EXPERT KNOWLEDGE OF ELASTOMER COMPONENTS

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Indispensable Rubber Tests Yesterday and Today

A review of over 100 years of testing history from the point of view of the new O-ring standard ISO 3601-5

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Summary

In 2015 the Shore A hardness was introduced 100 years ago and in 1916 W.C.Geer presented the heat aging of rubber to the public for the first time. The beginnings of tensile testing are even further in the past, while compression set testing is a more recent test method.
The new O-ring standard ISO 3601-5 (04-2015) shows that these test methods are still indispensable, even if partially modified. The first part of the lecture will focus on the history and development of these test methods (speaker Ulrich Blobner), while the second part (speaker Bernhard Richter) will explain why these tests in the new O-ring standard are still so important in today's practice.

1. Review of more than 100 Years of Testing History of Elastomers (U.Blobner)

The current technical literature on testing contains relatively little on the development and background of the most important basic test types (e.g. tensile test, hardness test, heat aging, compression set, etc.). What important steps did these tests undergo in their development? Why do some methods work with - at first glance - strange specifications or dimensions of the test equipment?
A deeper study of the history of these methods not only reveals curiosities, but also teaches a better understanding of today's testing methods or the ability to critically analyze them at certain points.

1.1 Tensile Test

Until systematic and reproducible tensile tests were carried out, many important scientists theoretically approached this field, including Galileo Galilei and Thomas Hooke in the 17th century. One of the earliest instruments for testing and measuring tensile strength was developed by the Dutch scientist Peter van Musschenbroek (1692-1761).
Charles Goodyear discovered vulcanization in 1839. In the early days of the use of rubber, people were particularly fascinated by two of its properties: its watertightness and its high elasticity. Tensile tests were necessary to better define the latter. Nevertheless, it took 30 years from the discovery of vulcanization to the first scientific description of a tensile elongation test on rubber. In 1869, Emilio Villari, university professor in Florence, published a report on this in the Italian journal "IL Nuovo Cimento" [1]. At the end of his studies in 1864 Villari stayed at the University of Berlin among others and also with Heinrich Gustav Magnus. It is therefore understandable that his contribution on the elasticity of rubber appeared two years later in German in the "Annalen der Physik" [2] which made it available to a wider audience.
He found that rubber has three different elastic coefficients [3]: The smallest and first "elasticity coefficient" is approximately constant, the second and mean coefficient is a variable rapidly increasing coefficient, while the third and largest coefficient is again approximately constant.
Almost simultaneously, Stevart published his studies on the tensile-stretch behavior of rubber.[4]
A major problem of the early tensile tests was the fixing and marking of the specimen in the testing machine. Villari used rubber cords. "The thread to be tested was folded over at both ends to form two small eyelets; the lacing was done while the thread was strongly tensioned. (...) The lengths were measured on two characters made with ink [sic!] on the rubber; to keep the characters sharp and fine, the thread was stretched strongly and applied cleanly with a feather dipped in ink. After the ink had dried and the rubber had been relaxed, the characters appeared black, even and very delicate, as one would not have otherwise achieved". [5]
In the following years, more often simple strip samples were used, but most of them cracked at the clamping points. In addition, many fundamental questions had to be clarified, such as whether the force at the moment of tearing should be related to the initial cross-section or the one tapered by the elongation. But already at the turn of the last century, this was clearly decided in favor of the first variant, as was usual for other materials (e.g. iron) at that time. [6]
In a detailed and scientifically exemplary investigation by the Royal Materials Testing Office (forerunner of the BAM (Federal Institute for Materials Research and Testing)) in Berlin in 1909,

MEMMLER and SCHOB investigated various rod shapes (including dumbbell shapes) and compared the results with those of standard rings. This report is still able to set standards in its thoroughness today, among other things, the different rolling directions of the test plates have already been taken into account and a very sophisticated system of conical centering rings has been used, which enabled right-angled cutting edges when punching out the standard rings. This treatise would certainly not have been possible with such precision had it not been for the congenial cooperation with the test equipment manufacturer Louis Schopper in Leipzig.

