

# High-Force DMA 503 *Eplexor*<sup>®</sup> Series up to $\pm$ 500 N

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Dynamic Mechanical Testing Systems

Analyzing & Testing



# Smart Material Testing UP TO HIGH FORCES

More than 40 years of experience in R&D for premiumquality dynamic mechanical analysis

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Successful introduction of materials and products onto the market is always preceded by a great deal of testing to determine suitability in day-to-day applications. Temperature, time, frequency, load level and other parameters change the viscoelastic properties of polymers and composites.

- How are your products influenced by different temperatures or moistures?
- How do the material properties depend on mechanical loading or different frequencies?
- What is the lifetime of the material under extreme stress conditions (cycling, hysteresis, etc.)?

NETZSCH instruments can answer these questions and more using our trend-setting technology in the field of material development and design. Our product line – designed by experts for experts – offers cost-effective and competitive solutions for nearly any experimental task.

The DMA 503 *Eplexor*<sup>®</sup> instrument series is in line with a variety of standards including DIN 53513, ISO 6721/1, ISO 6721/4, ISO 6721/5, ISO 6721/6, ISO 4664, ASTM D4065, and ASTM D4473.

### Dynamic Mechanical Analysis

The Dynamic Mechanical Thermal Analyzer applies forced periodic loads to the sample and analyzes the phase shift between this primary excitation and the material's response. The response of an ideal elastic system (e.g., spring) on a sinusoidal load at a given frequency is of the same frequency and exactly in phase with the excitation. The situation changes in a real system: A phase shift ( $\delta > 0^\circ$ ) between the primary excitation and response of the same frequency occurs in the case of linear visco-elastic materials (e.g., polymers); see figure 1.

Elastic and non-elastic properties inherently describe the dynamic mechanical performance of the material. The storage modulus E', the real part of the complex modulus E\*, represents the elastic component; the loss modulus E'', the dissipated part, is the imaginary part. Depicted in the complex plane, the loss and storage modulus are the projections of the complex modulus onto the real and imaginary axis (figure 2). The tangent of the angle between the real axis and the complex modulus (E\*) represents the phase shift (tan  $\delta$ ) between the two.

#### DMA Results and Testing Possibilities

- Dynamic modulus
- Damping factor (tan δ)
- Young's (static) modulus
- Frequency dependence
- Temperature dependence
- Glass transition
- Secondary transition
- Master curve
- Hysteresis
- Relaxation and retardation
- Creep testing
- Aging behavior
- Fatigue
- Predictive testing
- Immersion test
- Tests under controlled humid atmosphere





From honey to steel − the DMA 503 Eplexor® analyzes the mechanical properties of all kinds of materials.

Figure 1: Viscoelastic properties in the complex plane; various material classes exhibit significant differences in phase shifts Figure 2: Measured parameters: damping ( $\delta$ ), force and strain amplitudes

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# DMA 503 *Eplexor*® Series MODULAR DESIGN – ALL SET FOR THE FUTURE



#### Set for the Future

The DMA 503 *Eplexor*<sup>®</sup> series allows for easy upgrading the system with a variety of force sensors and strain extensions as well as furnaces. For non-stop operation, a fully automatic sample changer is available. The different DMA 503 *Eplexor*<sup>®</sup> versions are operated without a hydraulic pump system – thus providing energy-efficient operation.

#### Wide Deformation Range at High Resolution

Strain is accurately measured by two contactless optical sensors. This allows nanometer resolution throughout the entire range of up to 80 mm static displacement and up to  $\pm$  6 mm dynamic amplitude.

### Two Independent Drives for Static and Dynamic Force

Just as with the base DMA 503 *Eplexor*<sup>®</sup>, the DMA 503 *Eplexor*<sup>®</sup> *HT* instrument operates with two independent drives for static and dynamic force. Application of this principle is the only way of utilizing the entirety of the broad range of force when carrying out DMA measurements.

#### Broadest Temperature Range

By combining the high-temperature furnace up to 1500°C with a cooled low-temperature furnace, a range from -160°C to 1500°C can be covered in one DMA instrument. The furnaces are easily interchangeable by the user.

