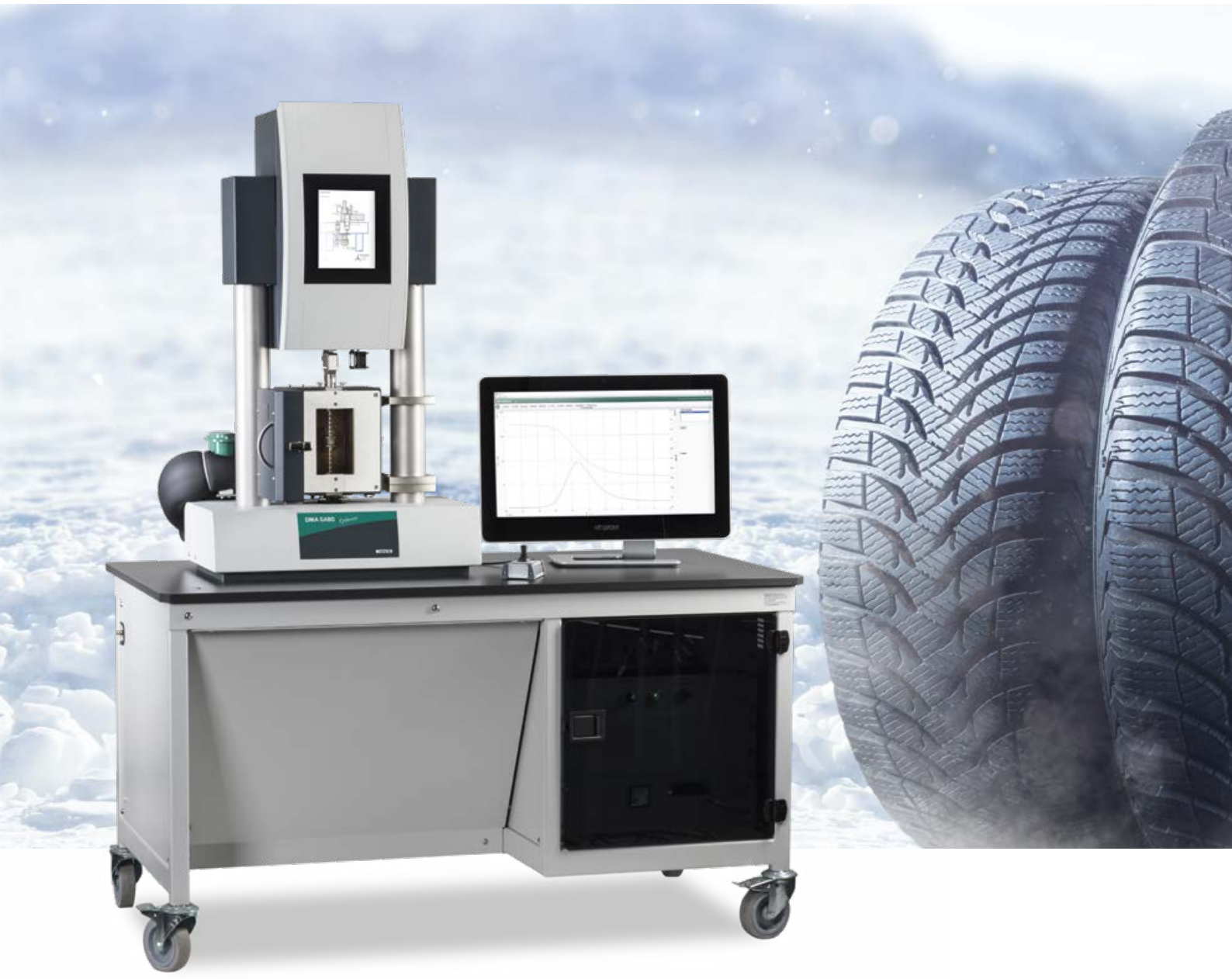
NETZSCH

Proven Excellence.



GABO DiPLEXOR®

Simultaneous Dielectric (DEA) and Dynamic Mechanical Analyzer (DMA)
Methods, Instrumentation, Applications

Analyzing & Testing

GABO DiPLEXOR®

The Perfect Combination of DEA and DMA



* DiPLEXOR® only registered in Europe

With simultaneous DMA and DEA, the dynamic mechanical and dielectrical properties of carbon black-filled rubber compounds can be analyzed.

