

## Monitoring Damage Development of Static Sealing Elements Using the Simultaneous Dynamic Mechanical and Dielectric Analyzer DIPLEXOR®

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1 DMA GABO EPLEXOR® (instrument) with special sensors for dielectric dynamic mechanical measurements

### Introduction

Sealing elements are used in technical applications to prevent mass transfer between two components or auxiliary chambers. The desired property profile is achieved primarily through a variety of design options. Besides the polymer and necessary additives, the filler used also plays a crucial role in establishing a sealing element's characteristics such as compressive strength, thermal and chemical resistance.

The sealing elements undergo continuous changes in operating and environmental conditions. They are subject to natural, thermo-oxidative or mechanical aging processes and must be replaced after a certain time. The condition for cost efficiency is that a sealing gasket should be used over its entire service life. This means that the sealing element should not be replaced too early, in order to save on unnecessary acquisition costs, and not too late, in order to prevent leakage damage.

Damage development in sealing elements can be detected by the integration of several control micro-systems. Most of these are associated with high costs and engender a high degree of complexity in the overall structure.

### A Seal Monitors its Own Wear

A solution that can be realized more easily is the use of intelligent monitoring systems. As necessary part of any technical elastomer composites, reinforcing filler can be also electrically conductive. When those electrically conductive fillers are mixed into the rubber matrix, the sealing element becomes electrically conductive above a system-specific percolation threshold when an electrical voltage is applied. The current changes in dielectric conductivity are in accordance with the state of its filler network, and hence the damage in the sealing element.

