EXPERT KNOWLEDGE OF TESTING METHODS FOR ELASTOMERS

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Testing of Compression Set (CS-Test)

Basics of Testing Techniques and Recommendations for Practical Application

Authors:
Dipl.-Ing. (FH) Ulrich Blobner,
Dipl.-Ing. Bernhard Richter

Applicable Test Standards:
ISO 815-1 (Issue 09-2014), ASTM D395 (Issue 2014)

Elastomers are not ideally elastic materials. For example, if a seal is deformed for a long time span, it will not return completely to its original shape after the applied deforming force is removed, If this deformation takes place with applied heat this effect is particularly distinct even if the temperature limit - typical for the given polymer - is not exceeded.
In the standard test of compression set an elastomer test piece with exactly defined dimensions is compressed to a predetermined percentage (usually 25%) in a special device and placed in the stressed state for a certain time (often 24 hours) to a laboratory oven. After the stress is removed (= release of the deforming force), the remaining height is measured and the compression set in percent is calculated. This article deals with the testing of compression set only at elevated temperatures, with the discussion and evaluation of results as well as their meaning for practical applications.

1. A short historical overview of the development and standardization of the test method for measuring compression set.

The determination of the compression set was standardized in Germany for the first time in December 1940 in the DIN 53 511 Blatt 3 Kreuzausgabe[1] ("*Prüfung von Gummi Elastisches Verhalten von Weichgummi gemessen nach Druckbeanspruchung mit bestimmten Größe der Zusammendrückung*" ("*Testing of elastic behavior of soft rubber by measuring its stressing by pressure of certain magnitude of compression").*

In the standard works written in German during the first half of the 20th century the expression "Compression Set" ("*Druckverformungsrest*") does not appear. However, methods similar to compression set measurement were investigated already in the 19th century although with mostly other issues than those existing in the compression set testing today.

Pioneering compression tests were performed and published already starting in 1871 by STÉVART[2]. He compressed rubber rings with rectangular cross section and with different diameter to height ratios. He varied these parameters and observed their effects on the cross-sectional geometry (bulging, buckling etc.). By that he could demonstrate the importance of the test piece dimensions in compression of a ring with a rectangular cross section. This discovery, of course applies also to current testing of the compression set and already for many decades there are officially required dimensions of test pieces. These dimensions are almost identical in ISO and ASTM standards so that the results from both of these standards are comparable.

In the German standard publication on testing of elastomers „*Der Kautschuk und seine Prüfung*"[3] ("*Rubber and Its Testing*") published in the year 1910 on pages 214 et seqq. an apparatus is shown for the measurement of the permanent set of ring test pieces. The extension was not brought about by a predetermined extension path but by using load weights. Whether the testing was performed also at elevated temperatures is not apparent in this literary source, although it is highly improbable because the subject of aging of rubber materials in hot air appears for the first time in the USA only about 1916.

In the "*Handbuch der Kautschukwissenschaft*" ("*The Science of Ruber", translated into English in 1934*) from 1930 there is a rather detailed description of so-called stretching tests, which show certain similarity with the today frequently used tensile set test[4], although without the influence of temperature.

As of „*Druckprobe*"("Pressure Test"), which can be seen as a kind of forerunner of the compression set test, it is discussed by MEMMLER and SCHOB: "The determination of the elastic behavior by measuring the deformation set after long duration of compression, analogous to the tension test, is seldom used in pure material research, but is specified now and then in tests for packing rings and similar soft rubber rings."[5]. From this citation it is obvious that

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[1] A *Kreuzausgabe* (=crossed edition) was the second version of a standard that was usually issued twice in the same year because of a small error. This practice was carried out until the end of the 1960s. By doing that it was possible to avoid the purchase of the new issue of the standard and write down the minute changes by hand.

[2] STÉVART in: Bull. Musée Ind. Belg. 59, 1871, pp. 5-15 and 63; 1873, pp. 5-15

[3] HINRICHSEN, F.W. and MEMMLER, K.: Der Kautschuk und seine Prüfung, Verlag von S.Hirzel, Leipzig, 1910

[4] The tensile set testing is in fact an independent standard testing procedure although it is related to the compression set test. On some finished parts the tensile set can be easier performed than the compression set and can therefore be used as an alternative
For that see also our expert technical article on tensile set on our website.
https://www.o-ring-prueflabor.de/files/fachwissen-zugversuch_10_2014.pdf

[5] MEMMLER, K.(Editor.): The Science of Rubber („Handbuch der Kautschukwissenschaft"), Reinhold Publishing

compression tests are used mainly for the determination of elastic properties but not of cross-linking properties.

In the „*Handbuch der Gesamten Kautschuktechnologie*" ("*Handbook of The Total Rubber Technology*") [6] published in 1935 under the subject of compression tests, only references to the above-mentioned sources and one reference to an English language citation can be found.

On the other hand, remarks and articles regarding this subject could be found much earlier in the American and British literature. Thus the American ASTM standard for compression set was published for the first time already in 1934[7] and consequently six years earlier than the corresponding DIN standard.

The fast and strongly growing automotive industry needed relatively early elastomer parts for damping vibrations. This circumstance was presumably the driving force for a greater in-depth search for a testing procedure suitable for the behavior of elastomers under pressure. To this day, for that reason, also in the USA, testing of compression set is done not only on a constant compression path but also and more frequently, as an alternative with a constant compression force (such as, for example, testing of engine mounts).

An extensive discussion of the compression set testing is that by ABBOTT[8] published already in 1930 but it paid particular attention to the compression by a constant load. The ground-breaking catalogue or checklist for the compression set testing has been answered and fulfilled in nearly all points by contemporary standards.

The understanding that the compression set test is also a simple and outstanding testing method for determination of the degree of cross-linking of an elastomeric part was not yet likely in the USA of the 1930s common knowledge. Thus CARPENTER wrote in 1937 only that "the high temperature increases the effect of plastic flow and gives some accelerated aging, which usually tends to reduce the power of recovery of the specimen".[9] It appears therefore that the idea of the possibility to examine the degree of cross-linking by testing the compression set has gained acceptance only later. Now back to the German DIN standardization cited at the beginning: The direct successor of DIN 53 511, Blatt 3 (Part 3) issued in December 1940, became in July 1960 DIN 53 517 („*Prüfung von Kautschuk und Gummi Bestimmung des Druck-Verformungsrestes*") ("*Testing of Raw Rubber and Vulcanized Rubber, Determination of Compression Set*"), which was then superseded in March 2000 by DIN ISO 815 („*Bestimmung des Druckverformungsrestes bei Umgebungs-, erhöhten oder niedrigen Temperaturen*") ("*Determination of Compression Set at Ambient, Elevated and Low Temperatures*").[10]

The current valid ISO and ASTM standards are introduced and commented on in a greater detail in Section 3.

Corporation, New York, 1934, p .564

[6] See: HAUSER, E.A.: Handbuch der gesamten Kautschuktechnologie, Union Deutsche Verlagsgesellschaft, Berlin, Volume 1, pp.125-126.

[7] ASTM International: Designation: D395 – 14 (Approved July 1, 2014): Standard Test Methods for Rubber Property – Compression Set, p.1, Footnote 1

[8] ABBOTT, Franz D.: The Testing of Automotive Rubber Parts Assembled under Compression, Part I – Deflection under Compression und Part II – Compression-Set and Some Special Tests in: Industrial and Engineering Chemistry – Analytical Edition, publ. by The American Chemical Society, Easton, PA., Issue 2., April15, 1930, pp.145-159

[9] CARPENTER, Arthur, W.: Physical Testing and Specifications in: DAVIS, Carroll, C. and BLAKE, John T. (Editors): The Chemistry and Technology of Rubber, Reinhold Publishing Corporation, New York, 1937, p. 807

[10] These pieces of information have been compiled with the help of the German National Library Leipzig, the Technical Information Library, Hannover and the DIN - Standards Committee for Materials Testing (NMP).

