

Sample holder for single/ dual cantilever

When the temperature range approaches softening, cantilever bending is the mode of choice for evaluating polymers.

Shear sample holder

This sample holder is used to determine the shear modulus, G. Typical applications include rubber, gels, and other materials with a consistency between pastes and high-viscosity liquids. The dual sandwich sample can be conveniently prepared away from the instrument and simply placed in the sample holder just prior to measurement.



Sample holder for measurements on pasty samples in compression with insert



Probes for penetration/compression (1 mm, 3 mm and 30 mm)

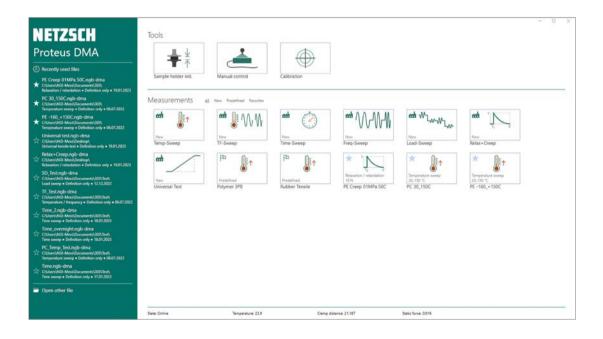


The probe with free alumina disc is particularly well-suited for compression measurements on specimens with uneven surface such as foams

Proteus® Software

The latest *Proteus*[®] measuring software version comes with many useful features for the DMA 303 *Eplexor*[®]. New specimen grips can now be initialized directly from the start screen and automatically determine the sample length.

Need to test a new specimen before the actual measurement? The manual mode allows you to apply static and dynamic loads to get a first impression of the sample. What's more is that the management of templates has been completely reconceived. In addition to templates for all types of measurements, you can also create your own predefined measurements or favorites and launch them directly from the start screen.



Features of the Measurement Software

- Calibration routines: Force calibration, empty system, system stiffness and phase calibration
- Manual control of static and dynamic loads, temperature and gases
- Multiple programmable sweeps may be combined in a segment program
- Online control of measurement with free configurable graphic or additional list of signals

Advanced Analysis Features

Results are obtained in *Proteus*[®] analysis and compared with all other measuring methods. In order to remain compatible with the extended application range of the DMA 303 *Eplexor*[®], the analysis options have also been extensively expanded. For example, any signal can now be used for the X-axis – whether it is a load sweep, tensile test or Cole-Cole plot, the user can always generate the optimum graph.

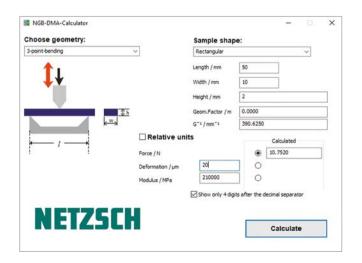
Of course, further features such as peak or onset determination or a linear fit are available. In addition to these generic features, special evaluations such as WLF master curve or determination of the activation energy with an Arrhenius plot can also be used.

Features of the Analysis Software

- 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
- Export graphics with evaluation results to clipboard or to common formats such as EMF, PNG, BMP, JPG, TIF or PDF
- Data export
- Special plots such as Cole-Cole, Arrhenius or Stress-Strain
- Arbitrary definition of the X- and Y-axis in the plot
- Determination of the WLF master curve
- General analysis of curves, detection of peaks, onset, inflection point and linear regression

The DMA Calculator – Finding Suitable Entry Values Quickly

The DMA Calculator is a flexible and unique tool for quickly calculating relevant DMA measurement parameters such as modulus, deformation or force values, thus avoiding multiple measurements. This is useful for both better interpretation of the results and for finding the best measurement setup for the material.



SMART ANALYSIS SOFTWARE AND QUALITY CONTROL

Identify

Database for Measurement and Material Identification

Identify is a unique software tool within the thermal analysis field for the identification and classification of measurements via database comparison. In addition to allowing one-on-one comparisons with individual curves and literature data, it can also check whether a particular curve belongs to a certain class. These classes can consist of curves of the same material type (material identification) or of reference curves for Pass/Fail testing (quality control).

