BAM

Bundesanstalt für Materialforschung und -prüfung

Sicherheit in Technik und Chemie

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LOW TEMPERATURE PROPERTIES OF RUBBER SEALS

RIEG „Low Temperature Webinar“

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BAM 3.4 Safety of Storage Containers

www.bam.de

Outline

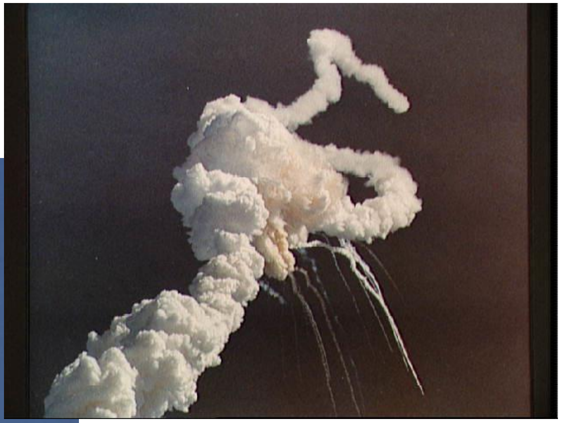
1. Motivation
2. Seal application
3. Methods / Results
   - Thermal analysis
   - Compression Set
   - Component tests: static/"dynamic"
   - Summary of results
4. Conclusion

1. Motivation:
- use of seals at low temperatures

Quellen: www.Wikipedia.org; www.siempelkamp.de; www.chemanager-online.com

1. Motivation:
- prevent accidents

Catastrophic failure of a seal causes explosion of Space Shuttle Challenger (1986):



Quelle: www.Wikipedia.org

Quelle: NASA, Challenger accident after launch in: S86-38989 (Ed.), 1986.

1. Motivation for BAM

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MATERIAL

physical / chemical properties

- Hardness
- Stiffness
- Glass Transition Region
- Decomposition Temperature
- Permeability Coefficient

?

- Compression Set
- Compression Stress Relaxation
- ?

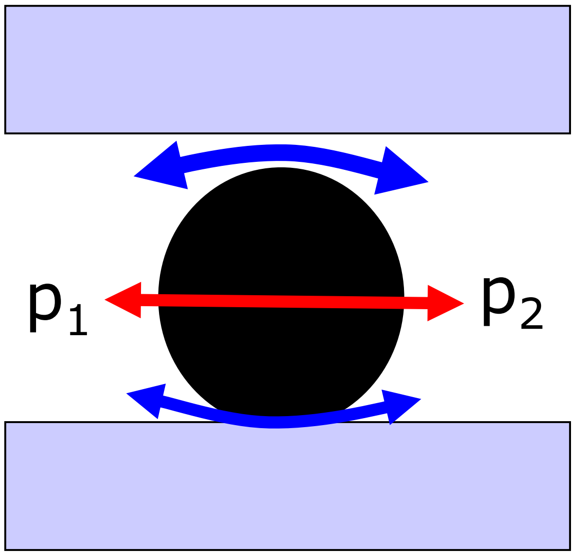
COMPONENT

function

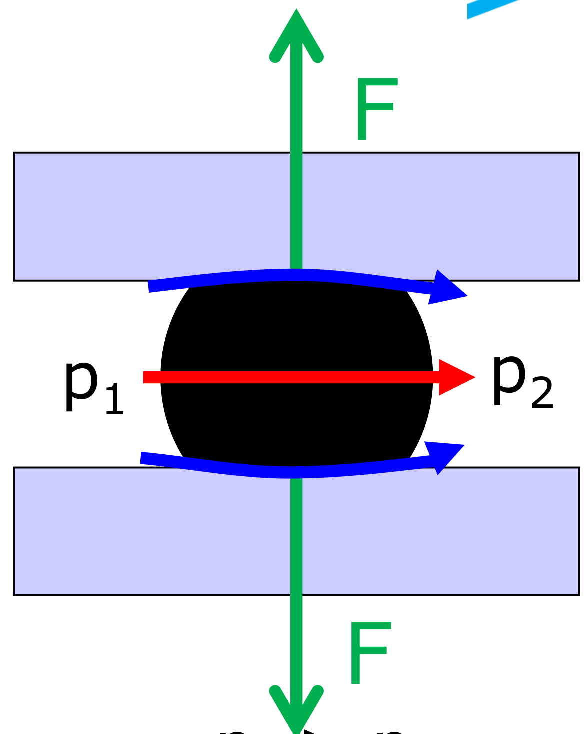
- Leakage Rate
- Application Conditions:
  - Groove Geometry
  - Temperature
  - Time
  - Media Influence
  - Irradiation

