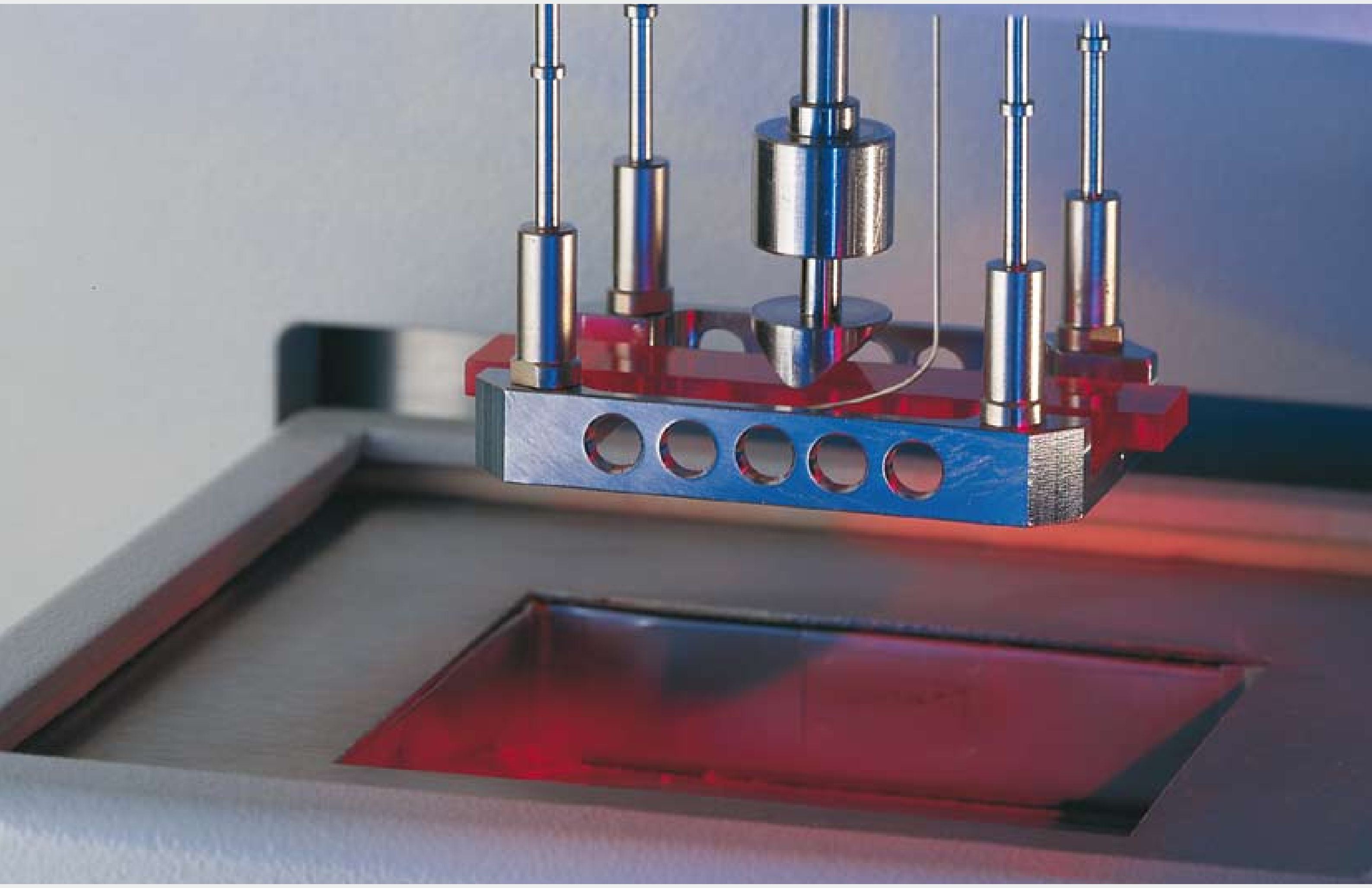
NETZSCH

Dynamic-Mechanical Analysis



Leading Thermal Analysis.

