EXPERT KNOWLEDGE

O-RINGS

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O RING PRÜFLABOR RICHTER

TESTING CONSULTING DEVELOPING

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Quality Assurance on O-Rings - How Is It Possible?

Important Information on the Theory and Practice of Hardness Test and Compression Set on O-Rings
(Short Version)

On the website of the O-Ring Prüflabor Richter there is also a long version of this article, which gives more detailed background information about the test methods and possibilities to influence the test result.

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For the user of O-rings, the question arises as to how he or she can check and ensure the quality of these inconspicuous sealing elements with reasonable effort.
With ISO 3601-5 (2015-04-01), for the first time an internationally valid standard is available to the user, which specifies material properties for various elastomer materials, both on test plates and - this is the special aspect - on the finished product O-ring, which correspond to a good state of the art.

1. What is the Typical Testing Practice?

Many companies carry out a simplified identity test, which includes density testing (protection against possible material confusion) and hardness testing.

In addition to compression set tests, tensile tests or short-term storage and swelling of O-rings can also be useful as qualification tests.
Where available, FTIR analyses and/or thermogravimetric analyses are also used to identify the formulation.

2. What Do Important Standards State About O-Ring Testing?

Usually test standards with test specifications refer to standard specimens which are manufactured from test plates in order to determine material characteristics as reproducibly as possible. Every seal manufacturer ensures the highest quality in the manufacture of test plates so that the best compound properties can be measured. However, finished parts are vulcanized differently and their degree of cross-linking is not defined or guaranteed by data sheets.

The standardization in the "O-ring country" USA was groundbreaking for the finished part testing of O-rings. The first edition of **ASTM D 1414** (current edition 2015) was published as early as 1956. This standard describes how the common test methods are to be performed on O-rings but does not specify target values.

ISO 3601-5 (2015-04) (Suitability of elastomeric materials for industrial applications) does not focus on test methods, but on materials for O-rings and their suitability for industrial applications. The test methods used to check the nominal values required in this standard refer to ASTM D 1414 described above and other standards such as ISO 48 (hardness) or ISO 815-1 (compression set).

3. Possible Causes for Poor and/or Fluctuating Qualities of O-Rings

Depending on the polymer type, different crosslinking systems can be used, which demand different process control requirements in production. For example, fluctuations in the degree of crosslinking of sulfur-crosslinked materials (e.g. NBR, EPDM, CR) can be compensated by subsequent tempering, whereas fluctuations in peroxidically crosslinked NBR, HNBR and EPDM materials cannot be compensated or can only be compensated to a limited extent because they are not sufficiently temperature-resistant for effective post-curing by tempering.
There are also **NBR** grades in the hardness range of 70 ShA with up to 20% plasticizers by weight. Such types tend to shrink during application, as the plasticizer can be washed out by hydraulic or paraffinic oil, for example.
While NBR elastomers in O-rings are predominantly sulfur-crosslinked, **EPDM** materials are predominantly characterized by a peroxide crosslinking system, since sulfur crosslinking considerably limits the service temperature of EPDM O-rings. Since EPDM can be swell with mineral plasticizers, (and even up to 50% oil-stretched EPDM polymer types can be obtained) the finished compound of such a polymer can contain up to 30% by weight of plasticizer at 70 ShA. Such compounds show a high long-term settling behavior (recognizable by compression set) and they can also shrink as the oil can be dissolved out by silicone oil (e.g. in sanitary applications) or hot water and evaporate due to heat. In addition, a compound with a high plasticizer content in combination with peroxides is more difficult to vulcanize since the effect of the peroxides is partially neutralized by plasticizers. Furthermore, with peroxide crosslinked

