Prediction of the functional limits of seals at low temperatures

Voraussage der Funktionsgrenzen von Dichtungen bei tiefen Temperaturen

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Summary:

Proving the required functionality of seals at low temperatures usually involves considerable effort. If the functional test reveals that the required targets are not met, this not only involves costs, but often also jeopardizes or postpones entire development projects. This raises the question of how realistic low-temperature limits can be determined in advance. This presentation will show that this can be done surprisingly well with conventional cold test methods if the type of stress is sufficiently known, using standardized types of stress. However, the exact nature of the stress can shift the failure limit of the seal at low temperatures up to 20K. And here the use of FE analysis offers new possibilities that are still far too rarely applied. By using appropriate models to simulate viscoelasticity, the limits of seals under the influence of dynamic gap- and pressure-changes could be predicted much more precise, and thus seal housing and loading scenarios could be much better adapted to the potential of available seal materials and seal geometries.

Zusammenfassung

Ein Nachweis der geforderten Funktionalität von Dichtungen bei tiefen Temperaturen ist in der Regel mit einem erheblichen Aufwand verbunden. Stellt man dann im Funktionstest eine Verfehlung der geforderten Ziele fest, geht es nicht nur um Kosten, sondern häufig auch um die Gefährdung oder Verschiebung kompletter Entwicklungsprojekte. Daher stellt sich die Frage, wie im Vorfeld bereits realistische Tieftemperaturgrenzen ermittelt werden können. Dass dies erstaunlich gut mit kon-

ventionellen Kälteprüfverfahren möglich ist, wenn die Art der Beanspruchung hinreichend bekannt ist, zeigt dieser Vortrag anhand von standardisierten Beanspruchungsarten auf. Allerdings kann die exakte Art der Beanspruchung bis zu 20K die Versagensgrenze der Dichtung bei tiefen Temperaturen beeinflussen. Und hier bietet der Einsatz der FE-Analyse neue Möglichkeiten, die noch viel zu selten ausgeschöpft werden. Durch die Verwendung geeigneter Modelle zur Abbildung der Viskoelastizität können die Grenzen der Dichtheit unter Einfluss von dynamischen Spalt- und Druckänderung noch viel präziser vorausgesagt und damit auch Einbauräume und Belastungsszenarien viel besser auf das Leistungspotential verfügbarer Dichtungswerkstoffe und Dichtungsgeometrien abgestimmt werden.

Introduction

Seal design in the low-temperature range requires a lot of seal-knowledge and experience and as well elastomer expertise. The following list identifies the most common problem areas:

- Every elastomer freezes at a certain temperature and loses its recovery behavior. The selection of sealing materials becomes particularly difficult below an application temperature of -30°C
- Almost within each elastomer family, such as NBR, EPDM or FKM, there are very large formulation- and polymer-differences with regard to freezing behavior
- General information on the low-temperature limit on material data sheets is not meaningful and reliable without specification of standardized low-temperature test methods, or even misleading.
- The different types of stresses at low temperatures in terms of pressure and gap movements can explain differences in leakage temperature of up to 20 Kelvin for the same seal.
- In some cases, it is not communicated clearly enough between the customer and the supplier whether the stated lower temperature limits refer only to storage or to a sealing function.
- The importance of the housings of seals is usually underestimated. Surface finish of the sealing surfaces, diameter clearance and the degree of deformation of the seal can be the famous "tip of the scales" in the case of poor design, which then leads to significantly premature leakage in cold conditions
- Aging of the seal due to operating conditions can shift the low temperature limit to higher temperatures, as well as shrinkage due to plasticizer extraction could do.
- The possibilities of FEA simulation to simulate sealing behavior in cold conditions are hardly applied in practice.

The most important "old school" test methods for demonstrating the limits of rubber elasticity at low temperatures.

Since the gap bridging capacity of the seal decreases at low temperatures, it can follow a change in pressure and gap more and more slowly. This loss of elasticity can be represented by almost any number of physical effects or test methods; 23 different possibilities are shown here in /1/. The challenge for the user is to evaluate the results of the respective test methods for his own application. Thermomechanical test methods, in which a specimen is deformed at room temperature, then brought to test temperature and then stress-released, are certainly the closest to a sealing application. Therefore, the two most informative test methods for sealing applications will be explained here, namely the compression set test at low temperatures /2/ and the TR test/3/. With the aid of these results, it is possible to map the broad performance spectrum of elastomer formulations with respect to low-temperature behavior and thus define minimum requirements for sealing materials for behavior at low temperatures. These are characteristic values determined under quasi-static conditions. They allow only general conclusions about application limits, based on empirical values for typical degrees of deformation, gap dimensions, and rates of pressure and gap change. Only testing in the application then provides the application-specific performance limits of the seal ("trial and error" principle) under real conditions with regard to the dynamics of the pressure buildup and the gap-change.