- develop methods for investigation
- define proper criteria (material selection, ageing evaluation)
- evaluate seal performance

2. Seal application



$p_1 = p_2$



$p_1 > p_2$

leakage rate Q : $Q = Q_{perm} + Q_{trans}$

$Q_{perm} = P * A/d * (p_1 - p_2)$

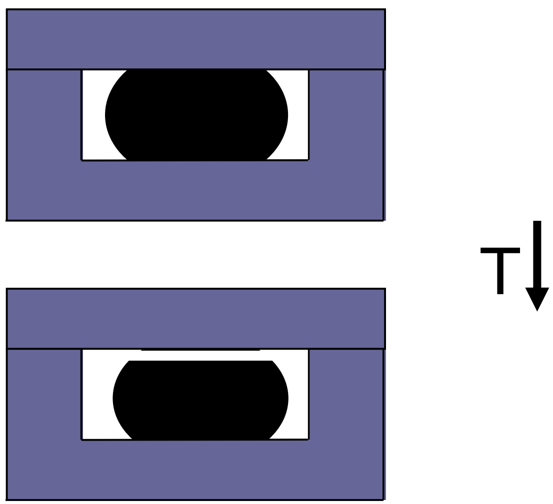
Q_{trans} = f(material contact, pressure difference, medium,...)

2. Seal application:

- Behaviour at low temperatures

1. case:

During cooling a leakage appears under static load:



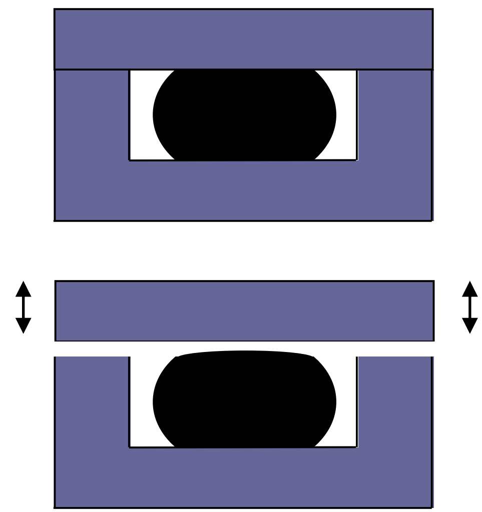
cause: $\alpha_{Metall} \neq \alpha_{Elastomer}$

⇒ the seal contracts in the groove

⇒ below a critical temperature this can not be compensated by recovery

2. case:

A leakage appears under dynamic load:



cause:

The velocity of the spontaneous elastic recovery is temperature dependent.

⇒ the leakage can close with time

2. Seal application:
- Processes at low temperatures

Glass-rubber transition:

- is a typical process in polymers/elastomers
- correlates with (intra-)molecular mobility
- above glass-rubber transition molecules are flexible
- below glass-rubber transition molecules are stiff

Crystallisation:

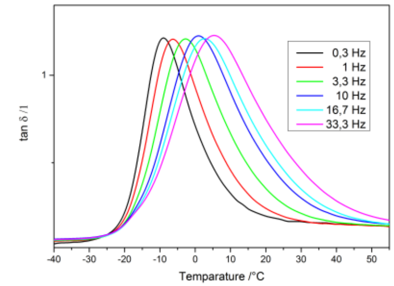
- crytallites act as additional crosslinks
  => they stabilise the deformed state
- causes density increase/volume reduction

⇒ but, where is the temperature limit?