DMA 242 C

-170°C to 600°C

Dynamic-Mechanical Analysis - DMA 242 C

With **D**ynamic **M**echanical **A**nalysis (**DMA**) it is possible to make a quantitative determinationof the mechanical properties of a sample under an oscillating load and as a function of temperature, time and frequency (DIN 53513, DIN EN ISO 6721, DIN 53440, DIN-IEC 1006, ASTM D 4065, ASTM D 4092, ASTM D 4473, ASTM D 5023, ASTM D 5024, ASTM D 5026, ASTM D 5418).
Most materials are both elastic (stiff) and viscous (damping behavior), i.e. they are **viscoelastic**. Thus they yield to an applied load, partially through viscous flow, with a permanent deformation. At the same time, the mechanical behavior is a function of temperature, time, degree and type of load. Structural transformations (e.g. glass transitions, secondary relaxations, cross-linking) are seen in considerable changes in the thermal and mechanical properties, which are demonstrated with the DMA. The DMA is more sensitive as compared to the DSC (Differential Scanning Calorimetry), especially for the investigation of relaxation processes.

Dynamic-mechanical analysis provides the user with extensive, practical information:

- Operable temperature ranges for application andprocessing (glass transition temperature, onset of softening and brittleness)
- Design data concerning stiffness and damping properties (modulus values, damping factor)
- Data on the composition and structure of polymers and polymer blends (compatibility)
- Curing, Vulcanization
- Ageing

DMA 242 C - significant and dependable information for

- research and development
- quality control
- quality assurance
- failure analysis


DMA 242 C

DMA 242 C - Your Versatile Analyzer

The versatility of the DMA 242 C can be seen in the **variety of sample holders**. Thus, depending on the type and consistency of your samples, you can carry out precise investigations of viscoelastic properties covering a **wide modulus range** of **10^{-3} MPa** to **10^{6} MPa**.

- **Polymers**
  thermoplastics
  thermosets
  elastomers
  composites
  lacquers and coatings
  adhesives
  fibers and films
  pastes
  biopolymers
- **Pharmaceuticals**
- **Food**
- **Ceramic Materials**
- **Glasses**
- **Metals**

Tests run in the temperature range from **-170°C** to **600°C** provide you with important data on your material. Dynamic, isothermal and stepwise temperature control make it possible to design flexible temperature programs suited to your particular problem.

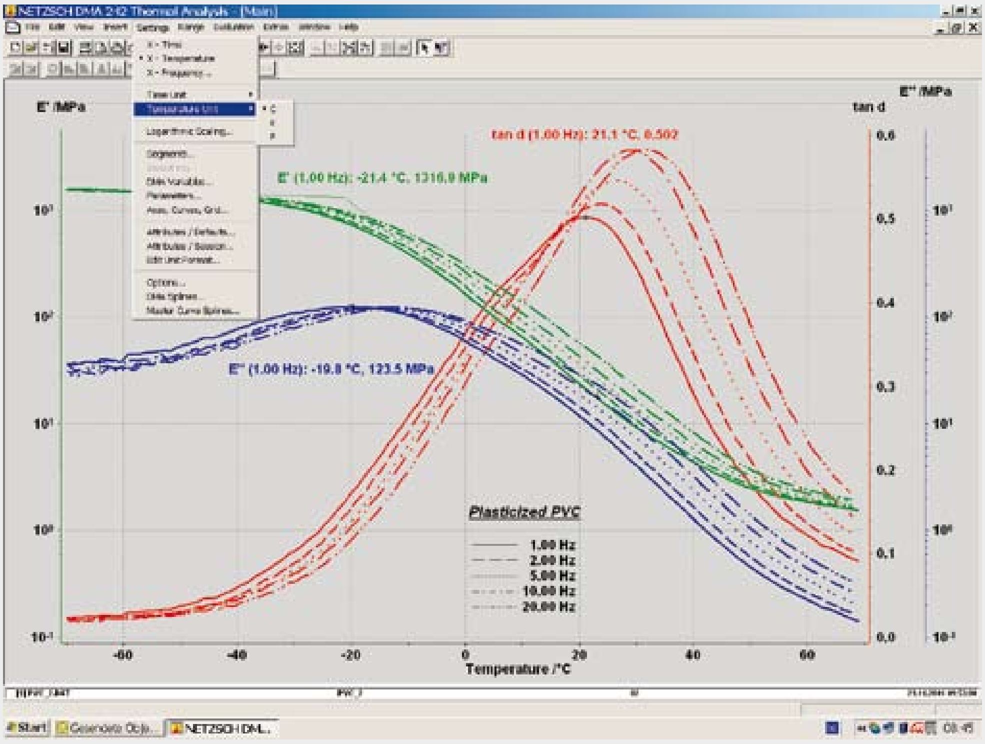
The sample is subjected to a **defined, forced oscillation**. A sinusoidal force (input signal) affects a deformation of the viscoelastic sample (output signal). This also produces sinusoidal, but phase-shifted, oscillation which is then recorded. These oscillations are reduced to a signal of extremely low noise level in the DMA controller by means of **FOURIER Analysis**.

