TECHNICAL PAPER

Compression Set — what do I need to know?

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Compression set testing, as outlined by ASTM D395 and ISO 815 is often quoted on material datasheets, often as a single value at one time and temperature. Once it has been established exactly what this value represents, it is important to give careful consideration to how this can be appropriately applied to a given sealing application, and how exactly it is linked to sealing efficacy.

Compression set testing, measures the amount of recovery in a rubber sample following the removal of a strain, usually the application of a 25% deflection, applied for a given time at a set temperature. The intention is to give an insight into the retention of the reaction forces to the strain imparted by hardware and the ability to form a seal. The results obtained are often clouded by the presence of so called 'cold set' a phenomenon which is discussed in detail below.

What is compression set?

Compression set is a laboratory test focusing on the ability of an elastomeric button sample to recover after the removal of an applied deflection. The procedure is defined by ASTM D395 and ISO 812, most typically the deflection is from the application of a nominal strain of 25%, ASTM D395 also specifies testing under a constant force; however, this is very rarely used. Figure 1 gives a schematic of a typical set-up for the case of constant deflection.

The time and temperatures at which the samples are exposed for are not defined; however typical times and temperatures for various material types are given in Table 1.

	Temperature (°C)	Time (hrs)
EPDM	125	24
NBR	100	24
HNBR	150	24
FKM	200	24
FFKM	200	72

Table 1 — Typical times and temperature of compression set, by material family

Table 1 shows typical times and temperatures that are usually stated on a datasheet however longer times and different temperatures are often also stated to provide a broader understanding of how the behaviour of a material evolves with time and temperature.

Following exposure, the seal is immediately removed from the jig and after 30 minutes at standard temperature its height (thickness) is measured. Compression set is then calculated as using the formula in Figure 2.

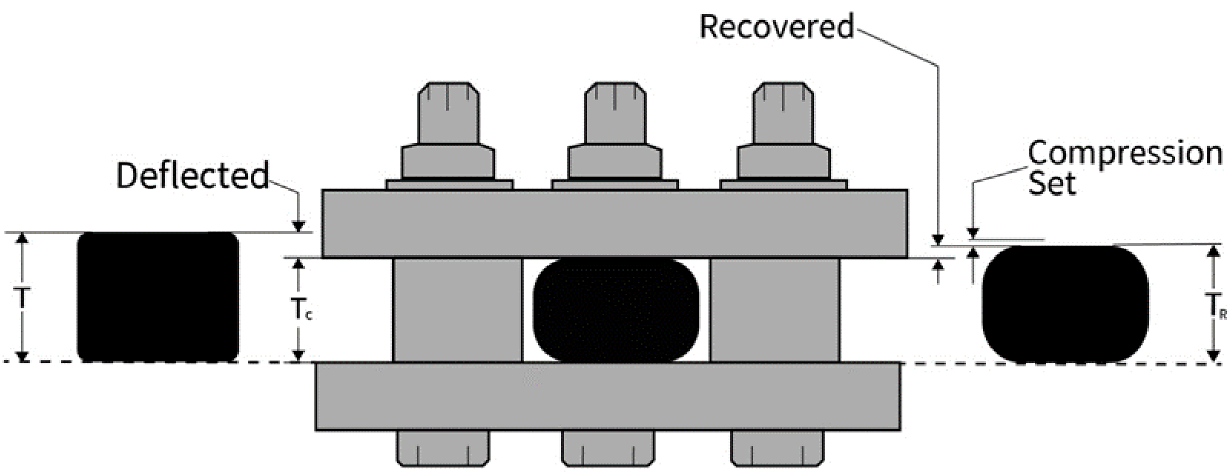

Figure 1 — Typical compression set up

$$\text{Compression Set} = \frac{T_I - T_R}{T_I - T_C} \text{ X } 100$$

T_I = Initial thickness
T_C = Compressed thickness
T_R = Recovered thickness

Figure 2 — Formula for calculating compression set

This means that complete recovery would be 0% compression set, while no recovery would be 100%, the lower the compression set value, the more recovery, the better a result. It should be noted that ISO 815 also allows the full jig to be cooled to room temperature before removal. The cooled button is then left before a measurement is taken after 30 minutes recovery; this would introduce significant amounts of 'cold set' — a phenomenon which will be discussed in detail in a later section.

