Hochschule Aalen

Qualitätsprüfungen an Kunststoffen und Elastomeren

- mechanische und thermoanalytische Prüfungen

-

Quality assessment of polymers and polymeric parts

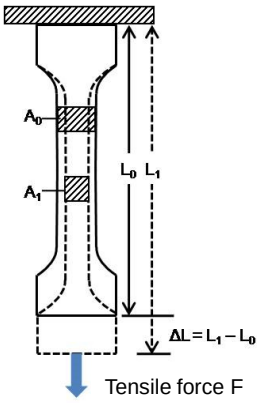
Prof. Dr.-Ing. Achim Frick

Aalen University, Institute of Polymer Science and Processing (iPSP)
Aalen, Germany

Tensile Test – Principle

Hochschule Aalen

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:

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

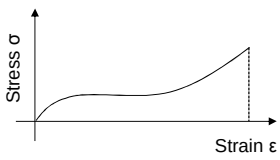
Assuming constant volume:

$$L_0 \cdot A_o = L_1 \cdot A_1$$

True stress:

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

Stress strain curve:

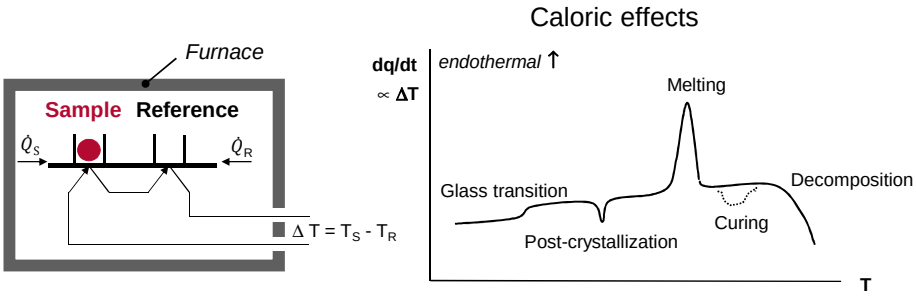


[Frick A., Stern C.: Praktische Kunststoffprüfung, Hanser, München, p. 304-307 (2011)]

DSC – Principle

Hochschule Aalen

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



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

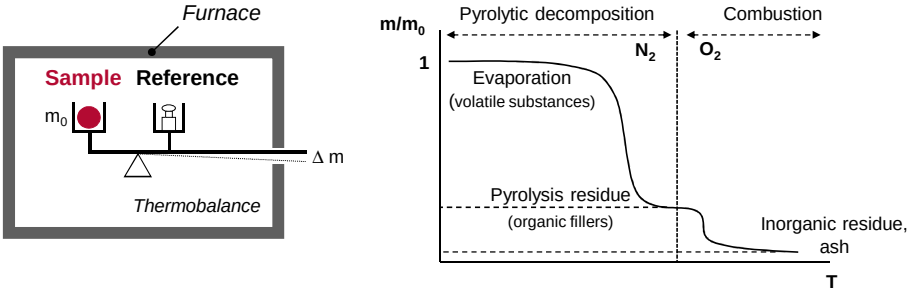
$$c_P \propto \frac{\dot{Q}}{m} = \dot{q} \qquad \text{with } \dot{T} = \text{const.}$$

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

TGA – Principle

Hochschule Aalen

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



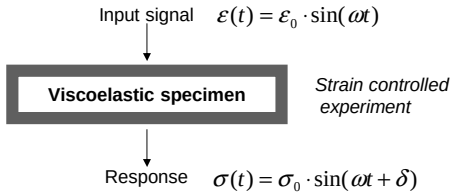
$$m_{rel} = \frac{m}{m_0}$$

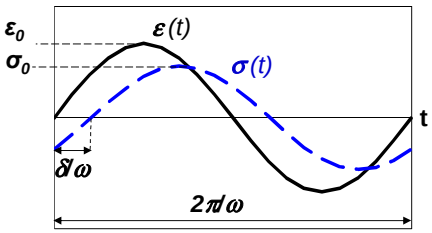
[Ehrenstein G. W., Riedel G., Trawiel P.: Praxis der Thermischen Analyse von Kunststoffen, 2. Aufl., Hanser, München, p. 150-160 (2003)]

DMA – Principle

Hochschule Aalen

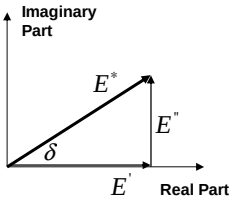
Measurement of input signal to output signal as a function of temperature and time for determining stiffness and damping

