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Testing of nitrile rubber joints for a deep submergence vehicle

Davies Peter [1,*], Le Gac Pierre Yves [1], Ciausu Viorel [2], Gallois Herve [3]

[1] IFREMER Centre de Bretagne, 29280, Plouzané, France
[2] IFREMER, Centre de Méditerranée, 83500, La Seyne-sur-Mer, France
[3] GENAVIR, 83500, La Seyne-sur-Mer, France

* Corresponding author : Peter Davies, email address : peter.davies@ifremer.fr

Abstract :

This paper describes an experimental study of the long term sealing performance of nitrile rubber joints on a manned deep ocean submersible operating down to 6000 m depths. An initial formulation showing large compression set is discussed first. Then results from long term laboratory testing of a second formulation, aged in water for 10 years, are presented. Much lower compression set and higher sealing forces are observed. Based on these results a testing methodology for the prediction of the long term sealing performance of nitrile O-rings is proposed. Results from laboratory tests are compared with those measured on joints aged for up to 10 years in service.

Highlights

► Compression tests performed after immersion over a period of 10 years are reported. ► A laboratory prediction methodology for long term sealing behavior is described. ► Relationship established between compression set and sealing force: ► Leakage criterion proposed. ► Laboratory test results are compared with results from an in-service underwater application and indicate a reasonable long-term correlation.

Keywords : nitrile, compression set, underwater, sealing, O-ring

1. INTRODUCTION

Elastomeric seals and joints are often critical components in underwater structures. They are subjected to a combination of mechanical loads and the marine environment for long periods. Evaluation of the durability of elastomers for marine applications has been studied by various authors. For example, the influence of seawater immersion on different mechanical properties has been discussed [1-3], while Gillen and colleagues have examined accelerated test methods for sealing joints in some detail [4-6]. The loading of interest in a sealing application is compression, and the property which is most commonly used to follow changes in joint behavior is compression set. This is the permanent deformation after unloading and provides an indication of remaining elasticity after prolonged compression. Such tests have been performed for many years, and various test standards exist for compression set; the most popular are ASTM D395 [7] and ISO 815 [8]. The former has been used for over 40 years. Jahn and Bertram published a study on nitrile rubber using this test method in 1973 [9], performing tests over a wide range of temperatures. Compression set measurements have also been used to evaluate sealing systems for NASA docking and berthing systems for space missions [10]. The data given on material datasheets usually correspond to the ASTM standard procedure B (performed under displacement control). A disc sample is compressed in a fixture, typically by 25% of its height, and heated for a number of hours. The fixture is then cooled uncompressed for 30 minutes before measurements are taken. Turland [11] has discussed these tests and indicated the following limitations:

- The test methods do not account for temperature and pressure variations in service.
- Tests are often performed at high temperatures to accelerate effects.

He concludes that as a result, compression set results often bear no relation to the actual sealing application. The shape of the samples, usually discs in the standard methods, may also be quite different from the application seal. There are few alternative tests but Jaunich et al [12] suggested performing compression set tests in DMA (Dynamic Mechanical Analysis) equipment, to provide additional information on time and temperature dependence of compression behavior. Joubert et al also examined the time dependent behavior and proposed a linear viscoelastic model of the compression set test, as part of a study of the elastic recovery of crosslinked networks [13].

There have been various laboratory studies to investigate factors which influence permanent deformation after compression. For example Mostafa et al. [14] studied NBR and showed that compression set increases with increasing carbon black content. Rattanasom et al. [15,16] examined the influence of various fillers on properties of natural rubber vulcanizates and found an increase in compression set as filler content increased. They suggested that this may be caused by poor filler dispersion or filler-rubber interactions. Several studies have examined the influence of curing conditions; for example, Ehabe et al. [17] showed that the reduction in compression set of natural rubber based vulcanizates was exponential with extent of cure and could be directly related to network modifications. Sensitivity of the compression set test to cross-linking conditions was one of the reasons for early attempts to automate this test [18]. Mohaved et al. [19] examined different cure systems for nitrile rubbers. They noted a strong sensitivity of compression set to sulphur content. Unlike natural rubbers, nitrile rubbers do not crystallize, and are more dependent on fillers to improve their mechanical properties. This study concluded that inclusion of carbon black fillers resulted in less compression set than silica based fillers. Other studies on aging of nitrile rubbers include an investigation of accelerated thermal ageing on O-rings by Morrell et al. [20] which found that the main mechanism resulting in compression set was oxidative degradation. Qian et al. used compression set testing to estimate the lifetime of nitrile rubber aged in transformer oil [21], and Coons et al used this test to study the long term behavior of flexible foams [22]. Kömmling et al. [23] also examined accelerated aging of thick O-rings but were limited by the presence of heterogeneous aging, caused by diffusion limited oxidation effects. Peng et al. applied a time-temperature superposition to compression set data for HNBR [24] to estimate long term behavior from short term accelerated aging. However, apart from the work of Gillen and colleagues [4-6] few studies have compared accelerated ageing results to service experience of sealing devices.

