

## Qualitätsprüfungen an Kunststoffen und Elastomeren

- mechanische und thermoanalytische Prüfungen

*Quality assessment of polymers and polymeric parts*

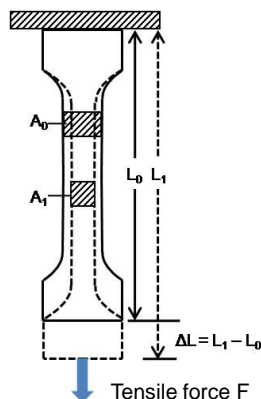
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### Tensile Test – Principle

**Determine mechanical properties, such as strength, stiffness, strain behavior, creep and relaxation behavior**



Nominal strain:

$$\varepsilon = \frac{\Delta L}{L_0} = \frac{L_1 - L_0}{L_0} = \frac{L_1}{L_0} - 1$$

Stretch ratio:

$$\lambda = \frac{L_1}{L_0} = \varepsilon + 1$$

Engineering stress:

$$\sigma = \frac{F}{A_0}$$

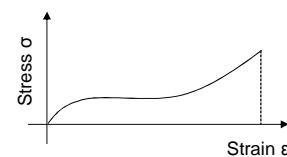
Assuming constant volume:

$$L_0 \cdot A_0 = L_1 \cdot A_1$$

True stress:

$$\sigma_w = \frac{F}{A_1} = \sigma \cdot \frac{L_1}{L_0} = \sigma \cdot \lambda$$

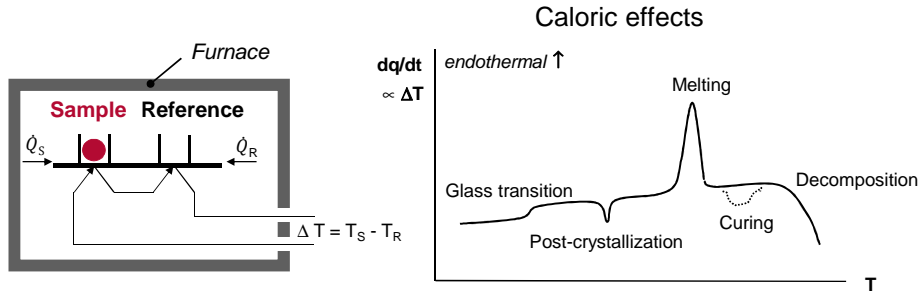
Stress strain curve:



[Frick A., Stern C.: Praktische Kunststoffprüfung, Hanser, München, p. 304-307 (2011)]

## DSC – Principle

Measurement of specific heat flow  $dq/dt$  of a sample as a function of temperature and time



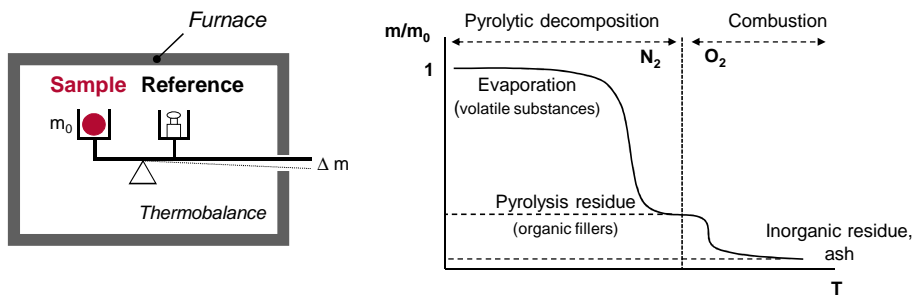
$$c_p = \frac{dH}{dT} \cdot \frac{1}{m} = \frac{dQ}{dT} \cdot \frac{1}{m} = \frac{\dot{Q}}{\dot{T}} \cdot \frac{1}{m}$$

$$c_p \propto \frac{\dot{Q}}{m} = \dot{q} \quad \text{with } \dot{T} = \text{const.}$$

[Frick A., Stern C.: DSC-Prüfung in der Anwendung, Hanser, München, p. 17-19 (2006)]