Why do we measure compression set?

In order to form a seal, there is a requirement for the elastomeric sealing material to produce a reaction force to applied deformation by the hardware, this sealing force is produced as a result of the cross links within the sample. As the sample is exposed to heat and media these bonds can break and reform in the compressed state, and/or completely new cross links can form in the compressed state, this will lead to a reduction in the sealing force and will reduce sealing efficiency. Compression set is an attempt to probe this reduction.

Typical compression set results

Figures 3-6 illustrate extended testing (56 days total with sampling points at 1, 3, 7, 14, and 28 days) for an HNBR, FKM, FEPM, and FFKM; for three temperatures, 100°C, 150°C, and 200°C.

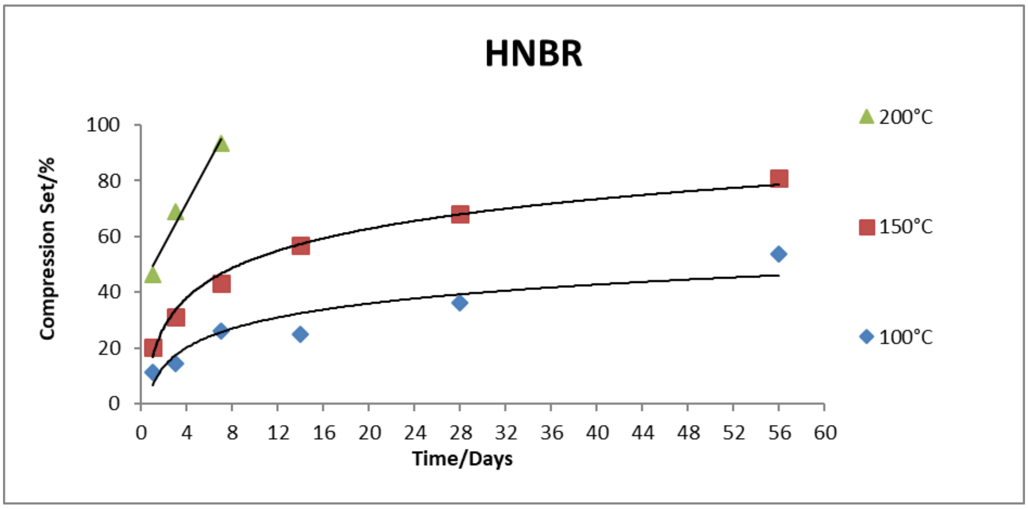

Figure 3 — Compression set testing for a typical 90 IRHD HNBR

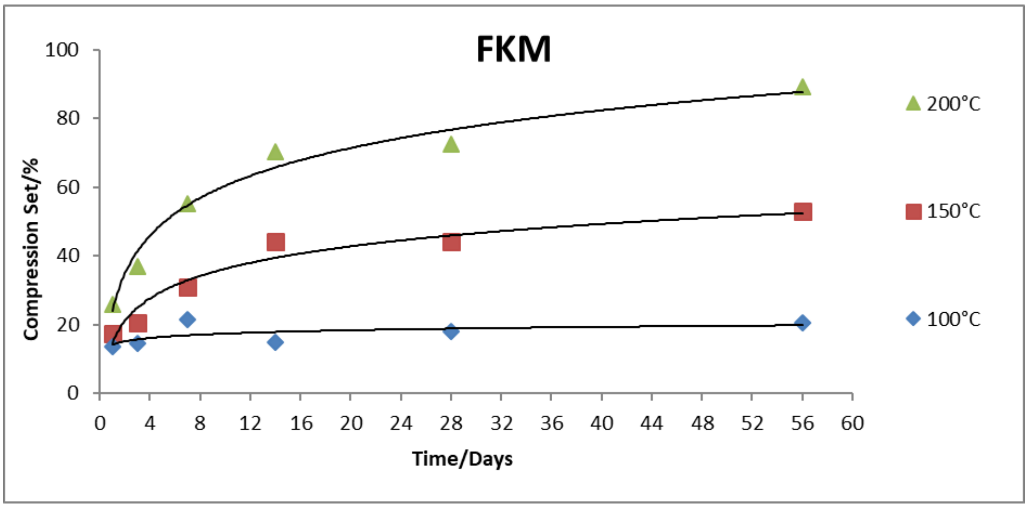

Figure 4 — Compression set testing for a typical 90 IRHD FKM

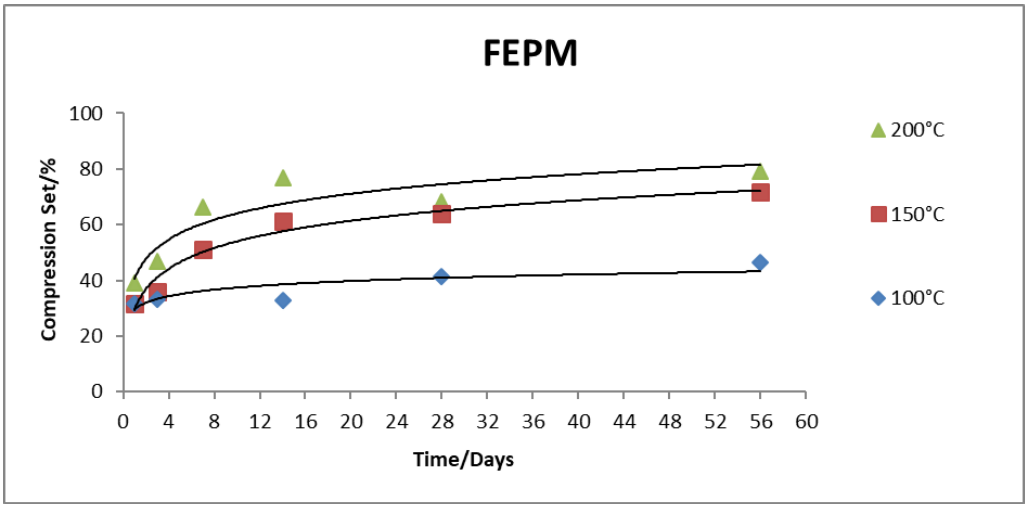

Figure 5 — Compression set testing for a typical 90 IRHD FEPM

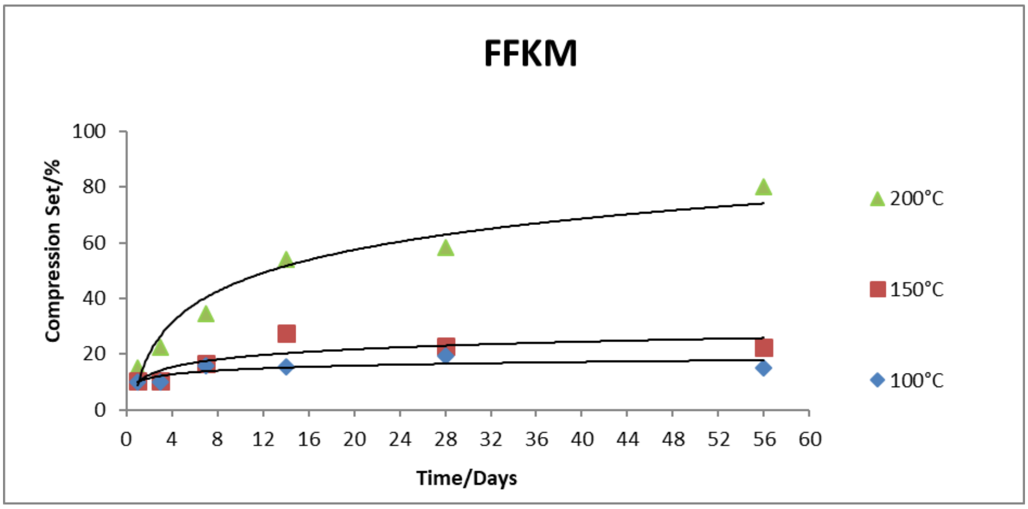

Figure 6 — Compression set testing for a typical 90 IRHD FFKM

There are several things to note from the above Figures. Firstly, that in most cases the set initially increases quite rapidly before plateauing; hence care must be taken if presented with only one data point; what is of greater use is taking the time to establish the longer term trend of the material at the temperatures of interest. The one exception is in the HNBR at 200°C, where the trend is in a straight line, and 100% set has been reached after just 7 days. If we consider the upper operating limit of HNBR of 180 °C the reason for this becomes clearer, the useful operating window of the material has been exceeded and the increased cross