The Ifremer Nautile deep sea submersible or deep submergence vehicle (DSV) is constructed from two titanium alloy hemispheres bolted together to form a sphere of 2.1 meters inner diameter, which contain the three crew members. The upper yellow parts shown in Figure 1a are composites covering buoyancy foam. The overall length is 8 meters, the weight in air is 19.5 tons. The sphere contains elastomeric joints, both to seal the entry hatch and between the titanium hemispheres (equatorial seal, Figure 1b).

(a) (b)

Figure 1. Example of deep sea manned submersible: a) Nautile, intervention to 6000 meter depth, b) detail of interface between hemispheres.

The DSV is subjected to a complete maintenance operation every 5 years, including replacing all the polymer components (PMMA view-glasses, hoses, and seals). When the equatorial joint was removed in 2005 after 5 years in service it showed a significant permanent deformation (compression set), Figure 2.

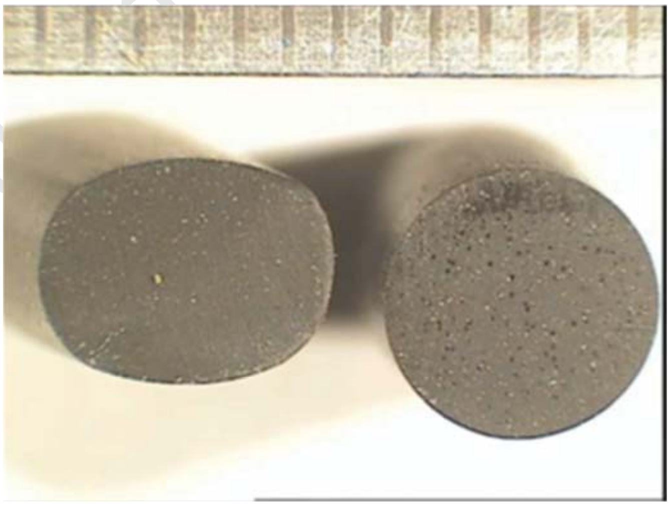


Figure 2. Nitrile rubber joint sections (Original). Millimeter scale.

Left: Joint after 5 years in service. Right: New joint before compression

A test programme was therefore put in place to examine aging effects, in order to quantify their influence on the compression set and sealing force. Two nitrile joint formulations were examined. The first was the grade used from 1995 to 2008 and showed large compression set (Figure 2). It will be referred to hereafter as “original”. This was replaced in 2008 by an improved formulation, which will be referred to hereafter as “new”. This paper describes the results from that test programme, including both laboratory tests over a 10 year period and comparison with results from tests performed on joints removed after service.

2 MATERIALS

The joints on the DSV sphere are all made from nitrile rubber (NBR). This designates a family of elastomers based on copolymers of butadiene (C_4H_6) and acrylonitrile (C_2H_3CN). The acrylonitrile content can be adjusted according to the application; it is increased to improve solvent, abrasion and temperature resistance. The butadiene configuration can also be modified (trans, cis…). Thus even without fillers there are many possible formulations of NBR. Table 1 shows the compositions of the original and new joint formulations. The glass transition temperatures, Tg, of the unaged joint materials were measured by calorimetry to be around -30°C. Hardnesses were measured to be in the range 70 to 80 Shore A.

Joint	Fillers, wt	Carbon black
Original 1995-2008	**23%** SiO_2, $Al(OH)_3$, $CaCO_3$	24%
New 2008-2018	**3%** $CaCO_3$	34%

Table 1. Materials tested and filler contents

Initially tensile tests were performed on both original and new specimens (loading rate 100 mm/minute). Figure 3 shows examples of tensile test plots for samples of both materials, both new and after aging in service on the DSV.