2. Meaning and Purpose of the Compression Set Testing

Compression set test is a relatively simple but also a diagnostically conclusive testing method that is performed for various purposes.

2.1 Comparative Evaluation of a Formulation

It should permit a comparative evaluation of a formulation (recipe), which means that compression set value represents as a measurable material characteristic the performance potential of the formulation in data sheets. For this purpose the testing times (22+2) h or (70+2)h according to ASTM D395B are often used. The test for this is performed on standard test pieces (usually the test piece B of the ISO 815 with the dimension approximately Ø13x6 mm). In many cases the found value of compression set may give indirectly the answer as to the cross-linking system used. Thus, for example, the peroxidically cross-linked EPDM elastomers exhibit significantly better results than those cross-linked by sulfur.

2.2 Testing of Finished Parts

The greatest practical importance of the testing of compression set pertains to the testing of finished products, in particular of the O-rings. It is not just about determining the formulation-specific characteristic value as shown in material data sheets but being able to provide a clear statement about the state of cure[11] of the finished part. However, the compression set gives barely sufficient statement about viscoelastic properties of the material. If the compression set value is not 10 to 30% higher than the formulation-specific characteristic value for (24-2) h (testing temperature = permissible 1000h-continuous temperature), it can be assumed that the state of cure is acceptable. Orientation values for a finished part (common industrial standard or good state-of-the-art) are shown in the Table 1, taken from ISO-Standard 3601 Part 5 (ISO 3601-5 (2015-04-01), Page 3, Table 2 “O-ring requirements“).[12]

[11] The compression set does not provide any statement about dynamic viscoelastic properties of the material but rather in the first place about the degree of cross-linking, which indirectly influences the viscoelastic properties under long-term static compression; for that, see:: RAHM, W.: Kurzzeitprüfverfahren für Gummi in der Qualitätssicherung in KGK Kautschuk Gummi Kunststoffe, Hüthig Verlag, Heidelberg, 48.Jahrgang, Nr. 9/1995, p..635

[12] In the ISO 3601-5 (Second Edition 2015-04-01 an error occurred during layout on the page 3 due to an erroneous shift in Table 2. In the second line the hardness values were assigned to the wrong base polymers. The correct assignments are as follows: To FKM, the hardness class (IRHD) 70, 75, 80, 90; to VMQ only 70; to EPDM-S 70 and 80; to ACM only 70. In the lines below no shift occurred, so that after correction and correct assignment the table can be used. The error will be corrected in the near future.

Base polymer		NBR				HNBR		FKM				VMQ	EPDM				ACM	Test method
Crosslinking system		Cross-linked with sulfur		Cross-linked with peroxide									Cross-linked with sulfur		Cross-linked with peroxide			
Hardness IRHD	°,CM	70	90	75	90	75	90	70	75	80	90	70	70	80	70	80	70	ISO 48 CM
Max. CS 24(+0/-2)h	%	35	35	30	30	40	50	25	25	25	30	35	30	35	30	30	40	ISO 815-1, method A
Testing temperature	°C	100	100	100	100	150	150	200	200	200	200	175	100	100	150	150	150	

Table 1: Excerpt from ISO 3601-5 (published 2015-04-01)

2.3 Verification of the Suitability for Application Technology

The testing of compression set should furnish the proof of the suitability of a material for the application technology. If the service life-temperature collective of the application is known, it is possible to determine the isothermal substitute service conditions (= chronologically shortened thermal service conditions at a constant temperature. This involves the use of simplified Arrhenius multiplicators (rule of thumb: 10 degrees Kelvin temperature increase = doubling of the reaction rate of aging). Then, when the finished part is tested using this isothermal substitute service conditions (time/temperature) it is possible to represent the application fairly close to reality by a laboratory experiment.

2.4 Determination of Propositions Regarding Long-term Behavior

At first sight it may appear that the test periods of compression set measurements between 6 and 18 weeks cannot provide any statement regarding the long-term behavior of seals over the entire lifetime of the product, which should provide sealing performance for years or in some cases even for decades. However, when the long-term behavior of the compression set is known at least at two and ideally three different temperatures and when the highest test temperature still allows undelayed aging (for example not delayed due to geometric effects, such as ratio of free surface and mass, as it is in case of an O-ring with large cross section-see Subsection 6.2.1), it is possible to construct straight lines for the assessment of the useful life.
A technical paper by Bernhard Richter on this subject can be accessed on our website at the following link[13]:
http://www.o-ring-prueflabor.de/download/Langzeitverhalten_von_O_Ringen.pdf

While the compression set testing describes how aging of the material affects its ability to recover, that is, how much the seal has set, or how much it can follow the change of the gap. There is also a possibility to describe the effect of aging on the deformation force. This is measured by compression stress relaxation; for this see also our pointers under this key word.

[13] This technical paper was published in a journal: RICHTER, Bernhard: "Lebensdauer von O-Ringen in: O+P „Ölhydraulik und Pneumatik", Vereinigte Fachverlage GmbH, Mainz, 42. Jahrgang, Nr.5/1998

This article deals with the compression set in hot air only. In a few cases, however, it is possible to obtain a more specific prognosis for a practical application if additional testing of compression set is done in liquid test medium, such as motor oil or transmission fluid and other). This is a new trend in newer standards especially in the vehicle manufacturing industry.

More information on this subject is in the advanced technical article by Bernhard Richter[14] available on our website:
http://www.o-ring-prueflabor.de/de/fachaufsaetze/bewertung-der-ergebnisse-von-bestaendigkeits-und-alterungspruefungen/

3. Important Currently Valid International Test Standards for Compression Set (CS)

There are various international test standards for the determination of compression set. In the following an overview of the two most widely used ones, namely ISO 815 and ASTM D395 will be given. As in the case of other material standards, the primary goal is the determination of certain material characteristic values for the description of the property of a specified formulation. For this purpose certain standard test pieces are then also prescribed (see more below in the text). The major practical importance of these standards is that they allow the determination of the degree of cross-linking of finished parts and the evaluation of the vulcanization process for finished parts. However, both above-mentioned standards describe „only“ the compression set testing on standardized test pieces but not on real seals, such as O-rings. The latter can also be subjected to the compression set testing but the results are based on short term tests (24 hours) and comparable with the standard test pieces assuming that the testing temperature does not exceed the permissible maximum 1000h continuous temperature load limit for the given material. This statement can be confirmed by a large number of our own laboratory test results. Thus, for example, the effect of the seal geometry becomes evident in longer continuous tests of compression set on O-rings (70 hours and more) (see Section 6.2.1).
Depending on temperature and time conditions, O-rings with larger cross sections yield better results than those with smaller cross sections since the free surface related to the mass decreases for larger cross sections and consequently more material mass in the O-ring core is available to resist the attack of the oxygen in the environment and of the aging.
The ISO 815-1 Standard in the current valid version from September 2014 deals with the compression set testing at elevated temperatures, while the Part 2 of this standard describes the compression set testing at low temperatures. The recommended compression of the test piece depends on its hardness. For the elastomer test pieces with the hardness ranging from 10 to 80 IHRD the prescribed compression is 25 ± 2%, for 80 to 89 IHRD 15 ± 2% and for 90 to 95 IHRD 10 ± 1%. There are two types of test pieces:

[14] This technical paper was published in a yearbook: RICHTER, Bernhard: *Beständigkeitsprüfungen von elastomeren Werkstoffen und Dichtungen* in: KIEFER, Sandra and BERGER, Karl-Friedrich (Editor): „*Dichtungstechnik Jahrbuch*“ (*Yearbook of Sealing Technology*“) 2012, ISGATEC GmbH, Mannheim, 2011, p.118 et.seq.