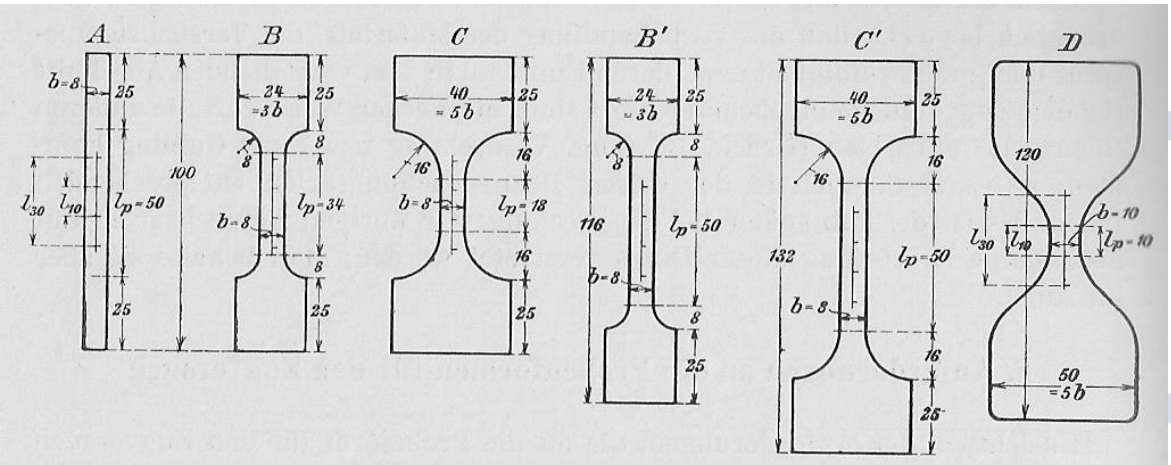

Fig. 1: Rod forms investigated by MEMMLER and SCHOB in 1909 [7]
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At the suggestion of Prof. Gustaf Dalén (1860-1936), Schopper's tensile testing machine, which was originally designed for paper and fabric, was set up for the purpose of tensile testing with rubber rings (...) "by attaching rotating rollers instead of clamping jaws in order to test the tearing of rubber rings". The force generator has a hydraulic drive and consists of a piston guided in a cylinder, which can be moved up and down by means of water line pressure (3 Atm.)" [8] The piston speed could be regulated by controlling the water flow. The force was measured with an inclination balance. Later, a line drawer was developed.

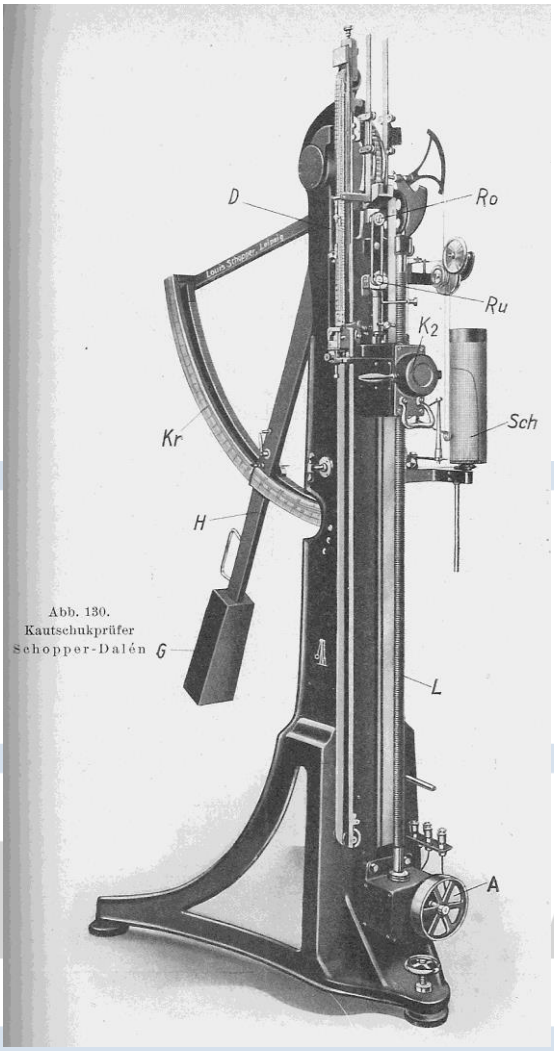

Fig. 2: Tensile testing machine according to Schopper-Dalén [9] (printed with kind permission of S. Hirzel Verlag, Stuttgart)

