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O-Rings in Hot Water and Steam – A Problem for Many Users

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This subject is defined by practical experience. Hardly any other operating media cause more problems with O-rings than water, steam or aqueous compounds, particularly at higher temperatures. Therefore, this paper will deal specifically with these issues by using practical examples.

Typical warm or hot water applications of O-rings are, for example, found in power plants and in process technology, such as secondary seals in mechanical seals of pumps. Other application examples are pumps, armatures and fittings in sanitation and heating technology as well as in long distance heating supply systems.

Examples of Damage on Failed O-Rings

First, the main causes of premature failure of O-rings in hot water and steam application will be demonstrated by pictures of failed O-rings. Poor compound quality and/or an incorrect degree of vulcanization of EPDM O-rings in Figure 1, or an insufficient resistance to hydrolysis of VMQ/FPM/FEPM or FFKM O-rings, see also in Figures 2 to 4.

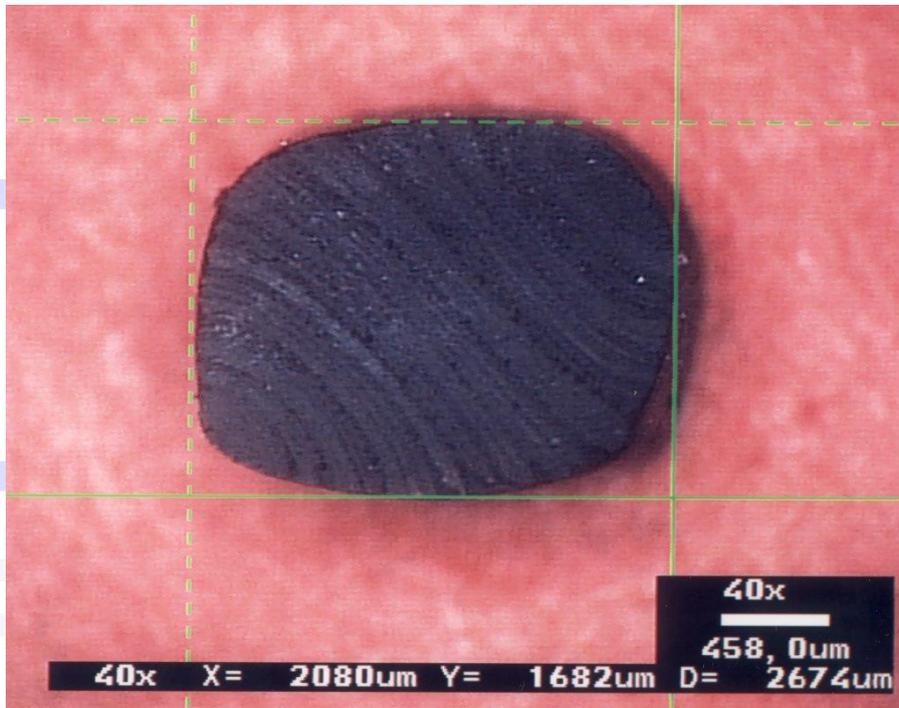


Figure 1: Severe permanent deformation of an EPDM O-ring after several months of service in a heating armature

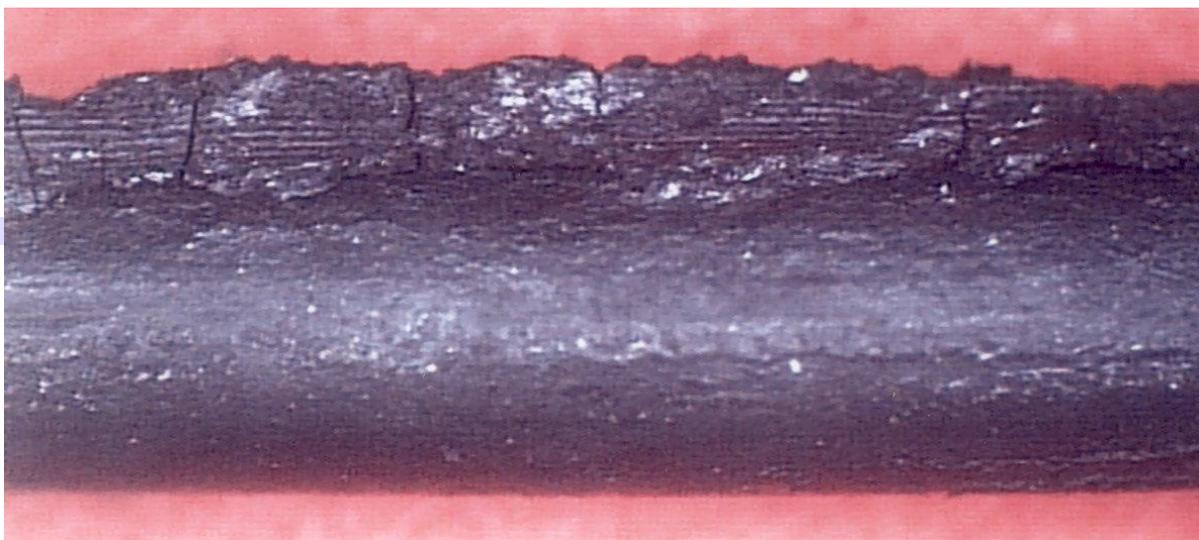


Figure 2: FKM O-ring destructed on the water side after a few months service at 130°C



Figure 3: FKM O-ring (Aflas) after a service of about 5 years in an armature of a district heating network at hot water temperatures up to 170°C (triangular groove)