linking in the compressed state has led to a large amount of set. To further this point let us consider the results we have for 100°C, we can see that the results of FFKM and FKM are similar, whereas those of HNBR as significantly worse. However, at 150°C, there are clear differences between the FKM and FFKM, representing the increase in thermal stability of FKM v FFKM.

One final point is the very high results for FEPM, they very quickly reach ~40% compression set, however these types of materials are often rated to 250°C and have been used successfully for many years. The large initial result is attributed to ‘cold set’

The effect of cold set

Elastomers are primarily made up of numerous long molecular chains, the ability of these chains to move is part of what leads to the elasticity; this movement requires energy, with more movement being possible at higher temperatures and less at lower temperatures. There is a point, known as the glass transition (T_g), at which there is no longer enough energy in the compound for any movement and the seal becomes glassy and brittle. This means that a lower T_g is evidence of greater chain mobility at any temperature, the reverse being true for a high T_g.

150°C			150°C to ambient		
From jig	After 30mins	After warming	From jig	After 30mins	After warming
0%	**3%**	**1%**	**45%**	**44%**	**0%**

Table 2 — Results from an investigation into cold set in HNBR

When a seal is exposed to stresses the chains will attempt to orientate themselves to reduce the applied stress, chains move to adopt the lowest stress position possible. This means that while the seal material is under stress during compression set testing the chains are moving to reduce the experienced stresses. It is possible that when the deformation is released and the temperature has reduced, that part of the observed set is not due to permanent formation of chemical bonds but as a result of chain movements that have taken place at high temperature, but do not have the required energy to relax at lower temperatures.