The provided NETZSCH libraries contain about 1300 entries related to different application areas such as polymers, organics, pharmaceuticals, inorganics, metals/alloys or ceramics. In addition to DMA, most of the data included are of the types DSC, TGA, STA, DIL/TMA and c_p , which can also be superimposed. Users can expand *Identify* as desired, adding unlimited amounts of their own data. In general, all database entries serve as a pool of results and useful measurement conditions.

AutoFvaluation

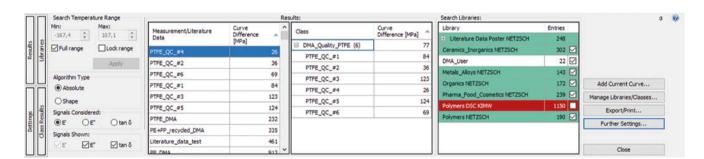
Objective and Fast Results

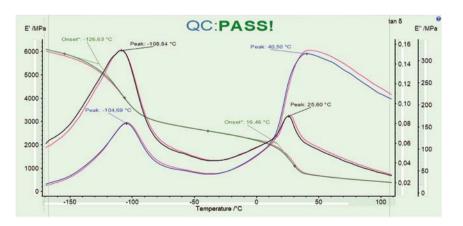
AutoEvaluation is a self-acting evaluation routine for DSC and TGA measurements. Fully self-executing and without user intervention, it evaluates onset temperatures in E' and peak temperatures in E' as well as tan δ at glass transitions. It's a time saver for every user.

Experienced users can take the automatic evaluation result as a second opinion – and, of course, modify or add calculations of values.

Report Generator

Each operator can easily create personal report templates – including logos, tables, description fields and plots. Several report examples are already included as templates within the *Proteus*® software.





This figure shows the comparison of a new DMA measurement (3-point bending, 1 Hz) on PTFE with previous measurements on the same material using *Identify*. The mean deviation of the new measurement is lower than a user-specified threshold, which is why the quality check is passed. The onset and peak temperatures shown were automatically evaluated by *AutoEvaluation*.

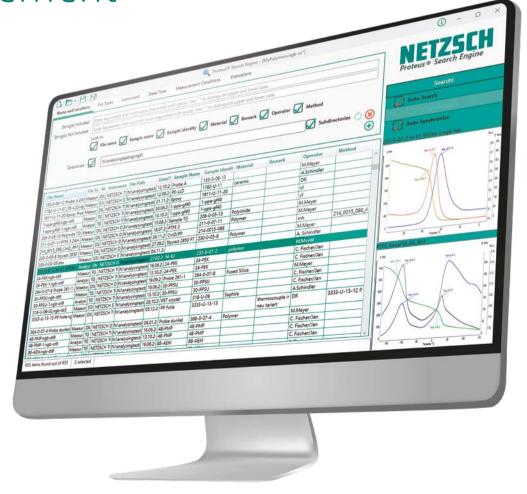
When working with measurement and evaluation data for different materials and different measurement setups, it is enormously helpful to be able to sort data by certain criteria. *Proteus® Search Engine* automatically synchronizes your measurement data with pre-defined directories and filters it in a matter of seconds. Previews of measurement curves or analysis statuses are available with just one click.

Users are able to create individual searches, for example "MyPolymers", and switch easily between different existing searches. This makes *Proteus® Search Engine* a very powerful data mining tool.

Filters can be set by such criteria as:

- File and sample name
- Remark and operator
- Instrument name
- File type
- Date of measurement
- Evaluated effects (onset and peak temperatures)

Proteus®Search Engine – Powerful Data Management

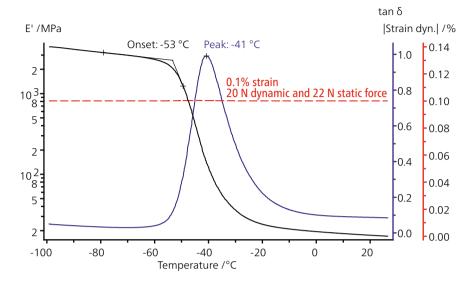


Applications

High Forces up to 50 N

50 N offers a wider range of options in experimental design than most table-top DMAs. This EPDM specimen – a synthetic rubber mainly used for sealings – was measured in tensile mode with a constant strain of 0.1% maintained across the complete temperature range from -80°C to 20°C. Modulus values beginning around 3 GPa are observed before the glass transition (Onset |E|: -53°C), whereas they go down to below 30 MPa at temperatures above -20°C.