- **Type A** (Ø = 29mm ± 0,5mm x h = 12,5mm ± 0,5mm)
and
- **Type B** (Ø = 13mm ± 0,5mm x h = 6,3mm ± 0,3mm).

If there are no test plates with the required thickness available, laminating of maximum three discs is permitted.[15] The use of a larger test piece is recommended for elastomers with a lower compression set, since the larger dimension allow a better accuracy.[16] However, it should be pointed out here that thereby the ratio of free surface to mass is not in any way comparable to typical finished parts such as O-rings. In the case of large test pieces only a relatively small amount of mass is in contact with the air around it and at elevated temperatures this allows only limited aging. This leads to the risk that the obtained test results will represent aging and/or resistance to relaxation of elastomeric materials that are markedly better than they actually are in real seals.
The compression of the test pieces occurs at room temperature (23°C). The standard also specifies the exact test duration, test temperatures, conditioning of the test pieces and requirements regarding the laboratory ovens. Particular attention has to be paid to the end of the test. There are three different methods to be considered:

Method A requires that the equipment be opened immediately after the withdrawal (that is, practically still at the testing temperature). The test pieces are then laid on a wooden bench and the height is measured after 30 ± 3 minutes.

Method B requires cooling down to room temperature in stressed state for 30 to 120 minutes. Then the mold is opened and after additional 30 ± 3 minutes the height is measured.

Method C requires that at the end of the testing time the testing equipment in the oven is opened and the test pieces should remain once more for 30 ± 3 minutes at the test temperature in the oven to recover. Then they are removed and after additional 30 ± 3 minutes of cooling down to the room temperature at which the height is measured.

The best (that means the lowest) compression set values are obtained from the Method C. The high temperature during the relaxation is causing a higher mobility of the molecular network and with it an easier resetting in the direction of original shape. The worst, that is, the highest values of compression set are obtained with using Method B since in that case the test pieces remain under load until they are cooled down to the room temperature and only then they are relaxed.

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[15] Some internal specifications of large automotive companies require compression set testing on laminated test pieces (2x2mm). One manufacturer requires for example that the 2mm thick test pieces for compression set test are cut out from the same sheet material as the test pieces for tear resistance testing. As it is evident in the Figure 10 of this article the optimum tear resistance of a material with a different degree of cross-linking than the optimum compression set. Because of this directive, the manufacturer of the compound has to settle on certain degree of cross-linking and cannot change it as he desires.
[16] See: BROWN, Roger: Physical Testing of Rubber, Springer-Verlag, New York, [4]2006, p. 213

The Method B with partially smaller modifications is preferred for the use in the automotive industry. Thus it is found again in the VW PV3330 (issued in July 2004) and the PV3307 (issued in August 2004) or in the DBL 6038 (issued in November 2011) by Daimler. Method B, however, also sometimes leads to some discussions due to widely diverging values. This has particularly something to do with the type of cooling. In this matter, it is possible to obtain a good reproducibility when the cooling is supported by a blower and the cooling time is fixed so, that regardless of the test temperature and the mass of the compression set test plates, the temperature of the laboratory of 23 ± 2 °C is reached always before releasing the test pieces. Then there are notably many discussions with customers of our laboratory when by doing that, the specified values are no longer attained. However, even an increase of the relaxation temperature of the test pieces by 5°C can improve the result by 10%.
The following Figures 1 to 5 illustrate the different cooling and relaxation procedures of the ISO 815-1 (Procedure A to C) and the Volkswagen compression set test instructions PV3307 and PV3330.

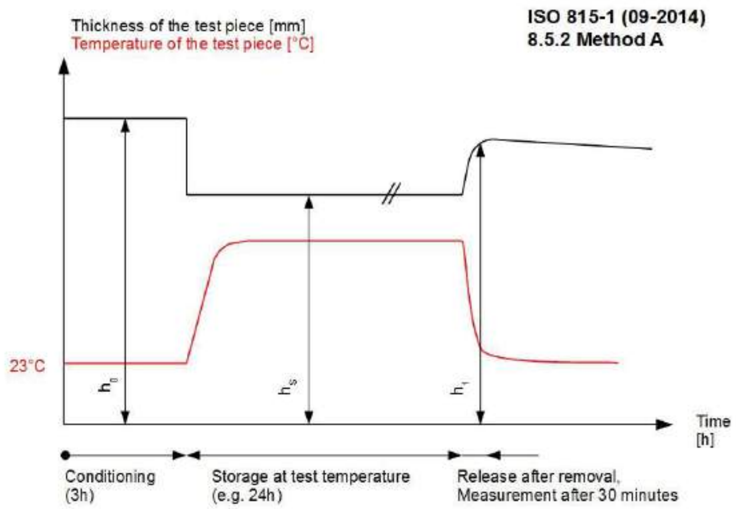

Figure 1: ISO 815-1, Method A,: final measurement 30 minutes after release

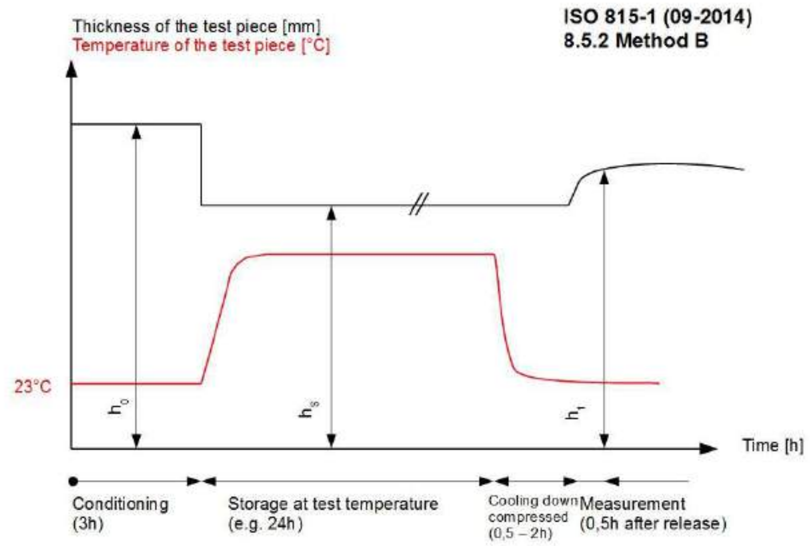

Figure 2: ISO 815-1, Method B: Cooling down to room temperature in the compressed state, final measurement 30 minutes after release: the method with the highest demand on the material

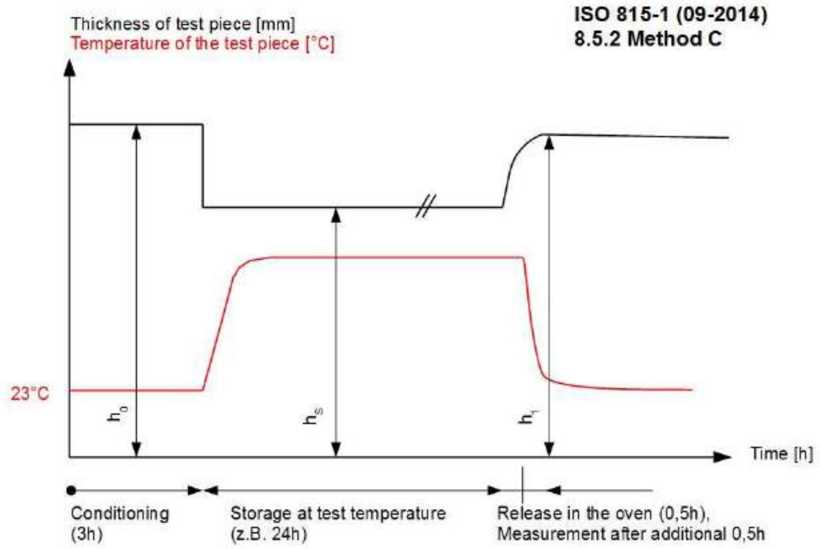

Figure 3: ISO 815-1, Method C: 30 minutes recovery in the oven at the testing temperature, final measurement 30 minutes after cooling down to room temperature, the method with the lowest demand on the material