#### **Rigid Design**

High temperatures place high demands on the flexural strength of the fixture within the hot-temperature zone. The DMA 503 *Eplexor® HT* instrument is equipped with sample holders made of sintered silicon carbide (SSiC), a dense and stiff ceramic that maintains this property even at 1500°C.

#### Variable Force Range

This one-of-a-kind DMA system offers the ability to install different force sensors to meet individual users' needs, with a total force range from 10 N to 2500 N. This allows for applications of the best possible conditions in order to optimize results. The force sensors can be changed easily by the operator with a minimum of effort.

### Chose From Three Different Dynamic Force Ranges in the DMA 503 *Eplexor*<sup>®</sup> Series

Force Ranges for DMA 503 Eplexor <sup>®</sup> and DMA 503 Eplexor <sup>®</sup> HT					
Dynamic	± 100 N (200 N)*	± 150 N (300 N)*	± 500 N (1000 N)*		
Static	1500 N	1500 N	1500 N		

\* Peak-to-Peak

# First-Rate Performance via Stiff Frame and Real-Time Operation

### DMA 503 Eplexor®

#### Unique by Virtue of Real-Time Operation and Digital Signal Generator

The DMA system allows for real-time operation.

The quasi-continuous frequency range with high data acquisition rate allows operation between 0.0001 Hz and 100 Hz. Frequency sweeps with high detection sensitivity are suitable for detecting even weak transitions and can be taken for master curve generation. For fatigue measurements, the maximum frequency can be extended to 200 Hz.

The optional digital signal generator for conventional and pulse DMA is a prerequisite for generating excitation signals of various shapes including pulses by harmonic synthesis. Predefined signal shapes (e.g., rectangular, saw tooth, Hanning, Hamming, Blackman, Blackman-Harris, or user-defined shapes) can be generated by optional software tools\*.

\* Please contact your local representative for more information



Schematic of the Eplexor®: force sensors are interchangeable

### DMA 503 Eplexor®

## THE PERFECT SAMPLE HOLDER FOR ANY APPLICATION

All investigations, characterizations and quality control tests conducted on a given material should be aligned with its intended application. The results obtained via measurements which closely parallel real applications allow for more accurate appraisals of a material's true performance.

A variety of sample holders in standard geometries (e.g., bending, tension) are available, as well as in dedicated user-defined geometries. Immersion systems are also offered, see page 9.



Compression



Tension



Tension for stiff samples



Shear



3-point bending



4-point bending



Dual cantilever



Tackiness

# HUMIDITY SIMULATION OF ENVIRONMENTAL INFLUENCES

Environmental conditions may cause water uptake and therefore play a decisive role in manufacturing processes and the storage of materials. The DMA 503 *Eplexor*<sup>®</sup> in combination with the HYGROMATOR<sup>®</sup> humidity generator is able to create a humid environment inside the test chamber to reveal the influences of environmental humidity on mechanical properties.

#### HYGROMATOR<sup>®</sup> – The World's First Climate Chamber for DMA

The HYGROMATOR<sup>®</sup> can adjust humidities from 5% rH to 95% rH in the temperature range between 5°C and 95°C. Measurements can be performed under stepwise humidity variation. Alternatively, linear humidity ramps can be set up to increase or decrease humidity. Stabilized conditions are possible up to 24 h.



Humidity Range: 5% rH to 95% rH

in a temperature range from 5°C to 95°C

### IMMERSION FOR TESTING SWELLING BEHAVIOR AND OTHER EFFECTS

The immersion bath allows for measurements in a liquid such as water or oil in combination with sample holders such as those for 3-point bending tests (picture on the right) or tension tests (picture on the left), in cases where evaporation and decomposition temperature of the solvent or liquid are not an issue.



Sample holder for tensile tests (left) and bending tests (right) in an immersion bath



Results of room temperature measurements on rubber: prestrain 20%, frequency 10 Hz; amplitude scans were applied after each aging step; E' significantly decreases with immersion time.