Simultaneous dielectric (DEA) and dynamic mechanical (DMA) analyses allow for the determination of dielectric material properties under different mechanical loads.

With this combination, structural and dynamic mechanical information can be obtained. The DMA part investigates the viscoelastic properties of substances such as carbon black-filled rubber blends and compounds. The DEA part analyzes the intrinsic structure and distribution of the cluster within the polymer.

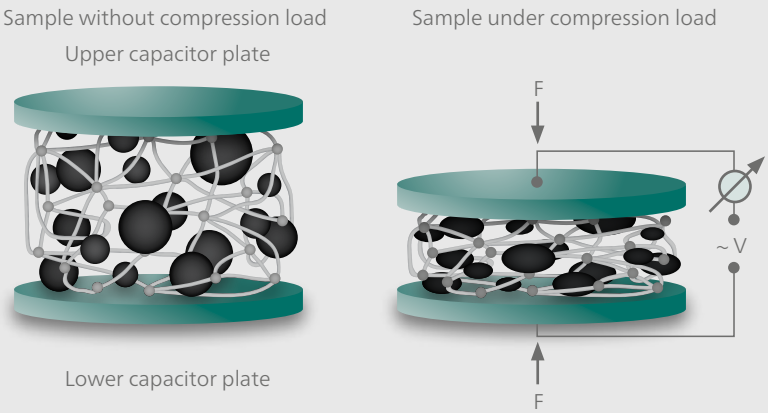
Only by combining DMA and DEA into a simultaneous analysis can one clearly understand changes in the structure, size and distribution of the cluster structure within the polymer matrix due to and during dynamic mechanical loading.

Dynamic mechanical specimen testing causes changes in the mechanical properties of a material. When the mechanical stress is halted, recovery processes are initialized which change the structure again. Only simultaneous DEA and DMA investigations allow for the monitoring of structural changes during dynamic mechanical sample loading processes.

What information does the GABO DiPLEXOR® provide?

Molecular mobility caused by thermal activation is influenced by external mechanical constraints, as are molecular transport and relaxation processes. Static and dynamic sinusoidal sample deformations applied within the scope of experiments change the complex dielectric function, ε*, systematically and allow for analyses of the activated internal migration. These processes can be viewed by monitoring the migration processes of the charge carriers within the sample driven by the electrical field.

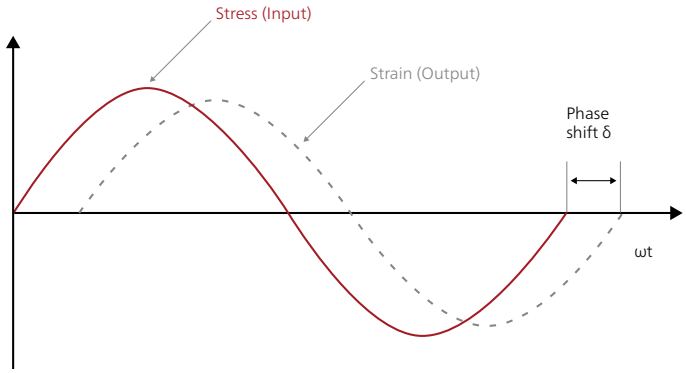
Simultaneous DMA and DEA



A vulcanized “virgin” rubber compound is one that has never been subject to mechanical constraints and differs in structure and morphology from its mechanically loaded counterpart.

In cases where a static (and/or dynamic) load is applied, the structure, size and location of the carbon black cluster changes; this can be analyzed by DEA.

The Dynamic Mechanical Analysis Method – DMA



DMA – Measurement principle

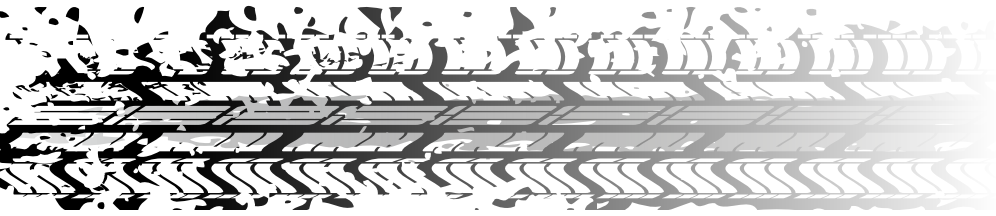
The DiPLEXOR® analyzes linear and non-linear material properties under loads ranging from very low to high.