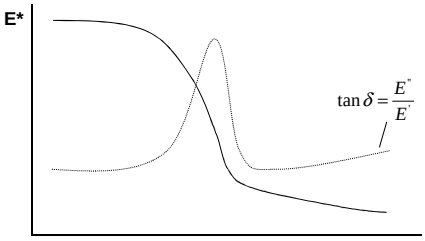




$$E^* = \frac{\sigma(t)}{\varepsilon(t)}$$

$$E^* = E' + i \cdot E''$$



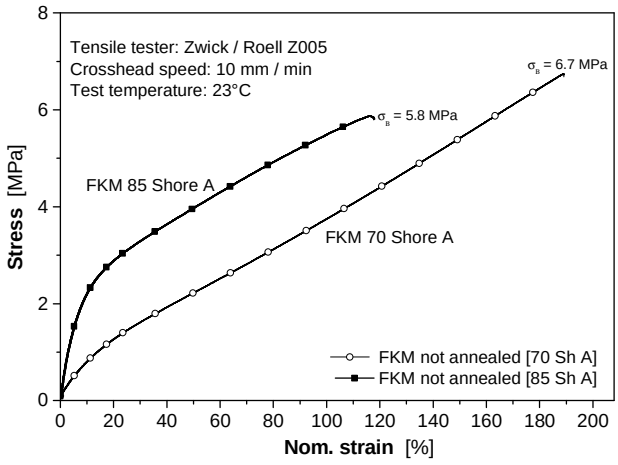


[Ehrenstein G. W., Riedel G., Trawiel P.: Praxis der Thermischen Analyse von Kunststoffen, 2. Aufl., Hanser, München, p. 255-263 (2003)]

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

Hochschule Aalen

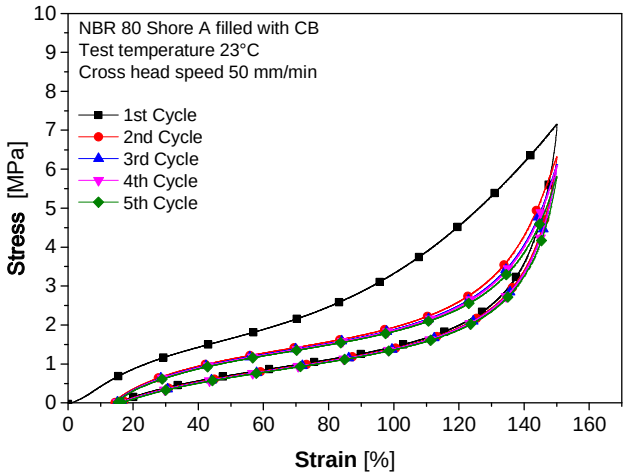
FKM different durometer hardness



Tensile Test – Cyclic measurements

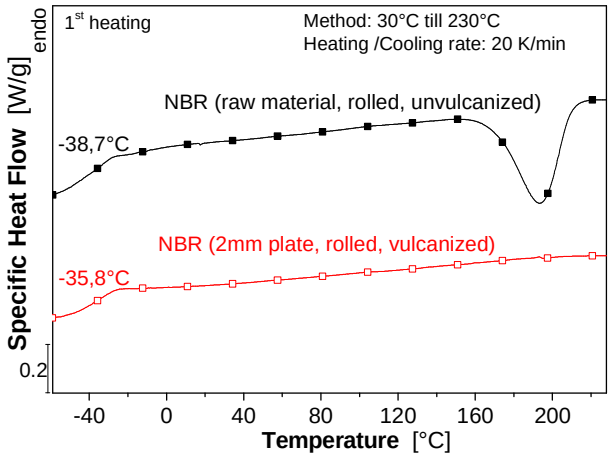
Hochschule Aalen

Variation of cycles



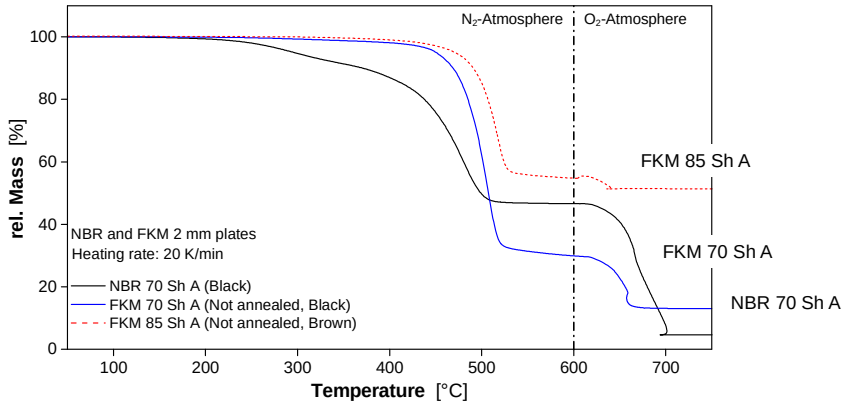
DSC – NBR (unvulcanized / vulcanized)