## TGA – Principle

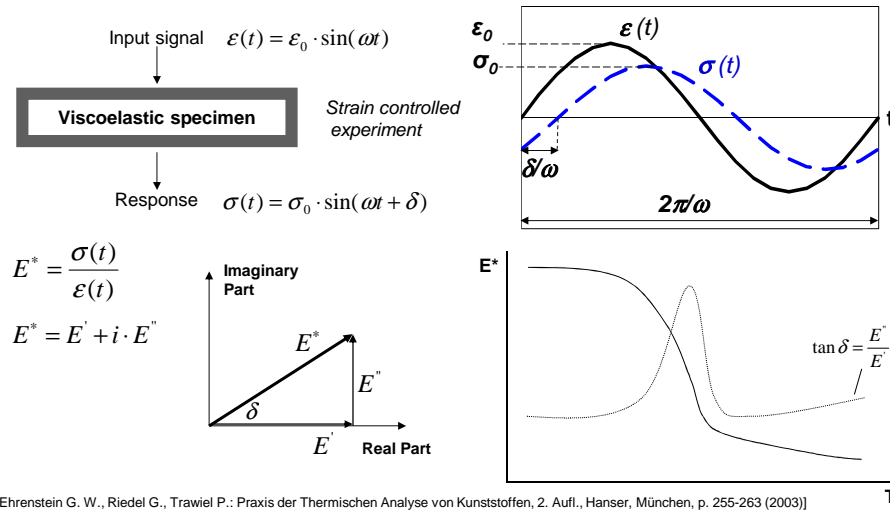
Measurement of mass change behavior of a sample as a function of temperature and time



$$m_{rel} = \frac{m}{m_0}$$

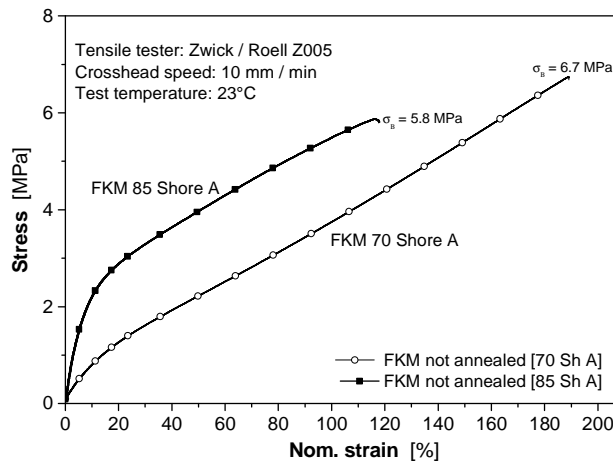
[Ehrenstein G. W., Riedel G., Trawiel P.: Praxis der Thermischen Analyse von Kunststoffen, 2. Aufl., Hanser, München, p. 150-160 (2003)]

Measurement of input signal to output signal as a function of temperature and time for determining stiffness and damping



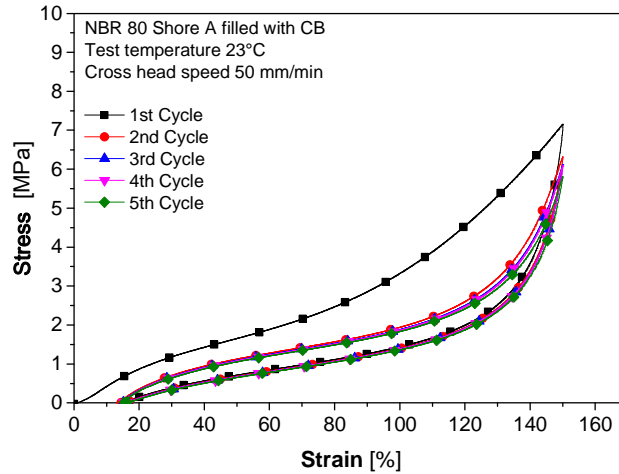
Tensile Test (TT) – FKM (70 / 85 Shore A)