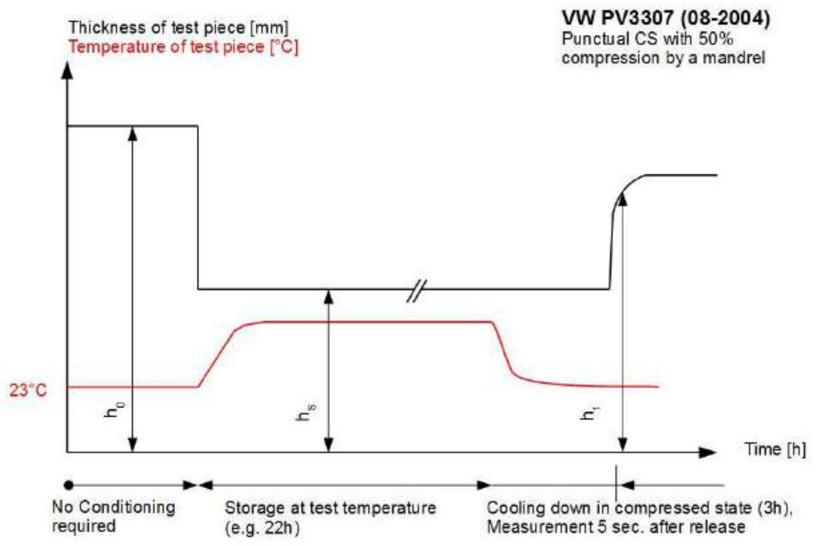

Figure 4: VW PV 3307: Very high compression by mandrel pressure and long cooling down in the stressed state, similar to the Method B, presented above. The required final measurement within 5 seconds after recovery is in practice very difficult to implement.

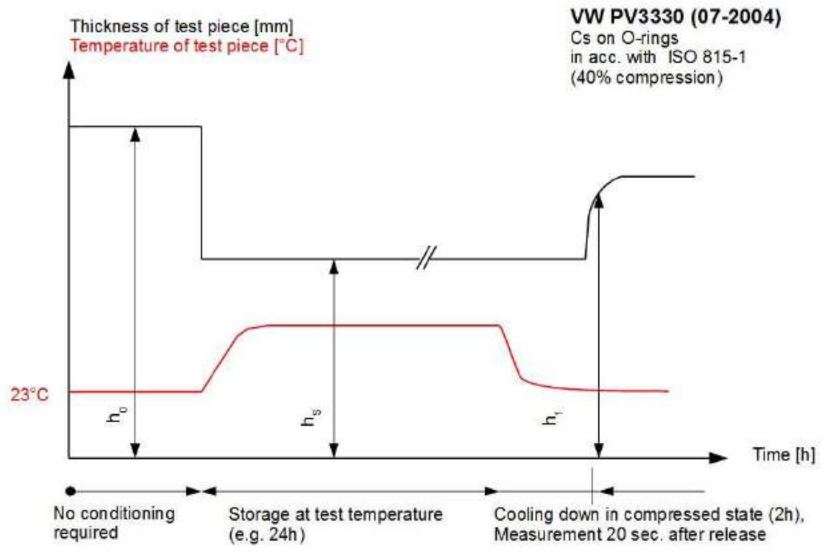

Figure 5: VW PV 3330: Test specification designated specifically for O-rings with high compression (40%) and longer cooling down in the compressed state, similar to the above presented Method B – The required final measurement after release follows 20 seconds after recovery.

According to ISO 815-1 the compression set of O-rings is calculated as follows:

$$CS = \frac{h_0 - h_1}{h_0 - h_S} \times 100$$

where

h_0 is the initial thickness of the test piece, in millimetres;
h_1 is the thickness of the test piece after recovery, in millimetres;
h_S is the height of the spacer, in millimetres.

From the formula it is obvious that the ideal elastic material would have a compression set value of 0%, which means that the material would spring back completely to its original height. A purely plastic material would have a compression set value of 100% (at the testing temperature) and in the practical application it would be the worst case, that is the seal will stay put and has consequently no residual seal force at all. In some cases it is possible to obtain a value greater than 100% because the test piece still cools down after recovery and consequently shrinks before the reference value h_1 is determined (Method A). In long-term compression set tests the shrinkage can be caused by the loss of plasticizers.

The ASTM D395 (issued in 2014) deals with the determination of compression set at elevated temperatures while the ASTM Standard D1229 deals with compression set at low temperatures. Thus the ASTM Standard Committee has not in this case followed the ISO practice, which in the compression set combines testing at elevated and low temperatures in one standard (ISO 815-1 und 2).[17] The Part A of the ASTM D395 treats the determination of the compression set under constant force that is applied to the test specimen (test piece) by means of a correspondingly strong pre-stressed spring. Since this method is scarcely used in Europe it will not be further discussed in this article. The Part B of ASTM D395 is about testing the deformation under constant compression. The required test specimen (test piece) 1A has dimensions identical to those of test piece Type A of ISO 815-1, small differences exist only in the thickness of the test piece 2B. The ASTM requires in this case the thickness 6,0mm ± 0,2mm, while the ISO 815 the height 6,3mm ± 0,3mm. However, these differences are categorized as negligible in their effect on the result. In a practical situation these required thicknesses of test pieces are not always adhered to. It is more important for a credible result that the compression be comparable, for example 25%.

At first glance it appears that there is a difference between testing times. While the ASTM among other things requires the time of 22 hours, in the ISO Standard it is 24 hours. Since in the daily laboratory practice the insertion times cannot be kept punctual to the minute, a tolerance is required. This amounts to in the ASTM Standard +2 hours and the ISO the requirement is -2 hours, so that even here there is a comparison for both standards. After the removal from the oven the test pieces have to be immediately released and placed on a wooden bench[18] for 30 minutes to cool down; after that the repeated measurement follows. The ASTM Standard offers only this single relaxation and cooling procedure, which matches the Method A of the ISO 815-1. In conclusion, it can be stated that by means of these both standards determined results can be definitely compared when the parameters of the tests (time, temperature, compression, and release/relaxation method) were chosen to be equal.

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[17] BROWN, Roger: Physical Testing of Rubber, Springer-Verlag, New York, [4]2006, p.. 214
[18] In the Standard a wooden plate is explicitly recommended because of its very low thermal conductivity.

4. Various Types of Test Equipment for Measuring Compression Set

Both above cited testing standards provide also instructions regarding the applicable kind of testing equipment. For this purpose strong and bend-proof metal plates which are screwed together for the desired height with the use of spacers. A typical design of testing equipment conforming to these standards can be seen in the Figures 6 and 7.

Figure 6: The O-Ring Prüflabor Richter GmbH owns numerous sets of tools for testing of compression set with base plates ground to different depths (see the lower plate in the picture), so that the insertion of spacers can be dropped)

Figure 7: Flat plates for compression set measurement with spacers. Compression set tools as used at the Prüflabor Richter for compression set testing in media. Their round shape allows their deposition into a glass beaker without problems.