3. Methods/Results:

Thermal Analysis: glass-rubber transition of e.g. FKM

method	T_g
DMA: E′-onset (1 Hz)	-26 °C
DMA: E′-inflection point (1 Hz)	-20 °C
DMA: E′-offset (1 Hz)	-14 °C
DMA: tan δ -peak (1 Hz)	-8 °C
DMA: E′′-peak (1 Hz)	-18 °C
DSC: Heat flow-onset	-23 °C
DSC: Heat flow-inflection point	-19 °C
DSC: Heat flow-offset	-15 °C

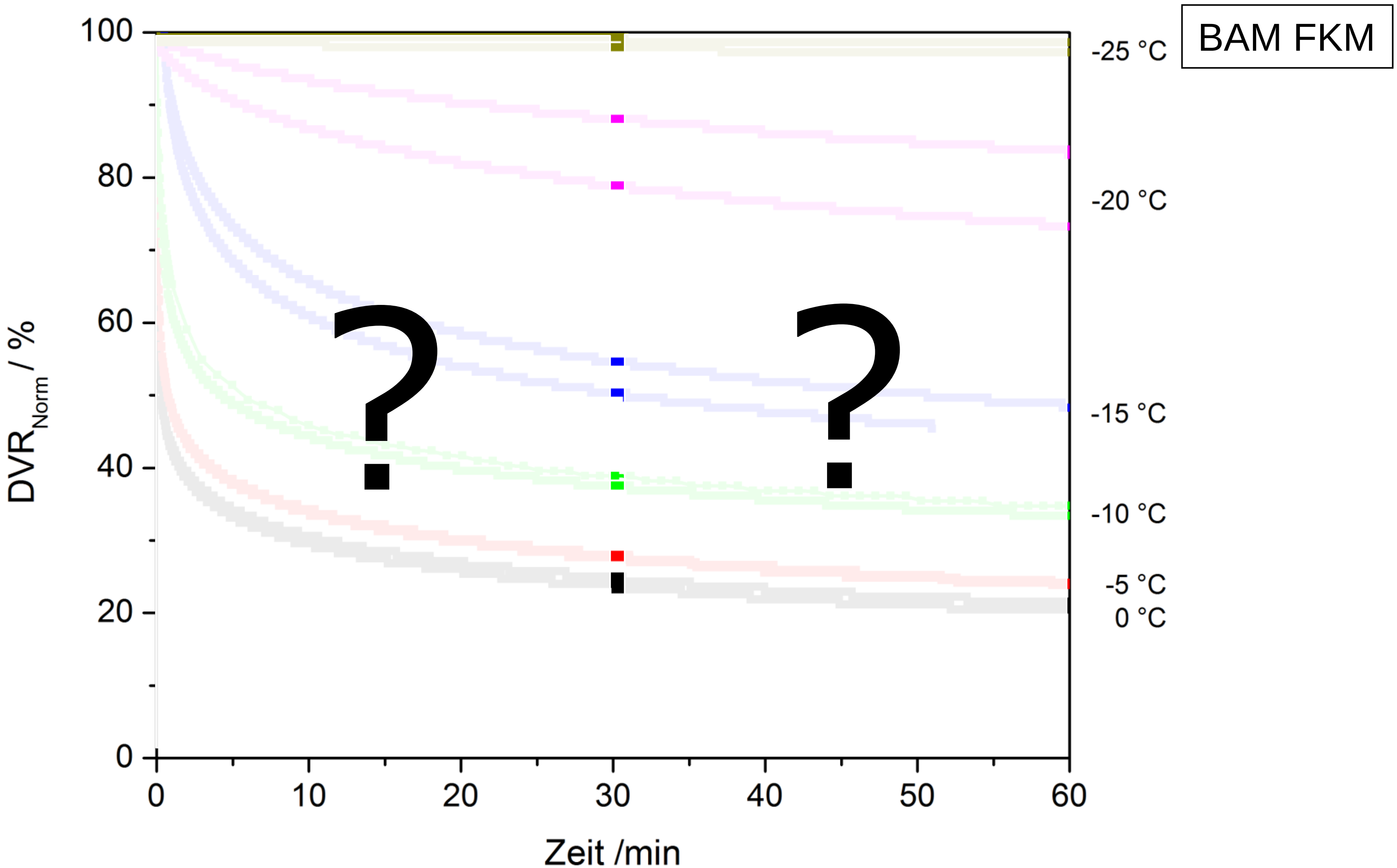


- ➢ the glass-rubber transition temperature (T_g) has to be defined: conditions and method of measurement, analysis method
- ➢ so far no direct correlation between the glass-rubber transition temperature and seal failure is known