The compression set test at low temperatures /2/

The test method that best represents typical sealing applications here, especially O-rings, is the compression set at low temperatures /2/. Here, a cylindrical specimen is usually deformed by 25% of its height, cooled, and then released at the test temperature. The result is the loss of recovery in % for a given relaxation time of 30 minutes, see Fig. 1. The advantage is that the test method is very close to the application. The disadvantage is the high sensitivity of the test method to erroneous measurements due to heat input and only limited temperature constancy in the chamber; the other disadvantage is that the result is obtained just for one temperature per (rather complex) test.

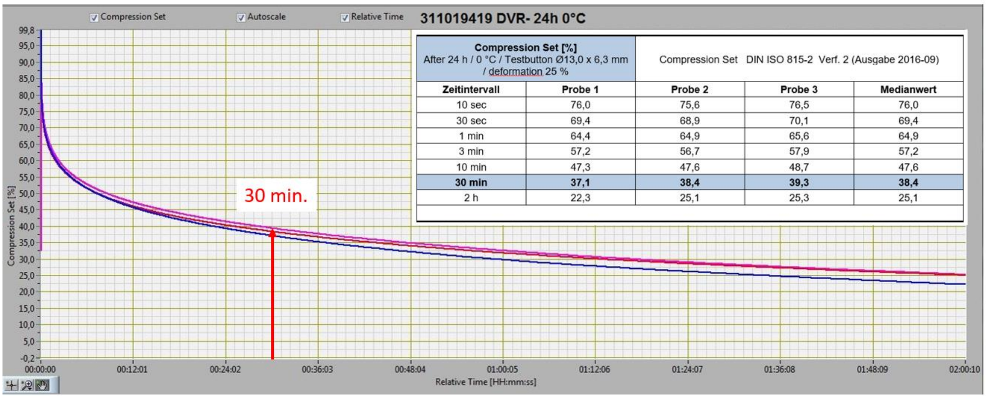


Figure 1: Example of a compression set test of an FKM 75 material (Cpolymer) according to ISO 815-2 at 0°C (24h)

Furthermore, this test only shows the result of a deformation reset after 30 minutes, even if the first reliable results can already be obtained after 10 seconds with the appropriate measuring device. This means that in terms of dynamics, this test method does not come close to representing the situation in a sealing application that leads to leakage. The great practical benefit of this test, however, is that it provides reliable criteria for functionality: as long as the result is less than 80%, the application (assuming typical housings) is considered safe up to the respective test temperature. Unfortunately, the available selection of elastomer materials that meet this conservative criterion at -30°C or even -40°C is very small. A significant upgrading of this method is possible when this test is performed by means of dynamic mechanical analysis, DMA (see below), see Figure 2. Due to the high-resolution displacement measurement in the DMA measuring chamber, the recovery behavior of a specimen can be measured in a loadable manner directly after stress relief. In /4/, a good conformity of the DMA method with the standard method (measuring time after 30 minutes) is shown. Two disadvantages of the standard method are thus eliminated by this method: By programming the test method, it can be carried out automatically at different temperatures, and on furthermore test results can be obtained from it after very short unloading times. As a result, an "old school" method has become a “cutting-edge” test method.