EPDM, an insufficient degree of curing, as with NBR, cannot be improved by post-tempering, see above. Vulcanization must therefore take place completely in the mold during processing. A reduction in the cycle time for economic reasons can result in a high loss of quality. Inadequate cross-linking can be proven very well with a compression set test. The primary purpose of compression set tests on O-rings is therefore not to identify the material properties of the formulation but to assess the manufacturing process of the supplier. Helpful guide values for O-rings made from peroxide cross-linked EPDM can be found in the requirements of ISO 3601-5, which also differentiates between the cross-linking systems in its target value specifications.
Fluororubbers **(FKM)** are mostly cross-linked bisphenolically. With regard to the degree of crosslinking, there are hardly any problems here. Moreover, the formulation of FKM compounds is much simpler (e.g. no use of plasticizers), so that the application of this material is much less critical than with EPDM. However, it should be noted that under high physical stresses, for example in the form of high pressures or abrasive stress and also in the case of long-term behavior at high temperatures, differences due to formulation and processing may very well be important.
With silicones **(VMQ)** there are often quality problems due to hardness fluctuations. These can be caused by post-processing O-rings (Mullins effect). Here it should be checked if this can have a negative effect on the application. Apart from this, VMQ materials are less critical elastomers for O-rings.

4. Hardness Test on O-Rings

The Shore A hardness as a material parameter refers to test plates, preferably with a thickness of 6 mm. If, on the other hand, the hardness is to be defined directly on O-rings, an additional test requirement or nominal value specification is required. Experience has shown that for O-rings with a cord thickness of approx. 3 mm or more, the material characteristic value of the formulation can be used as the nominal value (+/-5). Common for O-rings, however, is the IRHD microhardness according to ISO 48 (sub-process **CM** ("**C**urved surface"), "**M**icro" (indenter)). This hardness testing method is adapted to the Shore A method in such a way that the nominal value of the hardness of the material for O-rings from a cord thickness of 1.6 mm can also be used as the IRHD-CM nominal value (+/-5). For smaller cord thicknesses, it is common practice (ISO 3601-5) to extend the nominal value tolerance range to +5/-8 IRHD-CM degrees of hardness. However, hardness testing alone is often overrated. Conclusions are ascribed to it which it cannot or only insufficiently achieve: For example, hardness measurement can only give vague information about the degree of cross-linking of a gasket.

4.1 Useful Influencing Factors on the Hardness Testing of O-Rings

In the IRHD microhardness test, a ball (= indenter) with a diameter of 0.4 mm and a defined weight penetrates into the specimen. The penetration depth is a measure of the hardness. The indenter should contact the highest point of the O-ring cross-section. Due to the curved surface, the indenter ball can penetrate more easily than a flat or concave surface because it has to displace less material. Since a large penetration depth indicates a soft material, O-rings appear to be less hard than comparable test plates.

The problem is also addressed by DIN ISO 48, which introduces its own procedure designations for the measurement of curved surfaces and speaks of "apparent hardness". The CM sub-process is generally used for O-rings. For this reason, it is very important to determine with the supplier not only a hardness value for the test plates, but also for the finished O-rings. In order to measure exactly and reproducibly at the uppermost point of the O-ring cross-section, a centering device, or even better, a laser-guided table (see **Fig. 1 and 2**) should be used, especially for small cord thicknesses (e.g. < 2.62 mm).

Fig. 1: Stationary IRHD micro-tester with laser-guided table for exact hardness measurement of O-rings (CM method)