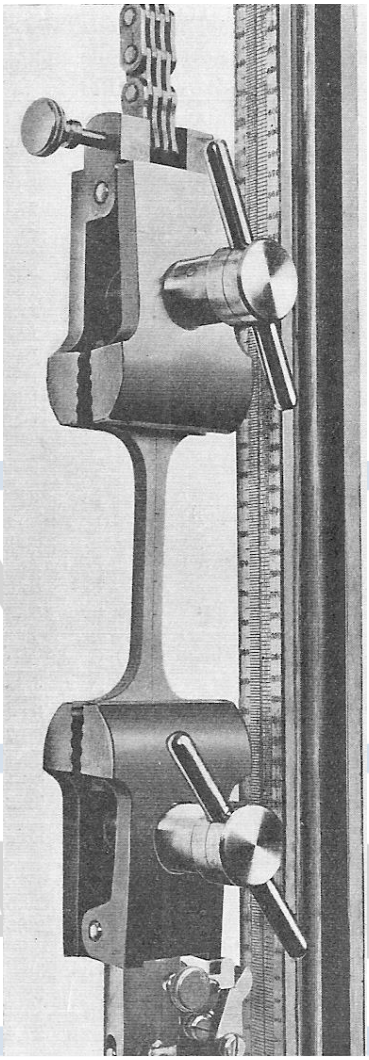

Fig. 3: Clamping jaws for thick shoulder bars [10] (printed with kind permission of S. Hirzel Verlag, Stuttgart)

At the beginning of the 20th century, the use of rings was particularly propagated in German literature. Before exact extensometers were developed, the following problems occurred when testing shoulder bars [11]:

- Easy slipping of the specimens out of the clamping jaws, re-clamping necessary during the tensile test
- Simultaneous observation of the two measuring points at the respective ends of the measuring length was very difficult for one observer.
- Inaccuracy due to widening of the measuring points during the tensile test
- Accurate reading of elongation at the moment of tearing was difficult, as breaks often occur unexpectedly

The problems described above did not occur with rings, since the inner diameter of the rings and therefore the elongation can be measured directly via the crosshead travel. However,

there are also criticisms of the ring test: the inner diameter is stretched more than the outer diameter of the ring.
In their study from 1909, MEMMLER and SCHOB found that ring specimens yield higher strength values than rod specimens. This principle no longer applies today. A glance at the test conditions makes this reversal understandable: MEMMLER and SCHOB used 6mm thick test rods and only 4mm wide rings. The ring width has remained the same in the standards to this day, while now almost only 2mm thick test rods are used. Due to the smaller specimen cross section and volume of today's test rods - in comparison to the standard rings - better results are achieved. [12]
In the following years, the tensile test method was further refined. As early as 1927, the following four areas were described in the literature [13], for which the tensile test can provide meaningful information:

- Evaluation of raw rubber
- Comparison of technical compounds
- Investigation of aging effects
- Ensuring a consistent quality in production

Already in 1928 tensile tests were carried out with a temperature chamber from SOMERVILLE and COPE. [14]
The shoulder bars have almost completely replaced the standard ring in today's testing practice - probably by the introduction of 2mm thick test plates, punching knives with exchangeable bendable blades and functioning clamping jaws.
This is also reflected in the current ISO 37 [15], the international standard for tensile testing of elastomers:

- Dumbbell bars should be preferred if the tensile strength of a material is to be determined. Standard rings give lower, sometimes much lower results than shoulder bars.
- For *elongation at break*, approximately the same values are obtained between tensile test bars and rings if the elongation of the rings is calculated as a percentage of the initial inner diameter of the rings and if the shoulder bars are cut at right angles to the orientations of the test plates (if any).
- For elongation under a given stress or for stress under a given elongation (= tension value), approximately the same values are obtained between tensile test bars and rings if the elongation of the rings is calculated as a percentage of the initial mean diameter and if the mean value of shoulder bars, which are cut parallel and perpendicular to the orientations, is determined.

1.2 Shore A - Hardness [16]

Since time immemorial, the hardness testing of iron products has been of interest to people. With industrialization in the 19th century, the demand for reproducible hardness testing methods for metals became even stronger. Around 1905 [17] Adolf Martens (1850-1914) developed the so-called indentation principle. Not only was the impression of for example a test ball measured, but the penetration depth and test force were recorded simultaneously. This method allowed more information to be obtained about the material than just its plastic behavior.

In the case of elastomers, unlike metals, permanent deformation cannot be measured after removal of an indenter. The extremely high elasticity of rubber, meaning its spontaneous re-deformation after loading, is probably one of the most important properties of this material.

At the turn of the last century, two important different principles for measuring the hardness of elastomers were developed, which were refined over the decades and are still used today:

1.2.1 Hardness Testers with a Load Weight

The indenter is usually loaded with a small pre-weight to ensure contact with the specimen. Then the actual load weight is added. The hardness value is read off after a specified test period (today usually longer than with spring-loaded devices). With this principle, the hardness value consists of the penetration of the indenter due to plastic flow and a second part caused by elastic deformation. While the test devices activated by spring force presented in the following more or less represent only the elastic indentation. [18]
Around the middle of the 20th century, there was a large number of testing devices that worked according to this principle. Today, the IRHD method with all its sub-variants (e.g. micro-hardness) has established itself in everyday testing. Only special industries still use special test methods.

