Behaviour of Elastomeric Seals at Low Temperature

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Abstract

The properties of elastomer O-ring seals (Viton, silicone rubber, EPDM) at low temperature have been investigated by measuring the gas leakage rate and the sealing force during thermal cycling between +20°C and -70°C. For all materials it has been found that at a well defined (critical) temperature the leakage rate sharply rises from permeation level to a high value which is determined by gas streaming through the leak path between the O-ring and the flange surfaces arising from thermal contraction of the elastomer in the glassy state. At the critical temperature the sealing force has been found to be zero or even negative due to adhesion between the elastomer material and the flanges. For all seals the critical temperature is well below the glass transition of the elastomer and also significantly below the temperature where the compression set becomes 100 %. Warming up the sealing system restores leak tightness. Low temperature cycles of elastomeric seals have been found to be entirely reversible.

1. Introduction

The use of elastomeric sealing materials at low temperature is limited by the transition to the glassy state which is accompanied by the complete loss of rubber elasticity. This problem has been of concern, e.g., for transport casks for radioactive material where a sufficient degree of leak tightness has to be ensured during transport at ambient temperatures down to -40°C (Weise et al. 1987, Burnay and Nelson 1991). Measurements of seal leakage as a function of temperature have shown large differences between the properties of the common elastomers Viton (fluoro rubber), EPDM (ethylene-propylene-diene-rubber) and silicone rubber. For all materials a well defined critical temperature was observed where the gas leakage rate rapidly rises by several orders of magnitude during cooling down. The aim of this work has been to investigate the influence of relevant parameters on the low temperature behaviour of sealing systems and to find a correlation between the (critical) temperature of leak formation and material data commonly used for characterizing the sealing elastomer. The gas leakage rate and the sealing force exerted by the seal on the flange have been measured simultaneously as a function of temperature. The compression set and the linear expansion coefficient of the elastomers were measured for describing their thermomechanical properties.

2. Experimental

Figure 1 shows a schematic representation of the experimental setup consisting of the sealing system in a temperature test chamber, the data acquisition system and the temperature control. Gas transmission through the sealing system is determined with a He-leak detector (HLT) or alternatively by measuring the gas pressure on the vacuum side at constant pumping speed. The temperature of the sealing system is computer controlled allowing very flexible programming of the temperature cycles. The sealing system (Figure 2) consists of a fixed flange which is connected to the vacuum system and the upper blind flange which is screwed to a force transducer measuring the sealing force as a function of temperature.

3. Results

Measurements have been carried out on O-ring seals of 42 mm inner diameter and 5 mm cord diameter. The compression of the elastomer cord was typically 25 % except in a series of measurements where the compression was varied keeping the other parameters constant. A representative measurement on silicone rubber is shown in Figure 3 where the sealing force F and the pressure in the vacuum system are plotted as a function of temperature. When cooling down from room temperature the sealing force decreases approximately linearly until at a certain point the force rapidly drops to zero and then changes its sign. Negative values can partly be attributed to the force exerted by the test gas (1 bar) on the upper flange which amounts to about 160 N. The other contribution may be caused by adhesion of the elastomer to the flange surfaces. At the temperature of maximum negative force the pressure sharply rises indicating the formation of a leak path between the O-ring and a flange surface. The gas leakage rate increases with decreasing temperature. When the temperature rises, the sealing force increases again and the leakage rate drops to the initial value at room temperature. Low temperature cycles can be repeated many times without any significant change in the behaviour of the elastomeric seal. The reversibility of leak formation leads to the same failure temperature (critical temperature T_{crit}) within a few degrees. For EPDM high negative values of the sealing force are observed at the critical temperature (Figure 4). The occurrence of leakage coincides with a sudden decrease of the absolute value of the force indicating a (partial) separation of the O-ring from the flange surfaces. For Viton and silicone rubber adhesion is less important. There are several parameters which influence the critical temperature of a sealing system. Generally T_{crit} decreases with increasing compression of the O-ring cord (Figure 5). At extremely high compression - typically above 70 % squeeze - Viton seals may be used even at cryogenic temperatures (Weitzel, et al. 1961). For standard high vacuum applications compression values around 25 % are commonly used. Therefore the results reported here were obtained for this standard condition. The upper temperature of the thermal cycle also influences the critical temperature of the sealing system. For Viton and EPDM, T_{crit} decreases with increasing upper temperature whereas no effect was observed for silicone rubber. The change of the properties of elastomers at low temperature may be characterized by different parameters which can be measured with standardized techniques (Nagdi 1988). The glass transition temperature is readily determined by measuring the linear expansion of a cylindrical elastomer sample. At the glass transition the linear expansion coefficient changes discontinuously, being significantly lower in the glassy than in the rubbery state of the elastomer. Another useful parameter is the compression set describing the ability of elastomers that have been compressed at room temperature and then subjected to low temperature, to recover from deformation when taken from the clamping device while still at the low temperature. 100 % compression set means that the O-ring completely retains its initial deformation after removal of the compressive force. At T_{100} the material loses its rubbery behaviour. If the deformation returns to half the initial value the compression set is 50 %. As an example Figure 6 shows a typical compression set curve as a function of temperature for silicone rubber exhibiting a rapid increase around -40°C when cooling down. Rubber elasticity is completely lost at T_{100} = -45°C. The glass transition temperatures, the temperatures for 50 % and 100 % compression set and the critical temperatures of the O-ring materials are compiled in Table 1.