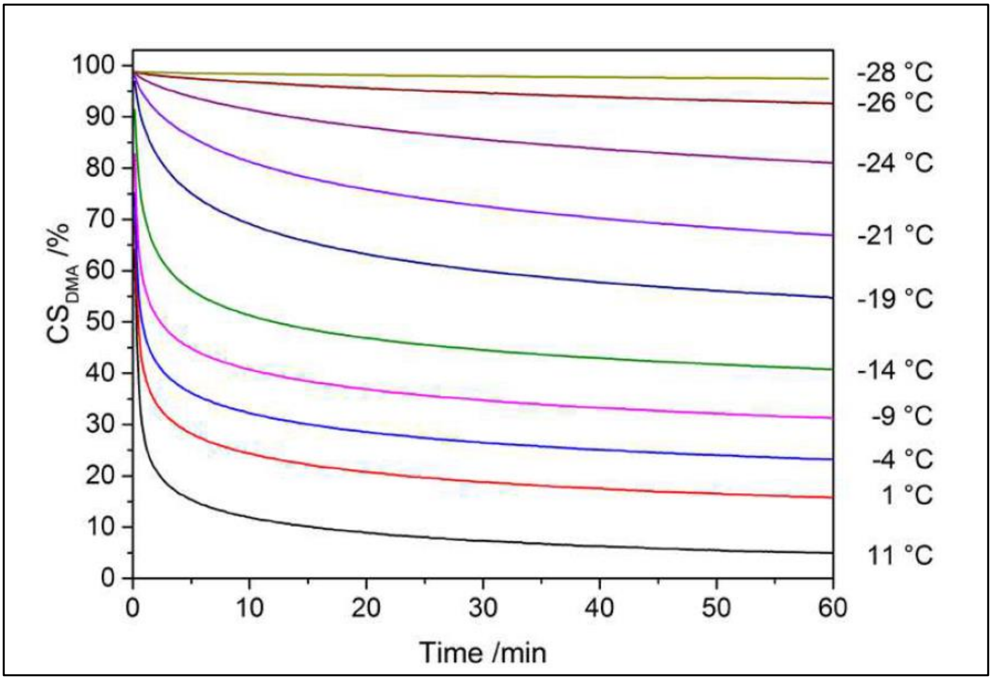
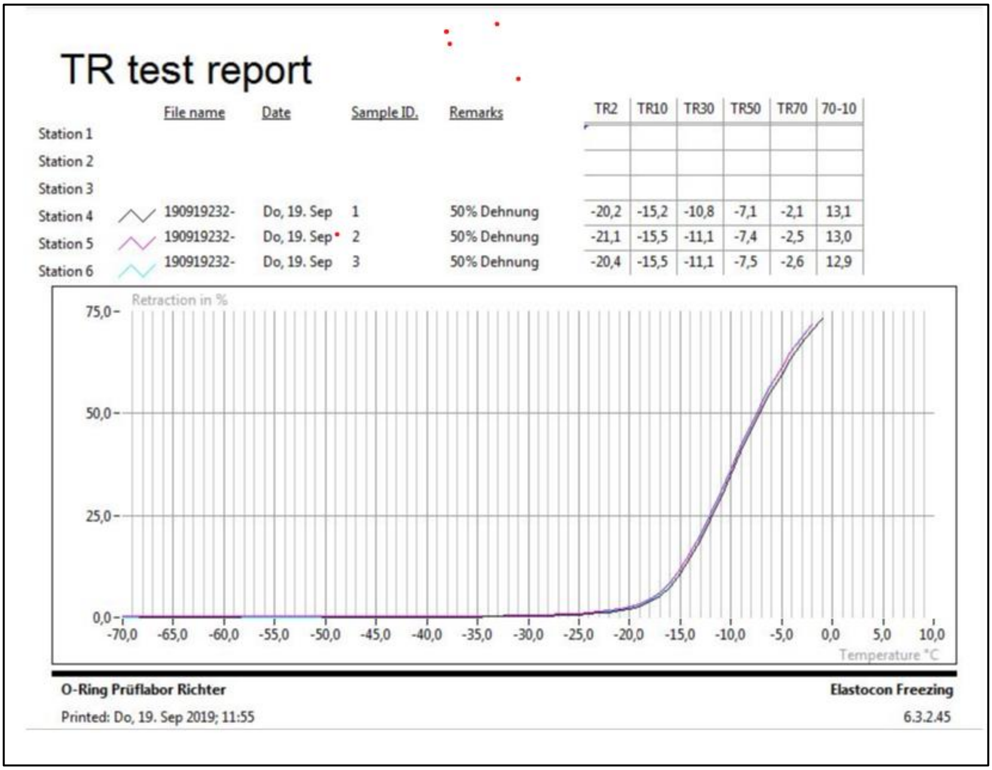


Figure :2: Recovery behavior of an FKM specimen at different temperatures as shown by the compression set (measured via DMA)/4/

The TR-Test

The abbreviation "TR" stands for Temperature Retraction, which means that it measures how a stretched rubber specimen, which is frozen in the stretched state, contracts again during subsequent continuous heating. The temperature at which the specimen has recovered 10% of its deformation is called the TR10 value. This test measures the recovery of its deformation from the completely frozen state to the recovery back to 70%, see Figure 3.



TR test report

	File name	Date	Sample ID.	Remarks	TR2	TR10	TR30	TR50	TR70	70-10
Station 1										
Station 2										
Station 3										
Station 4	190919232-	Do, 19. Sep	1	50% Dehnung	-20,2	-15,2	-10,8	-7,1	-2,1	13,1
Station 5	190919232-	Do, 19. Sep	2	50% Dehnung	-21,1	-15,5	-11,1	-7,4	-2,5	13,0
Station 6	190919232-	Do, 19. Sep	3	50% Dehnung	-20,4	-15,5	-11,1	-7,5	-2,6	12,9

Figure 3: TR test on an FKM 75 material, TR10=-15.5°C (median value)

Based on experience, the TR10 value represents a realistic limit temperature for seals under the influence of typical dynamic pressure- and gap-changes; for small pressure- and gap-changes, satisfactory gas tightness can still be achieved up to approx. 15K below the TR10 value.