# MEASUREMENTS IN PASSING

With the Automatic Sample Changer (ASC), the DMA 503 *Eplexor*<sup>®</sup> model becomes a fully automated testing system. These systems provide measurement results with the same or even higher level of reproducibility compared to manual sample changing but may be operated 24/7.

The ASC is offered in different configurations for up to 160 samples.



ASC gripper changing out the double shear sample holder

#### Automatic Sample Changer

You can select your required samples in tensile, compression, shear or bending. The Automatic Sample Changer (ASC) removes the samples from the magazine and places them into the temperature chamber at analysis position. All tests are run fully automatically. All the user has to do is select the test geometry, install the appropriate gripper, and start the system in automatic mode.

# TACKINESS EXTENSION

The tackiness extension for the DMA 503 *Eplexor*<sup>®</sup> and Gabometer<sup>®</sup> uses a patented procedure for the reliable measurement of the peel force of two adhesive components.



#### **Application Range**

Tackiness of "green tires", adhesives and paint layers.

#### **Special Features**

Adhesions are applied with a force of up to 500 N; adhesion forces are recorded with a resolution of 10 mN and up to 50 measuring points per second.



normal force 20 N, duration 20 s

Sample holder for tackiness tests



### The DMA 503 *Eplexor*® *HT* UNIQUE SYSTEMS FOR DYNAMIC TESTS ON METALS, GLASSES, CERAMICS OR COMPOSITES



### Two Temperature Chambers for the Broadest Temperature Range

With the DMA 503 *Eplexor® HT*, the user can choose between two different furnaces: a standard furnace which runs from -160°C to 500°C and a high-temperature furnaces which runs from ambient to 1500°C.

To take advantage of the full temperature range, a standard and high temperature furnace can be mounted together on the same system.

#### Designed to Investigate Metals, Glasses, Ceramics or Composites

The DMA 503 *Eplexor*<sup>®</sup> series up to  $\pm$  500 N uses a very stiff frame, which allows for direct compression tests on solids in strain-controlled measurements. The low compliance enables ideally suited conditions for studying even very stiff samples. The digital dynamic strain resolution amounts to 1 nm, which is advantageous for very precise measurements.

# Accessories for the DMA 503 *Eplexor*® *HT*

To ensure reliable measurement results, sample holders are available for a variety of sample geometries and temperature ranges.

- Compression holder: made of SSiC, up to 1500°C at 1000 N
- 3-point bending; 20- and 40-mm free bending length, made of SSiC, up to 1500°C at 500 N
- Tension holder: made of a high-temperature resistant alloy, up to 900°C

Sintered Silicon Carbide (SSiC) is a stiff, high temperature and thermal shock resistant material with high wear resistance. This makes it the optimum material for measurements up to 1500°C.

In addition, all sample holders associated with the DMA 503 *Eplexor*<sup>®</sup> 500 N system, such as double shear or single-/dual cantilever, can be used for lower temperature applications to 500°C.



3-point bending

Compression

Tension

# Applications DMA 503 Eplexor®

#### Influence of Carbon Black in Rubber

The *Eplexor*<sup>®</sup> is perfectly suited to investigate the influence of different filler recipes. In this example, Butyl Rubber (BR) filled with 50 phr carbon black is compared to BR without filler

in compression mode. Due to the carbon black content, the elasticity modulus of the filled BR is about 10 times higher than that of the pure BR at temperatures above 0°C. Both systems exhibit a very broad glass transition area covering a temperature range of about 50°C (half-width of the tan  $\delta$  peak). However, the tan  $\delta$  peak heights of the two systems are significantly different from one another.



BR in compression mode

Measurement parameters: -80°C to +80°C; static strain 4 %; dynamic strain ± 0,2 % at 10 Hz; sample dimensions: Ø 10 mm x 10 mm height

#### Payne Effect – Common Test for Rubber Materials

The Payne effect only occurs in filled elastomers and develops in the filler network. It causes a decrease in storage modulus and is a function of such filler-filler interactions as the breakage of physical bonds between filler particles. Once the strain is released, this becomes a reversing effect after some time. This plot shows the results of strain sweeps on a rubber material in double shear mode. The modulus decreases with increasing deformation due to the weakening filler-filler interactions.