The frequency range of **0.01 Hz to 100 Hz** includes up to 25 fixed frequencies, which can be selected individually or in any combination (multiple frequency). The **maximum force is 16 N** - i.e. 8 N dynamic and 8 N static.

The deformation amplitude (**approx. 0.1 µm to 240 µm**) as well as the sample position are controlled independently of one another. Thereby, constant contact between the push rod and the sample is guaranteed, even when the material softens greatly. This is imperative for the study of polymers, because the storage modulus frequently changes by several orders of magnitude during a measurement.

The patented displacement sensor (DE 4309530C2) with the new automatic ranging feature always guarantees the highest **resolution of the deformation amplitude up to 0.5 nm.**

32-bit NETZSCH *Proteus*® Software



DMA 242 C - Intelligent Technique

Dynamic

The oscillator converts an electrical signal into a force, which is applied to the sample via the push rod. The **broad oscillation frequency range** of 0.01 Hz to 100 Hz is made possible by the high degree of inherent rigidity and the stability of the instr-vedument. The position of the push rod registered by the displacement transducer shows the resulting deformation. The displacement transducer, which was specially developed for the DMA 242 C, measures the deformation with greatest sensitivity and, even at higher frequencies, with no distortion or time lag. The subsequent digital filtering, achieved by means of **FOURIER Analysis**, provides an **excellent signal/noise ratio**. This makes it possible to resolve even the smallest tan δ values.

Mechanical

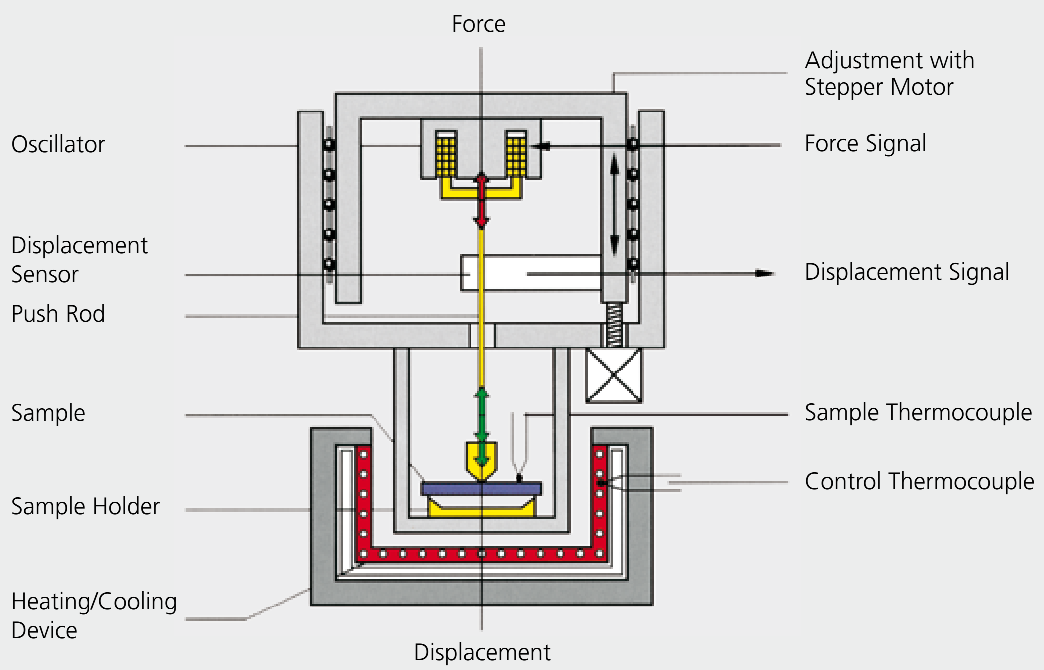
The **vertical design** and backward movement of the furnace into parking position provide free access for changing the various sample holders (three-point bending, single/dual cantilever bending, compression/penetration, shearing tension). Compensation is made for dimensional changes occurring during the measurement (expansion, shrinkage, creep) by moving the oscillator and the displacement transducer against the sample holder using a **stepper motor**. In tension, compression, penetration and threepoint bending the dynamic force is superimposed by a static preliminary force. Thus, even with great changes in the modulus, secure positioning of the sample is guaranteed. The static preliminary force is controlled according to the particular problem:

I. constant value (tension or pressure)
II. proportional (the initial stress on the sample is fit to the current modulus value)
III. combination of I. and II.

Of course, operation under constant static force is possible as well (TMA mode).

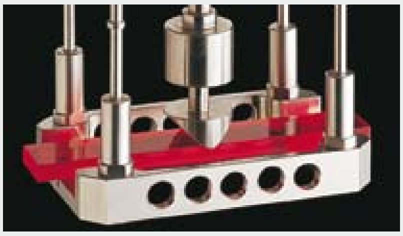
Thermal

The rectangular cross section of the furnace is fit to the sample geometry, such that the sample is **heated** constantly and **homogeneously**. The temperature gradient is minimized to ± 1°C over a sample length of 60 mm. The sample temperature is registered by a sample thermocouple and precisely controlled via the STC function (Sample Temperature Control). With controlled cooling using liquid nitrogen, temperatures to -170°C can be achieved with minimal consumption.



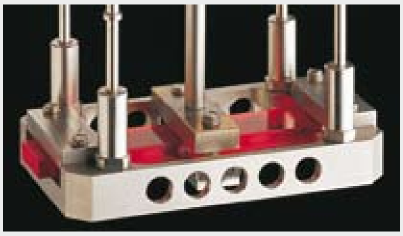
Application-specific Sample Holders

The DMA 242 C covers an extremaly broad range of sample geometries. Through the use of relatively large samples (max. sample length 60 mm in the bending mode), an excellent extrapolation of the results to conponent parts is achieved. By using a container, measurements in liquids are possible (immersion tests), Select the ideal sample holder* for your particular problem:



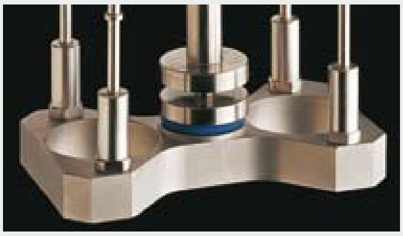
Three-point Bending

The sample is supported on two edges, while the cutting end of the push rod applies a load to the sample from above. The spacing between the two edges is in accordance with DIN 53457. Four different free bending lengths between 10 mm and 50 mm are available. This deformation mode is ideal for materials with a high storage modulus such as filled or reinforced thermoplastics and thermosets (composites or metals, alloys and ceramics).



Dual Cantilever Bending

The ends of the sample are tightly clamped. This arrangement is especially suitable for samples of mid modulus range (rubber and thermoplastics). Holders for three different bending lengths between 2 mm and 32 mm are available. By leaving one end of the sample free, measurements can be made in the **single cantilever bending** mode as well.

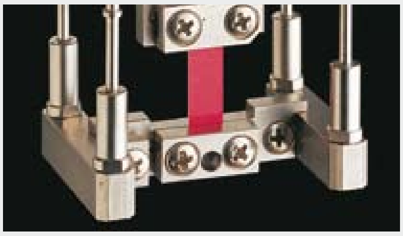


Compression

The sample (rubber, foams, biopolymers, pasty materials, etc.) is placed on a flat surface in the sample holder and pressure is applied from above with the plate-shaped end of the push rod. Different diameters up to 30 mm are available. By using a ceramic disk and a push rod of fused silica, a small temperature gradient can be achiev (especially for foams and rubber). If the end of the push rod is pointed rather than flat, the term is **penetration**. For example, this type of stressing is suitable for the investigation of coatings.