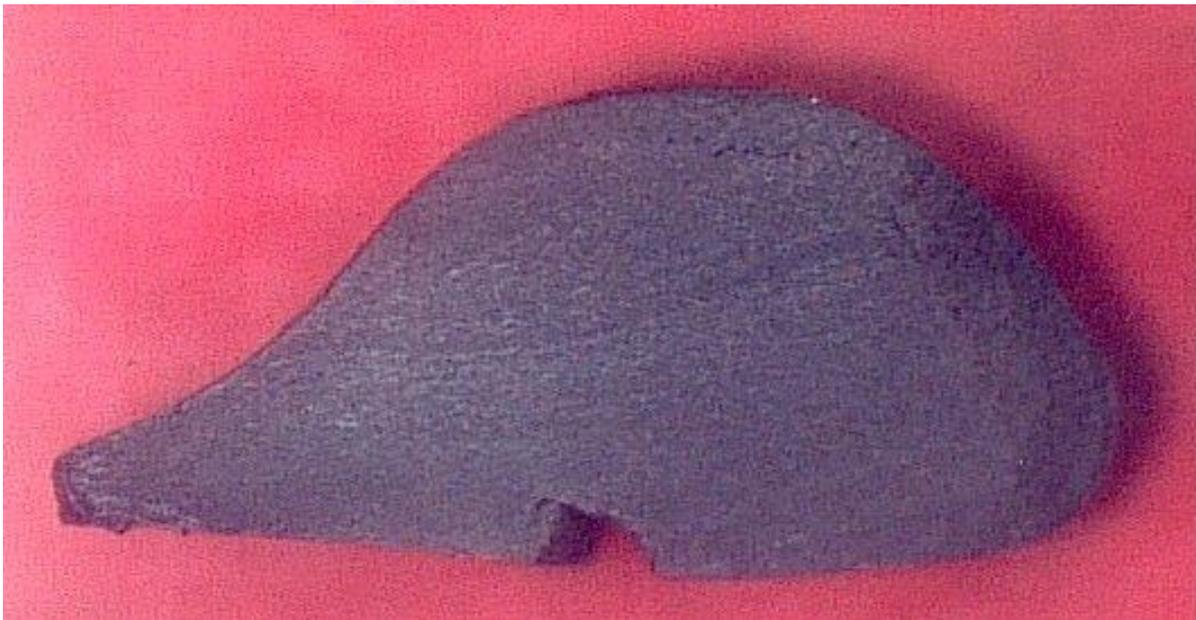


Figure 4: FKM O-ring after several weeks of use at 175°C in a press fitting and 175°C water temperature

These pictures prove that the effect of water at high temperatures can greatly reduce the service life of O-rings. Therefore it is necessary to carefully select O-rings with regard to their suitability to the service in question and to optimize the installation spaces of O-rings for each particular use.

Elastomeric Materials for Warm and Hot Water Applications

When defining applications in warm water with a permissible continuous temperature of at least 60°C and considering the service life requirements set by the users, it is possible to characterize material families for hot water applications, at least for each specific group of users, in principle as follows:

1. EPM-/EPDM-Elastomers
2. IIR-/CIIR-/BIIR-Elastomers
3. HNBR-Elastomers
4. VMQ-Elastomers
5. FEPM-Elastomers
6. FFKM-Elastomers
7. FKM-Elastomers

This general information will be extensively explained below. It is particularly important to note that effects based on variability in formulation and manufacturing methods prevent universally applicable statements. Therefore, for the definition of the technological suitability of O-rings for a given application, specifications regarding the formulation are essential to assure resistance to hot water and heat aging as well as an adequate degree of vulcanization of the O-rings. Both these influential variables, namely the formulation and degree of vulcanization, represent in the practical application important variables that have a defining effect on the service life of O-rings but are often underrated by users.

NBR O-rings are not mentioned here because they have been rated as unsatisfactory for long time service in warm water.

In the following sections only above-mentioned material groups will be ranked with regard to their resistance to hot water and important influential variables based on the specific compound will be pointed out.

EPDM /EPM Materials

No other elastomeric material has a higher potential with regard to hot water resistance than EPDM and EPM materials (see Figure 5).

The evaluated EPDM material „EPDM-1“ shows a superior behaviour even when compared to a tested FFKM material that was already optimized for hot water resistance. The degree of chemical change in both the polymer and network structures of a rubber material is best detected by the change in elongation at break and tensile strength of the material from its original state to that after storage in hot water for 200°C as shown. A very small change indicates a high degree of chemical stability, on the other hand, a large relative change indicates a strong impairment of the chemical structure of the material what becomes at first noticeable on an O-ring as a high permanent set.