Table 1 Characteristic data of the sealing materials

T_{crit}	temperature where gas leakage rises above the permeation level
T_{50}	temperature for 50 % compression set
T_{100}	temperature for 100 % compression set

Standard compression of the O-ring cord: 25 %

Upper temperature of the thermal cycle: room temperature

upstream pressure of the test gas: 1 bar (He or air)

O-ring dimensions: 42 mm ID, 5 mm cord diameter

Flat stainless steel flanges.

Viton 1...8 different Viton mixtures

Material	T_{crit} (°C)	T_{50} (°C)	T_{100} (°C)	T_g (°C)
Viton 1	-35 ± 2	-11	-21	-7
Viton 2	-20 ± 2	-1	-7	+1
Viton 3	-31 ± 2	-19	-27	-6
Viton 5	-44 ± 2	-23	-33	-23
Viton 7	-41 ± 2	-18	-33	-
Viton 8	-41 ± 2	-15	-33	-
EPDM	-61 ± 2	- 25	-47	-30
Silicone	-63 ± 2	-40	-45	-31

4. Discussion and Conclusions

The data in Table 1 show that the critical temperature T_{crit} where leak path formation occurs depends to a certain extent on the special compound of the elastomer. Various Viton compounds were tested with critical temperatures between -20°C and -41°C. By varying the mixture of the components the low temperature behaviour of the elastomer can be optimized (Jahn 1968). From comparison of the data it is evident that low temperature leakage occurs significantly below the glass transition. The sealing function is preserved even when the elastomer is already in the brittle glassy state as long as the sealing force is sufficient for maintaining a close contact between the elastomer and the flange surfaces. T_{100} may be used for assessing the applicability of sealing materials at low temperature though there is a considerable safety margin compared to the critical temperature. The low temperature properties of EPDM and silicone rubber are quite similar. For these materials no pronounced differences between different compounds were observed. From the point of view of low temperature performance silicone rubber is superior to Viton and EPDM but for vacuum applications the high gas permeation may be a severe disadvantage. EPDM has a lower gas permeability than silicone rubber and its behaviour both at low temperature and at elevated temperature above room temperature is favourable. Therefore EPDM might be a good compromise if in addition to good vacuum properties the low temperature behaviour is of importance. This work was supported by the German ministry of environmental protection and reactor safety under contract StSch 1081.

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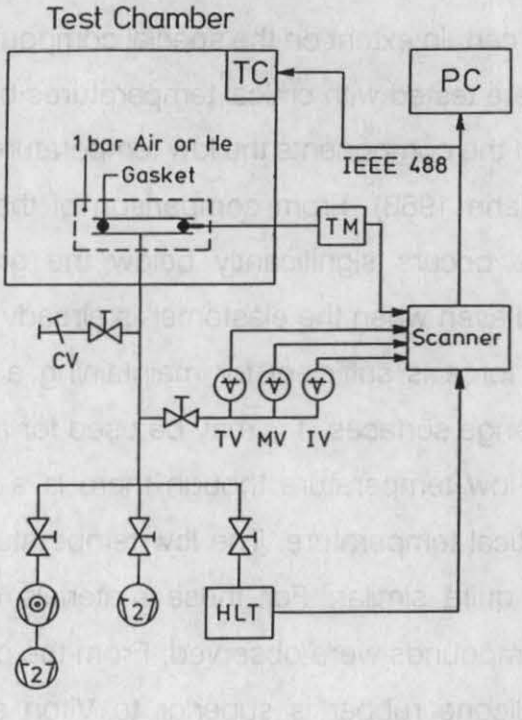

Figure 1. Experimental setup for measuring the gas leakage and the sealing force of elastomer O-rings at low temperatures.
HLT He-leak detector
TM temperature measurement
TC temperature control