The measured compression set has two components, permanent set as a result of chemical bonds which have formed while in the compressed state and prevent full recovery; and so called 'cold set' which is stresses introduced under high temperatures which cannot relax at lower temperatures. If a seal that presents with set is warmed in an oven then the portion that is cold set will relax, the effect is often observed in parts that have been allowed to cool in situ, be this before removal or as the result of a planned shutdown.

This cold set effect is demonstrated in Table 2, two sets of HNBR samples were placed into an oven, taken to 150°C and then held for 1 hour. One jig was removed, and the compression set calculated straight from the jig, after 30 minutes recovery, and again after 30 minutes unconstrained warming in an oven at 150°C. The second jig was allowed to cool to ambient temperature before compression set was calculated again directly from the jig, after 30 minutes, and again after 30 minutes unconstrained warming in an oven at 150°C. In both cases the samples were cooled after warming before a measurement was taken.

At 150°C almost no set was observed in any of the samples. The fact that set was at its lowest direct from the jig reflects high coefficient of thermal expansion of rubber, the reduction in height after 30 minutes reflects the contraction of the sample. In the sample that was allowed to cool within the jig significant set was observed direct from the jig with little recovery. However once the sample was warmed this set all but disappeared. The above is why the option to cool samples in a jig will produce very poor results as per ISO 815.