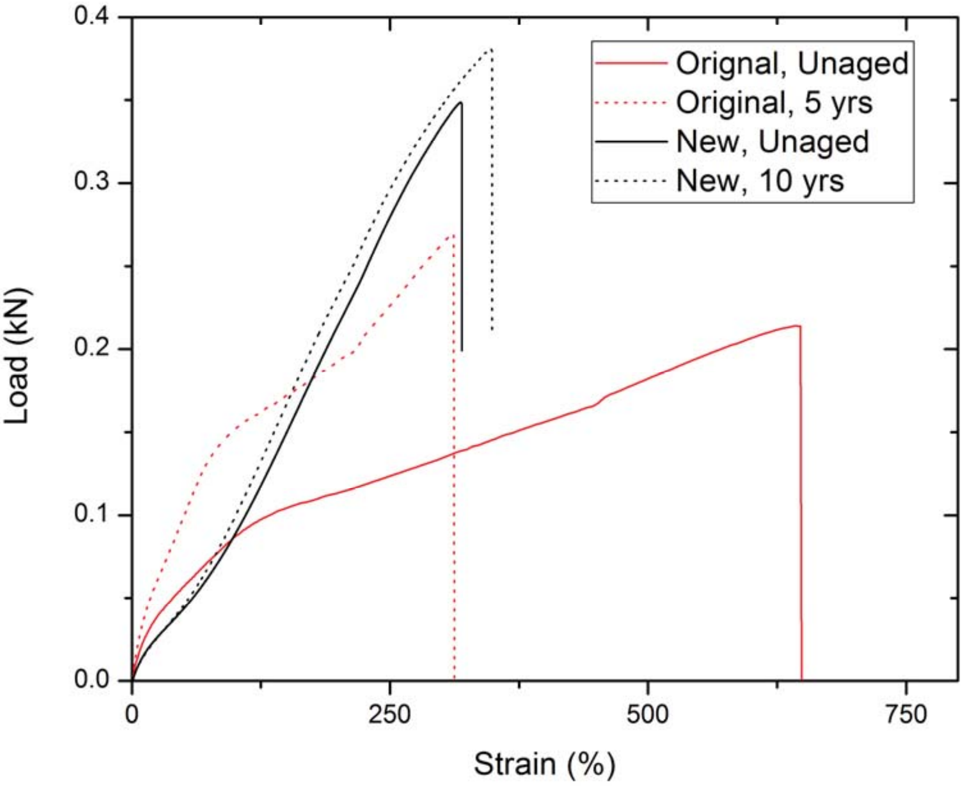


Figure 3. Tensile force-displacement plots for Original and New samples, before and after service.

While the initial slopes up to 100% elongation are similar for both materials before and after aging in service, it is interesting to note that the tensile properties of the new material are unchanged after service, whereas the original material became stiffer. These are both very high elongation materials under these test conditions, but it should be remembered that sealing materials of this kind are produced as a continuous rod. To obtain a circular O-ring joint of the correct dimensions requires assembly, with a self-bonding operation, and this is likely to be the weak point under tensile loading.

3 TEST METHODS

The role of the equatorial joint is to maintain sealing during the first tens of meters of immersion of the DSV. Beyond that depth metal-metal contact takes over. In addition to the compression loading the joint is exposed to sea water and a range of temperatures, up to

40°C on deck, down to 4°C at 6000 meter depth. In some cases, inspection of a shipwreck leaking oil for example, other chemicals may also be present.

A preliminary program investigated the influence of immersion in seawater, and UV exposure; little influence on tensile behavior was noted. Focus in the main study was therefore directed towards two main aspects:

- Measurement of the influence of time under load and temperature on compression set, and
- Measurement of sealing force after aging.

Special steel test fixtures were manufactured, Figure 4, in order to apply the same compression ratio as for the joints on the submersible (a compression displacement of 1.9mm), with the same recess profile, (see Figure 1b). Note that this compression is lower than the 25% recommended by the standard, but it was chosen as it corresponds to the application.

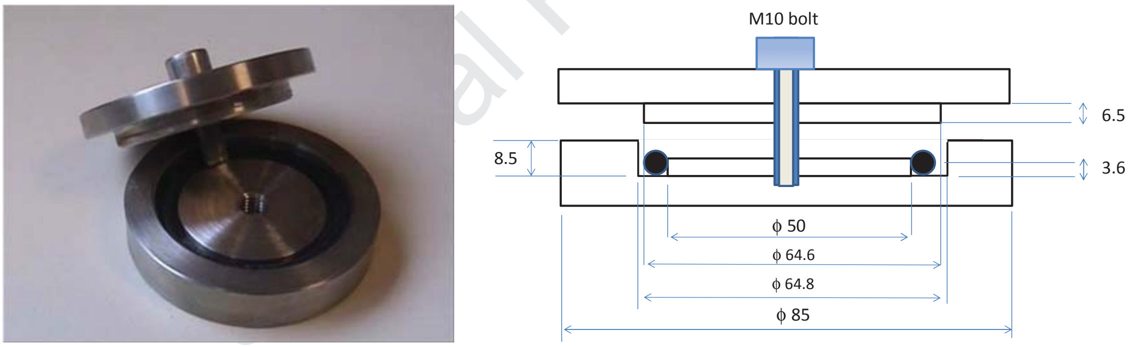


Figure 4. Compression test fixture. Joint internal diameter 50mm.