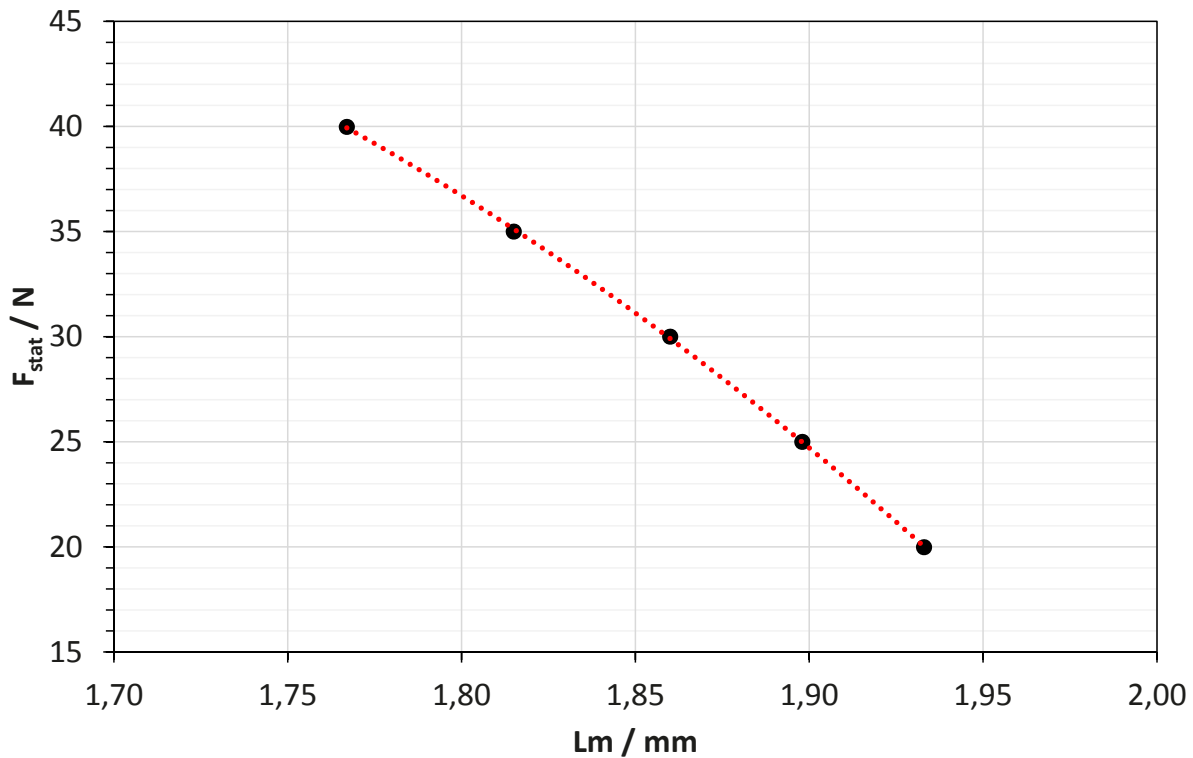
### Test Conditions

To illustrate the simultaneous mechanical and dielectric behavior of a sealing material and how the progression of mechanical damage can be characterized at the same time, a styrene butadiene rubber (SBR) filled with 70 phr carbon black (N 234) was prepared. The rubber matrix behaves as an insulator. The N 234 carbon black is electrically conductive because its surface area has a graphitic nano-crystallite structure. Here, it is important to note that the carbon black amount of 70 phr is above the percolation threshold, which is an absolute prerequisite for building up a closed filler network providing the necessary conductive paths.

The simultaneous mechanical and dielectric measurements were performed with the dynamic mechanical analyzer DMA GABO EPLEXOR® by NETZSCH (Figure 1)

\*DIPLEXOR® only registered in Europe

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2 Thickness variation of the SBR sample filled with 70 phr N 234 due to an increasing static load amplitude

which can be equipped with special sample holders and a dielectric controller – equipped with a broadband dielectric spectrometer (BDS) supplied by Novocontrol GmbH – in compression mode at room temperature. In this combination the device is also called DIPLEXOR®.

The compression clamps serve as electrodes. They are electrically isolated from the rest of the instrument in order to ensure that the dielectric properties of the SBR sample are the only aspect being measured.

The samples were 2-mm-thick cylinders with a diameter of 10 mm. The sample was coated with a very thin silver layer in order to improve contact with the electrodes and hence reduce the stray field. Dielectric spectra were recorded in a frequency range between 1 Hz and 105 Hz. The static force was increased from 20 N to 40 N in 5-N steps.

### Measurement Results

If the SBR sample is compressed with a defined static force, its thickness changes accordingly. Increasing the static load amplitude further reduces the sample thickness. This behavior is depicted in Figure 2. A change of up to 30% in thickness due to mechanical loading correlates