Filled elastomer in double shear mode

#### Time-Temperature Superposition

Rubber applications often involve dynamic stress over a broad frequency range. Investigations of the frequency dependence of dynamic mechanical properties are therefore often used to provide data for the development of proper rubber compounds. This plot shows a multi-frequency test result of a rubber loaded in tensile mode. Using Time-Temperature Superposition (TTS; red dots), a master curve can be calculated (blue curve; per the Williams-Landel-Ferry equation).



Measurement on a rubber material between -80°C and 80°C, 5 K interval, temperature was kept constant and a frequency scan was performed from 0.5 Hz to 50 Hz; frequency extrapolated above the GHz range.

### Temperature Sweeps

#### Thermal Treatment of PP

Temperature sweeps are useful to analyze the viscoelastic behavior of a polymer before and after thermal treatment. This plot shows the storage modulus, E', and the viscoelastic damping, tan  $\delta$ , as a function of the temperature of polypropylene. The damping behavior changes significantly due to the thermal exposure in the 1<sup>st</sup> sweep. The glass transition (peaks in tan  $\delta$  curves) shifts slightly to lower temperatures and exhibits a higher damping peak. The tan  $\delta$ shoulder between 50°C and 125°C disappears in the 2<sup>nd</sup> run.



Temperature sweeps in tensile mode: the  $1^{st}$  sweep (blue curves) is compared with the  $2^{nd}$  sweep (red curves). Static strain load: 1%; dynamic strain load: 0.1%, frequency: 10 Hz





#### Composite in Tensile and Bending Mode

A polypropylene (PP) sample with layered glass fibre structure [0/90/0/90/0/90/0] is measured in tensile and bending direction. The storage modulus in bending is higher than in tension over virtually the entire temperature range. This behavior is due to the asymmetric layer structure of the specimen. Since the outer fibers contribute to bending far more than the material in the center, the outer fibers in the direction of the load have a stiffening effect on the sample



Static sweep of PUR foam

Measurement parameters: RT; static strain: 0.5 to 70%; dynamic strain: 0.5% at 10 Hz; sample dimensions:  $18 \times 18 \times 20$  mm.

### Viscolastic Properties of Foam

An open cell PUR foam has been compressed from 0.5% to 70% static strain. Foams typically show a region of linear deformation at small strains, but as strains increase, the cells begin to collapse and the static stress curve flatens, accompanied by a decreasing dynamic modulus. Above 30% static strain, the slope of static stress and the dynamic modulus increase again, which is typically interpreted as further densification of the mostly collapsed cells. With the DMA, the static and dynamic properties of foams can be measured simultaneously for different static loads and temperatures, e.g., at temperatures.

# Applications DMA 503 Eplexor® HT

#### Bending Test on Bauxite\*

Bauxite is an aluminum ore and the basis for the production of metallic aluminum.

In order to characterize the mechanical behavior of bauxite, a bar of material was measured. The temperature was kept constant while increasing the static force by 85 N per second (i.e., approx. 510 N within 6 seconds); under these conditions, the sample exhibited approx. 30 µm of bending.

DMA instruments are not specifically engineered to handle destructive experiments. However, if such events were to occur, the unique blade spring system in the DMA GABO *Eplexor*® *HT* series offers the greatest possible degree of safety.



3-point bending mode; isothermal temperature: 800°C; static force: up to 500 N. The static force is shown in red; the deflection of the sample is shown in blue

\* The measurement was provided by Fraunhofer ISC in Bayreuth, Germany, to whom we extend our sincere appreciation.





### Temperature Sweep of Rubalit<sup>®</sup>\*

Rubalit consists of 96% aluminum oxide  $(Al_2O_3)$  and is frequently used in electronics industry. Its storage modulus is slightly lower compared to pure  $Al_2O_3$ . A temperature sweep uncovers the influence of additional phases at 787°C (Onset E') and 1006°C (Onset E').