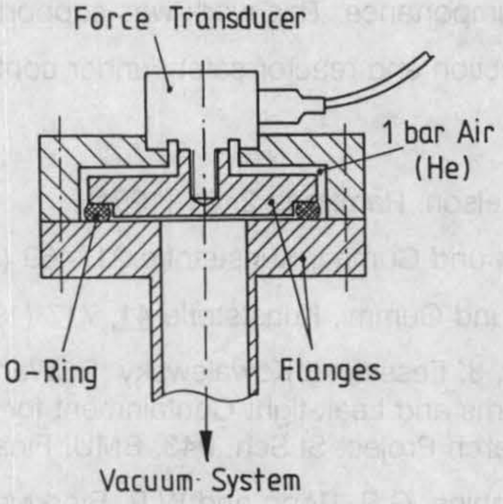

Figure 2. Details of the sealing system. O-ring: 42 mm ID and 5 mm cord diameter.

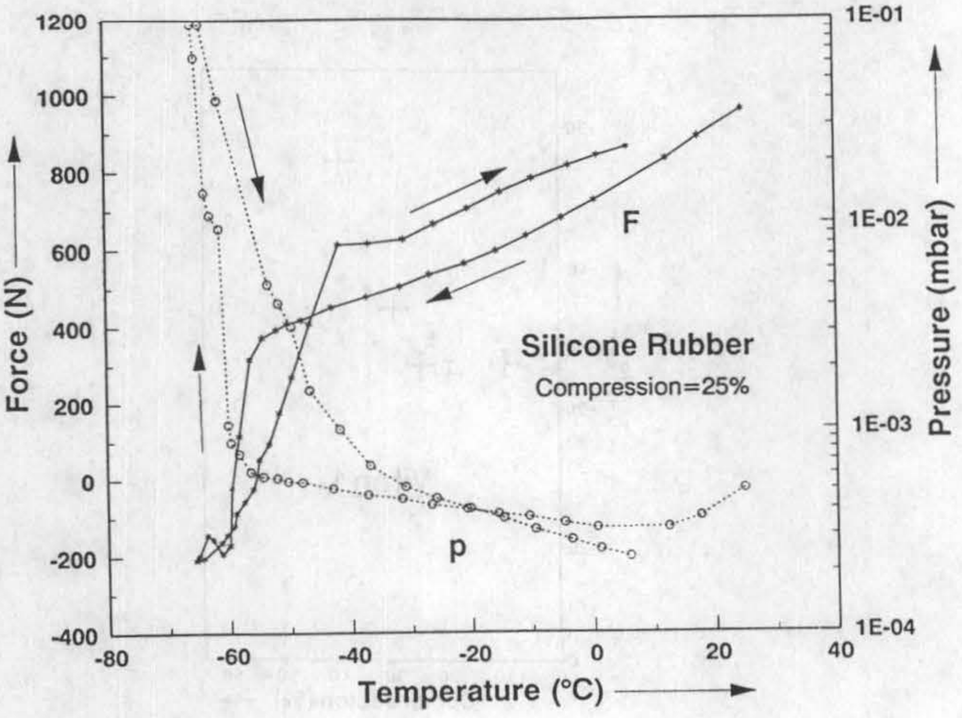

Figure 3. Sealing force F and pressure on the vacuum side (or leakage rate) as a function of temperature for a silicone rubber O-ring.