The phenomenon was further probed in the four materials for which compression set was reported above, through the following experiment. Samples of all four materials were held at 25% compression at typical ASTM D395 test times and temperatures; namely 24 hours at 200 °C for FEPM, FKM, FFKM, and 24 hours at 150 °C for HNBR. After this the compression set of one set of samples for each material was measured, as per ASTM D395, the sample was then allowed to recover unconstrained for 1 hour at test temperature, compression set was then reassessed. A second set of samples were allowed to cool for 30 minutes constrained before compression set was assessed, the sample was then allowed to recover unconstrained for 1 hour at test temperature, compression set was then reassessed. The results are shown in Figure 7.

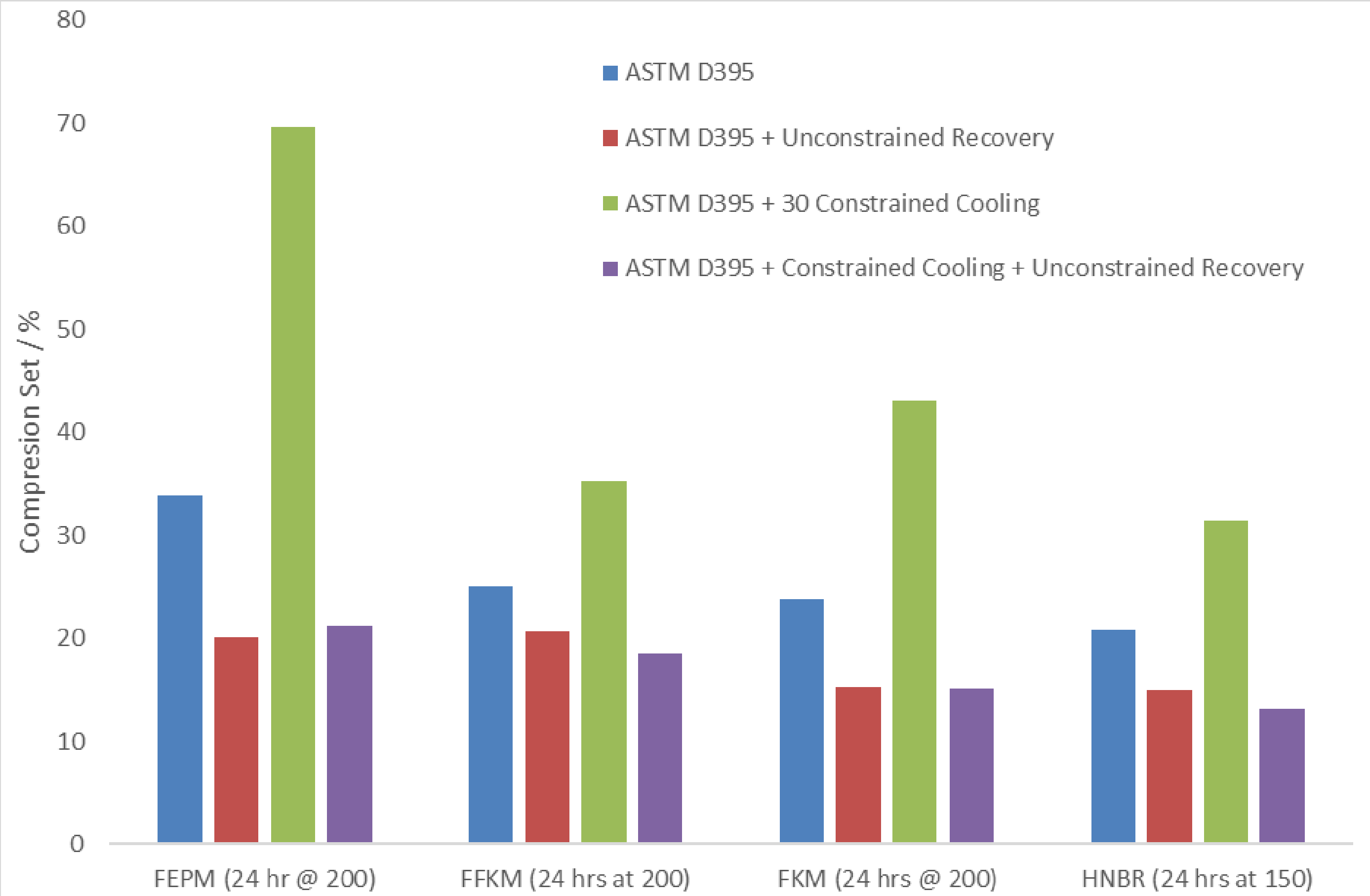

Figure 7 — Results from investigation into cold set

The results show that even for results measured as per ASTM D395 a degree of cold set was apparent in the result, this was most pronounced in FEPM with 40% of the observed set being attributable to 'cold set'. As would be expected the amount of set measured increased substantially when the samples were allowed to cool constrained, reflecting a higher number of chain movements which could not relax. In all cases it was observed that both samples exhibited broadly the same amount of set following unconstrained recovery, irrespective of intermediate cooling, this being the amount of set that was a consequence of permanent changes.