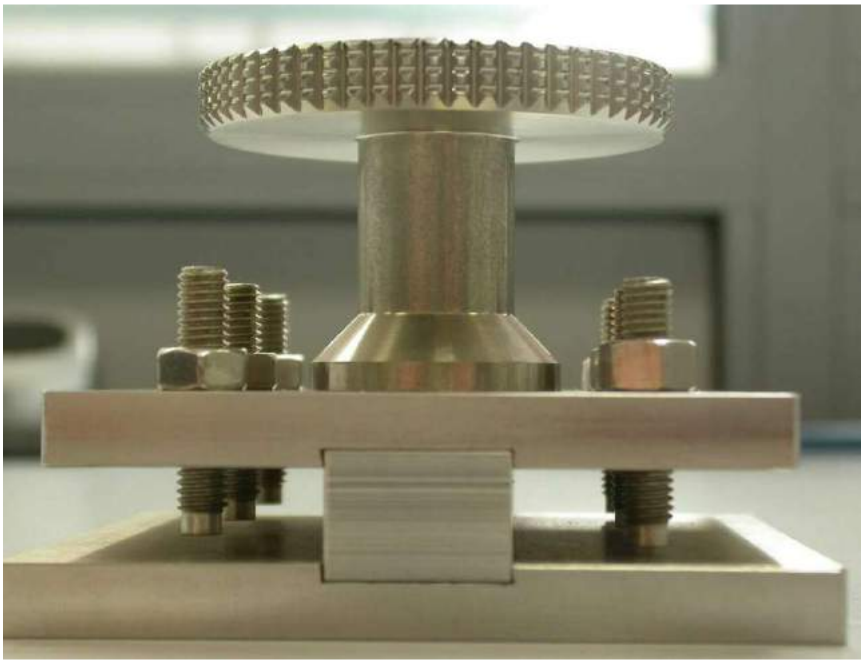

Figure 8: Compression set test fixture for VW-Test Procedure PV 3307 (published in August 2004) and VDA 675 218 (published in December 1992), produced by Gibitre Instruments, Bergamo, Italy

The Figure 8 shows a special fixture for the testing according to VW PV 3307. With this shown equipment the elastomer test piece is not loaded over the entire area but by a mandrel with 4,5 mm diameter and an edge radius of 0.2 mm. The use of this testing instrument has particular advantages in the testing of compression test of hoses, where the measurement can be done over the entire multilayer wall thickness.
If on a defined radius appear cracks on the test piece it is an evidence of a low tear resistance at elevated temperatures.
Particularly during the compression test of silicones (VMQ) at temperatures around 150°C or higher and fluoroelastomers (>200°C) it can happen – when the test pieces are poorly tempered – that they adhere to the test fixture and when the fixture is then opened, the test pieces are pulled up somewhat and this causes positive errors in results. In order to prevent its sticking of the test pieces to the mold, the use of release agents is specified in the standards. At the Richter test laboratory silicone oil is used in most cases and is applied only as a very thin coat by using a thin cotton cloth. In special cases, particularly for very high temperatures a very thin PTFE film is used, however its gauge has to be considered in the calculation of the compression set. Moreover it is important to make sure that there is no interaction with the elastomer. Another effect of the release agents is that they can make lateral deformation possible. If this kind of deformation is hindered, the stress state in the test piece changes and this can indirectly affect the test results.
A particular attention has to be paid to the production of test pieces. As a rule, they are prepared according to ISO 815-1 Type B (Ø = 13mm) by die cutting from 6mm thick test plates. Depending on the hardness and the type of material it is mostly impossible to obtain a rectangular cut edge for the support area (see Figure 9). Therefore O-Ring Prüflabor Richter has switched to cutting test pieces by a rotary knife.

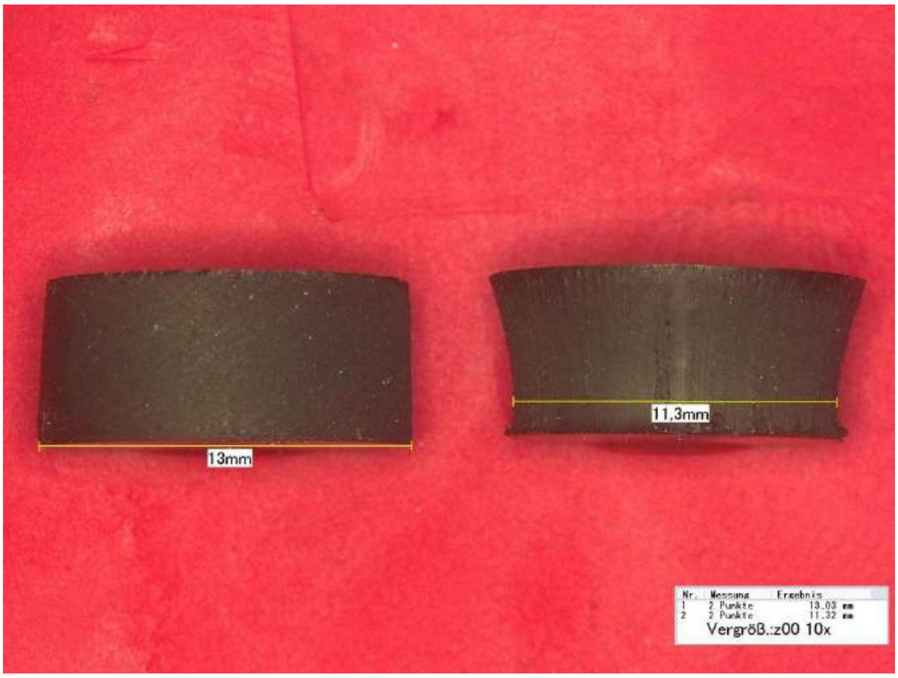

Figure 9: The die-cut test piece can be identified on the right side, the one, cut out by a rotary knife, is on the left. In the case on hand the smallest diameter of the die cut test piece (11,3mm) differs up to 13% from the specified size (13mm)! (Magnification 10x).

5. Performance Spectrum of Important Elastomers according to ISO/DIS 3601-5

The ISO 3601 Part 5 Standard was developed under the German project leadership and Bernhard Richter has also participated in the project with others. The current valid issue originated in April 2015. It replaced the ISO 3601-5 from the year 2002.
The ISO 3601 consists of five parts and deals with O-rings for sealing of fluids in machine building. In this standard among other things diameters, tolerances, installation situations, quality criteria, supporting and finally material specifications for O-rings in general industrial applications and the compounds used for them are specified. The latter compound specifications will be discussed in the following section. The requirements for the finished product namely an O-ring have been already cited above. Among the requirements for materials listed in the above ISO Standard the effects of the hardness and of the cross-linking system on the compression set are also taken into consideration. When these target values are met, the practitioner can be sure that he/she has received a state-of-the-art material.

The following table 2 [19], provides again an excerpt:

[19] The data were taken from: ISO 3601-5 Fluid Power Systems – O-Rings – Part 5: Specification of elastomeric materials for industrial applications, 2015-04-01, p. 4 et seq.

Test conditions	NBR 70 IRHD,M [100°C] cross-linked with sulfur	NBR 90 IRHD,M [100°C] cross-linked with sulfur	NBR 75 IRHD,M [100°C] cross-linked with peroxide	NBR 90 IRHD,M [100°C] cross-linked with peroxide	HNBR 75 IRHD,M [125°C]	HNBR 90 IRHD,M [125°C]	Test method
max. DVR, 72(+0/-2) h	40	40	40	40	40	45	ISO 815-1:2014, Method A
max. DVR, 336(+0/-2)h	60	70	50	60	60	70	ISO 815-1:2014, Method A

Test conditions	FKM 70, 75, 80 IRHD,M [175°C]	FKM 90 IRHD,M [175°C]	VMQ 70 IRHD,M [175°C]	EPDM 70 IRHD,M [100°C] cross-linked with sulfur	EPDM 80 IRHD,M [100°C] cross-linked with sulfur	EPDM 70, 80 IRHD,M [125°C] cross-linked with peroxide	ACM 70 IRHD,M [150°C]	Test method
max. DVR, 72(+0/-2) h	25	30	35	30	35	25	40	ISO 815-1: 2014, Method A
max. DVR, 336(+0/-2)h	40	45	55	60	60	40	50	ISO 815-1: 2014, Method A

Table 2: The compression set data, which are shown in this table draw up the input for compounds which were determined on test pieces of the type (Ø13mmx6mm). These test pieces are either cut out or die cut from test plates.