Linear Sharing

Two identical samples are pressured or glued in sandwich geometry between the flat surfaces of the sample holder and the push rod. The load is applied via the platelike extension of the push rod. Two types of sample holders are especially suited for soft materials such as rubber and adhesives.



Tension

The tension mode is preferable for the measurement of films and fibers or thin rubber sample. The lower end of the sample is held in place, whereas the upper end is clamped to the oscillating push rod.

* special deformation modes e.g. for viscous liquids or very stiff samples upon request

DMA 242 C - Advanced *Proteus*® Software

The **NETZSCH 32-bit *Proteus*® Software** runs with the **operating systems Windows® XP Professional or Windows Vista® Business and Enterprise** has multi-tasking capability and features:

- compatibility with standard computer systems
- a well-known user interface with most programs available on the market
- easy, user-friendly operation
- standard DMA evaluation routines
- quick reporting capability with graphic integration with text.

Clearly understood user input with pull-down menus helps you **program** your measurement. Here you select multiple programmable temperature segments (isotherm or dynamic) or temperature ramps with single or multiple frequencies, freely selectable forces and deformation amplitudes.

Calibration of the instrument, automated to the greatest extent possible, is also carried out with the help of the software:

- dynamic mass calibration
- empty system calibration
- system stiffness calibration
- rotation tuning (recommended for stiff samples)
- temperature calibration

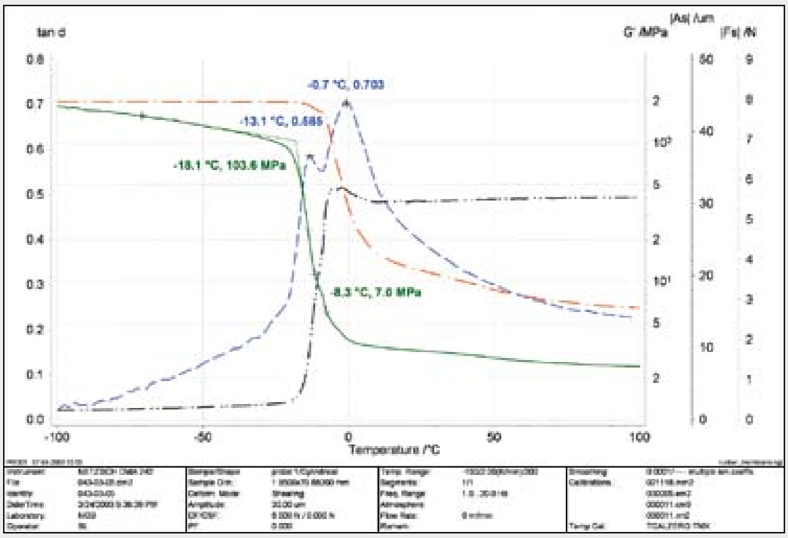
It goes without saying that **multi-tasking** (foreground/background) and **multi-module** operation for up to 4 instruments are integral parts of the software.

Extensive **evaluation routines** are available to you:

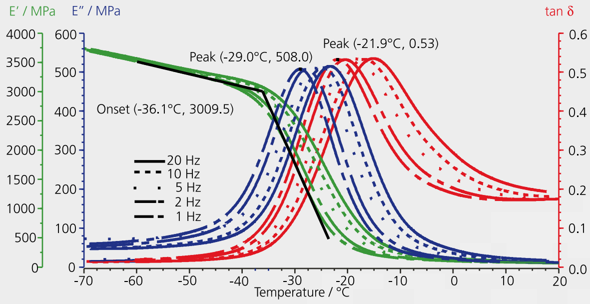
- online evaluation of the measurement in progress (snapshot)
- logarithmic or linear scaling of the resulting values (e.g. storage modu lus E´, loss modulus E´´ and damping factor tan δ) with up to 4 Y axes
- display of the static and dynamic force, deformation amplitude, changes in probe position, viscosity and compliance as a function of temperature/time and frequency
- **TMA**-mode (length change under static load) for creep tests
- adjustable curve properties (color, line thickness, line type) schaften (Farbe, Liniendicke, Linienart)
- 1st and 2nd derivative of every report parameter
- set value on every report parameter curve
- superposition of several measurement curves plotted as a function of frequency (master curve); extrapolation to user-defined temperatures and frequencies beyond the measurement range in accordance with the Williams-Landel-Ferry equation (**WLF**)
- calculation of the activation energy from the shift of the maximum of tan δ with respect to frequency and temperature and extrapolati on (**ARRHENIUS** analysis)
- **Cole-Cole diagram**
- determination of characteristic temperatures such as peak maximum, T_{onset}, T_{endset}
- **curve comparison** of up to 32 data files or segments
- graphical functions PIP (picture in picture) and FLIP (inverse PIP)
- grid overlay
- fast zoom functions
- output of results on any standard WINDOWS-compatible printing device
- storage of the analysis status (results and graphics)
- data export (ASCII, copy to clip board, Bitmap, Enhanced Metafile Format)
- E-mail support
- 7 different print languages

Graphic output with entire documentation

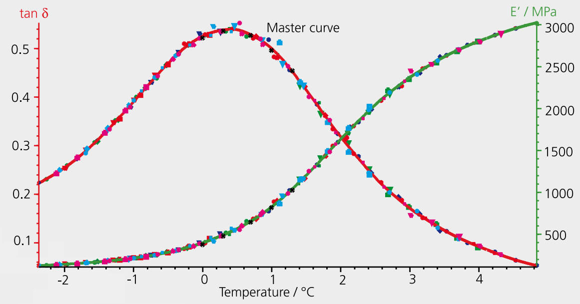
In addition to the data most frequently used in practice, such as storage and loss moduli and the loss factor tan δ, the software also allows the presentation of sample amplitude, static and dynamic sample force. The plot shows a multiple frequency test, evaluated at 1 Hz, for a rubber membrane.



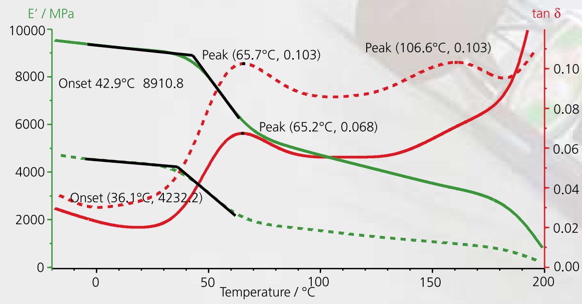
DMA 242 C - Wide-ranging Applications



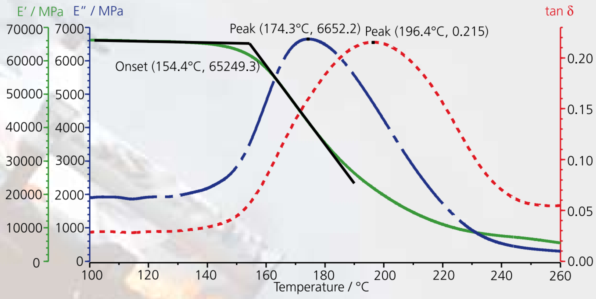
The influence of frequency is demonstrated with the example of an SBR rubber mixture. As expected, with increasing frequency, the T_g (evaluated at 1 Hz) is shifted to a higher temperature and higher E´ values are obtained (multi-frequency measurement in the dual cantilever bending at 2 K/min).



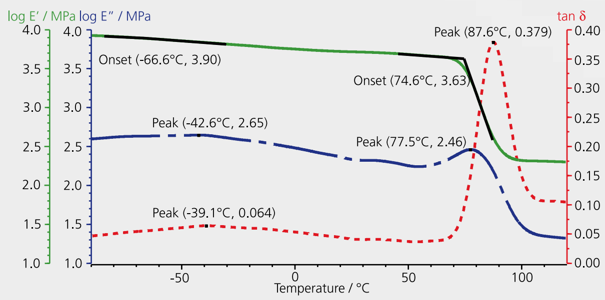
With a multi-frequency measurement, frequencies beyond the measurable range of the DMA can be achieved by using the superposition method.
Employing the Williams-Landel-Ferry (WLF) equation, e.g. E´- and tan δ values at a certain reference temperature (here -20 °C) can be extrapolated to 100.000 Hz.