1.2.2 Hardness Testers with Spring Force (short: Spring Pressure Testers)

The test force is transferred to the indenter by means of a spring. Albert Shore's durometer is undoubtedly the most prominent representative of this type. However, there were also variants of other manufacturers, partly with other indenters as used by Shore.

1.2.3 Albert Shore's Durometer Type A

1915 [19] is considered the official year of the introduction of the Type A durometer by Albert Ferdinand Shore (1876-1936). He was the founder of the company named in his surname "The Shore Instrument & Mfg. Co. Inc. based in Jamaica, New York. Shore's son Fred continued the business after his father's death. [20] Albert Shore was an active inventor, of whom more than 30 patents have been obtained for the most diverse areas of technology.
By patent searches, however, an earlier very similar invention for hardness testing could be proven, which was already registered by William F. Shore in 1911 and patented on 29 October 1912 under the number 1,042,721. William was presumably Albert's brother. [21] This document presents a pocket-sized round hardness tester that anticipates many features of the later round durometer ("round style"). The patent drawing even shows the truncated cone indenter, and the use of a sharper indenter for harder materials is suggested. The hardness scale ranges already from 0 to 100, but the movement of the pointer is from 100 to 0. The most serious difference to the durometer introduced in 1915 is the use of a helical compression spring instead of the later common leaf springs. This invention was especially designed for testing rubber, as described at the beginning of the patent. In addition, it has already been recognized that not only the hardness but also other properties of the material can be measured, such as flexibility and elasticity.

Why it came to the introduction of the "quadrant type" [22] durometer in 1915 with a much less accurate reading scale is no longer clear today. Perhaps Albert wanted to circumvent his brother's patent. When the round durometer with a more accurate reading scale was introduced in 1944, his brothers patent had already expired. However, after reviewing this patent, it becomes difficult to identify Albert F. Shore as the sole inventor of the Shore durometer.
The "quadrant type" durometer became very popular. In the following years, devices for softer and harder materials were developed, whereby the hardness and partly the contact pressure increases with the letter ascending in the alphabet (e.g. Shore D for plastics).
Finally, the company changed hands several times and "The Shore Instrument & Mfg. Co. Inc." seems to have gone out in the meantime.

1.2.4 Durometer in Germany

Soon Shore hardness testers were manufactured in Germany in great variety. In 1942 KLUCKOW [23] already described three Shore hardness testers from German production. All devices were still variations of the original "quadrant type" style of the US-American company Shore. The outer dimensions correspond approximately to a quarter-circle area. It can be assumed that the ±5 ShA tolerance which is still customary today for the specification of hardness values is still based on this rough classification of the initial time. Although many elastomer products can now also be produced in a hardness window of ±3 ShA.
Today, in addition to digital hand-held testing devices, there are still a few round analog devices with a 1 ShA scale division in everyday testing.

1.2.5 General Advantages of the ShA Hardness Testing Method

The great popularity of A.F. Shore equipment, which was not limited to the USA, was undoubtedly due to the variety of its advantages:

- Low weight
- Easy to manufacture
- Simple calibration and easy to repair
- Low purchase price
- Few moved and, in their design, very simple parts (much simpler than a mechanical wristwatch of that time)
- Easy to Use
- Testing on the finished part possible
- Non-destructive test method
- Easy to transport
- Due to the geometry of the truncated cone, a round and flat surface lies on the specimen at the beginning of the test. Compared to pointed indenters, this results in a lower sensitivity to different surface structures.
- For many years it had hardly any serious competition as a pocket tester. [24] The only "competitors" were unwieldy and difficult to operate large laboratory bench-top devices with load weights. Alternative pocket instruments with spring force did not emerge until the Shore Durometer was already established or often these alternatives were a more

or less accurate copy of the original A.F. Shore.

- The low sensitivity of the device sometimes produces a uniformity in the results, which was often wrongly attributed to the accuracy of the device. [25]
- The classification of the scale from 0 (very soft) to 100 (infinitely hard) is logical and comprehensible even for the layman rather than the usual indication of the penetration depth of the floor-standing devices with load weight.
- The handy tester was particularly popular among practitioners and users of elastomers. This is why, when ordering seals or other elastomer components, the hardness was defined using the ShA test method. [26]

1.2.6 Important Criticisms of Devices, Processes and Indenter Geometry

LARRICK [27] describes the problem in 1940 that if the pocket tester is pushed too hard, a soft elastomer can be pressed into the bore from which the indenter protrudes. This pushes the indenter upwards and incorrectly indicates a harder material. With the introduction of the round durometer in 1944 and an increased pressure foot, this danger was reduced because the user would now have to apply a much greater force to obtain the same negative effect as with the small pressure foot of the quadrant-style durometer.