⇒ additional tests are required: Compression Set / Component Tests

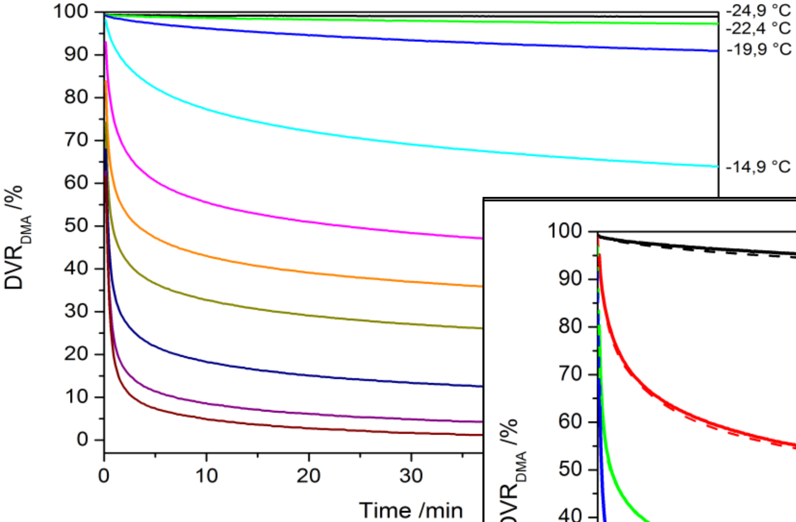
3. Methods/Results:

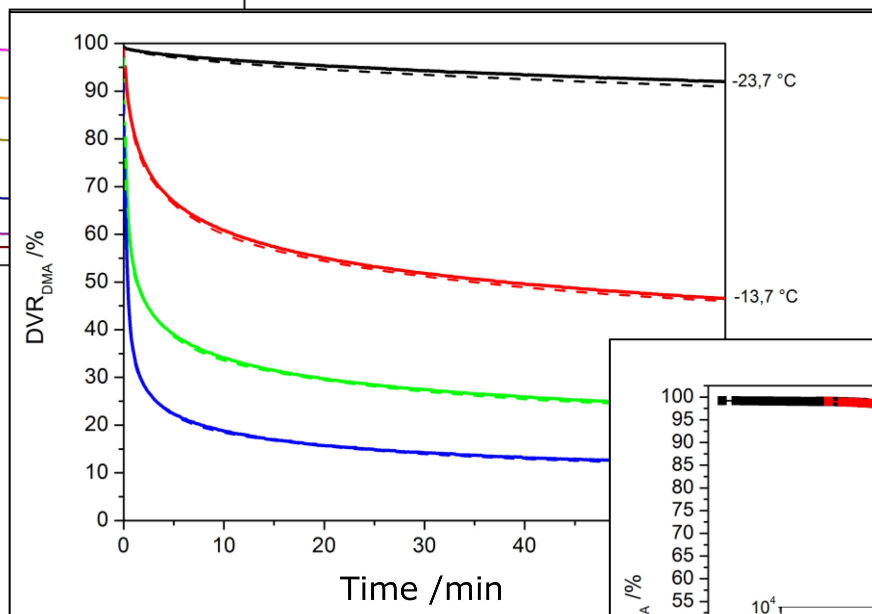
CS according to standard procedure (ISO 815-2)