\* Rubalit is a trademark name owned by Ceramitec

Rubalit in 3-point bending Measurement parameters: RT to 1200°C at 3 K/min ; static PF 1.5; dynamic amplitude: 30 µm with 10 N force limit at 5 Hz.

#### Bending and Softening of an Al<sub>2</sub>O<sub>3</sub>/Metal Compound\*

Heating a ceramic/metal compound with  $Al_2O_2$  as the ceramic component in the 3-point bending mode to 1450°C results in heavy deformation of the sample. Between RT and 1250/1300°C, bending of about 2 mm takes place, which corresponds to a static strain of approx. 40% (relative to the original specimen height). At about 1400°C (extrapolated onset temperature), bending becomes even more pronounced. This is due to softening of the sample resulting from the melting phase of the metal included.

The DMA 503 *Eplexor® HT* series is specifically designed to reach such high temperatures.



DMA test on  $Al_2O_3$  + metal; 3-point bending mode; heating rate: 5 K/min; static force: 50 N. The static strain is shown in blue; the deflection of the sample is displayed in red.

\* The measurement was provided by Fraunhofer ISC in Bayreuth, Germany, to whom we extend our sincere appreciation.

DMA 503 Eplexor <sup>®</sup> Series up to ± 500 N					
	100 N	150 N	500 N		
Dynamic force range	± 100 N (200 N)*	± 150 N (300 N)*	± 500 N (1000 N)*		
Static force range	1500 N	1500 N	1500 N		
Dynamic displacement	± 2 mm (4 mm)*	± 3 mm (6 mm)*	± 6 mm (12 mm)*		
Static displacement	Up to 80 mm; 45 mm with furnace	Up to 80 mm; 45 mm with furnace	Up to 80 mm; 45 mm with furnace		
Frequency range	0.0001 Hz to 100 Hz; for fatigue up to 200 Hz	0.0001 Hz to 100 Hz; for fatigue up to 200 Hz	0.0001 Hz to 100 Hz; for fatigue up to 200 Hz		
Heating	<ul> <li>DMA 503 Eplexor<sup>®</sup>: RT to 500°C</li> <li>DMA 503 Eplexor<sup>®</sup> HT: RT to 1500°C</li> </ul>	<ul> <li>DMA 503 Eplexor<sup>®</sup>: RT to 500°C</li> <li>DMA 503 Eplexor<sup>®</sup> HT: RT to 1500°C</li> </ul>	<ul> <li>DMA 503 Eplexor<sup>®</sup>: RT to 500°C</li> <li>DMA 503 Eplexor<sup>®</sup> HT: RT to 1500°C</li> </ul>		
Cooling	<ul> <li>AIC: -70°C bis 500°C</li> <li>LN<sub>2</sub>: -160°C to 500°C</li> </ul>	■ AIC: -70°C bis 500°C ■ LN <sub>2</sub> : -160°C to 500°C	■ AIC: -70°C bis 500°C ■ LN <sub>2</sub> : -160°C to 500°C		
Accessories	Automatic sample changer, humidity, immersion, purge gas				
Deformation Modes:	Compression, tension, bending, cantilever, shear				
Software	Master Curve, Segments, Creep/Relax, Load Sweep, Fatigue, Time Sweep, Temperature Sweep, Frequency Sweep, Temperature-Frequency Sweep Universal Test, Hysteresis, Pulse				

\* Peak-to-Peak

# Technical Specifications

The owner-managed NETZSCH Group is a leading global technology company specializing in mechanical, plant and instrument engineering.

Under the management of Erich NETZSCH B.V. & Co. Holding KG, the company consists of the three business units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems, which are geared towards specific industries and products. A worldwide sales and service network has guaranteed customer proximity and competent service 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.

DMA 503 *Eplexor* \* EN · 1224 · Technical specifications are subject to change

NGB.

NETZSCH-Gerätebau GmbH Wittelsbacherstraße 42 95100 Selb, Germany Tel.: +49 9287 881-0 Fax: +49 9287 881-505 at@netzsch.com https://analyzing-testing.netzsch.com