The early Shore devices were also criticized for their spring characteristics. The five different Shore A durometers examined by LARRICK [28] all required a lower preload (i.e. < 2oz./57 gr.) to display the value zero than what was specified for the devices. Also, to display the value 100, different weights were needed than the required 29 oz. (822gr.). Furthermore, there were also larger dimensional deviations at the different diameters of the truncated cone. These have a considerable influence on the results. Due to the narrow tolerances in today's standards, this influence is small, but not completely negligible.

It is also important to take a closer look at the device spring. According to SPÄTH it has two functions: Firstly, the spring applies the load to the test specimen, and secondly, the spring deformation is used to determine the penetration depth. The load exerted by the spring is not constant, but "depends to a large extent on the value to be measured itself". If the aim were to maintain a constant load, this instrument spring would have to be so large that the entire measuring stroke of the test probe does not exert a significant influence on the spring force. [29] If this requirement were met, it would logically no longer be possible to produce pocket-size hardness testers.

In the hardness test methods with load weight, the test pressure is kept constant, whereas in devices with a spring mechanism, the test pressure depends on the hardness of the specimen. SPÄTH criticized in 1956 that "the linear classification of the hardness scale values to the deformation of the instrument spring does not do justice to the concept of hardness" [30]. For this reason, the greatest care should always be taken when changes in Shore A hardness are interpreted in comparison with changes in other important properties (e.g. from the tensile test, compression set, etc.).

The arbitrary determination of the hardness scale is a very justified point of criticism, as it does not allow simple conclusions to be drawn about other mechanical material properties. According to the definition, 100 ShA corresponds to the hardness of a glass plate. However, the durometer measures rubber materials, which are usually many times softer. This results in an extreme compression of different hardness classes at higher hardness levels (> 90 ShA). Since the resolution is also extremely low in the lower range, it is recommended only to include

ShA hardnesses >20 ShA degrees of hardness in evaluations or to use a different test method for softer qualities. This arbitrariness in determining the Shore A hardness scale also explains why there is no linear relationship between the ShA hardness and the moduli of elasticity obtained from static load tests.
From very early on, the great inaccuracy of the ShA measurement method was criticized. Important steps in the direction of higher precision were among others:

- The introduction of the round durometer (incl. tripod), with a display scale in one step and a much higher resolution than the "quadrant-style" durometers
- The increasingly precise standardization of the test procedure (especially in DIN 53 505 and its successor DIN ISO 7619-1) over the past decades
- The introduction of digital displays
- Lastly, the increased precision of today's precision mechanical manufacturing

There were many suggestions to change the indenter geometry of the ShA pocket tester, be it for practical or scientific reasons. However, nothing could establish itself permanently. But to deal more profoundly with the associated arguments can lead to a better understanding of ShA hardness testing.
Already at an early stage, studies repeatedly pointed out the risk of wear of the truncated cone. Due to its geometry, this risk is higher than with hardness testers using spheres as indenters. It is therefore necessary to inspect the truncated cone at regular intervals under a microscope and to replace it if it shows signs of wear or if it leaves the specified dimensional tolerances.

1.3 Heat Aging [31]

The term "aging" describes a large number of processes in elastomers which lead to a chemical and physical conversion and degradation of vulcanized test specimens or finished parts. Several aging test methods have been developed which try to reproduce these damage mechanisms from the everyday use of sealing materials in the laboratory in a kind of "time-lapse".
The oxidation of natural rubber has been known for a long time and was already described by Spiller [32] in 1865. "He found that waterproof rubberized felt no longer had its closed structure and waterproof properties after six years. After extraction of the elastomer and evaporation of the residue, a resinous film was formed which differed from the usual characteristic elastomer film. This product was called "Spiller's resin" and the phenomenon that led to its formation was recognized as oxidation". [33]
BURGHARDT concluded at the end of the 19th century that "the amount of oxygen absorbed by or bonded to the rubber is a measure of the degradation experienced by the rubber". [34]
In 1885 THOMSON also noticed a clear oxidation by atmospheric oxygen at high temperatures. Therefore, he suggested to produce elastomer products under vacuum, which WEBER already recognized around 1900 as an unnecessary measure. [35]
The "Admirality Test", commonly used around the turn of the last century, subjected the specimens to one hour of "dry heat" in an "air bath" at 270°F (132°C) and three hours of "wet heat" in an autoclave at 320°F [36] (160°C). This test was not an aging test in the current sense, but was intended to investigate whether the compound was degraded by the addition of substitutes. [37]
In industry, however, there was a growing interest in obtaining information on the aging behavior of elastomers in fast motion.