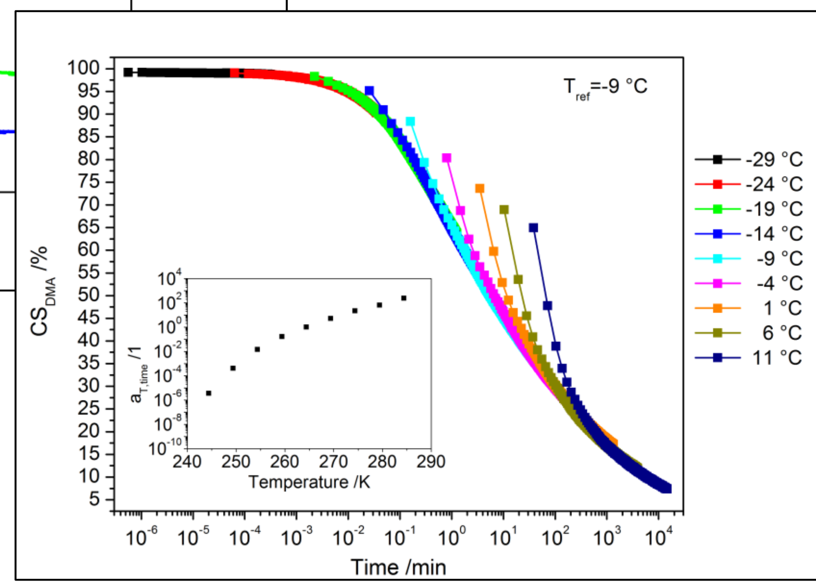
3. Methods/Results:

Compression Set by DMA equipment[1]