Water Resistance 504h/200°C Comparison of different Materials

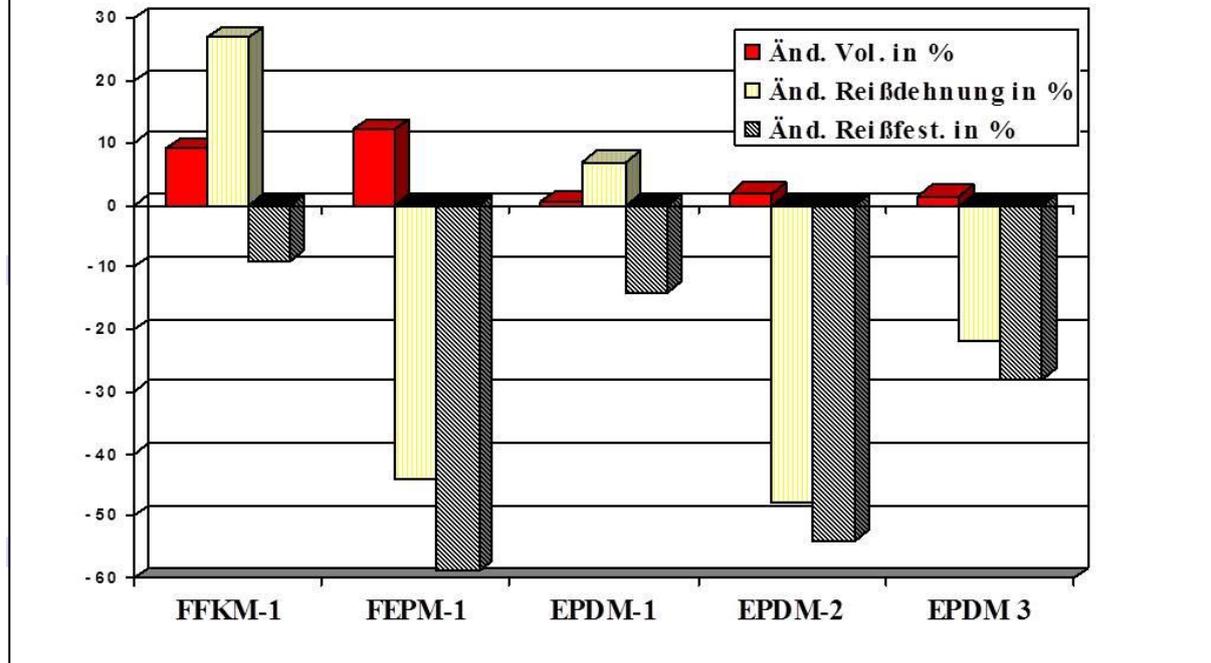


Figure 5: Comparison of hot water resistance of selected compounds at 200°C

(Translation key:

- Änd. Vol. .% = Volume change, %
- Änd. Reißdehnung in % = Change of elongation at break, %
- Änd. Reißfest., in% = Change of tensile strength, %)

Good EPDM materials can be used in hot water up to 200°C without any problems as long as free access of oxygen is prevented or at least greatly restricted. Extended time tests in water in an autoclave more than 2000 hours at 200°C on a temperature sensor, which was sealed by an EPDM O-ring, have shown that these O-rings were still reusable without any restriction after the test. In contrast comparable EPDM O-rings used as an autoclave seal show already after 1000 hours at 200°C a high permanent set and on the air side signs of a polymer degradation that already occurred; the surface of the O-ring was blackened with carbon black on the air side and the O-ring was considerably embrittled while on the water side it still exhibited a rubbery elasticity. Thus the use of EPDM materials is practically not limited by water resistance but by its relatively low resistance to hot air. Where besides watery compounds mineral oil based media can come in contact with the O-ring, the use of EPDM is out of question as seal material and polymers resistant to mineral oil must be used.

Differences in EPDM O-Rings

In no other elastomer family is the degree of freedom with regard to formulation design as great as in EPDM materials: The polymer architecture is of great importance (ethylene content from 40 to 90% and diene content from 0 to 8% are possible), the cross-linking system (sulfur or peroxide) and the plasticizer content (0 to over 30 is possible), the addition of antioxidants and antidegradants can increase their heat resistance markedly. Already two simple short-term tests on EPDM O-rings, namely the compression test after 24 hours at 150°C and the increase of hardness after heat aging for 70 hours at 150°C can reveal significant differences in EPDM O-rings. Thus optimally vulcanized O-rings made from a good quality formulation exhibit compression set values under 20%, also values up to 30% can still be considered as being good. Values between 55% and 100% as measured on commercial O-rings are considered either unsatisfactory or as not suitable for demanding O-ring applications. In terms of increase in hardness after the effect of heat (70 hours at 150°C) good EPDM O-rings show changes less than 6 points of hardness, poor O-rings in some cases show changes greater than 20 points of hardness with the initial hardness of 70 Shore A.

The most important components of a formulation can be determined, at least approximately, by thermogravimetric analysis. In that procedure a sample of the material (about 10 mg) of the analyzed O-ring is heated continuously up to the maximum temperature of 1000°C while measuring the relative weight loss with the increase in temperature. The evaluation of the curve allows the quantitative determination of the mix as vaporisable components (predominantly plasticizers), pyrolisable components (predominantly polymers), oxidisable components (predominantly carbon black) and nonoxidisable components, also referred to as ash residue.

In the case of typical static O-ring seals we can assume that they maintain their sealing function until the O-ring loses its initial load completely. This point can be illustrated in a laboratory on O-rings by attaining the value of a compression set of 100 %. In order to be able to draw conclusions from short-term tests as shown for example above and from formulation design, as easily determined by TGA analysis of O-rings, on the long-term compression set, 5 different positions of EPDM O-rings from different manufacturers were more thoroughly examined. Table 1 shows the found properties after different short-term tests. Table 2 shows the differences as found by means of the thermogravimetric analysis.

Formulation Code	A	B	C	D	E
Hardness, IRHD	81	67	77	75	74
Compression set					
24h/100°C, [%]	5,6	-	18,1	22,0	25,9
24h/125°C, [%]	7,2	12,4	33,7	39,9	47
24h/150°C, [%]	12,1	21,9	61,5	60,8	72,8
Circulating Air 70h/150°C					
Hardness Change, IRHD	+/-0	+14	+8	12	13

Table 1: Short-term test results on 5 positions of different EPDM O-rings (cross section $d_2=3.53$ mm)

Formulation Code	A	B	C	D	E
Vaporisable component (residues of monomers processing aids and plasticisers) [wt. %]	1,9	8,2	16,5	14,1	16,0
Pyrolysable components (mainly polymers), [wt. %]	61,7	45,6	44,6	44,0	41,7
Sum of organic components, total [wt. %]	63,6	53,8	61,1	58,1	57,7
Oxidisable components, ($T>650^\circ\text{C}$, O_2)	31,4	40,5	36,5	39,3	39,6
Nonoxidisable components, wt. %	5,0	5,7	2,3	2,5	2,7

Table 2: Quantitative determination of the main components of the O-rings analyzed by thermogravimetry (TGA)

The results from the long-term behaviour show a very strong difference between both peroxidically cross-linked O-rings (A+B) and the three cross-linked by sulphur (C+D+E). At 125°C in air these differences are expected to give at least a 10-fold greater service life of the O-rings made from the Formulation Code A versus the EPDM O-rings from the Formulation Code E. The order of the results from the short-term measurements of compression set is in a good agreement with the order of the long-term measurements. Both peroxidically cross-linked EPDM O-rings show in fact good short-term compression set values at 150°C, however Formulation B shows a distinctly higher proportion of low boiling components (see Table 2, vaporisable components) and probably a higher diene content in comparison with Formulation A (strong increase of hardness after 70 hours at 150°C) in the polymer, and this has a negative effect on the long-term behaviour.