On the example of a sulfur-cured NBR in the long-term compression set test (336 hours) the differences based on different hardness values are obvious. A material with a high hardness produces usually an increased, that is, worse compression set value since in this case the elastomer compounds contain a high proportion of fillers. Because of that, the cross-linked polymeric matrix, responsible for the elastic recovery, occupies a smaller proportion in the compound.
With EPDM the differences generated by different cross-linking systems are clearly identified. Although peroxide cross-linked EPDM is tested at higher temperatures than the one cured with sulfur, it can attain better compression set values.

6. Things to Know for the Interpretation and Evaluation of Results from Compression Set Tests

As important as individual material properties such as compression set may be, when evaluating this property it should always be borne in mind which additional properties of the formulation or finished part play an important role for the application (see section 6.1). If in certain applications in fact the compression set is the essential criterion for a safe sealing function - what is the case with many O-rings - it should be also understood what influences bring about a good, that is, a lower value of compression set (see Section 6.2 with subsections).

6.1 Effects of the Compression Set or of the Degree of Cross-linking on the Properties of a Material

As has been already mentioned briefly above, the compression set is an indirect measure of the degree of cross-linking of a material. The cross-link density has a decisive effect on the different important properties of a seal. There is no definite cross-link density at which all properties exhibit an optimum, but it is true that for each type of application a compromise can be found. This can be seen in the following illustration[20] (Figure 10):

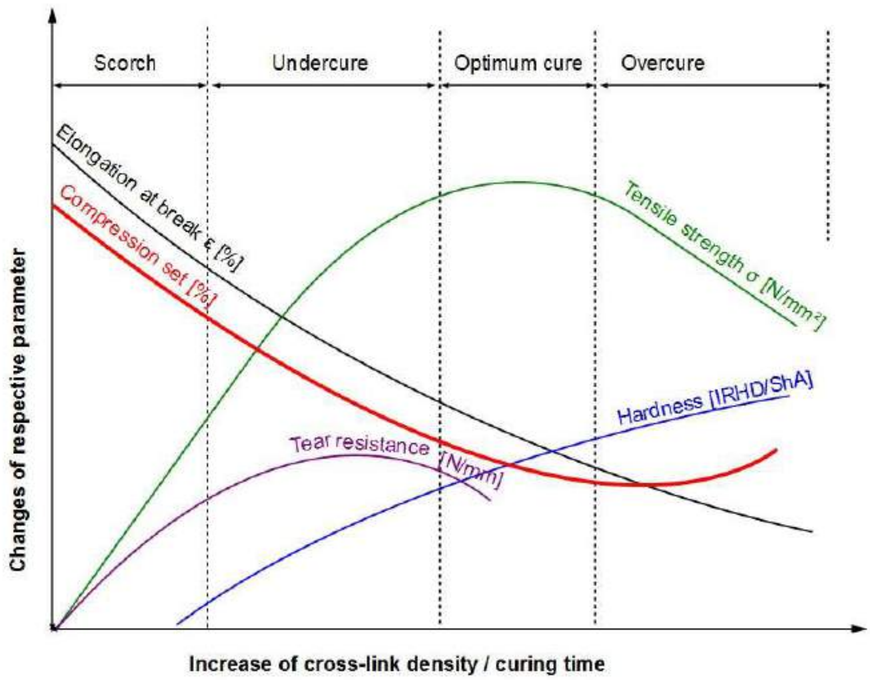

Figure 10: The compression set attains its lowest, that is, best value only towards the end of the optimum degree of vulcanization or at the beginning of the overcure zone.

6.2 Effects on the Test Result of the Compression Set

The result of the compression set value can be influenced positively or negatively by many factors. The familiarity with the factors that influence the results from compression set testing is important for arriving at stable and reproducible test values. This knowledge is particularly helpful when the test results are near the borderline of the specification requirement.

6.2.1 Effect of the Test Piece Geometry

Table 3 below shows that there is no effect of the cross section except typical uncertainty in measurements for NBR O-rings (NBR Formulation 1) with a good vulcanization degree after aging 22 hours at 100°C. The same is true about EPDM O-rings cross-linked with peroxide

[20] This diagram was drawn up with the help of following presentation and reworked: MATSCHINSKI, Paul (Editor): "*Roh- und Hilfsstoffe in der Gummiindustrie*" ("*Raw Materials and Additives in the Rubber Industry*") VEB Deutscher Verlag für Grundstoffindustrie, Leipzig, 1968, p. 171

after 22 hours at 150°C and for FKM O-rings after 22 hours at 200°C. On the other hand, the result after already 70 hours for the NBR Formulation 1 shows that O-rings with larger cross sections achieve better results.

The example with the Formulation 2 shows better compression set values for smaller cross sections after 22 hours at 100°C, which clearly hint that the thin cross section (1,78 mm) is vulcanized better than the thick cross section (6,99mm).

These results demonstrate that for checking the degree of vulcanization of finished parts 24 hour test times should be used preferably because with that the effect of geometry, that is the thickness of the test piece has only a negligible effect on the result. (This is true for test temperatures up to 1000 hour long-term temperature, the service limit of the material).

	NBR (Formulation 1)			**NBR (Formulation 2)**		
Cross section [mm]	1,78	3,53	6,99	1,78	3,53	6,99
CS after 22+2h, at 100°C [%]	10,4	12,8	9	11,4	15,7	18
CS after 70+2h, at 100°C [%]	23,8	23	16,8	24,3	24,2	28,9

Table 3: Effect of the cross section on compression set

In the following diagram (Figure 11) it is obvious that the der O-ring with the smallest cross section (d2 = 1,78 mm) shows the worst compression set values. The critical compression set value of 90% is attained already after 205 hours. The best results are obtained from an O-ring with a cross section of 6,99 mm. The critical compression set of 90% occurs only after 1300 hours. This O-ring can be compared most likely with the dimensions of the test piece of the type B (Ø13 mm x 6,3 mm) from ISO 815-1. Having said this, it is evident that the compression set values determined on standard test pieces cannot be compared to values from O-rings and as a rule, they are better than those measured on real seals.

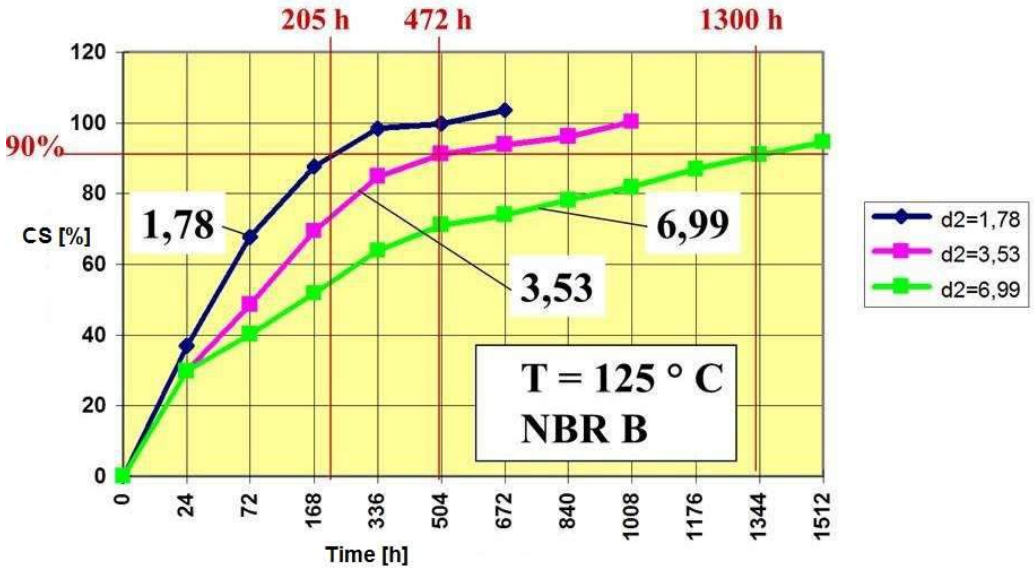

Figure 11: Effect of the cross section on the compression set of NBR at 125°C (long-term measurements of compression set) (Source of the diagrams: O-Ring Prüflabor Richter GmbH, Großbottwar)