$$y = y_\infty + \sum_i A_i e^{-\frac{t}{t_i}}$$

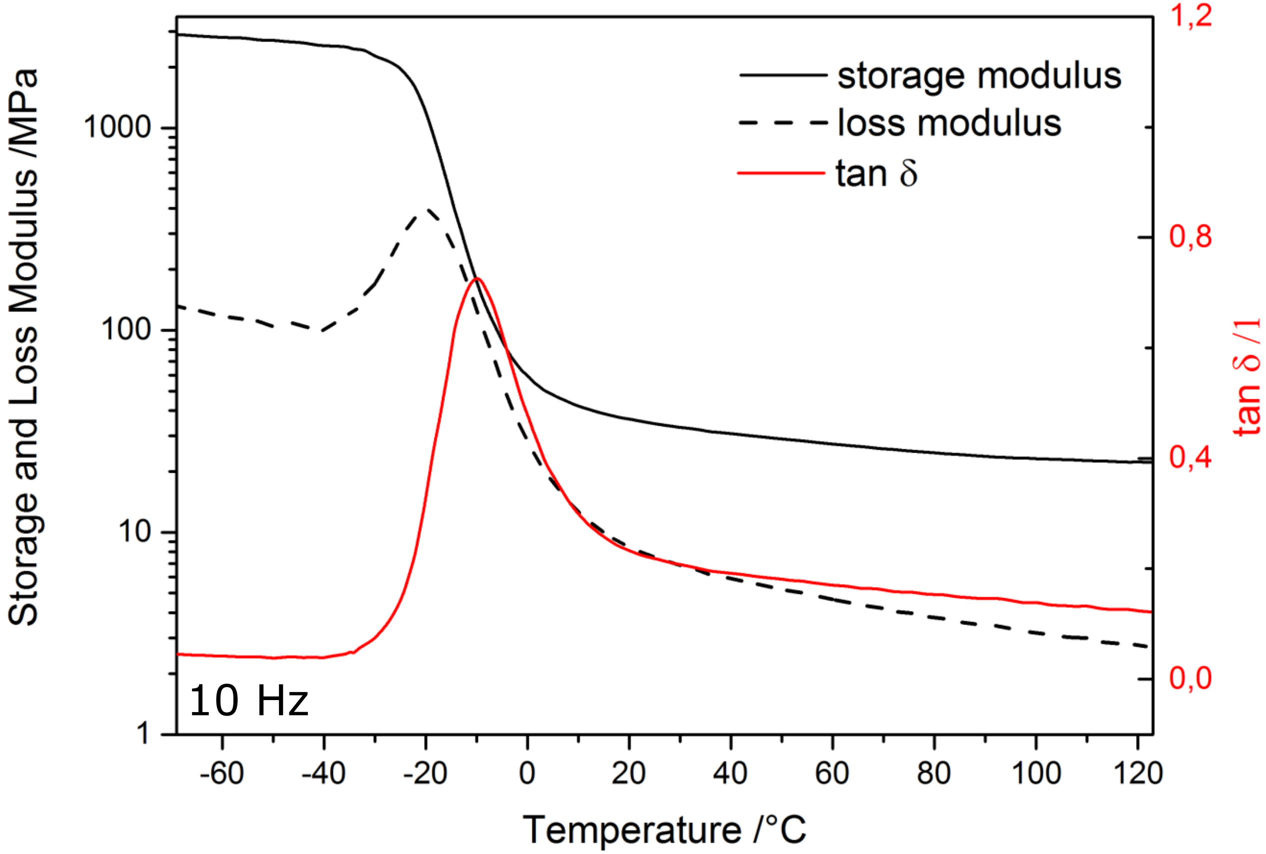


FKM

[1] M. Jaunich, W. Stark, D. Wolff; Polymer Testing 29 (2010) 815-823.

3. Methods/Results:
Component tests - material

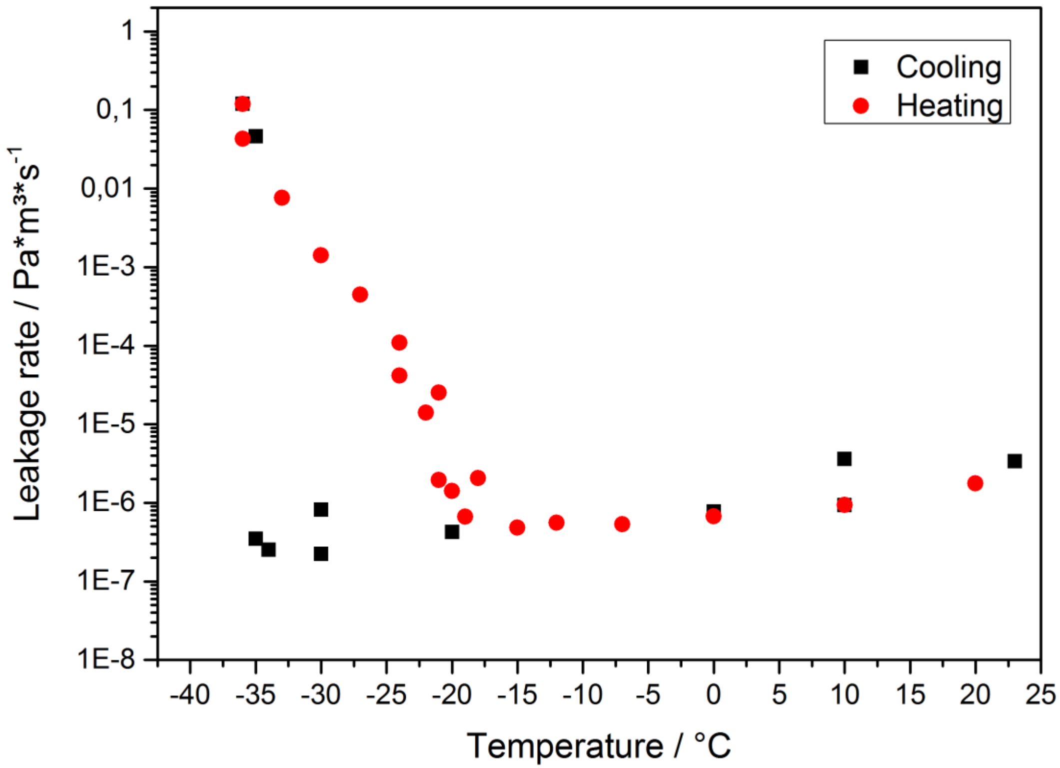
HNBR with 36 wt% ACN, app. 4 % residual double bonds, 80 phr Filler, 5 phr Plasticizer