The sulphur cross-linked formulations reveal consistently considerable proportions of plasticizers, and this, along with the cross-linking with sulphur, has a negative effect on the long-term behaviour. However, there are definitely also EPDM 70 O-rings, which in this case

contain still a considerably higher proportion of plasticizers and therefore can be even worse than Formulation E.

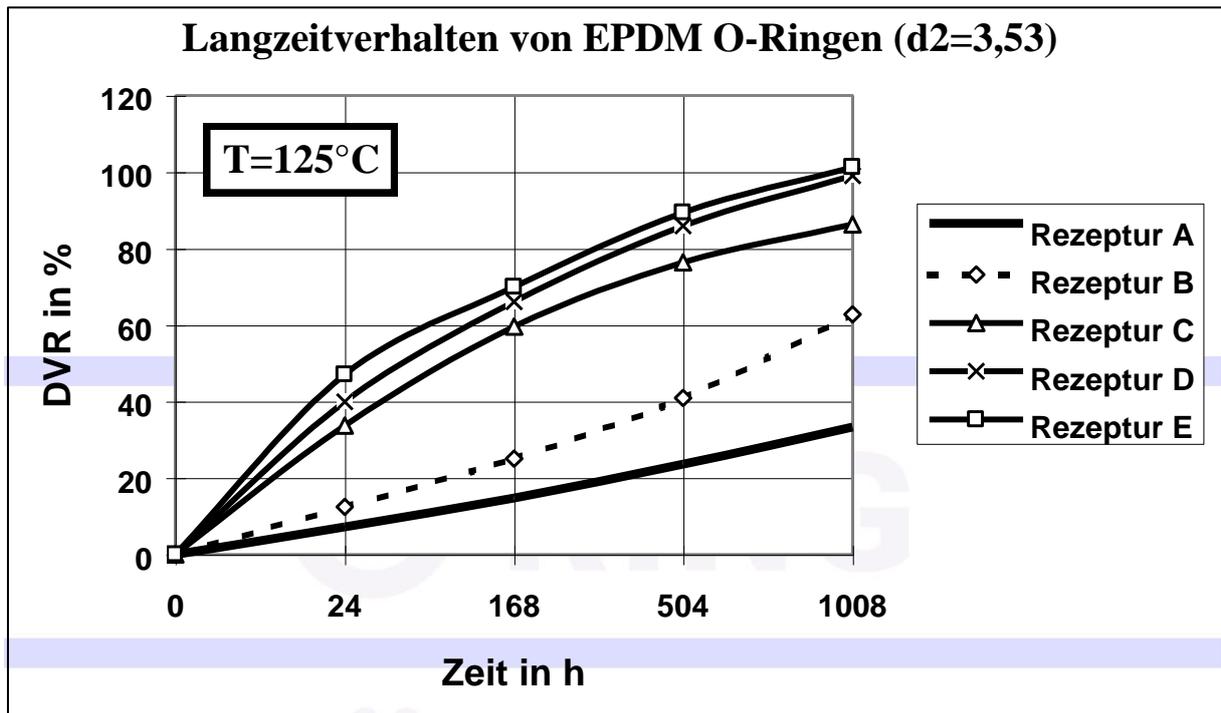


Figure 6: Long-term behavior of compression set of 5 tested EPDM O-ring positions
(Translation key:

- Langzeitverhalten von EPDM O-Ringen = Long-term behaviour of EPDM O-rings
- DVR = Compression set (CS)
- Zeit = Time
- Rezeptur = Formulation)

Process-Related Factors

For all elastomeric seals vulcanization conditions are very important for the achievement of a good degree of cross-linking. The high cost pressure on O-rings, particularly in the mass production, leads to an extremely tight curing time in the mold. In some cases the vulcanization degree of partially cross-linked O-rings can be still significantly improved by post-curing. This is practically successful in the case of FKM O-rings; also sulphur cross-linked EPDM O-rings can be improved considerably by relatively low temperature post-curing. On the other hand, peroxidically cross-linked EPDM O-rings require higher temperatures for the post-curing. However, in this case there are limits imposed by the temperature resistance of the O-rings. Therefore for the peroxidically cross-linked EPDM O-rings considerably longer curing times in the mold are required than those for sulphur cross-linked O-rings. In addition, the sensitivity to temperature fluctuation in the mold of the former is higher.

Therefore the good quality of the formulation (i.e. values shown in the Data Sheet) alone is not a guarantee for good EPDM O-rings. For that, it is necessary to provide guidelines for the maximum acceptable compression set values from production O-rings to the supplier. This is already nowadays implemented by many sensitive endusers.

Proposal for a Purchase Specification for EPDM O-Rings

As explained above, the quality of the service life of O-rings results from the quality of the formulation and the quality of the manufacturing process and can be represented as a product (see Figure 7). The most important influencing factors on the quality are the polymer alone, the content of plasticizer (as small as possible) and the cross-linking system. The most important production-related influencing factors on the material properties are the vulcanization (cross-linking process) and post-curing conditions. The verification of the formulation quality is performed usually only once, namely during the first article inspection, but for that purpose it should include the complete spectrum of important properties. Besides the basic physical properties (e.g. hardness, specific gravity, possibly also tensile strength and elongation at break) the high temperature behaviour (permanent set and heat aging), the behaviour at low temperatures (e.g. compression set) and the swelling in reference media (e.g. acetone + redrying) should be tested. After the formulation is tested once on production O-rings and validated still the typical identification characteristics of the formulation should be defined.