In the period that followed, various laboratories carried out attempts to artificially simulate the aging of elastomers. Dr. W.C. Geer's method, which he had developed in the laboratories of B.F. Goodrich Co. since autumn 1907 and which he presented to the public for the first time in September 1916 [38] at an ACS symposium on "The Accelerated Life Test of Rubber Goods", had gained acceptance. GEER used pressureless circulating air furnaces. The term "Geerofen" was common in Germany until the 1960s, but is hardly used today. In the meantime, the term warming cabinet or aging furnace is usually used. During storage, GEER paid particular attention to a sufficient supply of hot fresh air circulating in the oven.
In 1916 he described his test conditions as follows: The specimens were placed in the furnace at 160°F (71°C) for a period of two weeks. Every day 3 specimens were taken, which then rested at room temperature for 24 hours until they were tested for tensile strength and elongation at break. Finally, the results were recorded in a diagram documenting the degradation of the material over time. In addition, specimens subjected to natural aging over long periods of time were compared with those from hot-air aging. GEER produced diagrams for natural aging with a monthly scale and for artificial aging with a daily scale. Since the curves were quite similar here, the test method could be classified as realistic.
Later, the "Geer aging test" was understood to mean the aging of the samples "in a drying cabinet with circulating air at 70° for 7, 14, 28 days (...) in most cases, after which the stress values are determined. [39]
While in the 1950s storage in pressure vessels (e.g. Bierer Davis bomb: storage in compressed oxygen at 21 bar) was still common practice, it no longer plays a role in today's day-to-day testing, since this form of aging deviates greatly from the reality of most sealing applications.
In 1952 GILLMAN and HAINES [40] proposed an aging test in a "water bomb". The samples were stored in an autoclave in water containing a strong oxidizing agent such as potassium chlorate. Samples of the liquid were taken regularly to check its oxidizing effect. However, this test method no longer appears in today's literature.
In the meantime, the aging test, which goes back to W.C. Geer, has completely established itself in today's testing practice alongside the less common cell furnaces and is described in detail in the following standards: ISO 188 (Edition 10-2011), DIN 53508 (Edition 03-2000), ASTM D 573-04 (Reapproved 2010). In the current DIN 53508, the preferred test conditions are the 7 days at 70°C which have become rare today and which presumably can still be traced back to their "inventor" Dr. W.C. Geer.

1.4 Compression Set [41]

Today, compression set testing is used for the comparative evaluation of formulations, for testing finished parts, for checking the technical suitability of elastomers and for obtaining information on the long-term behavior of sealing materials.
The determination of the compression set was first standardized in Germany in December 1940 in the DIN 53 511 Sheet 3 Cross Edition ("Testing of Rubber Elastic Behavior of Soft Rubber Measured After Compressive Stress with a Certain Compression Size").
The term "compression set" does not appear in the standard German-language literature of the first half of the 20th century. However, methods similar to compression set testing were already being investigated in the 19th century, although mostly with different questions than those mentioned at the beginning.