3. Methods/Results:

Component tests – static

HNBR

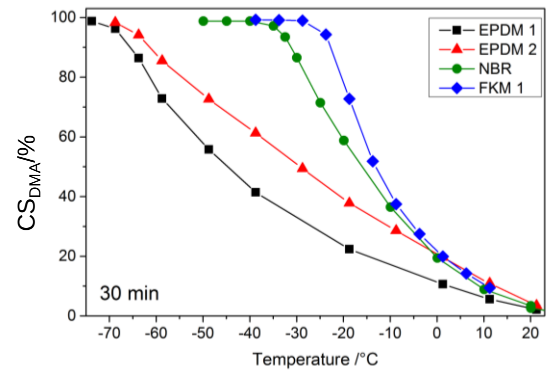


1. Step wise cooling
   →leakage rate increase at -36°C

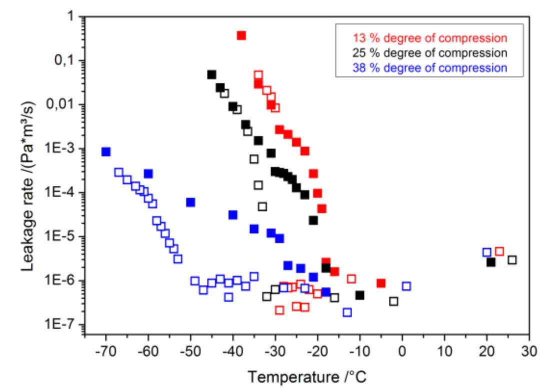
2. Step wise heating
   →leakage rate normalized around -18°C

3. Methods/Results:
- overview of results

Investigation of:
- temperature dependence of properties, e.g. Compression Set
- ➢ Description of temperature dependence



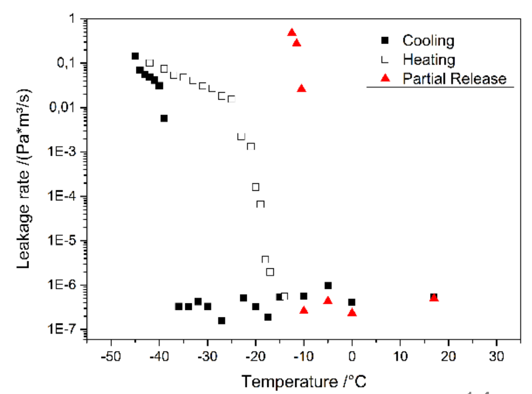
- sealing performance and influencing factors
- ➢ Supercooling for leakage path formation
- ➢ Influence of degree of compression



- Equipment for quick partial release

(quick: within a second; partial: from 25 % to 23 % compression (<0,2 mm))

- ➢ leakage path formation observed at much higher temperatures
- ➢ Correlation with recovery behaviour