If we return to the case of FEPM, the T_g of these types of materials is around 0 °C, this can be compared to ~-10 °C for a typical FFKM, ~15 °C for typical Type 2 FKM and ~-30 °C for a medium ACN HNBR. This means that we could expect the cold set effect to be more pronounced in FEPM type materials as the high T_g reflects low chain mobility.

There is another type of testing which seeks to return similar sorts of information as compression set, this being compressive stress relaxation (CSR) as covered by ASTM 6147/ISO 3384. What CSR seeks to do is give the actual force being exerted by a sample; the measurement can be continuous as shown in Figure 8(a) or non-continuous as show in Figure 8(b). The jig as shown in Figure 8(a) is placed within an oven with typically 25% compression applied to a rubber sample placed between the plates, a gauge returns the reaction force which is then recorded. In the case of non-continuous testing, jigs as shown in Figure 8(b) are placed within an oven and are periodically removed from the oven and the force required to compress the sample a small amount is used to calculate the force returned by the sample. Again, the results are typically reported as a percentage, but this is the percentage of the original force exerted now being experienced. A loss of 50% of initial force is often given as an end point of an experiment.

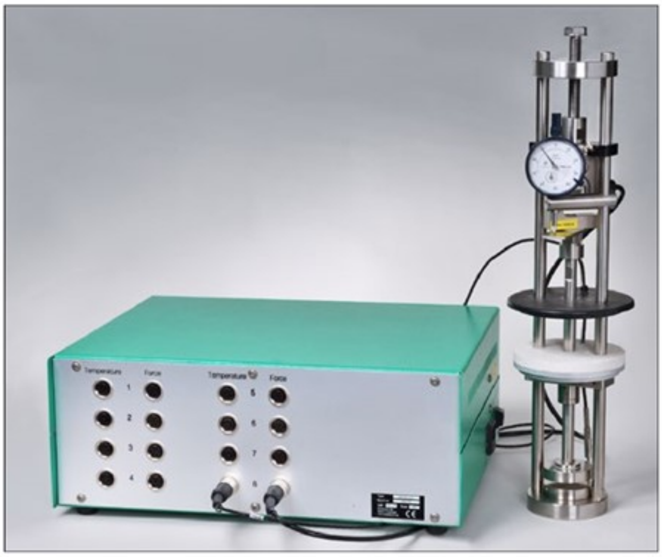

Figure 8(a) — Continuous CSR equipment

Figure 8(b) — Non-continuous CSR equipment

Shown in Figure 9 are CSR curves from continuous measurement for an FEPM material at 200°C, a range of FKM materials are shown for comparison.