FKM different durometer hardness

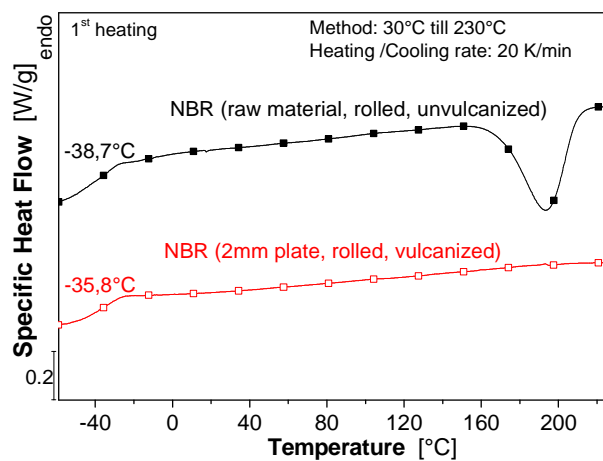


## Tensile Test – Cyclic measurements

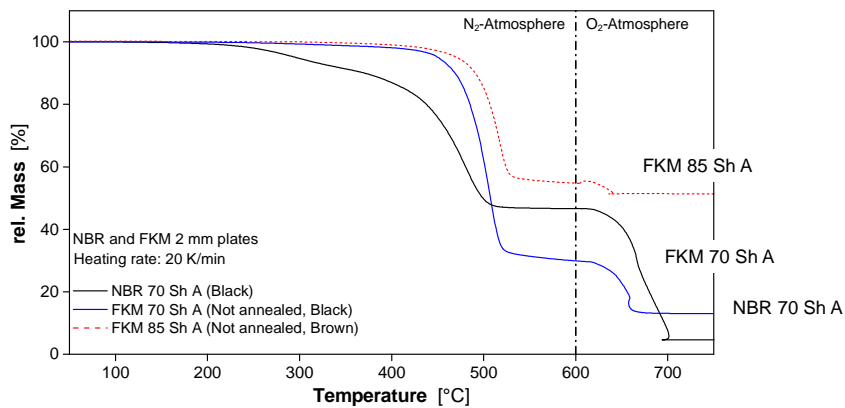
### Variation of cycles



## DSC – NBR (unvulcanized / vulcanized)

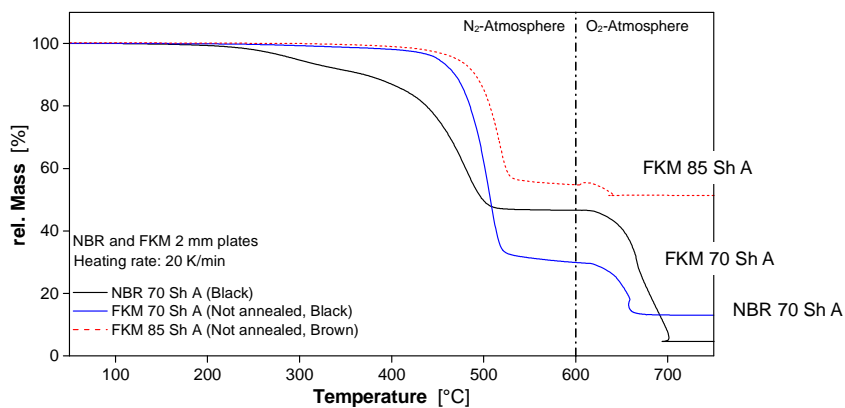


## TGA – NBR and FKM identification



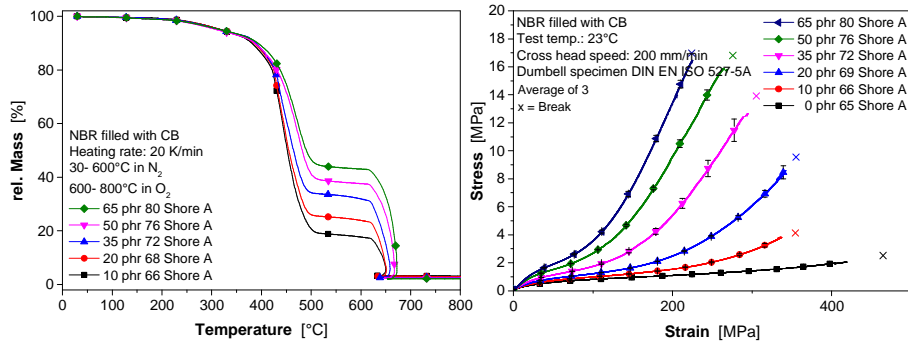
Sample [°C]	Relative mass loss [%]					
	100°C	150°C	200°C	250°C	550°C	750°C
NBR 70Sh A (black)	0.04	0.20	0.7	2.1	53.2	95.4
FKM not ann. 70Sh A (black)	0.00	0.04	0.1	0.3	68.8	87.0
FKM not ann. 85Sh A (brown)	0.24	0.18	0.1	0.1	44.3	48.7