Different types of stress on seals at low temperatures

In order to determine realistic low-temperature limits, it is first necessary to define how the gasket is actually stressed in the cold. For this purpose, a distinction can be made between three different load groups

Static, pressureless sealing system *without* pressure- and gap-change

This stress represents the most common type for static seals. It is essentially pressureless, corresponding leak tests usually take place at approx. 1 bar internal pressure, and no gap changes occur. The sealing effect is sufficient as long as there is still contact between the seal and the mating surface. The contact forces play a minor role with this type of seal. The decisive criterion for the sealing function is not the flexibility or elasticity of the seal, but the physical contact or a tolerable minimum gap between the seal and the housing.

This sealing gap must not be more than a few µm. In the case of seals in the low-pressure range (< 2 bar), mere contact of the seal with the sealing surface is sufficient to prevent leakage.

It should be noted, however, that in the case of cold-induced embrittlement, pressure activation of the seal is not or hardly possible.

Corresponding leak tests at DuPont /5/ indicated that a gas tightness of 10-15 Kelvin below the TR10 value is possible in the present stress case. If a compression set was measured instead of the TR10 value, a gas tightness 10-15 Kelvin below the temperature whereas the compression set is 80% can still be achieved for this type of stress. This is probably the most common type of stress on a static seal, for example as a housing seal for an electronic component.

Static sealing system with constant pressure *without* pressure- and gap-change

In this type of stress, a static seal is subjected to a constant pressure of at least 50 bar at room temperature, that means the seal is pressure-activated, and then cooled down. In the cold, it does not undergo any pressure or gap changes. As a functional criterion, the seal must still be in contact so that the sealing surface pressure is maintained. In this case, tightness up to more than 20 Kelvin below the TR10 value is possible (see table 1). This is a typical example of a static O-ring seal for a gas tank, which is cooled down in a pressurized condition. This however is not a typical sealing situation.

Sealing system *with* pressure- or gap-change (pressurized static seal or dynamic seal)

This case presents the greatest challenge to a sealing system. As a functional criterion, in addition to the contact of the seal, there must still be a residual elasticity. So the seal can be deformed by the pressure, that means activated, or follow a gap-change in a reasonable time. For dynamic sealing systems, then, in the cold, the gap dimension that occurs and the surface quality, especially also the surface structure, have a significant influence on the lower functional limit. In application practice, the TR10 value has proven to be the lower limit temperature. This is confirmed for pressurized seals at low temperatures in test results published by JAMES WALKER & Co. Ltd., see Tab. 1 /6/

	Sealing-Stress-Case	HNBR LT [°C]	FKM LT [°C]	FKM ULT [°C]
TR10-Temperature		-36	-31	-40
Tightness up to	Constant high pressure	-53	-54	-55
	Pressurized at low temperature to 50 bar	-	-31	-41
	Pressurized at low temperature to 100 bar	-41	-31	-41
	Pressurized at low temperature to 175 bar	-	-40	-45

Table 1: Results of low-temperature tightness tests with different types of stress on special elastomer compounds that are particularly flexible at low temperatures

Cutting Edge: Simulation of cold behavior using DMA data-based models

If rubber elasticity is to be described from an application engineering point of view, this can be done, for example, by means of the following characteristic features:

- Rubber elasticity means that, compared to ideal elasticity, there is always a delay between force and displacement, and thus the recovery behavior after a change in gap is always delayed.
- This delay decreases at elevated temperatures and increases at low temperatures.
- similarly, the deformation resistance increases at low temperatures
- as the deformation rate increases, the deformation resistance also increases.

By means of a DMA (dynamic mechanical analysis), rubber samples can be deformed within a frequency spectrum (e.g. 0.01 Hz to 100Hz) and temperature spectrum (e.g. -100°C to 200°C) specified by the test instrument. The deformation behavior can thus be described for a defined deformation via the required deformation stress and the phase shift between force and displacement. If the available frequency spectrum is now screened in a meaningful way over the available temperature range, which is interesting from an application point of view, in a so-called multi-frequency analysis, a set of curves is obtained, which can be summarized by certain mathematical methods. This also makes it possible to "master" deformation states that can no longer be determined physically. The special feature of this test method is that it can be used to test and describe rubber materials as they are stressed in sealing applications. This means with high deformation rates and rather small gap-changes. These characteristic values obtained under conditions close to the application can then be incorporated into FEA analyses that additionally take into account all other boundary conditions relevant to the application. This means that it can be determined at a very early stage of development to what extent the existing sealing materials are suitable for the new applications. If necessary, sufficient countermeasures can be taken at an early stage, such as stiffening critical components to limit gap widening or rubber compounds with improved low-temperature behavior.