Photo (left) and Diagram showing cross-section and dimensions (in mm) (right)

Samples were compressed in the fixtures then immersed in deionized water (rather than seawater, in order to avoid corrosion of metallic fixtures) at different temperatures (25, 40, 60 and 80°C). For the original joint material one fixture was immersed at each temperature, while for the longer tests on the new material at least two fixtures were immersed at each temperature. Fixtures were removed from water, unloaded, and left in a temperature (21°C) and humidity (50%) controlled laboratory for 30 minutes before measuring the thickness of each joint at four points around the ring. The compression set (permanent set) in % was calculated as:

$$100\,\frac{(t_o - t_t)}{1.9}$$

with t_o the mean initial measured thickness in millimeters of the ring, nominally 5.5 mm, t_t the mean thickness measured after aging time t.

Sealing force was then measured by compression of the aged joint between two flat platens on an Instron™ 5566 test frame with a 10 kN load cell. The force corresponding to an overall displacement value of 1.9mm (including set) was recorded. Loading rate was 2 mm/minute.

Other identical fixtures were fixed to the outside surface of the sphere of the DSV for 5 years, and were subjected to the same conditions during diving and storage as the joint in the sphere. Compression set and sealing force were then measured on these in the same way.

A complete equatorial joint removed from the Nautile DSV after 10 years in service became available in 2018 and was subjected to the same tests.

In order to examine the tightness of the joint a helium detection device, an Alcatel ASM 121h model, was employed, Figure 5. This is more sensitive to leaks than visual observation of water drops and should provide a conservative leakage criterion.

Figure 5. Helium leak detection equipment

A special compression fixture was designed, which allows a vacuum to be created in the central joint cavity inside the O-ring before filling it with helium while the detector could be moved around the periphery to check for leaks, Figure 6. The applied load on the joint could then be reduced until leak detection.

Figure 6. Helium detection during compression loading of joint in test fixture.

4 RESULTS and DISCUSSION

First, the original material was characterized by immersion of compressed joints in water at different temperatures. Periodic measurements of thickness and sealing force were made over a 2-month period. Then the new material was tested in a similar way but over a ten year period.

4.1 Original material behavior

Images of sections of the original O-ring material, Figure 2, after 5 years in service indicated a compression set of around 53%. In order to study how this might evolve over a longer period, specimens were aged at higher temperatures in water for 2 months, and their compression set was measured periodically. A master curve was then constructed, Figure 7, by applying a horizontal shift to elevated temperature data until they fell on a single curve. This allowed the time to reach a given compression set value at 25°C to be estimated. These results indicate that the time to reach 100% compression set, and hence lose all sealing force, might be less than 5 years. An alternative material was therefore sought.

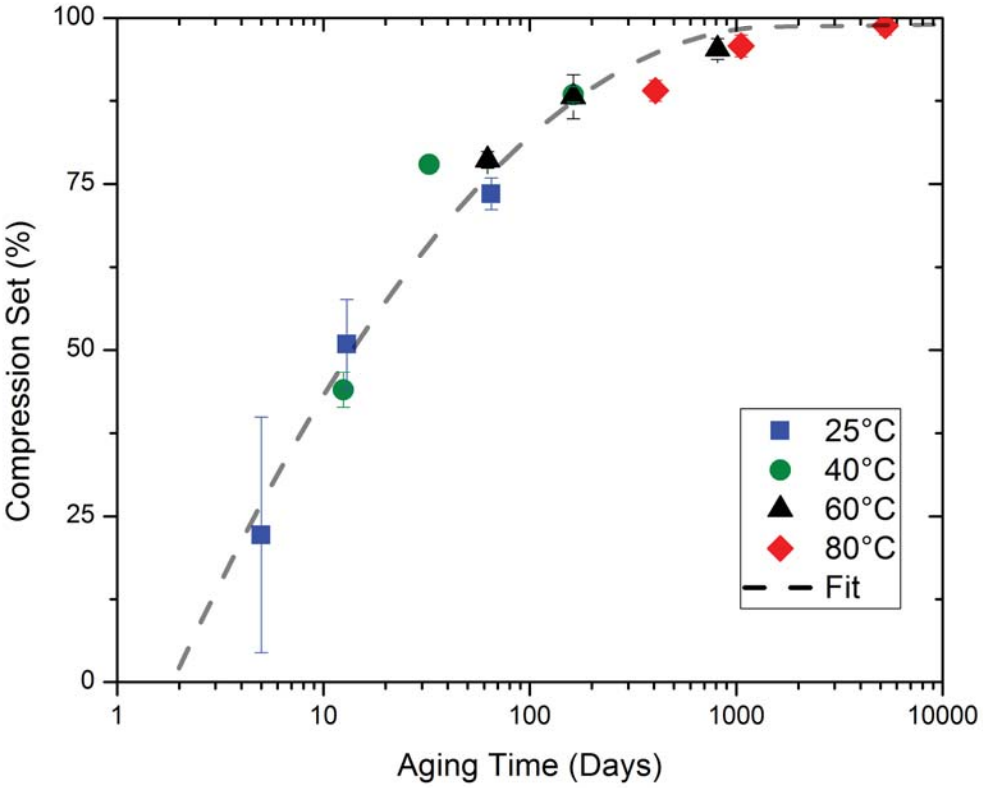


Figure 7. Master curve at 25°C of compression set values, produced by horizontal shift of data from higher temperatures. Dashed line: Second order polynomial fit. Error bars show minimum and maximum values.