The first scientific study on the behavior of rubber under pressure appeared in 1856 [42]. At that time rubber was a much-wanted material for the buffers of the still young railway.
The compression tests of STÉVART [43], who published about it from 1871, were groundbreaking. He compressed rubber rectangular rings with different diameters and heights at room temperature. As he varied these parameters, he observed the effects on the cross-section geometry (bulging, buckling, etc.). In the case of a rectangular ring, he was thereby able to prove the importance of the sample dimensions in compression tests. This insight also applies equally to today's compression set and for many decades there have been specified specimen dimensions which are almost identical between ISO and ASTM standards and therefore lead to comparable results.
In the German standard work on elastomer testing "Der Kautschuk und seine Prüfung" [44] from 1910, an apparatus for measuring the permanent strain of ring specimens is presented on page 214ff. The strain was not caused by a predetermined strain path, but by load weights. Whether the test was also carried out at elevated temperatures is not apparent from the reference, although it is very unlikely, since heat aging of rubber materials was not described until 1916 in the USA.
The Handbook of Rubber Science of 1930 describes in more detail so-called stretch test specimens, which show a certain similarity to the tensile set used today, although without the influence of temperature. MEMMLER and SCHOB write about the "compression test", which can be regarded as a kind of precursor of the compression set: "The determination of elastic behavior by measuring the deformation set after prolonged exposure to compression forces, analogous to the stretch test, is hardly used for pure material tests, but can be found here and there in acceptance regulations for sealing rings and similar soft rubber goods". [45] This quote shows that compression sets were primarily used to determine elasticity properties, but not cross-linking properties.
In the "Handbuch der Gesamten Kautschuktechnologie" [46] (Handbook of the Entire Rubber Technology) of 1935, pressure tests contain only references to the above-mentioned sources and one English reference.
In American and British literature, on the other hand, one finds explanations and treatises on the subject much earlier. The American ASTM standard on compression set was published for the first time in 1934 [47] and therefore 6 years before the corresponding DIN.
The fast and rapidly growing automotive industry in the USA required vibration-damping elastomer components relatively early on. This circumstance was probably the driving force behind the in-depth search for test methods for the behavior of elastomers under pressure. For this reason, testing of the compression set is still carried out in the USA not only using a constant compression travel, but also more often alternatively using a constant compression force (e.g. as useful for testing engine bearings).
A comprehensive treatise on compression sets can be found as early as 1930 at ABBOTT [48], but with special attention to compression under a constant load. The groundbreaking catalogue of requirements [49] and the checklist for compression sets contained in this article has been answered and fulfilled in almost all points by the regulations of today's standards.
The realization that compression set is also a simple and excellent test method for determining the degree of crosslinking of an elastomer component was probably not yet common knowledge in the USA in the 1930s. In 1937 CARPENTER wrote only that "the high temperature [during compression set] increases the effect of plastic flow and allows a certain accelerated aging, which usually leads to a reduction of the re-deformation force of the

specimens". [50] This knowledge about the possibility of investigating the crosslinking state by means of DVR seems to have only been gained later.
In the meantime, compression set testing has become an essential part of the series of standard physical testing methods for rubber. With the international standard ISO 815 (09-2014), there are now clear requirements for this test method to be carried out reproducibly worldwide.

2. Why are These "Old" Tests so Important in the New O-Ring Material Standard ISO 3601-5? (B. Richter)

2.1 The New O-Ring Standard ISO 3601-5

Before the new edition of the O-ring standard is briefly presented, the most important functions of a standard should again be pointed out:
The user should be sure that the standard described represents a good state of the art. Furthermore, it should be possible for different manufacturers to manufacture these standardized products at reasonable costs. These two important aspects of a standard are reflected in the new edition of ISO Standard 3601-5.
Since O-rings, like other elastomer seals, also have a multiplicative relationship between formulation quality and processing quality, which means that one has no value without the other, the greatest challenge of the new standard was probably to define both formulation requirements and specifications for the degree of cross-linking of the O-rings and to find a consensus on this at an international level. In addition, however, it was also necessary to determine to which groups of materials the standard should actually refer.
This point was solved as follows (see Table 1 of the standard, NB: The number behind the elastomer is the hardness in IRHD,M):

- NBR 70/90-sulphur cross-linked (S)
- NBR 70/90 peroxide cross-linked (P)
- HNBR 75/90
- FKM 70/75/80/90
- VMQ 70
- EPDM 70/80- sulfur cross-linked (S)
- EPDM 70/80- peroxide cross-linked (P)
- ACM 70

This was a total of 16 materials defined, whereby it is certainly possible to discuss whether a Sulphur-crosslinked EPDM material or an ACM material belongs to the standard repertoire of O-ring materials, whereas an FVMQ and FFKM material are missing.

2.1.1 Hardness Specifications in the New ISO Standard 3601-5

However, the most important aspect of the standard is then found in Table 2 of ISO 3601-5, in which hardness values are now defined directly at the O-rings for all the material families

described above, as well as compression set values. Therefore, these two tests described above represent the basic framework of this standard, but not as a material characteristic, as is known to the typical user, but as a finished part test. All this for cord thicknesses from approx. 1 mm upwards (smallest dimension according to ISO 3601-1 0.74x1.02 mm).
With regard to hardness measurement (IRHD according to ISO 48 M or CM), it should be noted that with the state of the art for O-rings from 1 mm cord thickness, a laser-guided measuring table (as offered by several hardness tester manufacturers) enables a person-independent, well reproducible hardness measurement. In addition, Table 2 of the standard for O-rings with a cord thickness of < 1.6 mm permits a hardness range (+5/-8) extended by 3 points downwards. Experience has shown that the geometry-related influence of the lower material displacement on O-rings with small cord thicknesses when the hardening needle penetrates is compensated for by a material that is more densified and cross-linked during vulcanization, also due to the higher ratio of the free surface to the mass of the O-rings.