Possible applications of DMA data for the simulationof the cold behavior of seals

Part 1- Verification of the simulation-model using "old-school" test-methods

The purpose of this simulation is not primarily to replace established "old-school" material testing methods, but to use these results to check the simulation model and the recipe-specific inputs from the multi-frequency analysis. However, it must be taken into account that the two "old school" test methods presented tend to evaluate high restoring deformations. The gap changes, however, which ultimately trigger leakage, are typically between 0.01 and 0.05 mm, i.e. considerably smaller compared to the recovery measured after stress relief by the classic cold test methods.

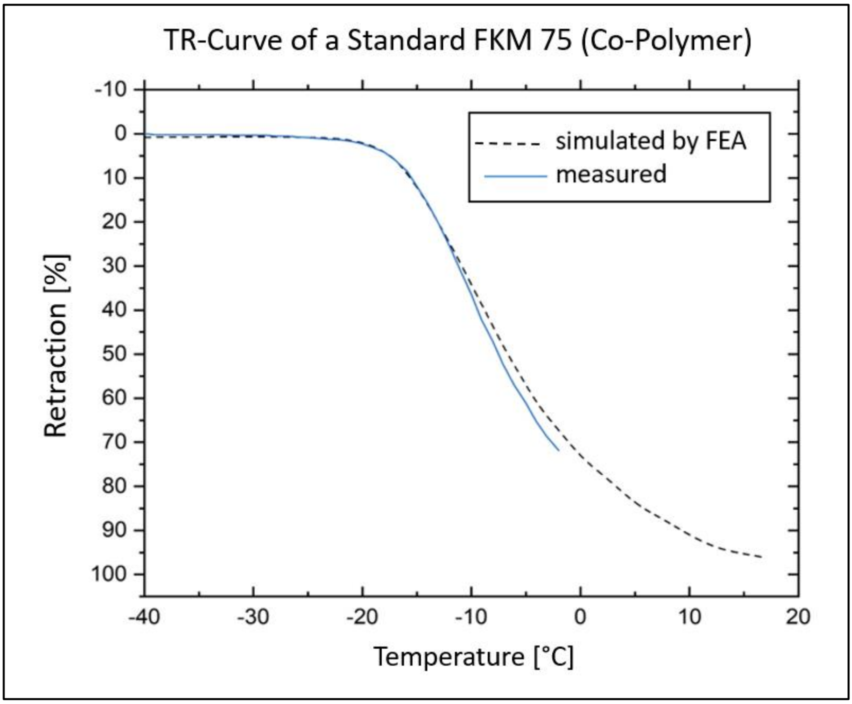


Figure 4 shows the complete recovery curve from the TR test to 70% recovery for a FKM 75 copolymer.

Figures 4 and 5 show the comparison between the measured and calculated results. The simulation has been done by Dr. Manfred Achenbach.

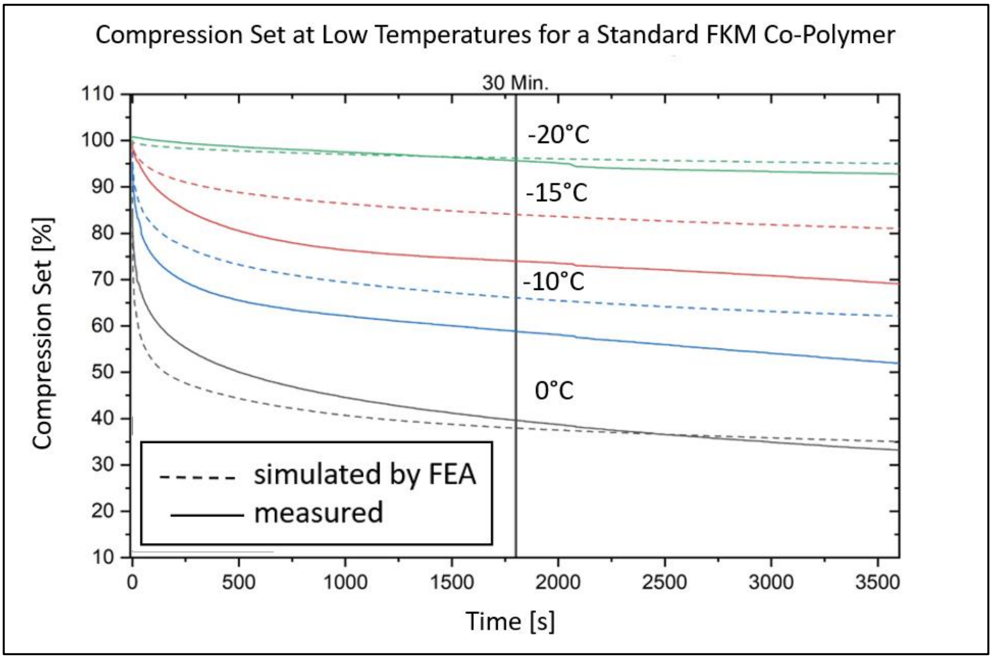


Figure 5 shows the compression set at low temperatures at four temperatures for an Standard FKM (Copolymer)