It should be noted that in the laboratory the compression set of this original material reached 50% after a couple of weeks at 25°C; this is a similar value to that measured on the sample shown in Figure 2, which was aged in service for 5 years. There are several factors which could explain this difference in time to reach a similar permanent set value. First, the joint removed from the DSV in the South of France took some time to reach the laboratory, so it had time to recover completely before measurements were made. The measurements made during the laboratory aging study were all taken 30 minutes after unloading the joint; these values would tend to be lower after a longer time. Second, the aged joints were held in a water bath at 25°C. The aging temperature in service can be considerably lower, descending to around 5°C in the deep oceans. This would slow the permanent set.

4.2 New material behavior

Samples of the new material were immersed in de-ionized water in 2009 and are still immersed today. This has provided considerable information, not only on how compression set and sealing force change with time under load but also on the influence of accelerating temperature. Figure 8 shows raw data from 10 years of immersion at 25°C and 80°C, and compares values for the new material with those for the original O-rings.

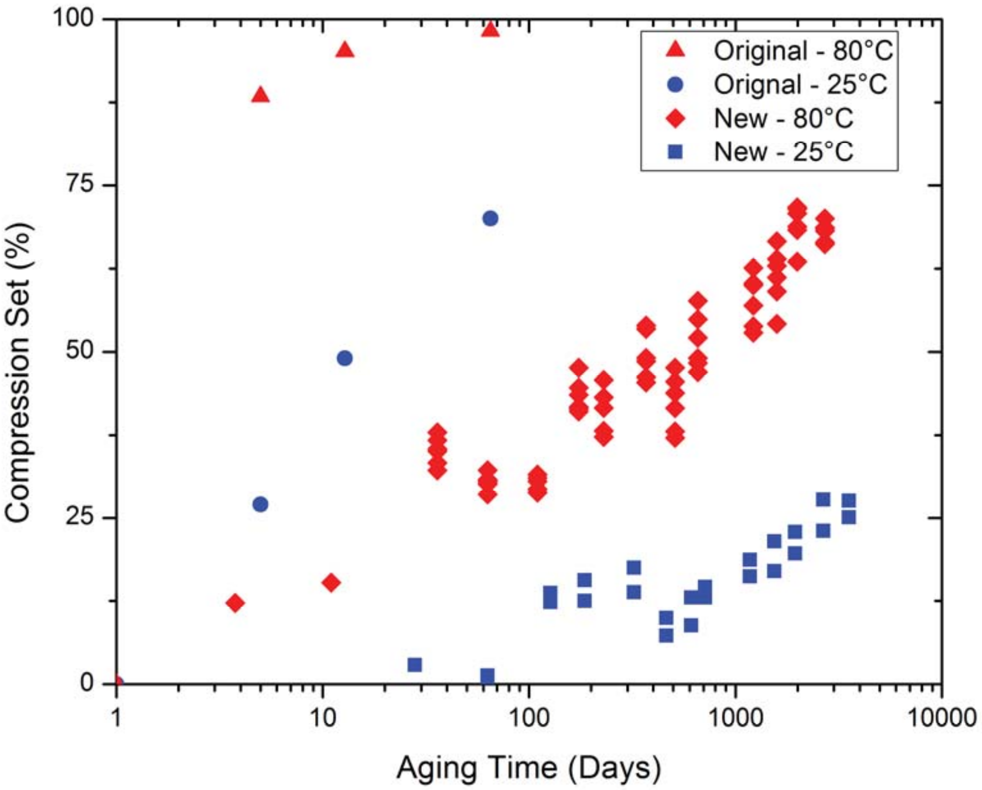


Figure 8. Comparison between compression set values for Original and New joints, after immersion at 25°C and 80°C.

It is clear from this figure that the new joint formulation shows considerably lower compression set after 5 years than the original material. The next question to address is whether the compression set and sealing force can be predicted for long periods. As for the original material, Figure 7, test fixtures were immersed in water at four temperatures (25, 40, 60 and 80°C) and an example of the measured compression set values is shown in Figure 9 below. There is some scatter in the measurements; it should be remembered that each point represents the mean of four micrometer measurements, and that these measurements were made over a period of ten years, but there is a clear increasing trend. To a first approximation a semi-log relationship can be applied to the data at each temperature.