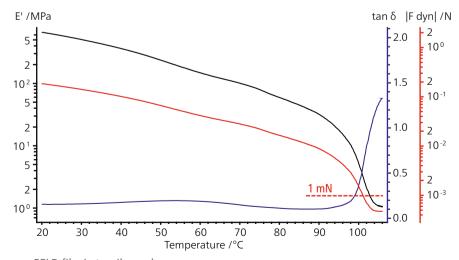
With a force range of 50, a constant level of elongation can be achieved. An amplitude or force control as commonly used in table devices, is not required. The force spectrum allows for constant test conditions and, in addition to application-oriented loads, also enables analysis of highly rigid material in various measurement modes.



EPDM in tensile mode Measurement parameters: -80°C to +20°C at 3 K/min, 0.1% dynamic strain at 10 Hz; free length: 25 mm, cross-section: $3.3 \text{ mm} \times 1.9 \text{ mm}$

Small Forces down to 1 mN

It can be challenging to analyze viscoelastic effects over the entire temperature range using only a single setup, particularly when a material undergoes a significant change from a stiff to a rather soft and viscous state. In this case, besides the maximum force, even the smallest detectable signal plays a role. This measurement was carried out on a 30-µm thick PE film with a constant amplitude of 50 µm. When reaching the melt, the PE film becomes very soft, with modulus values under 2 MPa resulting in dynamic forces smaller than 1 mN.

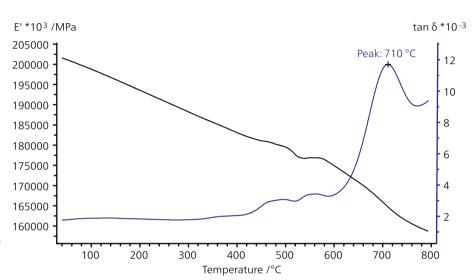


PELD film in tensile mode Measurement parameters: -20°C to +105°C at 3 K/min, 50 μ m dynamic amplitude. at 1 Hz; free length: 25 mm, cross-section: 4.7 mm x 30 μ m

High Temperatures to 800°C

Along with low-temperature applications, the DMA 303 *Eplexor*® allows for testing medium- and high-temperature materials, e.g., steel and ceramics, up to 800°C. Inconel 625 is a nickel-based superalloy that is known for its excellent resistance to corrosion in high-temperature environments. It is used, for instance, in exhaust systems or gas turbine blades, where its temperature-dependent stiffness is of crucial importance.

The specimen was measured in 3-point bending to 800° C. The measured modulus agrees very well with the literature values obtained by tensile tests. A significant effect can be seen in tan δ at 710° C accompanied by two minor effects between 450° C and 600° C, which are most likely related to the precipitation hardening phenomenon in Ni-based alloys. This also contributes to better understanding of high-temperature materials and their heat treatment.

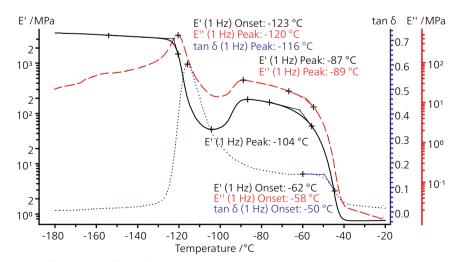


Inconel 625 in 3-point bending
Measurement parameters: 25°C to 800°C at 5 K/min, 40 µm dynamic amplitude at 1 Hz; bending length: 40 mm, cross-section: 1 mm x 9 mm

Low Temperatures down to -170°C

Precise knowledge of the glass transition temperature of sealing materials is indispensable for their proper application.