Dynamic Mechanical Analysis (DMA) measures the viscoelastic properties of polymers, elastomers, rubber compounds, etc., during a controlled temperature and/or frequency program.

During the test, a sinusoidal force F (stress σ) is applied to the sample (input). This results in a sinusoidal deformation (strain ε) (output). The mechanical frequencies usually range from 10^{-3} Hz to 100 Hz. The sample's response to the load (strain or force) is measured; it exhibits a time delay (phase shift).

Stiffness and phase shift depend on the material and its physical state (temperature, load, frequency, etc.). The dynamic material properties are represented by the complex modulus E* = E' + iE", which itself depends on the structure and morphology of the material.

High-power servomotors and strong electrodynamic shakers generate the sample load. Force and strain sensors record the signals at high resolution.



DMA Measurement Information

- Stiffness and damping properties
- Phase transitions
- Aging effects
- Polymerization/cross-linking
- Curing/post-curing
- Cracking propagation
- Changes in the physical state due to external constraints like temperature, frequency, irradiation or mechanical loads (e.g., compression or tensile stresses)
- Glass transition temperature of highly cross-linked, amorphous or semi-crystalline polymers, elastomers, rubbers and composites
- Creep and relaxation
- Stress and strain sweeps
- Frequency sweeps
- Temperature sweeps

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The Dielectric Analysis Method – DEA

DEA explores the dielectric structure of materials by applying a sinusoidal voltage to two electrodes (electrical capacitor). The frequency of the sinusoidal voltage ranges from 10^{-2} Hz up to 10^{7} Hz.

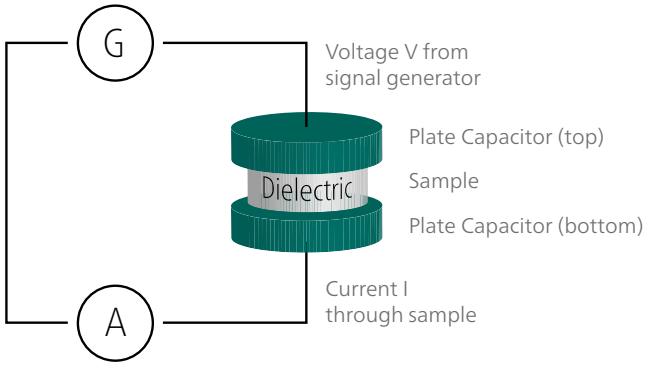
A sinusoidal alternating voltage with variable frequency (up to the MHz range) is applied to the upper plate of the compression holder. The upper and lower holders act as a perfect plate capacitor. The specimen (e.g., elastomer) acts as the dielectric. An AC current flows through the specimen and is measured. From the adjusted voltage and the recorded current, the complex sample impedance can be calculated.

The phase shift depends on the material and its condition. An evaluation of the recorded signals allows for calculation of the complex dielectric function $\varepsilon^* = \varepsilon' - i\,\varepsilon''$.

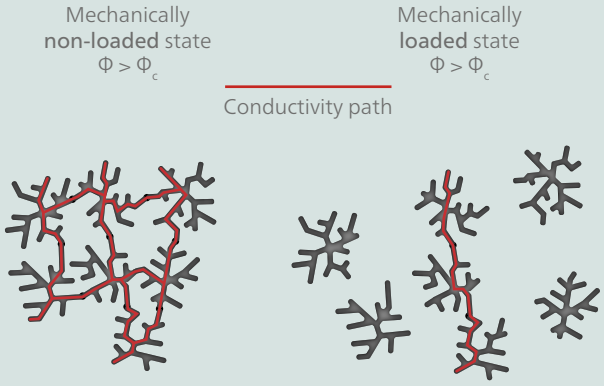
This function describes the dielectric material behavior and depends on factors such as time, frequency, temperature, molecular mobility within the material, the macroscopic orientation of the polymer chains, electromagnetic fields, and applied mechanical load (e.g., pressure and tensile stresses).

DEA Measurement Information

- Electrical conductivity
- Permittivity
- Information about cluster structure
- Information about carbon black density



DEA Investigates Flocculation of Carbon Black



Φ_c: critical percolation concentration

Source: D. Steinhauser et al., Deutsches Institut für Kautschuktechnologie e.V.

Free charge carriers on the surface of conductive fillers – e.g., carbon black – within a rubber matrix are polarized in an alternating electric field.

The oscillating electric field induces dynamic migration processes in the charge carriers along the surface of the carbon black particles. Larger carbon black clusters require longer migration times than smaller carbon black clusters. Relaxation times differ with cluster and particle size.