The agreement for the TR10 test is excellent, for the compression set at the 30 minute value good (-20°C and 0°C) to satisfactory (-10°C and -15°C). However, considering only the beginning of the curves relevant to the sealing function, the agreement is good at all four temperatures.

If this approach is now extended to other materials, such as EPDM 80 and NBR 70, the agreement is not quite as good, see Figure 6. The rubber elasticity is too complex for the standard DMA-based model used to accurately represent all the effects of a wide variety of polymer families or structures simultaneously for both large and small deformations. Thus, "old school" materials testing can help to reveal model inaccuracies. There is no doubt, however, that this approach is very well able to mapping the performance limits of seals at low temperatures under dynamic loading under a wide range of boundary conditions, and thus to setting the course in good time for the right material selection or for adapted boundary conditions.

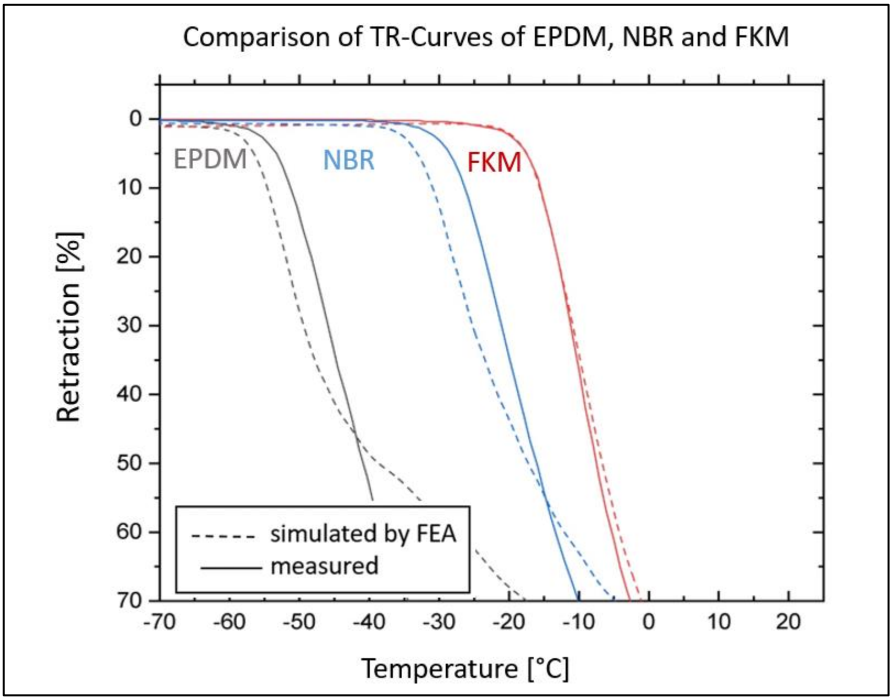


Figure 6 shows complete recovery curves from TR testing to 70% recovery for an FKM 75 copolymer, an NBR 70- and an EPDM 80-material.

Part 2- simulation of an application

In a simulation carried out by Dr. Achenbach, it is shown how a FKM O-ring deformed by 20% with a TR10 value of -15°C reacts to a gap-change of 0.05 mm within 0.05s at different temperatures. While at 20°C the O-ring can follow the gap-change, see Figure 7, at 0°C a short lift-off from the outer contact surface already occurs, see Figure 8. At -10°C the gap is closed only about halfway after 2 seconds, see Figure 9, and at -20°C the O-ring can´t longer follow the gap-change at all, see Figure 10