Hochschule Aalen



TGA – NBR and FKM identification

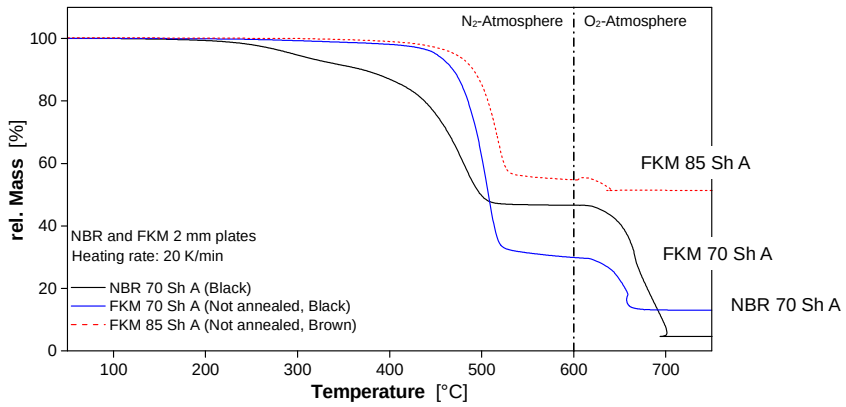
Hochschule Aalen



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

Hochschule Aalen

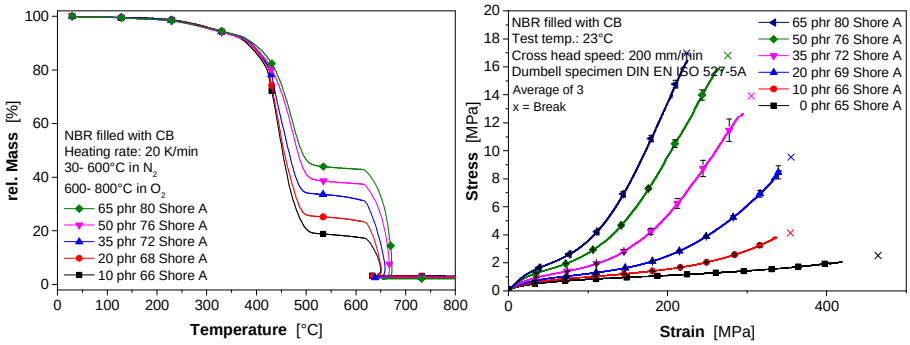


Sample [°C]	Relative mass loss [%] 100°C	150°C	200°C	250°C	550°C	750°C
NBR 70Sh A (black)	0.04	0.20	0.7	2.1	53.2	95.4
FKM not ann. 70Sh A (black)	0.00	0.04	0.1	0.3	68.8	87.0
FKM not ann. 85Sh A (brown)	0.24	0.18	0.1	0.1	44.3	48.7

Achim Frick Institute of Polymer Science and Processing ‚iPSP' 10

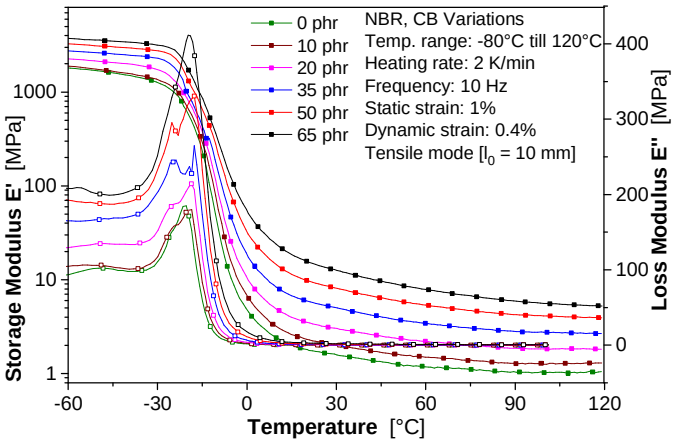
TGA / TT – NBR (influence of different carbon black content)