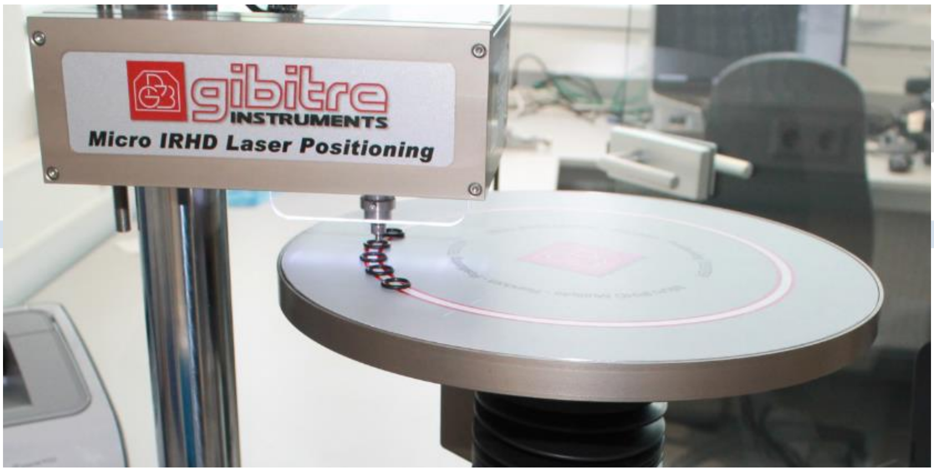

Fig. 2: Laser-guided rotating table for automated IRHD-CM measurement of a larger number of O-rings

Hardness measurement on O-rings is recommended but should be combined with other methods such as density and DVR testing. This is the only way to obtain reliable information on the formulation identification and quality of an O-ring.

5. Compression Set Test on O-Rings

In the compression set test, an elastomer component is usually compressed by 25% and then stored at an elevated temperature. Typical test times are 24^{-2} and 72^{-2} hours. After removal from the oven and removal of the compression, the height difference of the component is determined from the measurement before and after the test and related to the deformation height. The more a gasket has "settled", the higher and worse the compression set result [%] will be.
ISO 3601-5 requires ISO 815-1 (Method A) as the test method to be used.

Fig.3: Compression set test tools with base plate ground to different depths (see lower plate), the use of spacers is not necessary.

Fig. 4: Plane plates for compression set measurement with spacers: there are three O-ring sections on the base plate.

"The greatest practical significance of compression set testing can be found in the testing of finished parts, especially O-rings (see **Fig. 4**). The purpose here is not to determine the formulation-specific characteristic value as found in material data sheets, but to be able to provide information about the degree of vulcanization of the finished part."[1]

[1] BLOBNER, U. und RICHTER, B.: Druckverformungsrestprüfung (DVR-Prüfung): - Prüftechnische Grundlagen und Empfehlungen für die praktische Anwendung, Internetveröffentlichung, 06/2015, S.4 (http://www.o-ring-prueflabor.de/files/fachwissen_druckverformungsrestpruefung_06_2015.pdf)

5.1 Useful Factors Influencing Compression Set Testing of O-Rings [2]

Depending on the test temperature and the time, the cord thickness of O-rings can have a considerable influence on the result. Thousands of compression set tests carried out in the O-Ring Prüflabor Richter have shown that at 24h test time and at an appropriate test temperature (= permissible continuous temperature) the influence of the cord thickness is no greater than other random influences (measurement uncertainty during height measurement, planarity of the test tool plates (see **Fig. 3**), temperature and time fluctuations). Other important influences besides the test temperature and the test time are of course the degree of deformation (e.g. 25%, 10% or 40%) and the type of cool-down (in the relaxed state = method A or in the still deformed state = method B).

6. Conclusion

Due to ever increasing quality requirements - also for the mass-produced O-ring - it is for seal users not only sensible, but also necessary to clearly define the quality of O-rings and to control it appropriately. ISO 3601-5 provides good assistance in this respect in terms of target values and test methods.

It is most likely the most frequently performed physical test methods in incoming goods are the measurement of density[3], hardness and compression set. These test methods are considered simple and generally known. The importance and significance of compression set testing as a finished part test, on the other hand, is usually underestimated. If these test methods are implemented correctly and consistently, the user can ensure consistent quality despite global and economical procurement. However, this does not release the user from the obligation to adequately verify the basic suitability of the formulation used and to check or qualify the supplier with regard to his processing and quality competence.

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[2] Ausführlichere Informationen zu den Einflussfaktoren: Ebd., S. 15-21

[3] Ausführliche Informationen zur Dichteprüfung: Siehe: BLOBNER, U. und RICHTER, B.: Identitätsprüfung: Übereinstimmungen finden; Internetveröffentlichung, 03/2014, (http://www.o-ring-prueflabor.de/files/fachwissen_identit__tspr__fung_03_2014.pdf)