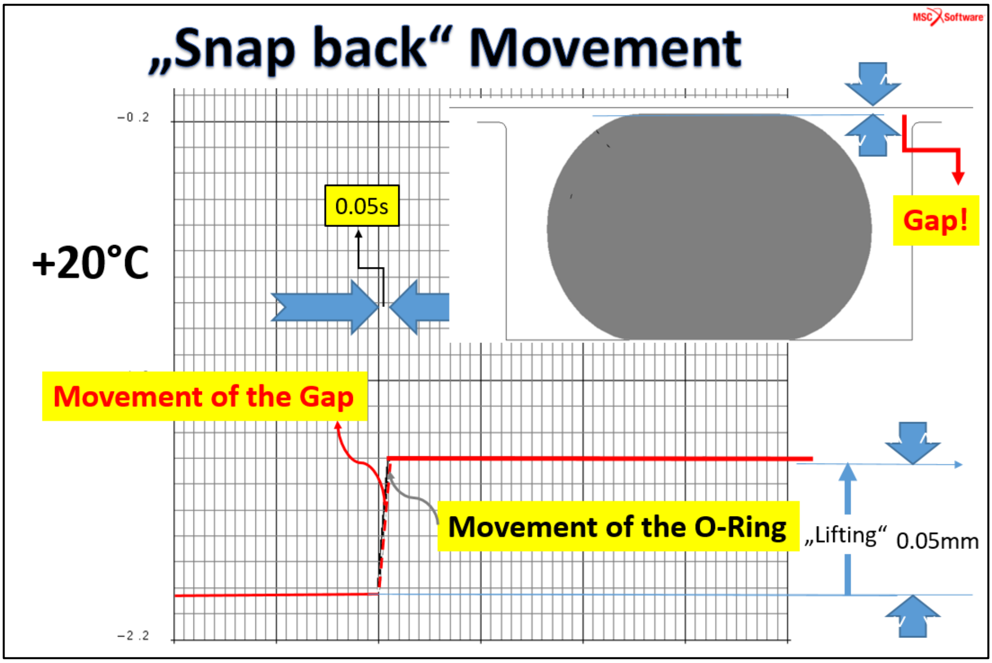


Figure 7-“Snap back movement of an FKM Standard O-Ring (Copolymer) at 20°C

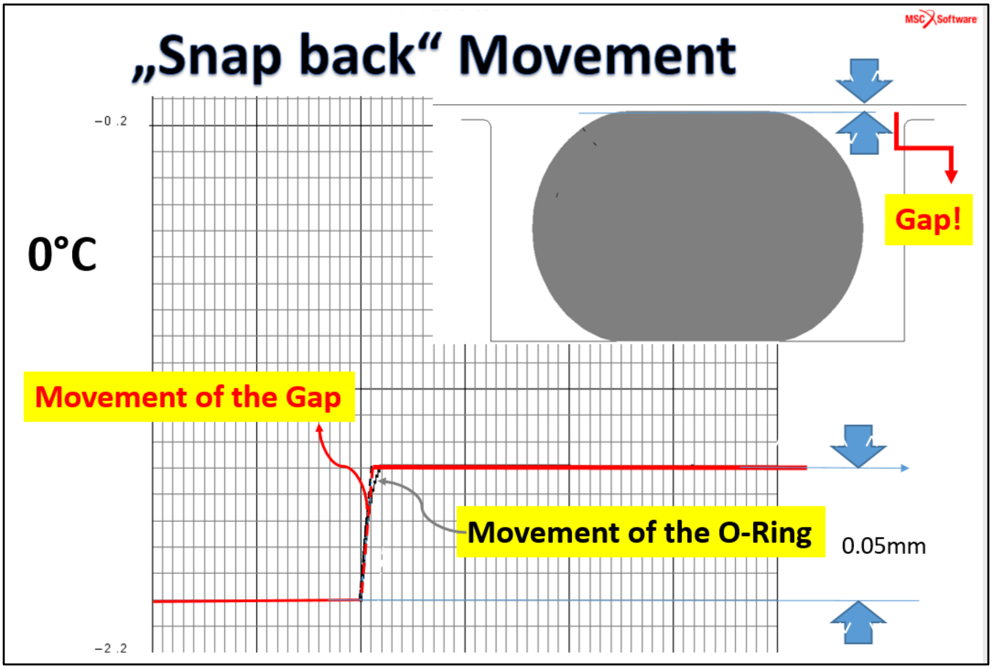


Figure 8-“Snap back movement of an FKM Standard O-Ring (Copolymer) at 0°C

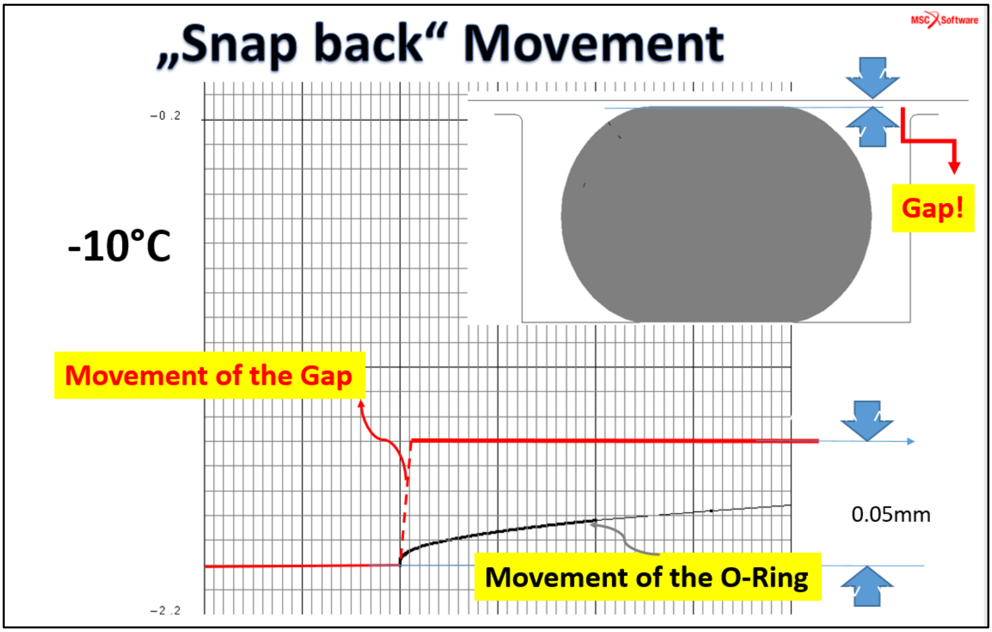


Figure 9-"Snap back movement of an FKM Standard O-Ring (Copolymer) at -10°C

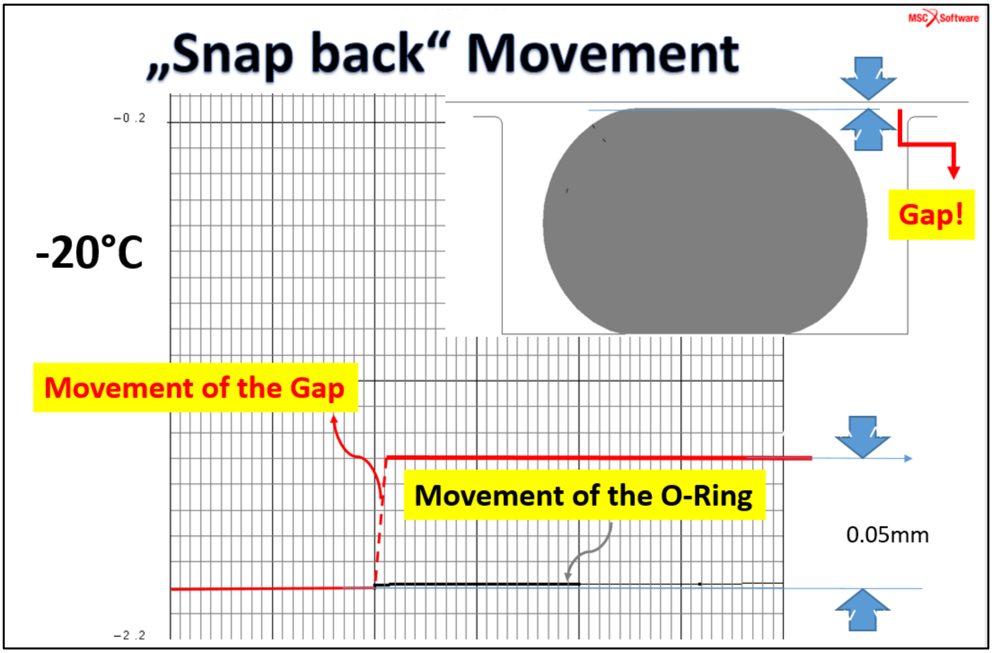


Figure 10-"Snap back movement of an FKM Standard O-Ring (Copolymer) at -20°C

Summary

The low-temperature behavior of elastomer seals, especially O-rings, can be predicted with sufficient accuracy by means of frequently used and proven thermomechanical low-temperature tests, provided that the stresses are clearly defined and the housings comply with the relevant regulations. It is then up to the user to specify the materials with sufficient accuracy. In addition, however, it is possible to determine low-temperature limits of elastomer seals for any seal geometries as well as gap and compressive stresses by means of DMA data-based FEA simulation. This can minimize development times and costs and lead to better solutions

Literature cross-references

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/4/ Dietmar Wolff, Mathias Jaunich, Ulrich Probst und Sven Nagelschmidt, BAM Berlin, Understanding the Long-term Behavior of Sealing Systems and Neutron Shielding Material for Extended Dry Cask Storage, PSAM 12.Juni 2014, Honolulu Hawaii

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