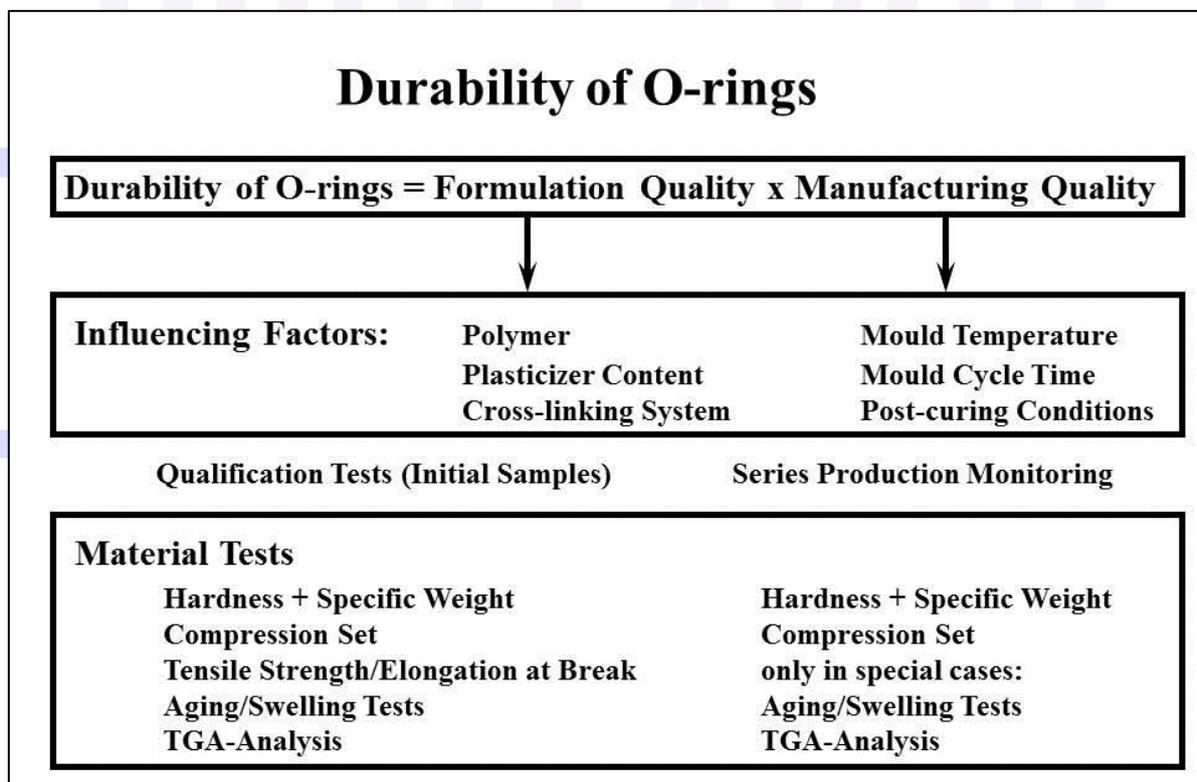


Figure 7 describes the most important material tests for the definition of important material properties.

This can be done with the hardness and the specific gravity, but with the TGA analysis (see above) the formulation can be characterized considerably better.

For inspection of production deliveries, that is, for typical incoming goods testing, as a rule, it is enough when the formulation is once roughly identified (hardness and specific gravity), and when a satisfactory degree of vulcanization is also proven by the measurement of compression set. Regardless of the above, naturally, the adherence to dimension tolerances and to the demanded surface finish of the O-ring should be monitored (see above).

From that it is possible to derive simple but effective order specifications regarding the material properties of O-rings. Table 3 gives an example how this can look like for peroxidically cross-linked EPDM O-rings.

Material Testing	EPDM peroxide cross-linked
Hardness DIN ISO 48 M, IRHD, referred to the nominal value in the formulation	+/- 5
Specific weight, DIN 53 479, permissible deviation from average value specific to the formulation, [g/cm ³]	+/-0,02
Compression set 24h/150°C, DIN ISO 815-1, [%] Compression 25 %	30 max.
Compression set 24h/150°C, DIN ISO 815-1, [%] Compression 25 %, cooling down to 23°C in compressed state	50 max.
Immersion in acetone, DIN ISO 1817, 24h/23 °C	
Volume change, [%]	0 to 10
Subsequent redrying 22h/100 °C	
Volume change, [%]	-3 max.
Hardness change, [IRHD]	+6 max.
Circulating air, 70h/150 °C, DIN 53 508	
Volume change, [%]	-3 max.
Weight change, [%]	-3 max.
Hardness change, [IRHD]	+6 max.
TGA analysis, ISO 11358-1	
Maximum proportion of vaporisable components, [%]	5 max.
Maximum deviation from initial samples regarding vaporisable components, pyrolisable components (polymer) und oxidisable components (carbon black), [wt. %]	+/- 2 max.
Maximum permissible deviation from initial samples regarding the inorganic residue, [wt. %]	+/- 1,5 max

Table 3: Proposal for a finished part related order specification (material requirements) for peroxidically cross-linked, unplasticized EPDM O-rings from the upper performance spectrum.

Depending on the requirement specification of the application it can naturally appear as meaningful to reduce the quality of the O-rings for cost reasons. However this should be done in a controlled manner by widening the individual proposed specification limits. Particular requirements in the application can equally make amendments necessary, for example, for application in hot water at temperatures over 150°C, the resistance to hot water should be ensured by an experiment with an adequate immersion test in hot water. However, just to assure to good state of the art for general industrial application, it is recommended to apply ISO 3601-5 (2015) EPDM P 70 or EPDM P80, where as well O-ring requirements (compression set, hardness) and recipe-requirements are defined.

Butyl O-Rings (IIR/CIIR/BIIR)

Butyl rubber is also good for applications using hot water. However, the unsaturated main chain in the polymer results in reduced performance potential of this elastomer regarding resistance to aging in comparison with that of EPDM rubber. Moreover, butyl rubber has a distinctly higher swelling in water than EPDM and the processability on the other hand is more difficult. Therefore butyl rubber O-rings (only special formulations) are used in hot water applications only occasionally.