This silicone sample shows a distinct transition at -120°C, followed by recrystallization starting at -104°C, resulting in an increase in both modulus values. At -62°C (onset E'), the crystals begin to melt and the material exhibits its well-known pasty behavior.



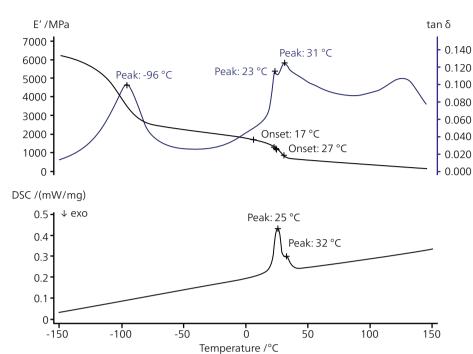
Silicone in tensile mode
Measurement parameters: -180°C to 0°C at 2 K/min, 80 μm dynamic amplitude at 1 Hz; free length: 11.5 mm, cross-section: 0.96 mm x 6.4 mm

Homogeneous Temperature Distribution

The resolution of a DMA depends on sensor precision, homogeneity of the temperature distribution in the furnace and accuracy of temperature control.

A comparison of DSC and DMA measurements on PTFE samples shows that the *Proteus®* software can combine different characterization methods. The DMA measurement depicts a clear glass transition at -96°C (peak tan δ), while the glass transition temperature (change in heat capacity, Δ c_p) is too small to be detected in the DSC curve.

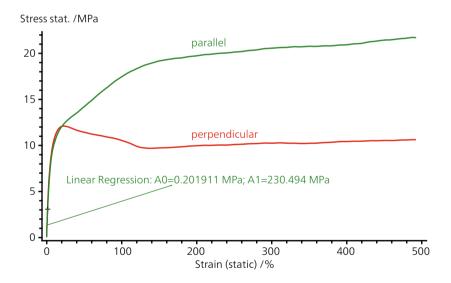
Uniform temperature distribution in the furnace of the DMA 303 $Eplexor^{\circ}$ leads to the clear separation of the tan δ peaks at 23°C and 31°C.



PTFE in 3-point bending
Measurement parameters: -150°C to +150°C at 2 K/min, 60 μm dynamic amplitude: at 1 Hz; bending length: 40 mm, cross-section: 1 mm x 8 mm

Universal Testing

By means of of the "Universal tensile testing" measuring program, tempered uniaxial tensile tests can be realized. Two samples of a PE film were tested to an elongation of 500% to analyze the direction-dependent material behavior (parallel and perpendicular to the manufacturing direction). For elongations of less than 10%, both samples show an almost linear behavior between stress and strain, whereby strictly speaking, differentitation would have to be made between the linear and non-linear viscoelastic behavior. In the subsequent non-linear flow range, plastic flow and stretching of the material occur. The maximum strength values are higher for polymer chains that are aligned parallel to the load direction.



PE film in tensile mode Measurement parameters: Universal tensile test up to 500% with a strain rate of 20%/s; free length: 5 mm, cross-section: 0.05 mm x 7 mm

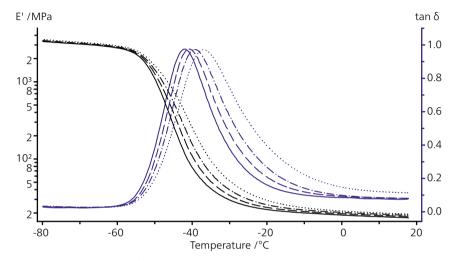


Generating a Master Curve

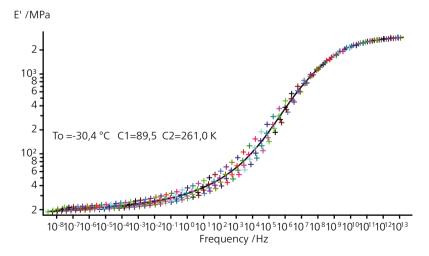
Generation of a master curve via time-temperature superposition makes characterization of viscoelastic properties at frequencies that are significantly beyond the measurable range possible. By using this technique, viscoelastic values at extremely high or low frequencies can be predicted.