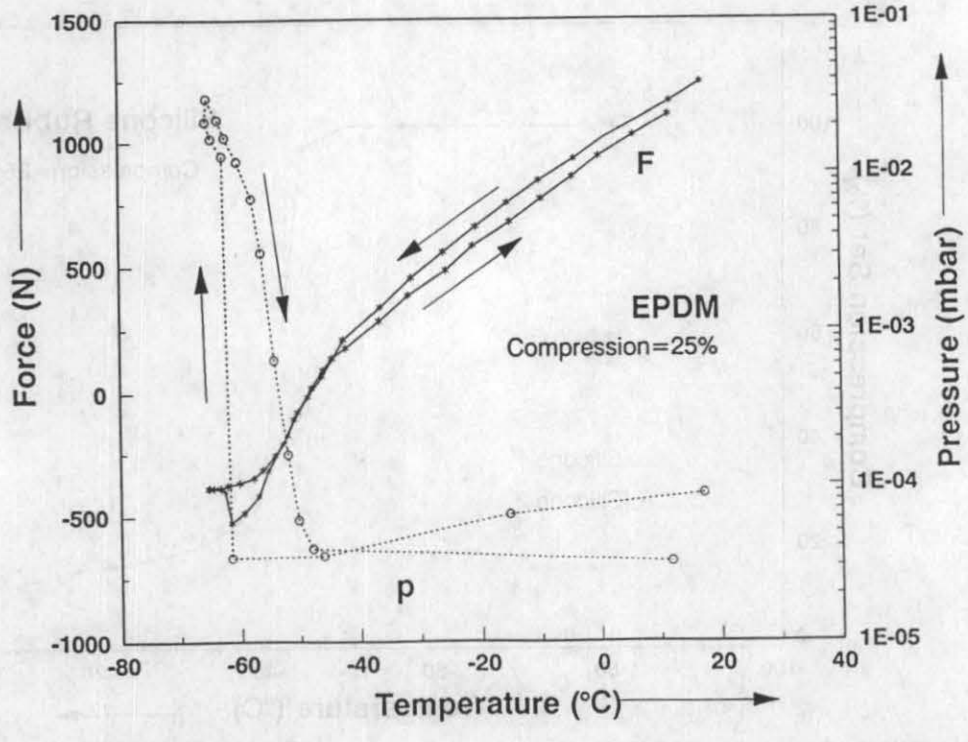

Figure 4. Sealing force F and pressure on the vacuum side (or leakage rate) as a function of temperature for an EPDM O-ring.

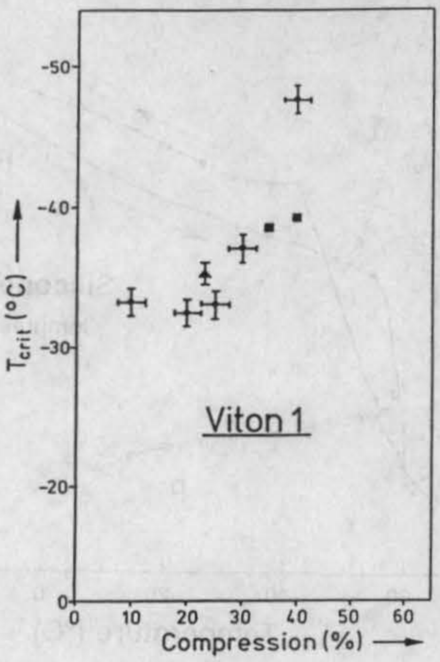

Figure 5. Temperature of leakage occurence as a function of the compression of Viton O-rings.

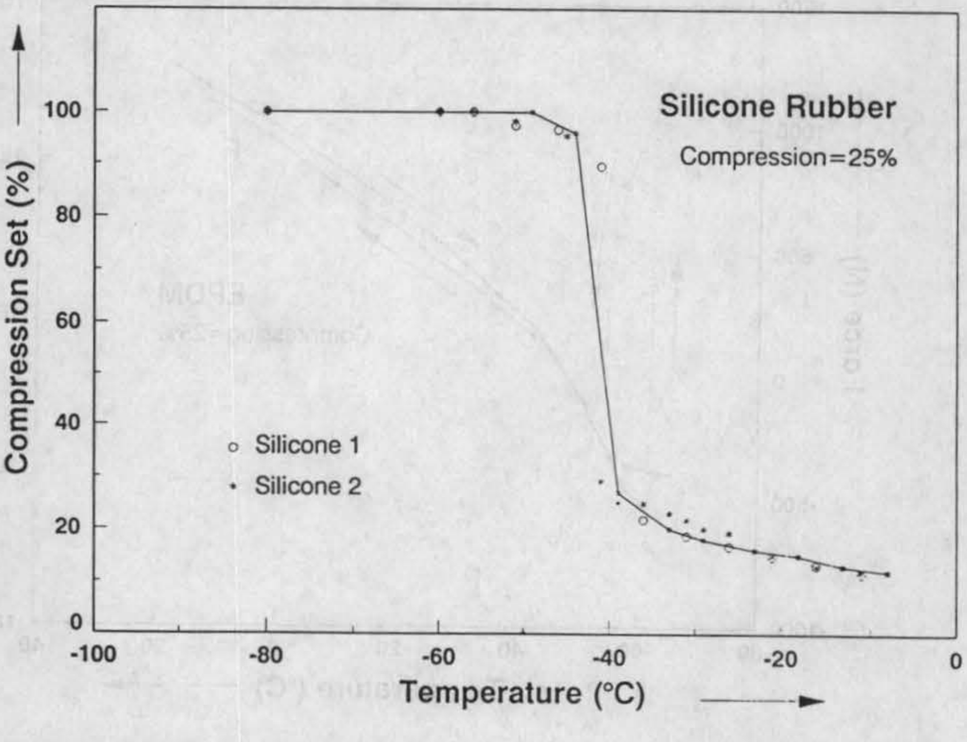

Figure 6. Compression set of silicone rubber as a function of temperature.