In testing the FEPM has performed the best, retaining the highest proportion of its initial sealing force. The next best performers were the terpolymers, followed by the copolymer. In compression set testing the trend would typically be reversed, however the above CSR testing contains no component of cold set as all the measurements are taken at the test temperature. This further suggests that a factor in the poor performance of FEPM materials in compression set testing is the fact that although every effort is made to dissemble the jigs at the test temperature, some degree of cooling will have taken place and the results are greatly influenced by its poor chain mobility, and cold set effect.

Issues with compression set testing

Alongside the difficulty of being able to separate cold set effects from the permanent, irreversible formation of new crosslinks under compression; all the testing shown above took place in air, under laboratory conditions with no applied pressure. As with a lot of rubber testing although some useful comparative data has been given, it can be hard to apply this directly to application conditions.

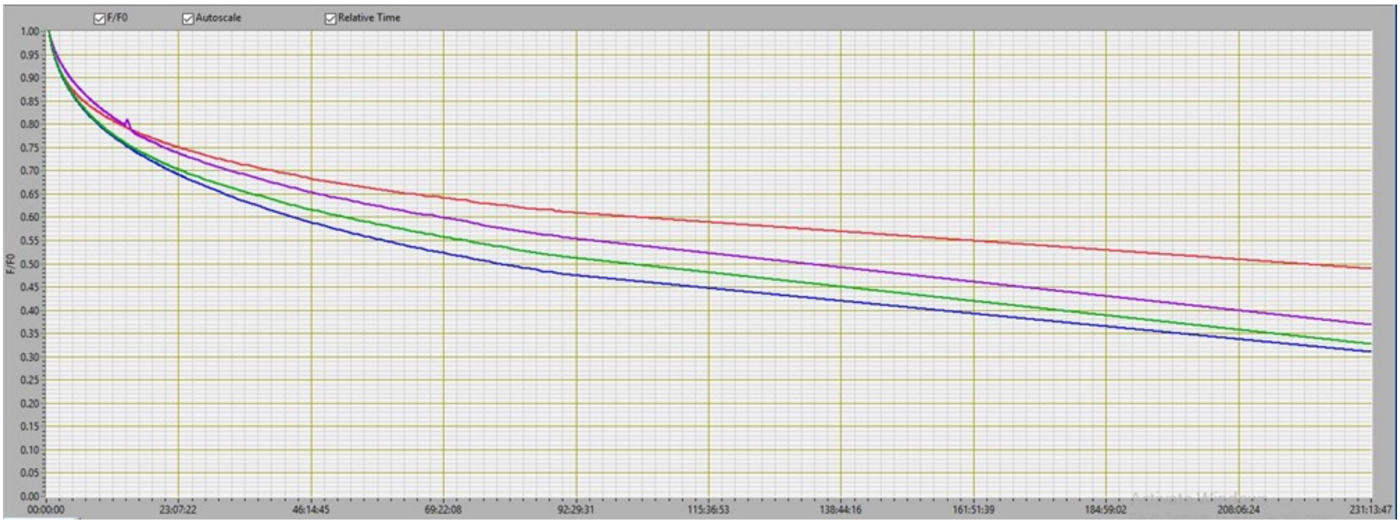

Figure 9 — CSR curves for FEPM (red), FKM Terpolymers (green and purple), and FKM co-polymer (blue)

As an example of why this can be an issue, consider the following: A seal user wished to more accurately reflect application conditions so requested compression set testing be carried out in a sour fluid as below:

Vessel Contents	10% Water Phase 60% Hydrocarbon Phase 30% Gas Phase
Gas Phase Composition	25% H_2S 5% CO_2 70% CH_4
Hydrocarbon Phase Composition	70% Heptane 20% Cyclohexane 10% Toluene

The exposure temperature was 100°C and time was 72 hours.