Above the percolation threshold, the carbon black density is very high and the filled rubber matrix becomes conductive in an alternating electrical field; many migration paths are available (image on the left).

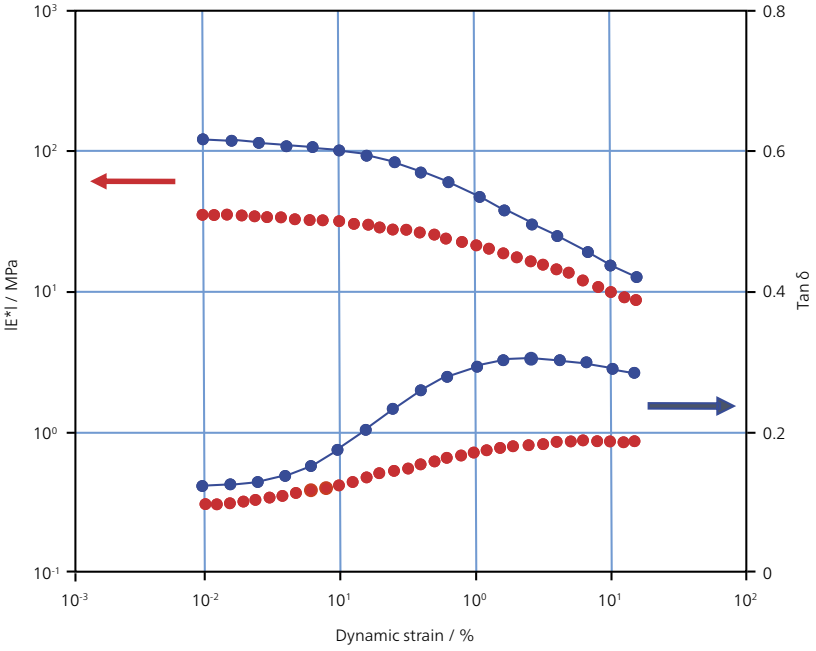
In the case of static and/or dynamic-mechanical loading, carbon black clusters are destroyed and the migration path density is thus reduced (image on the right). As a result, conductivity and permittivity decrease as load increases.

APPLICATION EXAMPLES

How Does a DMA Measurement Determine the Suitability of a Seal for Use?

A seal achieves optimum functionality when, for example, it returns to its original position quickly after being subjected to a compression process. This requires high elasticity/low damping (low tanδ) and a high restoring force (high modulus of elasticity, E*) of the material in order to avoid leakage.

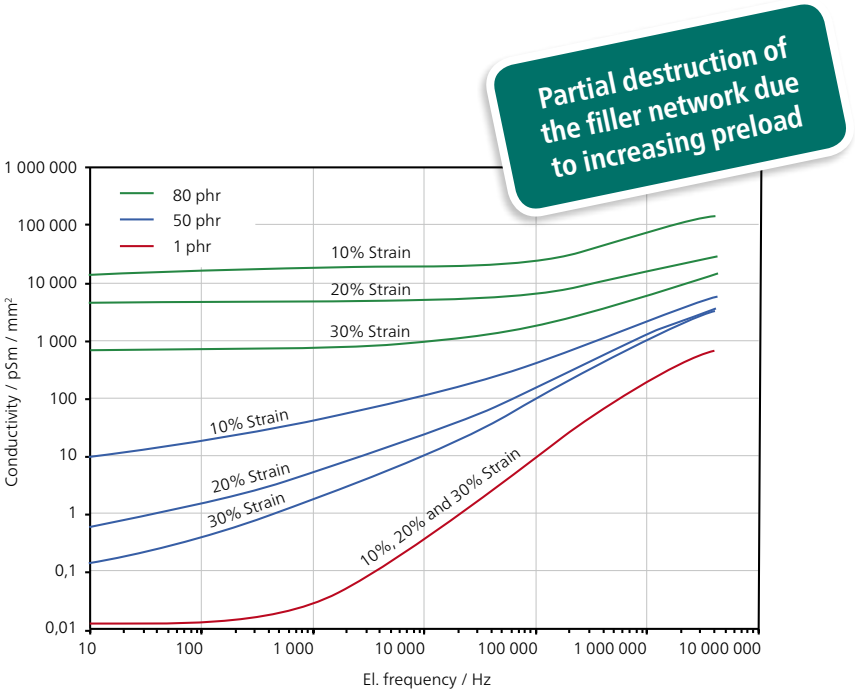
In the plot, two elastomers exhibiting differing degrees of suitability are compared. The "blue" seal only indicates a high E* and low tanδ at small dynamic amplitudes. If the dynamic stresses increase, this material fails as a seal. In such a case, the damping increases sharply, and the seal rises too slowly and thus cannot prevent leakage. The red system, on the other hand, remains much more elastic at higher amplitudes. It also works with larger deformation amplitudes.