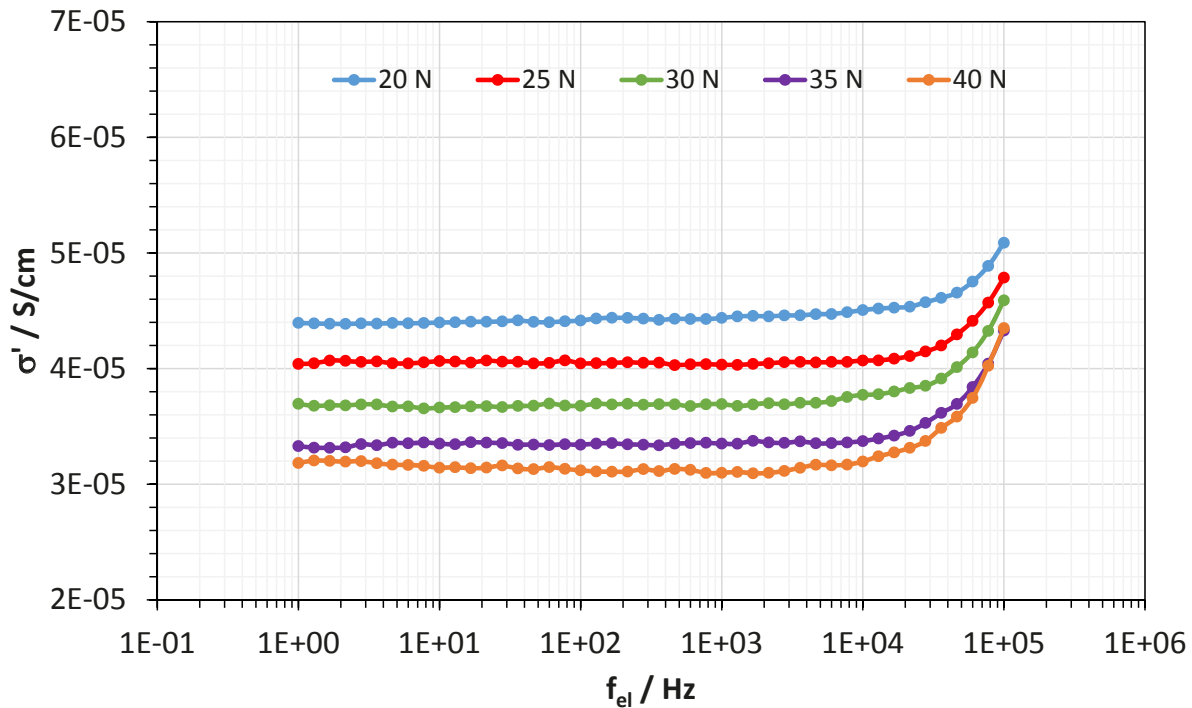
quite well with installation procedures for seals in real applications.

Increasing the mechanical loading increases the internal friction within the SBR sample due to diffusion processes as well as displacement or orientation of filler particles in the direction of compression. The filler network is progressively destroyed and the sample stiffness decreases. Therefore, the damage progression is associated with a gradual decrease in the density of the conduction paths within the sample.

An additional application of an alternating electric field,  $\mathbf{E}(\omega)$ , generates an electric current within the SBR sample because the free electrical charge carriers gain the ability to move along the surface of carbon black clusters, which form continuous conduction paths from one side to the other. The electric current density,  $\mathbf{J}(\omega)$ , is proportional to the electric field applied, as per the following:

$$\mathbf{J}(\omega) = \sigma^* \cdot \mathbf{E}(\omega)$$

where  $\sigma^*$  is the complex dielectric conductivity and  $\omega=2\pi f$  is the angular frequency. The complex conductivity,  $\sigma^*$ , represents a measure of the transported charge per unit of time.



**3** Variation in the real part of the complex dielectric conductivity,  $\sigma'$ , of the SBR sample due to a varying static load in a frequency range between 1 Hz and  $10^7$  Hz at room temperature

Variation in the real part of the complex dielectric conductivity,  $\sigma'$ , due to an increase in a static load is shown in Figure 3.