A problem often encountered when testing in media other than air is that it is often not safe to remove test jigs from exposure vessels at the test temperature, so the jigs are often cool when they are disassembled. This means that care must be taken so that cold set does not obscure the true result. It is for this reason that Precision Polymer Engineering recommend that when jigs have had to be removed from the test media while cool, the samples are warmed back to test temperature constrained, if possible, or unconstrained, before reverting to the standard method.

In the testing outlined above the low temperature FKM had a compression set of just 4%.

The addition of the warming step was a concern, as it is a large deviation from the established test method. So, the following validation was carried out:

- Compression set in air for 72 hours at 100°C, as per ASTM D395: 14%
- Compression set in air for 72 hours at 100°C, jigs cooled to ambient: 21%
- Compression set in air for 72 hours, jigs cooled to ambient, unconstrained buttons warmed to 100°C for two hours: 12%

The results show that allowing the jigs to cool and then calculating compression set would have returned a result with considerable amounts of cold set, and that the results as per the standard did include a small amount of cold set effect. However, the results from tests 1 and 3 were very consistent, and the additional warming step was not considered to have returned an unduly favourable result.

A final point of interest is that the result in air is significantly worse than that in the sour fluid. Although initially surprising it is worth remembering that 20% of air is oxygen, a highly reactive species. A fluid may at first glance be considered more aggressive based on its effect on the human body as opposed to the chemical bonds in elastomers.

Key points and conclusions

- Compression Set is the recovered height of a rubber sample after the removal of an applied strain, usually through the application of a constant deflection of 25%, as per ASTM D395 (ISO 815). 0% is fully recovery, while 100% is no recovery at all.
- Another related method is compressive stress relaxation (CSR) [ASTM 6147/ISO 3384]; this measures the retained force being returned by a sample compressed by typically 25%.
- Compression set is often reported on datasheets; however, the time scales are often very short. When longer time scales are used it is often that although the set initially increases rapidly it then reaches a plateau.
- There will be a point at which the set continues to rapidly increase and no longer plateaus, this will be at, or around, the upper operating temperature limit of the material for a given fluid.
- To fully understand the behaviour of a material it is often necessary to carry out testing over a range of times and temperatures.

Key points and conclusions cont.

- The observed set has two components, the most concerning is permanent addition of crosslinks in the compressed state which prevents fully recovery. The second is so called cold set. This is where chain movements that were possible at higher temperatures are not able to relax out at lower temperatures and is most pronounced in samples that are allowed to cool while compressed. Elastomers with poor chain mobility such as FEPM are most susceptible to cold set.
- When seals are heated unconstrained, any cold set present will relax out leaving only permanent set. It was shown through experimentation that increasing 'cold set' was related to poor chain mobility (T_g is ~0 °C in FEPM as opposed to ~-15°C in FKM), further it was shown that the underlying set due to permanent change was consistent.
- FEPM materials are often considered to be poor in compression set testing as they can take on around 40% set after just 24 hours, whereas FKMs would be closer to 20%. However, CSR shows that the retained force in FEPM is higher than that of FKMs. This further suggests that the poor chain mobility leads to significant cold set effects in standard compression set measurements.
- Compression set is usually measured under laboratory conditions, at a constant temperature in air without any applied pressure; this can make the results difficult to apply to real world environments.
- Where attempts are made to replicate real world exposure, it can be hard to make compression set measurements while avoiding the effects of cold set as safety considerations can make the removal of samples whilst warm difficult.
- The large oxygen component of air can make it a deceptively aggressive media. In fact, an FKM performed better in compression set testing in a sour fluid than it did in air.
- Care must be taken to properly assess testing results and apply them usefully to seal selection and compound assessment.

Talk to your seal supplier to make sure you have the correct data to make informed decisions on your sealing system. For the most critical applications it is prudent to consider a bespoke testing regime.

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