How can the above-mentioned results be now explained theoretically? The rate of chemical reactions increases exponentially with the temperature as expressed by the Arrhenius Equation. This means for the aging by the exposure to heat and oxygen that an O-ring ages without restriction only then, when it is adequately in contact with oxygen If the ratio of free surface and mass predetermined by the geometry (cross section or cord size) then the supply of oxygen is restricted in dependence on the geometry. Long-term compression set experiments with NBR O-rings have shown that this effect of geometry at 80 °C is not having yet any significant importance, but already at 100°C a considerable effect is recognizable (approximately four times greater oxygen requirement compared to that at 80°C) and that at 125°C is severe (about twenty five times greater oxygen requirement than that at 80°C). However, the Arrhenius Law is valid only when there are sufficient amounts of reaction partners available. Here the Arrhenius Law shows that at 100°C the aging reaction runs four times faster than at 80°C, thus it will happen only when at 100°C also four times more oxygen is available than it was at 80°C.
At temperatures up to about 20-25°C below the permissible long-term upper temperature limit (1000 hour - criterion) for O-ring materials the effect of the cross section is very small. For NBR these temperatures up to about 80°C, for peroxide cured EPDM up to about 125°C, for FKM up to about 175°C und for VMQ up to 150°C.
Now, what do these findings mean for practical applications? In critical applications – when the installation situation allows it – an O-ring with larger cross section is preferable. Beyond that the results from compression set tests performed on standard test pieces should be critically scrutinized with regard to their transferability onto O-rings when these compression set data were determined at higher temperatures than those stated above.

<u>*6.2.2 Effect of the Percentage of Compression*</u>

As already mentioned above when introducing the Testing Standard ISO 815-1 the compression of (25±2) % is the most widespread standard for the determination of compression set. For harder materials the compression is reduced correspondingly. However, there are also specifications from different users of O-rings, which require higher compressions, such as, for example, 30%, 40% or 50% (see VW Specification PV3330).

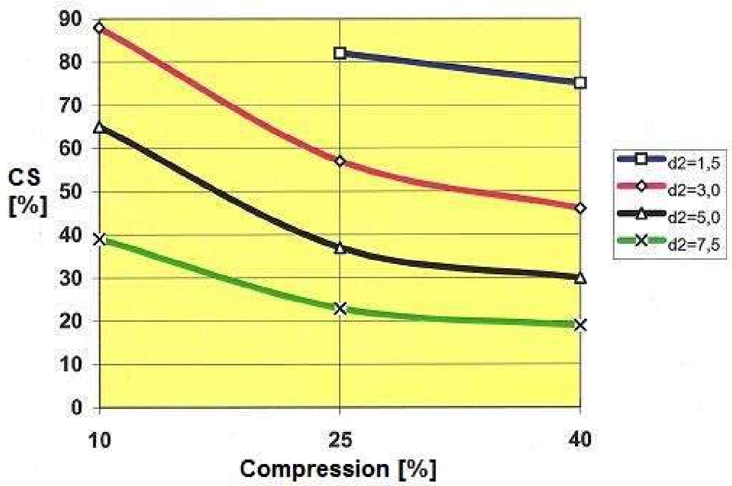

Figure 12: Effects of the compression and cross section on the compression set of O-rings, made from an HNBR sample formulation, after 7 days (168 hours) at 150°C[21]

[21] MAGG, H., Bayer AG, Seminar „O-Ringe in Kraftfahrzeugen“ on October 7.1997 in the Haus der Technik, Essen

In general, it can be said that with increasing compression (up to about. 35-40%) compression set data can improve and that on O-rings with compressions under 10% considerably worse results are obtained than at 25%. Therefore the mounting spaces of the O-rings should be chosen so that the minimum compression of 10% is maintained. At too high compression and high temperatures there is a risk of interior stress cracks. This risk increases with increasing cross section. Therefore here critical deformations of large cross sections are substantially lower than those of smaller cross sections. From experience it is known that for O-rings with a maximum cross section of 6,99 mm, with usual hardness values (maximum 90 Shore A) and with the 25% compression, the danger of blowing out due to internal stress cracks is small, provided that the permissible continuous service temperatures (see remarks in chapter 6.2.1) are not exceeded.

6.2.3 Effect of the Recovery Temperature of Test Pieces

As already described above and illustrated by Figures 1 to 3 the ISO 815-1 allows three different recovery methods (Section 8.5.2 in the standard: methods A, B, C)
In the method A the test pieces are released immediately after their removal from the oven, while in the method B they are removed from the oven and then cooled down in a compressed state for 30 to 120 minutes to the room temperature. The last method B is the most demanding for the elastomer compound and it usually produces the worst results. Materials that exhibit the lowest elasticity at room temperature (for example fluoroelastomer or polyacrylate rubber compounds) show the largest differences between data from methods A and B. These materials spring back relatively faster in warm state than at room temperature.[22]
This means for practical applications that materials with a very low elasticity at room temperature or low temperatures in dynamic systems with large temperature fluctuations are suitable only for a limited use. However, at permanent high temperature the use is less problematic.

6.2.4 Effect of the Test Medium

This article deals only with the testing of compression set in hot air, nevertheless it should be briefly noted that compression set tests in other test media yield better data than in pure ambient air contact. This has to do with the fact that the corresponding contact medium protects the seal from oxygen and resulting rapid aging. However in exceptional cases there are contact media that are more aggressive than air, here the picture is then opposite.
This subject is discussed in a professional article published on our website: http://www.o-ring-prueflabor.de/de/fachaufsaetze/bewertung-der-ergebnisse-von-bestaendigkeits-und-alterungspruefungen/
For practical applications this means that in many cases a seal, which is completely surrounded by a medium will have very likely a longer service life.

[22] See, NAGDI, Khairi: *„Gummi-Werkstoffe*, *Ein Ratgeber für Anwender“* (“Rubber Materials *A Guide for Practitioners")* Ratingen, ² 2002, p. 300

6.2.5 Effect of Special Seal Designs on the Reproducibility of the Measurement

In order to obtain reproducible test results from complicated seal geometries (that is, not O-rings) such as, for example, flange seals, precise test methods must be established. In the test conditions, it is recommended here to specify the point at which the section to be tested is cut out of the seal and to precisely record the often difficult positioning of the specimen in the test plan of the incoming goods inspection. Either a special device for taking up and holding of the piece to be tested has to be acquired or for flange seals a crossing point of three or four seal arms is cut out so that the test piece can stand safely and independently in the compression set testing equipment.
Of course, sections from flange seals can be tested lying flat, however there are often problems caused by supporting studs, concave shape, knit lines and the very low height; these factors reduce accuracy and reproducibility. In general, it is recommended to work out such special test plans in cooperation with the manufacturer of the seal. This facilitates the communication if problems arise.
For practical applications this means that – where absolutely necessary – individually test methods should be established. However, a clear communication and agreement between all participants (manufacturer, customer and possibly the engaged test laboratory) regarding these deviations from the standard conditions is crucial. The O-Ring Prüflabor Richter has an extensive experience with compression set testing on finished parts. A widely used method for profile seals is measuring approximately plane parallel profiled disks and marks the measured points. In comparison to standard test pieces and O-rings, however, this can lead to greater measurement uncertainties.