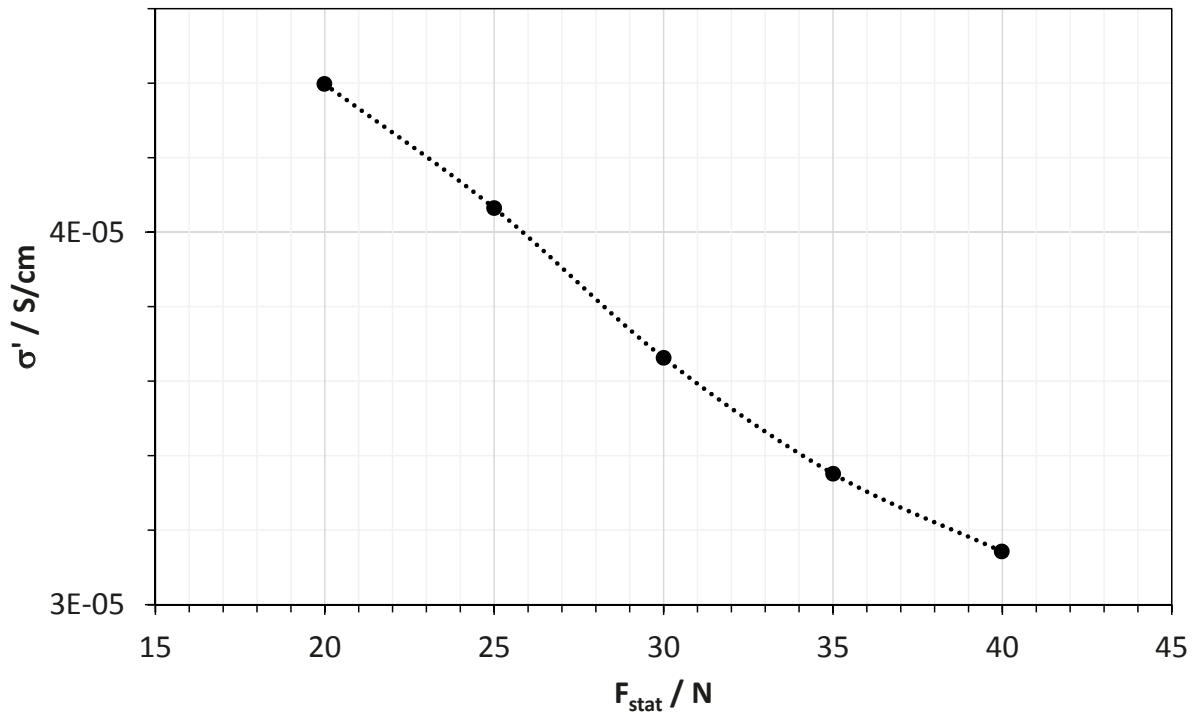
At frequencies up to 2000 Hz,  $\sigma'$  is frequency-independent and reaches a plateau value known as DC-conductivity. At higher frequencies,  $\sigma'$  becomes frequency-dependent. This area is called dielectric dispersion because the variation in the electric field is not associated with an instantaneous change in the sample polarization.

Obviously, the real part of the complex dielectric conductivity,  $\sigma'$ , decreases over the entire frequency range

as the static force is increased, as a consequence of progressive destruction of the filler network. This fact is correlated to a reduction in the conduction path density which occurs throughout the entire SBR sample due to mechanical destruction processes caused by the static load applied.

Therefore, the variation in  $\sigma'$  during the operational life of an elastomeric sealing material can be used as a smart way of monitoring the actual damage state. This behavior becomes more evident when the variation in the real part of the complex dielectric conductivity,  $\sigma'$ , that is due to varying static load is examined at a given dielectric frequency,  $f_{el}$ .

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4 Variation in the real part of the complex dielectric conductivity,  $\sigma'$ , of the SBR sample filled with 70 phr N 234 as a function of the static force at a dielectric frequency,  $f_{el}$ , of 10 Hz

Figure 4 illustrates this dependence at a dielectric frequency,  $f_{el}$ , of 10 Hz.

Figure 4 confirms the relationship between the increasing static loading and decreasing complex dielectric conductivity. This is attributed to the density decrease in the conduction paths within the SBR sample and allows for the monitoring of the actual state of damage of the filler network.

### Conclusion

Dynamic mechanical analysis (DMA) is the main quality control system for technical products under mechanical load. Dielectric analysis (DEA) further supports the development process for technical products. The very large

available frequency range (as compared to DMA) permits an in-depth molecular understanding of the inner dynamics. This valuable insight into a material's microstructure allows conclusions to be drawn – with minimal effort – about the actual state of damage of a finished technical product during active operation, when electrically conductive fillers are used.

It was shown that the current changes in dielectric conductivity are in accordance with the state of its filler network, and hence the damage in the sealing element.

The DIPLEXOR® 500 N offers a unique advantage: It permits characterization of the dielectric properties of sealing elements under high mechanical load, in order to determine first their properties and later their actual performance during operation.