HNBR-Elastomers

HNBR (hydrogenated nitrile rubber) is sometimes referred to as oil resistant EPDM because of its good resistance to hot water, however this is true as related to the middle performance spectrum of EPDM elastomers and not to their highest performance potential. Beyond that, this is not correct with regard to the presence of many possible additives in water (e.g. corrosion inhibitors) or regarding the behaviour at low temperatures. Moreover, the entire range of offered HNBR elastomers reaches from a partially hydrogenated grade with 80% saturation, which can be as usually cross-linked by sulphur, all the way to a fully hydrogenated grade with saturation higher than 99%, which can be cross-linked only by peroxide because of its very low number of double bonds. Between these, there is a wide spectrum of grades as to their resistance to aging. It should be also noted that fully hydrogenated HNBR elastomers alone are true alternatives to EPDM elastomers but cost several times more than these. Therefore HNBR elastomers remain as the main choice for the use in pumps or fittings where oil resistance is required and where the requirements for heat resistance still allow their use. Currently, this is predominantly the case for cooling water applications in the automotive industry (in utility vehicles). Where a higher resistance to aging is required, fluorinated elastomers (FEPDM, FKM, and FFKM) are the proper choice.

Silicone-Elastomers (VMQ)

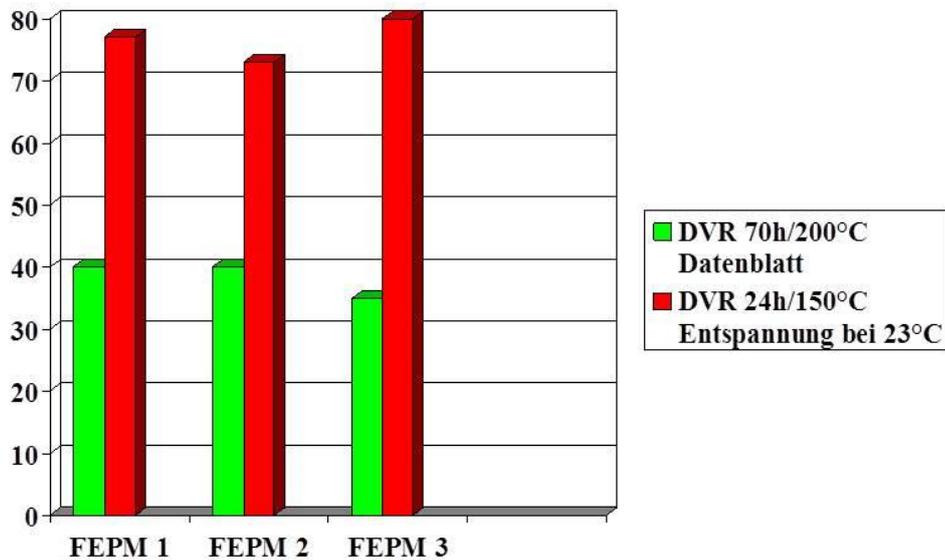
Silicone elastomers have the advantage of a very good resistance to hot air combined with an excellent flexibility at very low temperatures and a considerably lower polymer price in comparison to fluoroelastomers. They can be easily coloured and are also available in hardness values 60 or 50 Shore A and even below that, without sacrificing their heat resistance; this cannot be said about EPDM materials. On the other hand, the water resistance of silicone elastomers is often overrated. The effects of hydrolysis become apparent fastest in compression set tests in hot water and hot water/Glycol mixtures. Thus, for example, in a comparison test on O-rings after 500 hours at 125°C in a water/Glycol mixture even the silicone O-rings specially optimized for hot water use had a compression set of 60-70 % and the O-rings were strongly softened (typical signs of occurred polymer degradation) after the test, while the EPDM O-rings tested parallel yielded a value of 15% without any signs of occurred polymer degradation. Therefore, the use of silicone O-rings in hot water is limited mainly to the continuous temperature range up to 100°C.

FPM Elastomers (Aflas)

Aflas polymers contain monomers from EPDM (propylene) and FKM (tetrafluoroethylene) and have the advantage that the benefits of EPDM materials, namely a good resistance to hot water, and the benefits of FKM materials, namely a good heat resistance (in circulating air), are united into a greater part. One drawback of Aflas materials is their poor resistance to low temperatures, which after a high-temperature service and a subsequent cooling down to room temperature in deformed state leads to a high permanent set, see Figure 8 „CS values after 24h/150°C relaxation at 23°C.“ However, this permanent deformation is to a high degree reversible by exposure to heat, see Figure 8 „CS Values 70h/230°C Datasheet“. However, this property can be problematic in all applications with frequent changes of temperature particularly if the temperatures drop under room temperature. A changeover to better low temperature resistant types (bisphenol cross-linked type MZ) is possible only on the expense of the resistance to hot water. An additional drawback of these materials in comparison to FKM materials is their considerably worse processability and this greatly limits the availability of Aflas O-rings on the market. However, what ultimately makes these materials interesting for the user is that in comparison with FFKM O-rings they are considerably less expensive and in comparison with peroxidically cross-linked FKM elastomers they have better water resistance. Thus the main application field of these O-rings is there where even good EPDM materials are no longer usable and FFKM elastomers simply not affordable.

Indeed, the resistance of these materials to hot water is not as good as that of the best EPDM elastomers, (see Figure 5 above), and this is also proven by the damage patterns in Figure 3. Table 4 shows a comparison of an Aflas O-ring from the upper performance spectrum with a standard FKM O-ring (no special hot water resistant grade).

FEPM-Materials in Comparison Compression Set Behaviour



Result: Consistently high set after cooling down to 23°C

Figure 8: Set behaviour of FEPM O-rings, relaxed once cold (23°C) and once warm (200°C)
(Translation key:

- DVR Datenblatt = CS Data sheet
- DVR Entspannung bei 23°C = CS relaxation at 23°C)

Comparison of FEPM/FKM(Standard) at high temperatures and in steam/hot water

Conditions	FEPM-O-Ring	FKM-O-Ring
70h/230°C in air	36 %	69 %
168h/220°C in air	50 %	73 %
70h/110°C in water	36 %	48 %
70h/230°C in steam	43 %	> 100%

The shown values pertain to certain formulations and cannot be generalised.