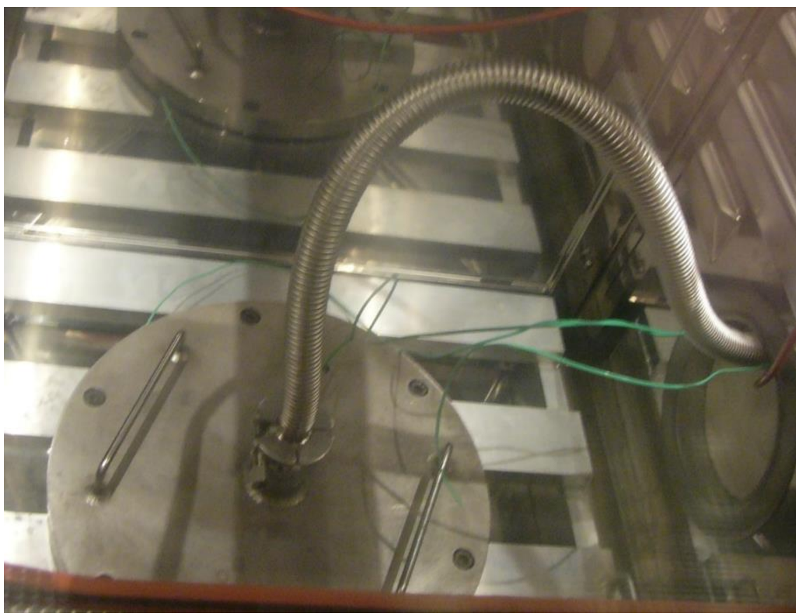
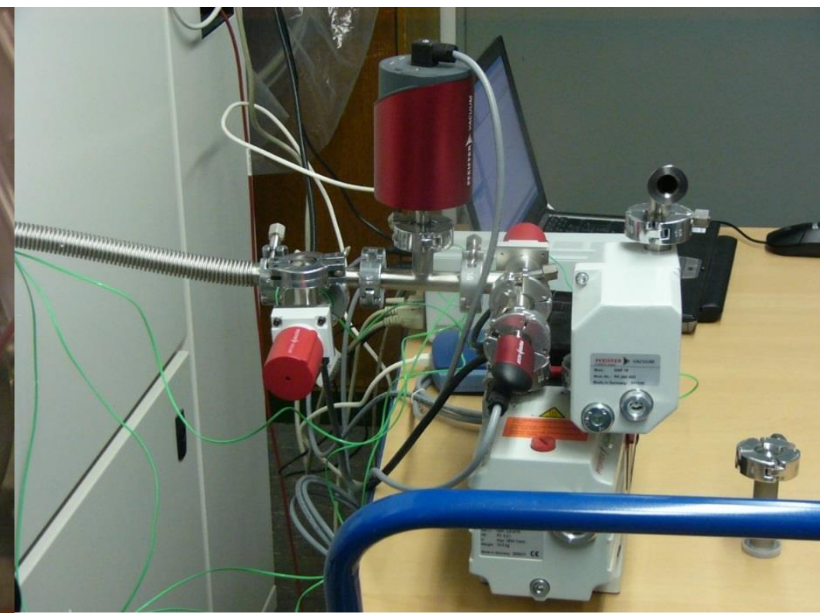


4. Conclusion

- Elastomers show a complex material behavior
- Structure-property and property-performance correlations are helpful for material selection
- Low temperature performance is strongly influenced by the glass rubber transition
  - Under static conditions functionality could be given even below glass transtion
  - Already small deformations can lead to leakage in the temperature region of glass transition, even above!

  and possible crystallization of the material
- Failure temperature is strongly influenced by application conditions and the tightness requirements
- Lower Tg does not necessarily result in lower failure temperature

Thank you for your interest!

Contact:

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Selection of papers focussed on low temperature behaviour:

- M. Jaunich, W. Stark, D. Wolff, A new method to evaluate the low temperature function of rubber sealing materials, Polym. Test, 29 (2010) 815-823.
- M. Jaunich, W. Stark, D. Wolff, Comparison of low temperature properties of different elastomer materials investigated by a new method for compression set measurement, Polym. Test, 31 (2012) 987-992.
- M. Jaunich, W. Stark, D. Wolff, Low Temperature Properties of Rubber Seals, KGK-Kautsch. Gummi Kunstst., 64 (2011) 52-55.
- M. Jaunich, D. Wolff, W. Stark, Low Temperature Properties of Rubber Seals - Results of Component Tests, KGK-Kautsch. Gummi Kunstst., 66 (2013) 26-30.
- T. Grelle, D. Wolff, M. Jaunich, Temperature-dependent leak tightness of elastomer seals after partial and rapid release of compression Polym. Test, 48 (2015) 44-49.
- T. Grelle, D. Wolff, M. Jaunich, Leakage behaviour of elastomer seals under dynamic unloading conditions at low temperatures, Polym. Test, 58 (2017) 219-226.
- A.G. Akulichev, B. Alcock, A.T. Echtermeyer, Elastic recovery after compression in HNBR at low and moderate temperatures: Experiment and modelling, Polym. Test, 61 (2017) 46-56.
- A.G. Akulichev, A.T. Echtermeyer, B.N.J. Persson, Interfacial leakage of elastomer seals at low temperatures, International Journal of Pressure Vessels and Piping, 160 (2018) 14-23.