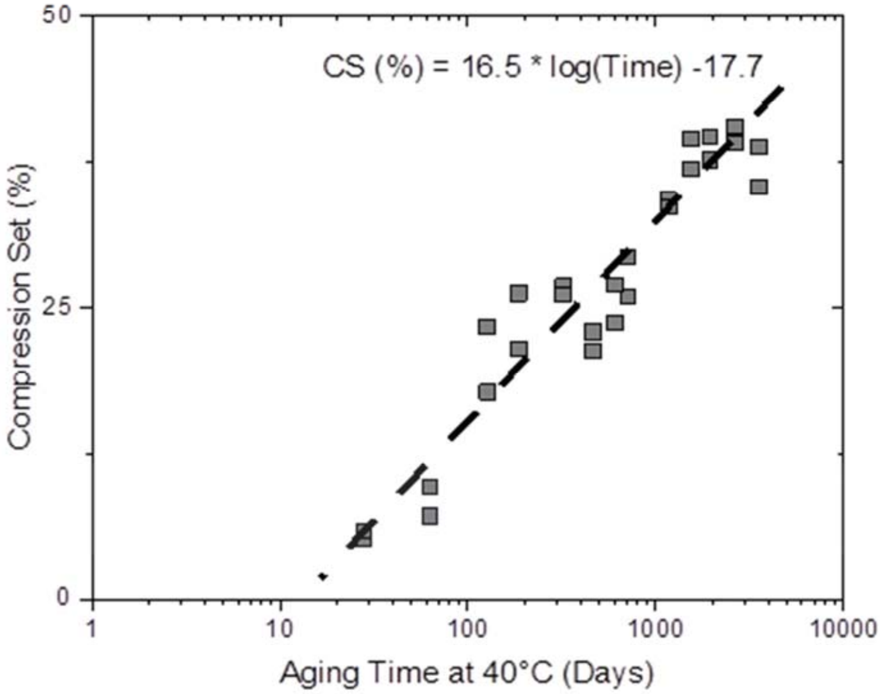
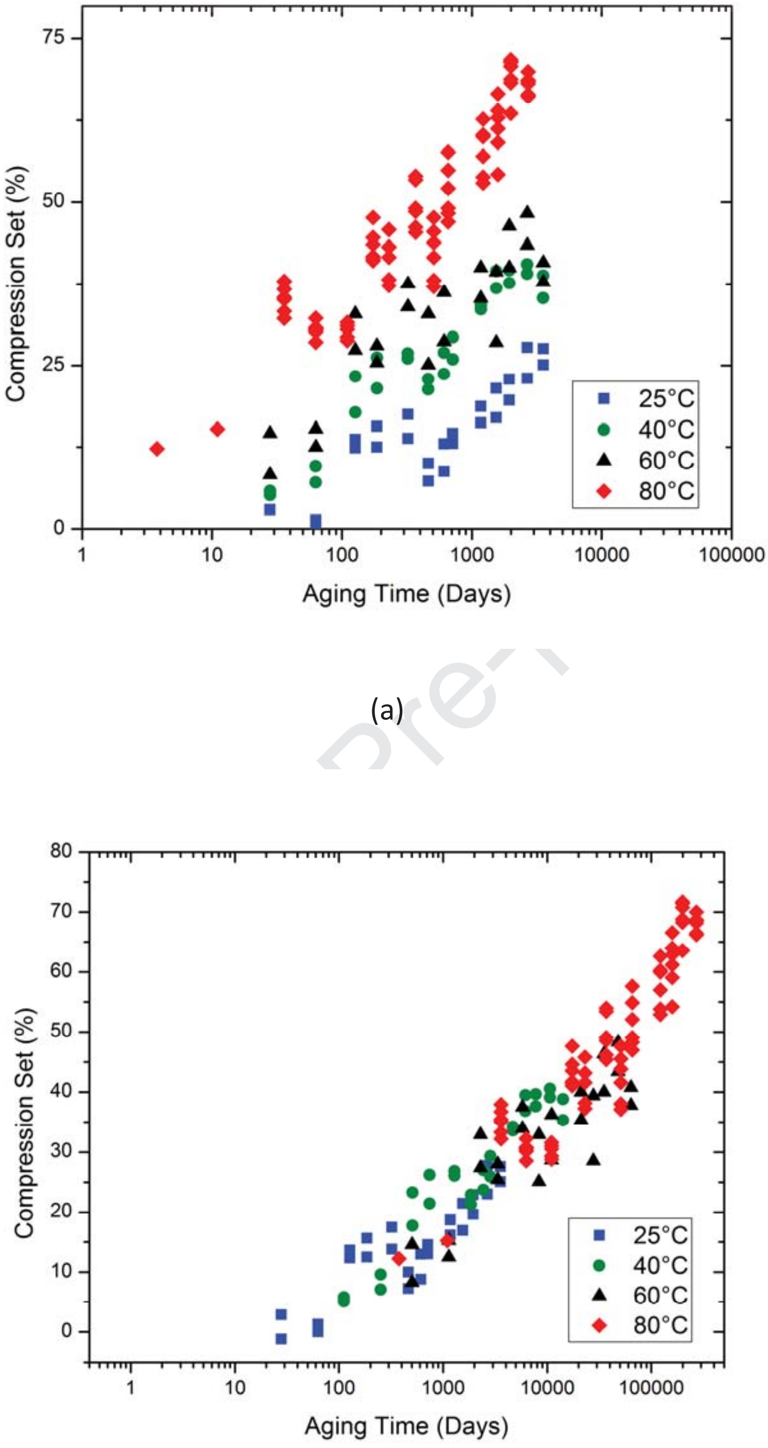


Figure 9. Example of compression set data, new joint, 2 samples, 40°C, 10 years.