Stiffness and damping of two different elastomeric systems depend on the applied dynamic strain.

Influence of the Filler Content

This plot exhibits tests on HNBR samples with different levels of carbon black N550 content. Each sample was compressed by 10%, 20% and 30%. The conductivity increases with increasing filler content. However, conductivity decreases with increasing static deformation. In the case of a negligible carbon black content level (1 phr*), any filler-induced influences on the conductivity are lacking as there is no filler present (red curve). This changes at 50 phr carbon black (blue curves). This concentration is closer to the percolation limit, as in the case of 80 phr carbon black (green curves). At 50 phr, the strain dependence of the conductivity is higher than at 80 phr carbon black.



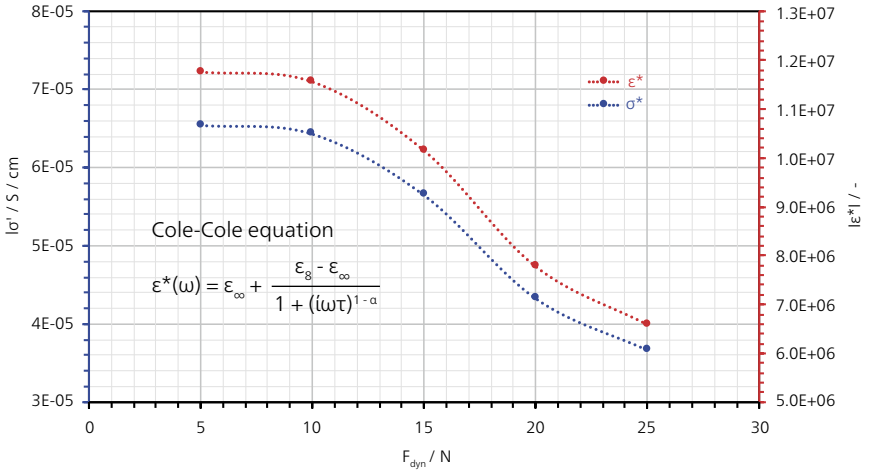
DiPLEXOR® measurements on HNBR with different levels of carbon black N550 content: 80 phr, 50 phr and 1 phr in compression mode at 25°C.

*phr = parts per hundred rubber

Permittivity Provides Information About Carbon Black Structures

This plot shows the influence of the dynamic force on an SBR sample: An increasing dynamic force results in decreasing conductivity and permittivity.

Based on the Debye equation, the semi-empirical Cole-Cole, Cole-Davidson and Havriliak-Negami equations were developed. They allow for simulation of the permittivity. With the support of computer-aided fit programs, these mathematical techniques can identify the most important fitting coefficient: the relaxation time, which indicates the size of the carbon black structure.



SBR 70 phr* N 234 at F_{stat} 30 N, f 10 Hz and f_{el} 10 Hz; conductivity and permeability versus dynamic force amplitude varying from 5 N to 25 N in steps of 5 N

Key Technical Specifications

GABO DiPLEXOR® – Standard Configuration of Simultaneous DMA and DEA	
Dynamic force range	up to ± 500 N
Static force range	up to 1500 N
Dynamic strain	up to ± 1.5 mm (3 mm*); optional: ± 6 mm (12 mm*)
Frequency range	0.001 Hz to 100 Hz; optional: 0.0001 to 200 Hz
Dielectric broadband analyzer	Dielectric impedance spectroscopy for ε* and σ*
Frequency range of dielectric broadband analyzer	0.01 Hz up to 10 MHz
Temperature	RT to 500°C; optional: -160°C to 500°C
Optional accessories	HYGROMATOR®, UV accessory

Additional configurations are available; please see DMA GABO EPLEXOR® brochure or ask your local representative.
* Peak force (peak-to-peak)



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The NETZSCH Group is a mid-sized, family-owned German company engaging in the manufacture of machinery and instrumentation with worldwide production, sales, and service branches.

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