Hochschule Aalen



DMA – NBR (influence of filler content)

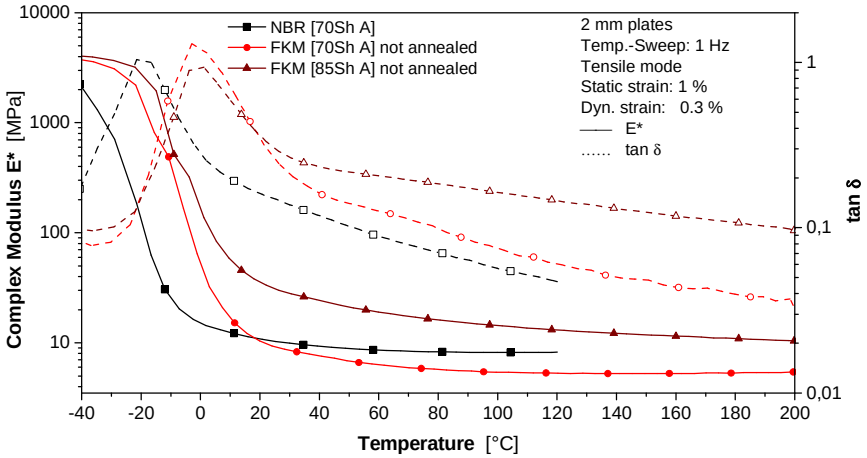
Hochschule Aalen



Härte	*Rußgehalt*
Shore A	*phr*
80	*65*
:	*:*
65	*0*

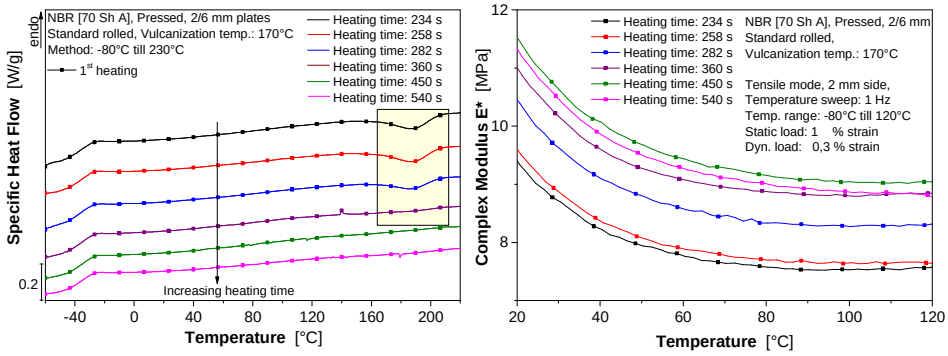
DMA – NBR and FKM (Temperature sweep, tensile mode)

Hochschule Aalen



DSC / DMA – NBR (Different vulcanization times comparison)

Hochschule Aalen



ΔH= Enthalpy; T_c= Curing temperature; T_{ce}= Extrapolated curing temperature; w_p= Peak width h_p= Peak height.

Conclusion I

Hochschule Aalen

State of the art of rubber material characterization:

- **Durometer hardness** → Shore hardness
- **Tensile test** → Strength at break, Strain at break (Modulus σ (ε = 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

Hochschule Aalen

Thermal analysis techniques provides multi-point information.

DSC

- Operating temperature limits (Tg)
- Vulcanization behavior / kinetics (ΔH)
- 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

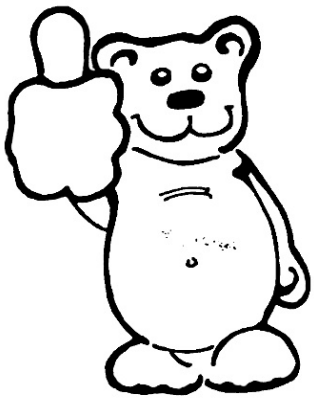
DMA

- Determination of stiffness (E* = E' + i·E'')
- Determination of damping behavior (tan δ) for f = const., ε = const.
- Mullin effect (Dynamic strain-sweep)
- Indication of possible operating temperatures (Tg)
- Frequency dependent stiffness at reference temperature (Master curve)

Geometry dependent

Prüfdienstleistungen und Entwicklungen für Kunden

„PETZ“ .....



Steinbeis Transferzentrum
Polymer Engineering (PETZ) - Aalen

Leiter: Prof. Dr.-Ing. Achim Frick

--> Email: Achim.Frick@hs-aalen.de
--> Tel.: 07361 / 576-2171, -2169