Based on these values and similar data for 60 and 80°C an empirical reference curve can then be constructed by horizontal shifts onto the 25°C data, Figure 10. This allows the compression set to be estimated at 25°C at any time.



(a)

b)

Figure 10. a) Compression set data at different temperatures. b) Compression set master curve for 25°C immersion, using all measurements for new joint (no data excluded).

There is some scatter on the master curve and the exact shift values are subject to interpretation, but it is interesting to note that the shift factors used to construct Figure 10b can be plotted on an Arrhenius plot and a reasonably linear fit is obtained, Figure 11.

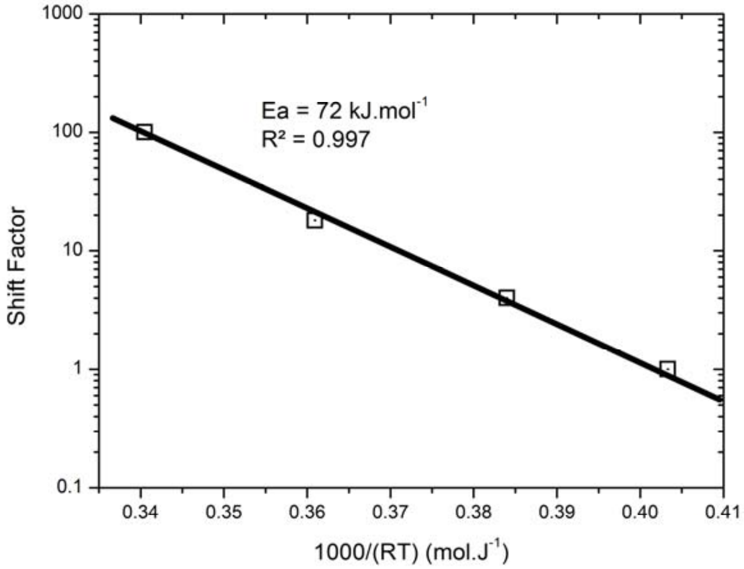


Figure 11. Arrhenius plot of horizontal shift factors used to construct the master curve in Figure 10b.

The activation energy value of 72 kJ/mol is similar to the range of values often obtained in studies on similar elastomeric materials. For example, Budrugeac found activation energies for the degradation of various NBR formulations in the range between 67 and 96 kJ/mol [25]. Buckley and Roland found an activation energy for oxidative degradation of a particular NBR O-ring of 82 kJ/mol, a value which did not vary for different immersion liquids [26].

The compression set is easy to measure in the field, and allows materials to be compared easily, but the key parameter of interest for a O-ring is the sealing force. This was measured at the same time as the compression set, throughout the aging period. It should be noted that these measurements, both set and sealing force, are not simple; the material has been compressed for up to 10 years, so when it is released it requires a long time to reach a stable value. Gillen et al [6] have examined temperature cycles and overstraining in order to accelerate stabilization. Figure 12 shows examples of force-displacement plots, recorded initially and after 10 years in water.