2.1.2 Compression set requirements in the new ISO Standard 3601-5

t=24h	**NBR S 70**	**HNBR 75**	**FKM 70/75/80**	**EPDM P 70/80**
Test-T [°C]	100	150	200	150
Comp. set max, [%] (d2 min.=2.62mm)	35	40	25	30
Comp. set max, [%] (d2 < 2.00 mm)	40	45	30	35

Tab. 1: Compression set nominal values of the most important O-Ring Materials acc. to ISO 3601-5

t=336h	**NBR S 70**	**HNBR 75**	**FKM 70/75/80**	**EPDM P 70/80**
Test-T [°C]	100	125	175	125
Comp. set max. [%]	60	60	40	40

Tab. 2: Compression set nominal values of the most important O-ring materials according to ISO 3601-5 (standard test buttons B)

In the compression set test on these small cord thicknesses, it must of course be considered that greater random scattering can occur here due to the higher measurement uncertainties during height measurement, which has also led to a 5% increase in the nominal values for cord thicknesses of less than 2 mm. In addition, many years of experience in the O-Ring Prüflabor Richter have shown that the limit values given in Table 2 are at least 10-15% (absolute) higher than what is technologically possible in large quantities and under consideration of random scatter. A nominal value of 24h/150°C of <35% for a peroxide cross-linked EPDM 70 O-ring, for example with a cord thickness of 1.5 mm, does not represent a particularly strict limit value. In the O-Ring Prüflabor Richter, such O-rings have been tested over many years with results of approx. 10-20%, and not by the "premium O-ring suppliers", but rather by those from the second row.
However, the material properties hardness (IRHD) and compression set are of course also required as specifications for the description of the formulation qualities, which can be found in Tables 3 to 10 of ISO 3601-5. The decisive difference in these tables, however, is that these requirements refer exclusively to standard specimens, which are ideally vulcanized test specimens, usually produced in a laboratory. This also includes the standard O-ring 24.99 x

3.53, which may be used as an alternative to the usual standard specimens for hardness, compression set and tensile test. Particularly remarkable about the target values for the compression set is the fact that, in addition to the short-term specifications for 24 and 72 h, target values are also defined over a test period of 2 weeks. After all, a good short-term compression set is no guarantee of good long-term performance. Influences of the polymer (e.g. diene content in EPDM), the viscosity of the polymer or the plasticizer content in the formulation can lead to considerable differences between elastomer materials with the same short-term behavior. Therefore, the user of O-rings is well advised to apply this standard also with regard to the formulation specifications.

2.1.3 Specifications for the Tensile Test in the New ISO Standard 3601-5

The tensile strength also reflects properties which not only indicate the load limits but also, to a limited extent, the long-term behavior. The above-mentioned influence of a low viscosity of the polymer or a low average molecular weight leads to lower tensile strength values as well as the inappropriately high quantities of plasticizers in the compound. Tensile strength is therefore an important criterion for the quality of a compound, even if the load limits themselves are only important in a few applications, such as dynamically stressed O-rings or applications with high pressures.

ISO 37	**NBR S 70**	**HNBR 75**	**FKM 70/75/80**	**EPDM P 70/80**
Tensile Strength [MPa]	12	16	10	10
Elongation at Break [%]	250	200	150	150/120

Tab. 3: Required tensile strengths of the most important O-Ring materials according to ISO 3601-5 (standard test rods)

2.1.4 Specifications for heat aging in the new ISO standard 3601-5

The additional testing of hot-air storage mentioned above is now, so to speak, the third type of assurance of compound quality because the above-mentioned influences of the polymer and plasticizers can also have a negative effect on heat resistance. At the same time, the user gets a realistic idea of how much the material changes under the influence of high temperatures. For this purpose, specifications are made after 70 and after 168 hours.

t=168h	**NBR S 70**	**HNBR 75**	**FKM 70/75/80**	**EPDM P70/80**
Test-T [°C]	100	150	200	150
Change in Hardness [IRHD,M]	max.+10	max. +10	max. 6	max. 12/10
Change in Tens. Strength [%]	+/-25	+/-25	+/-15	+/-40
Change in Elong. at break [%]	+/-40	+/-30	+/-25	+/-50

Tab. 4: Required heat resistance of the most important O-Ring materials according to ISO 3601-5 (standard test rods)

It can therefore be seen that these above-mentioned material tests are still of great importance today, and it would be unthinkable to imagine the operational practice of seal users without them.
For the sake of completeness, it should also be mentioned at the end that the new ISO 3601-5 also provides limited requirements with regard to swelling resistance and low-temperature flexibility, which is partly explained in detail under [51]. This standard therefore contains appropriate limit values for all important material properties, which is expected to lead to a high level of acceptance among users.

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