Table 4: Comparison of an FEPM O-ring from the upper performance spectrum with a standard FKM O-ring (not a hot water resistant grade)

FFKM Materials

The very high price of FFKM materials is a criterion for exclusion for many hot water applications. Once this acceptance limit is overcome, there are still additional obstacles to reach a good hot water sealability: On the one hand the use of only special hot water resistant FFKM formulations makes sense (in no case should be used a FFKM material cross-linked by triazine), as shown in Figure 9, on the other hand these O-rings are considerably more sensitive with regard to the formation of stress cracks due to higher temperatures and high compressions, as shown in Figure 10. Therefore for these O-rings particularly for those with cross sections from 3,5 mm reduced compression values should be provided, which means that the installation spaces of the O-rings may need to be modified somewhat. Generally, for O-rings a compression target value between 30% (for nominal cross section 1,78 mm) and 20% (for nominal cross section 6,99 mm) is considered adequate, this should be reduced somewhat for the use of FFKM O-rings (by about 10 percentage points to 20 or 10% respectively)

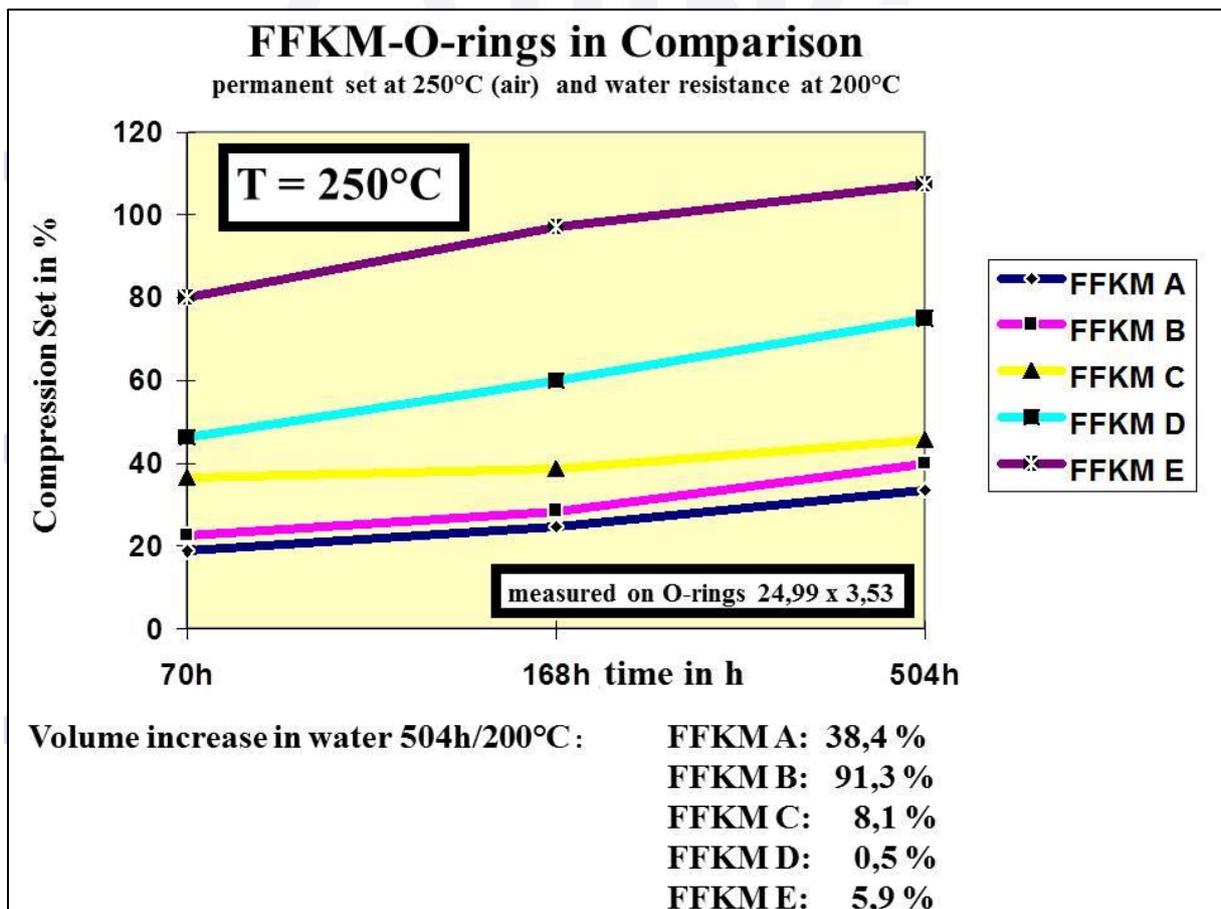


Figure 9: Resistance of different FFKM formulations in hot water (volume changes) or in air (compression set values)

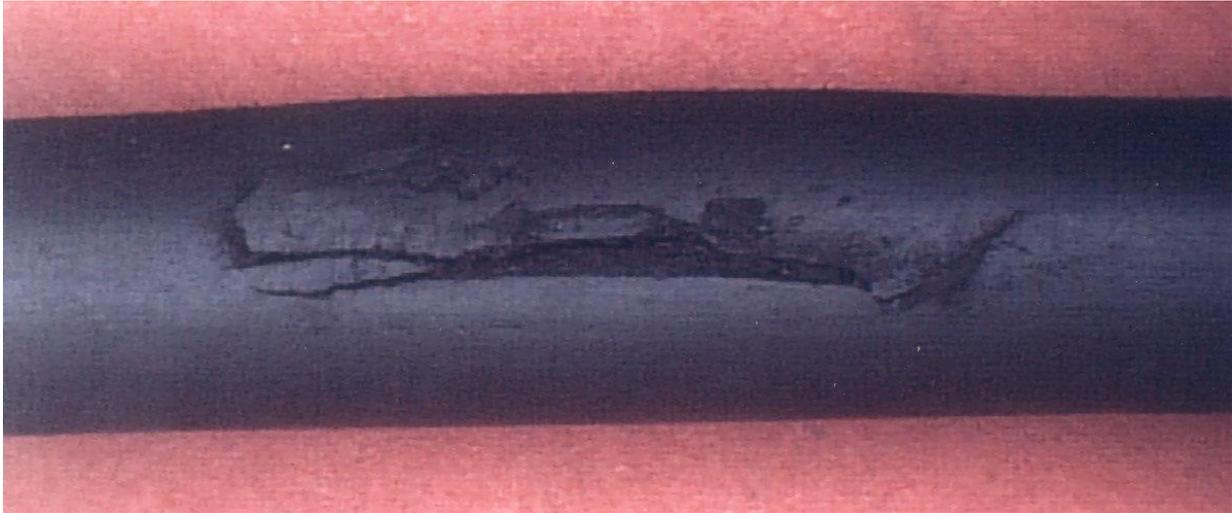


Figure 10: FFKM O-ring after service in hot water at 200°C, split due to a too high compression