6.2.6 Effects of the Processing

The properties of elastomer products are affected sustainably by the modular network. On the one hand, always a characteristic network structure is formed and on the other hand the network density, that is the number of cross-links as well as their length[23] are equally important.
This modular network is critically dependent on two factors, namely for one thing on the quality of the formulation and for another thing on the quality of processing. Both these factors combined are the measure of the durability of a seal which can be reflected by means of the results from the compression set testing.

Durability (compression set) = Formulation quality x Processing quality

This multiplicative linkage means that a high formulation quality, that is, a good compound, remains ineffective when the elastomer seal is not vulcanized under the correct conditions during its manufacture. In contrast to optimally vulcanized test pieces from test plates, seals are often cured within a fraction of the time. Therefore the data sheet information has significance only for formulation quality but not for the quality of the finished product.

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[23] See:. RAHM, W.: „*Kurzzeitprüfverfahren für Gummi in der Qualitätssicherung“* in: KGK Kautschuk Gummi Kunststoffe, Hüthig Verlag, Heidelberg, 48.Jahrgang, Nr. 9/1995, p..634

Many compounds are today fully vulcanized already in the mold, however the omnipresent high cost pressure is causing occasionally that the cycle time and consequently the vulcanization time is shortened. This means saving in the wrong place with occasional disastrous consequences. However, there are also compounds requiring longer vulcanization time, which cannot be implemented economically during the injection molding process. These seals are left in the mold until they reach dimensional stability and are subsequently heated in a special tempering oven post-curing (additional cross-linking). This additional operation can easily cause problems, since it can be either forgotten or incorrectly executed for the reason that it is not automatized as easily as the injection molding process. If this process step is omitted the seal will very likely fail during its use. This error can be mostly recognized and proven without problems by means of the compression set test.
The following processing study (Tables 4 and 5)[24] from Parker Hannifin GmbH, performed with peroxide cross-linked HNBR O-rings, shows on the one hand the high sensitivity of the compression set value to too low mold temperatures (Table 4), on the other hand these diagrams document that a strong undercure of O-rings (mold temperature T = 170 °C) cannot be detected by measuring the hardness (Table 5), since the variation of hardness values caused by the production process conditions lay almost consistently within a bandwidth of 10 hardness points. The resolution and respectively the sensitivity of the compression set measurement is here much higher than that of testing of hardness. This is particularly obvious during inspection and comparison of data at the mold temperature of 170°C. When inspecting only hardness data, a clear increase can be identified. At first glance it could be assumed that this is a sign of improving of the degree of cross-linking. However, when comparing all the compression set results one would have to observe with surprise that for all three cycle times the compression set data were 100% or more[25], a clear sign of undercure, which in practical application would lead to a certain failure of the seal.

Mold-temperature	**CS after cycle time 60sec.**	**CS after cycle time 120 sec.**	**CS after cycle time 180 sec.**
170°C	108%	103%	100%
190°C	95%	75%	47%
210°C	39%	35%	30%

Table 4: Effect of the processing parameters on compression set (24h at 150°C) from an HNBR O-ring with dimensions 19,3mm x 2,4mm (Source of the data: Parker Hannifin GmbH)

[24] The procesing study was publically presented by Bernhard Richter in the seminar „Dichtungswerkstoffe für O-Ringe“ (“Seaing Materials for O-Rings“) , Haus der Technik , "Anwendung und Instandhaltung von Gleitringdichtungen" on 29./30.November 1995

[25] For that see also explanations of compression set results >100% in Chapter 3 of this technical paper.

Mold-temperature	Hardness after cycle time 60sec.	Hardness after cycle time 120 sec.	Hardness after cycle time t 180 sec.
170°C	58 IRHD	63 IRHD	66 IRHD
190°C	64 IRHD	68 IRHD	71 IRHD
210°C	67 IRHD	69 IRHD	69 IRHD

Table 5: Effect of the processing parameters on hardness (24h at 150°C)
From an HNBR O-ring with dimensions 19,3mm x 2,4mm
(Source of the data: Parker Hannifin GmbH)

Thus when this is transferred to practice, it means that primarily compression set measurements on seals alone provide some information about the state of cross-linking of the seals and that the hardness of the seals or more specifically, the shape stability of the seals indicate the undercure very inaccurately. Furthermore, it is recommended to train the own company employees that are involved in purchasing, quality insurance, and application to learn the basics of elastomer technology. It is also important to explain to them that the importance of hardness measurements is often overrated and can occasionally lead to risky incorrect assessments and decisions. A better knowledge is helpful in faster resolving discrepancies with the manufacturer and ultimately find more economically optimal solutions.

6.2.7 Effect of Usual Fluctuations of the Test Results from Different Measurements or Laboratories (Repeatability (r) / Reproducibility (R))

The ISO 815-1 (issued 09-2014) contains in Annex A the information regarding precision of the test procedure. The data were determined in an interlaboratory test programme (TP) in the year 1986. Determined were the repeatability (r) and the reproducibility (R) based on the test piece of the Type A and Type B produced from three different materials.
The results show that the different materials but also the dimensions of the test pieces cause substantial differences so that it is not possible to speak about one certain general value of the repeatability and reproducibility of the data from compression set measured on elastomers.
It should be added that, tests for the Annex of an ISO Standard have to meet the highest requirements. Only selected, long known and perfectly mixed and processed reference elastomers are used and these are tested under ideal conditions. Such a high precision is for the most part not attained with the use of materials from the normal everyday production.

	Mean value [%]	Within one laboratory		Between two laboratories	
Precision		***r***	***(r)***	***R***	***(R)***
EPDM	14,8	3,3	22	4,5	30
NBR	24,4	4,3	18	7,7	32

Legend:
r = Repeatability in measurement units
(r) = Repeatability in percent (relative)
R = Reproducibility in measurement units
(R) = Reproducibility in percent (relative)

Table 6: Precision data for the compression set measurements on test piece Type B (Ø (13±0,5)mm and thickness = (6,3 ± 0,3)mm) after 30 minutes (Method A) at 100°C, 24 h and 25% compression[26]

This means for practical applications that variations in the ± 2%-points on standard test pieces and ± 3%-points on finished products (O-rings) are still within the area of measuring accuracy. It is in fact common (but not conforming to standards) to report compression set data with one decimal place. It is recommended not to overvalue this decimal place as to its importance. If, for example, a specification allows the maximum value of compression set to be 30% and you receive an offer of a material with a compression set value of 29% and another with 25%, it is recommended to accept the latter even when both meet the specification on the first sight. Thus with the better compression set value you will have in the future a greater safety of having the product with the compression set value that is within the range specified by the standard.

7. Conclusion

Testing of compression set belongs besides the measuring of hardness, density and tensile tests to the important simple and basic testing procedures in the elastomer industry.
It is the youngest basic testing procedure and the entire informative value that is contained in it, has been fully recognized only in the last decades and to some extent and to this date, it is not utilized to the fullest extent.
The compression set testing also enables a comparative evaluation of formulations and reassessment of technological suitability, as well as the determination of a statement regarding long-term behavior of materials or finished parts. With proper knowledge and control of all important factors affecting the test result, it can also be used for many elastomer-based components (such as O-rings) and this is where it can mainly provide easily determinable and well reproducible statements regarding the quality of the processing.

[26] The data were taken from: ISO 815-1 (published 09-2014), translation of the terms with the help of DIN ISO 815 (published 03-2000)