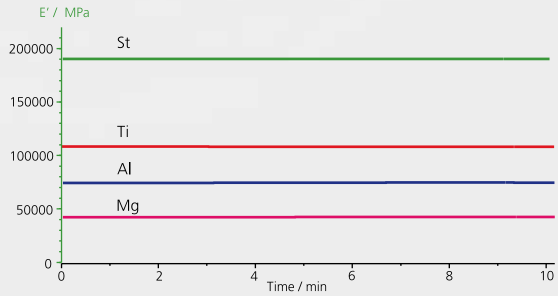
A 30% glass fiber-reinforced PBT (parallel and perpendicular to the fiber orientation) was measured in the 3-point bending mode at 1 Hz and 2 K/min. Considerably higher values for the stiffness and the onset of the E´ decrease (43°C) are obtained for the parallel-oriented type (straight line). The tan δ values are accordingly lower. The tan δ peaks are at the same temperature.



A carbon fiber-reinforced epoxy resin was measured in the 3-point bending mode at 1 Hz and 3 K/min. Even at a temperature of 140°C, the stiffness values are of the same magnitude as those for aluminium. Glass transition starts at 154°C (extrapolated onset). The E´´ (174°C) and tan δ peak (196°C) are also characteristic values for the glass transition temperature.



The polyester fiber tested in the tension mode shows relaxations in the low-temperature range, which can be evaluated as E' onset, E´´ peak or tan δ peak.
werden können. Die Glasumwandlung beginnt bei
Glass transition starts at 75°C. The storage modulus decreased from approx. 4.200 MPa to 200 MPa.



The single cantilever mode with a special sample fixture in combination with a free push rod is preferable for the determination of the bending modulus of metals. Presented in this figure is the modulus series of sheet-metal strips of steel, titanium, aluminum and magnesium at room temperature and 1 Hz.

DMA 242 C - Technical Data and Worldwide Service

Modes of deformation
- three-point bending
- single/dual cantilever bending
- shearing
- compression/penetration
- tension
- special deformation modes upon request
- creep / relaxation
- stress / strain sweep
- TMA mode

Sample size
e.g. for bending
length: max. 60 mm
width: max. 12 mm
thickness: max. 6 mm
(dependent on deformation mode, details upon request)

Modulus range (E′)
10^{-3} MPa to 10^{6} MPa
(dependent on deformation mode)

Frequency range
0.01 Hz to 100 Hz

Measuring range tan δ
0.00006 to 10

Controlled stress (force)
e.g. for bending
max. 16 N
static: 8 N
(tension or procedure)
dynamic: ± 8 N

Controlled strain (amplitude ranges)
max. 240 µm
Autoranging with highest resolution (nm range)

Heating and cooling system
Temperature range:
-170°C to 600°C
Cooling time:
20°C to -150°C in 10 min
(cooling agent liquid nitrogen)
Heating rates:
0.01 K/min to 20 K/min
Temperature homogeneity:
± 1 K (for sample length 60 mm)
Thermostatic control (optional)

Atmosphere
inert and reactive purge gases (non-toxic, non-flammable, non-explosive)

Unique gas sealing

Immersion test capability
for all sample holders

Photo-DMA (for UV-curing)

Simultaneous DMA-DEA

Temperature control
TA System Controller
TASC 414 for temperature programming and sample temperature and real-time program voltage adjustment (PID and STC), data acquisition, IEEE interface, control functions for cooling

Fourier analysis
Optimizes signal for maximum sensitivity and noise reduction.

Automatic calibration routines

User friendly help system

NETZSCH *Proteus*® Software runs with the operating system Windows® XP Professional or Windows Vista® Business and Enterprise

Technical data subject to change

NBG · DMA 242 C · E · 1000 · 0709 · LH

Leading Thermal Analysis.

NETZSCH

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