## TGA – NBR and FKM identification

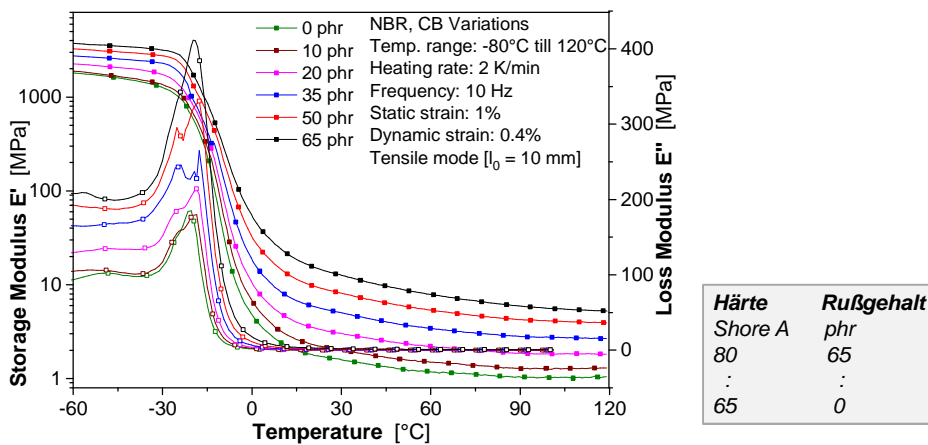


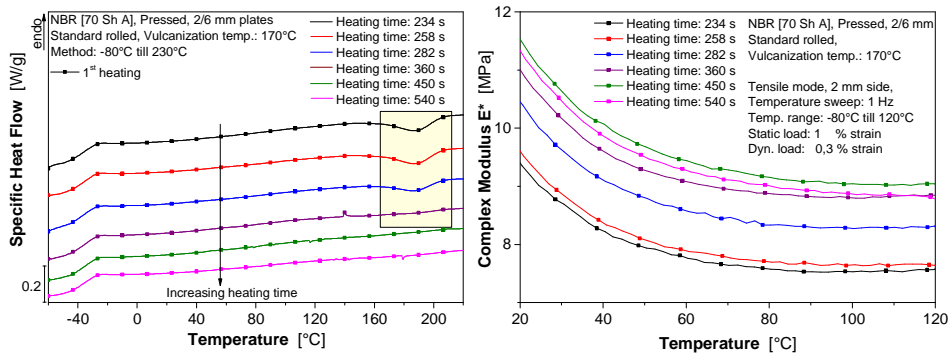
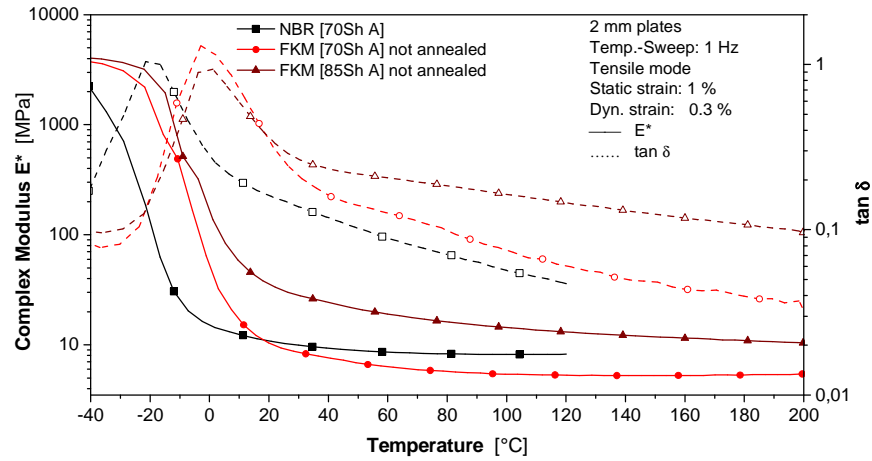
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TGA / TT – NBR (influence of different carbon black content)



DMA – NBR (influence of filler content)





$\Delta H$ = Enthalpy;  $T_c$ = Curing temperature;  $T_{c0}$ = Extrapolated curing temperature;  $w_p$ = Peak width  $h_p$ = Peak height.

## Conclusion I

### State of the art of rubber material characterization:

- **Durometer hardness** → Shore hardness
- **Tensile test** → Strength at break, Strain at break (Modulus  $\sigma$  ( $\epsilon = x \%$ ))
- **Glass transition temperature  $T_g$**

### Advanced Techniques → Thermal Analysis Methods (TA):

- **Differential Scanning Calorimetry (DSC)**
- **Thermo-Gravimetric Analysis (TGA)**
- **Dynamic Mechanical Analysis (DMA)**

## Conclusion II

Thermal analysis techniques provides multi-point information.

### **DSC**

- Operating temperature limits ( $T_g$ )
- Vulcanization behavior / kinetics ( $\Delta H$ )
- Differentiation between different material composition
- Melting, Post-crystallization and annealing effect on re-crystallization

### **TGA**

- Material identification by decomposition behavior (finger-print)
- Differentiate between annealed and non-annealed material
- Volatile / Plasticizers content
- Filler type and filler content identification

Geometry independent

### **DMA**

- Determination of stiffness ( $E^* = E' + iE''$ )
- Determination of damping behavior ( $\tan \delta$ ) for  $f = \text{const.}$ ,  $\epsilon = \text{const.}$
- Mullin effect (Dynamic strain-sweep)
- Indication of possible operating temperatures ( $T_g$ )
- Frequency dependent stiffness at reference temperature (Master curve)

Geometry dependent



Prüfdienstleistungen und Entwicklungen für Kunden

„PETZ“  .....

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