FKM Materials

O-rings made from FKM elastomers without any particular specifications are usually dipolymers cross-linked by bisphenol which stand out due to their low compression set values. This is true only for their behaviour in air. In water such O-rings will be strongly deformed permanently already after about 1-3 weeks at temperatures of 150°C. Therefore when using FKM O-rings in warm and hot water it is important to make sure that special formulations are used, that is, at least such that are cross-linked by peroxide. Even then, the use of FKM elastomers in hot water and steam is not recommended for temperatures above 150°C, when there is no adequate evidence of demanded service life backed up by test results.

Higher Leakage Risk in Cyclical Mode of Operation

In order to be able to assess the long-term sealing behaviour of O-ring seals it is necessary to observe not only the chemical changes in the O-ring caused by effects of temperature and medium but also the physical effects on the sealing location.

A consistent continuous temperature in comparison to a cyclical operation mode involves a markedly stronger chemical change in an O-ring, which means that the stress relaxation of the O-ring is considerably stronger than in the cyclical temperature service conditions since the reaction rate of chemical processes increases exponentially with temperature. This means that an O-ring subjected to a cyclic operation loses sealing force slower than an O-ring in an isothermal operation. However, this does not mean that because of this an O-ring in cyclic operation always seals for a longer period, since in addition to that the functional behaviour depends on the change of gap on the sealed area. Then in the operation mode at nearly constant temperature the O-ring will be minimally stressed in terms of recovery. This is because usually in a steady state and stationary service practically no changes of gap

occur, moreover, the O-ring is additionally activated by its thermal expansion. In other words, the ring will thus be used under ideal boundary conditions. As long as the O-ring does not lose volume due to the loss of plasticizers (this is why they should not be used in a formulation, if at all possible) it will seal even when the stress relaxation is well advanced. The reason: No gap, no leakage. It is quite different in a cyclical temperature operation mode: Masses und coefficients of thermal expansion of the components to be sealed cause that with each reduction of temperature on radially sealing O-rings a gap is formed that the O-ring has to span. In addition to that the O-ring suffers a considerable thermally induced shrinkage. In unfavourable cases the material can beyond that, already at room temperature, also lose sealing force due to a beginning "freeze". Consequently, the O-ring will be highly loaded as to its recovery behaviour at each drop of temperature, so that a leakage can occur even without the O-ring having to be relaxed to 100%. Practically, this means that a strong cyclical operation mode demands much more from the O-ring material than the isothermal mode. Therefore cyclical temperature service conditions, particularly when the lower temperature limit is at room temperature or below, create more possibility of leakage.

Effect of the Design of the O-ring Housing

By a clever design of O-ring installation spaces it is possible to greatly increase the time until an O-ring being used at extreme temperatures finally fails. Essentially, the O-ring has to be always sufficiently deformed (>10-15%), also involving the unfavourable tolerance positions, so that the used elastomeric material can also retrieve the available elastic recovery potential. Beyond that, further improvements can be achieved by the following additional hints mentioned below:

1. Axially sealing installation spaces should be preferred. The degree of deformation of axially deformed O-rings depends only on one tolerance (assuming a flat cover) while a radial groove results ultimately by the interaction of 3 dimensions. With this the degree of deformation of the O-ring in the flange groove can be better controlled. In addition to that, temperature fluctuations do not affect the depth of the groove as it is in the case of the radial groove. Notably in hot water applications where EPDM O-rings are used as seals, with an axial groove the air access can be better limited than with a radial groove. EPDM O-rings can have an outstanding hot water and steam resistance, however, ultimately the service life is limited by an oxidative attack on the air side. Limiting this effect means increasing the durability of the seal to the same extent.
2. When sealing radially, it should be done with minimal diametric play. If it is necessary for constructional reasons to provide radial O-ring grooves, then the size of the radial clearance of the components has to be sealed tightly, is important for two reasons: the smaller the sealing gap is, the less the degree of deformation of the O-ring varies (high deformation = good, very small deformation = poor), and the smaller the sealing gap is, the more strongly the amount of air is reduced. Air reduces the service life of EPDM O-rings considerably.
3. In radial seals the use of a double O-ring seal can be of advantage. This is true particularly about EPDM seals, since their hot steam and air resistance are so greatly

different. The first EPDM O-ring on the air side is, so to speak the „sacrificial ring“, which should keep off the air as long as possible from the second EPDM O-Ring. This set-up cannot be recommended in cases, where strong pressure surges can occur and as a result pressure can build up between these two O-rings.

4. The use of large cross sections, if at all possible, is recommended. "A lot helps a lot!" This also applies to the use of O-rings. The larger the cross section, the smaller is the ratio of the free surface to the mass of the O-ring. With that it is possible to extend its service life especially at very high temperatures.
5. Apply a large amount of grease. Because of the susceptibility of EPDM O-rings to aging by heat and oxygen, it makes sense to prevent the access of air by applying liberal amounts of grease. Of course, it is important to make sure that the grease does not affect the O-ring and continues to remain neutral. This is particularly important here, because EPDM has a high sensitivity to swelling by all derivatives of mineral oil and chemically similar materials.

O RING

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RICHTER