At increasing frequencies, the glass transition is shifted to higher temperatures – in other words, the material becomes stiffer. Based on this multi-frequency measurement (see upper plot), the *Proteus* software calculates the shifted master curve and the WLF coefficients shown in the lower plot. In this case, the behavior of storage modulus E' from 10-8 to 10-13 Hz is depicted at a reference temperature of -30°C.

Predictions of the material's behavior under long-term loading or for applications where high frequencies play a role – like sound insulation – are possible.



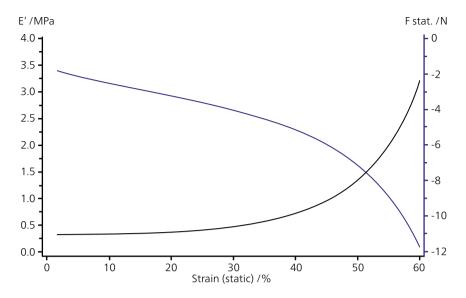
EPDM in tensile modeMeasurement parameters: -80°C to +20°C at 1 K/min, 0.1% dynamic strain at 5, 10, 20 and 50 Hz; free length: 24 mm, cross-section: 1.85 mm x 3.28 mm



Master curve of an EPDM sample at a reference temperature of -30 $^{\circ}$ C

The Effect of Different Levels of Strain

The degree of deformation of foam materials has significant influence on their stiffness and energy dissipation. A piece of open-cell foam was measured in compression mode at several static strains. Up to about 30-% strain, the static force changes slowly, but as the strain increases, the cells begin to collapse and the static force and dynamic modulus significantly increase. By introducing a new measurement mode, dynamic measurements with an increasing static force and thus rising global deformations can now be performed.



PUR foam in compression Measurement parameters: static load sweep up to 60% strain, 30 µm dynamic amp. at 1 Hz; specimen dimensions: 7 mm x 7 mm x 9 mm

FOR ANY APPLICATION Sample Holders for Different Modes

Sample Holder	Sample Dimensions			Applications
Single/Dual Cantilever	Free Bending Length*	Width (max.)	Height (max.)	
Standard	(2×)5 mm (2×)17 mm	13 mm 13 mm	10 mm 10 mm	Thermoplastics, elastomers
3-Point Bending	Free Bending Length*	Width (max.)		
Round-edged	10 mm 20 mm 30 mm 40 mm 50 mm 60 mm	13 mm 13 mm 13 mm 13 mm 13 mm 13 mm		Fiber-reinforced or highly filled thermoplastics
Tension	Length*	Width	Thickness	
Standard	30 mm	13 mm	5 mm	Films, fibers, thin rubber strips
Compression/ Penetration	Sample Ø (max.)	Probe Ø [mm]	Height (max.)	
Standard	15 mm	1, 3, 15	25 mm	Soft samples; e.g., rubber
Shear	Ø/Width/Height (max.)	Thickness (max.)		
Flat surfaces	15 mm	5	mm	Adhesives, elastomers

 $^{^{\}star}$ The samples must be greater in length than the free bending and free tension length values listed here.

	OMA 303 Eplexor®		
Temperature range	-170°C to 800°C		
Heating rate	0.01 K/min to 40 K/min		
Frequency range	0.001 Hz to 150 Hz		
Force range	From 1 mN to 50 N		
Maximum controlled amplitude	± 2.5 mm		
Static deformation	Up to 30 mm		
Cooling device	Liquid nitrogen: -170°C to 800°CAIC 80 air intracooler: -70°C to 800°C;		
Deformation modes	 3-point bending Single / dual cantilever bending Shear Tension Compression/penetration 		
Additional measurement modes	Universal testCreep/relaxationLoad sweep		
Sample geometries	Dependent on the deformation mode, e.g., for 3-point bending maximum sample dimensions: length: 60 mm, width: 13 mm, thickness: 10 mm		
Optional accessories	Immersion bathDielectric Analyzer (DEA)		

Technical Specifications

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 4,000 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 60 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.

Proven Excellence.

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