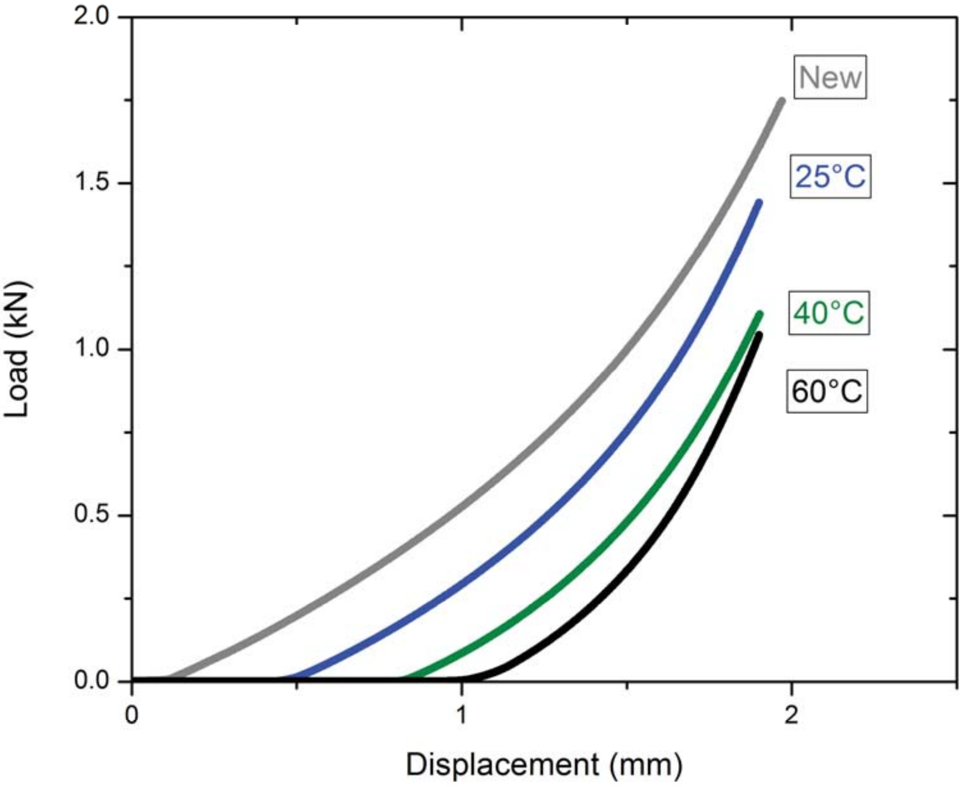


Figure 12. Examples of compression force-displacement plots for O-rings, both new and after 10 years under load in water at three temperatures.

From such curves the sealing force values corresponding to the 1.9 mm compression displacement were evaluated, and these are plotted versus the compression set values in Figure 13.

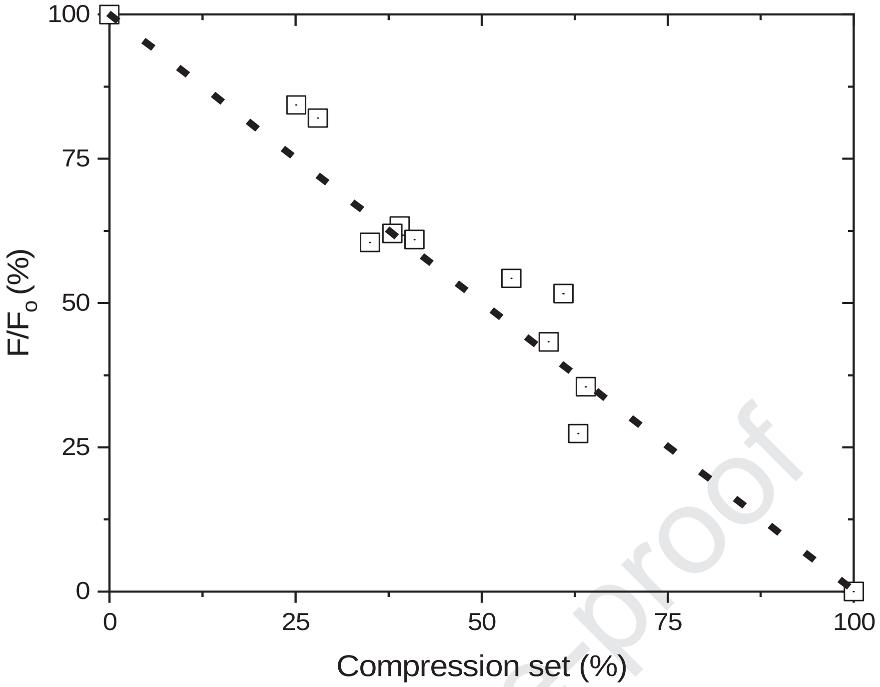


Figure 13. Sealing force versus compression set.

Gillen et al [6] indicated a linear correlation between stabilized sealing force and compression set for butyl rubber materials and, to a first approximation a similar relation can be seen here for this NBR formulation. This allows sealing force to be estimated from compression set data.

4.3 End-of-life criterion

Given that the compression set can be estimated and correlated with the sealing force, the next question when trying to predict the lifetime of a sealing device is “what is the criterion corresponding to loss of sealing ?” In the case of a DSV this would correspond to water entry, but in order to determine a criterion a helium detection device was employed, as described in section 3 above. This is more sensitive to leaks than visual observation of water drops and should provide a conservative criterion.

Results obtained with the fixture shown in Figure 6 indicate that leaks are only detected at very low sealing force levels. A similar conclusion was noted previously by Gillen et al [6]. In their case leaks were only detected at values below 10% of the initial sealing force. For the geometry and materials tested here no helium leaks were detected even with a joint aged to 80% compression set. This value might therefore be used as an end-of-life criterion. On this basis it is possible to estimate the time required at a given water temperature for the compression set (and hence the joint sealing force) to reach a level for which there is a risk of water ingress. The results in Figure 10b indicate that for the new joint material, and provided that only the compression set mechanism is considered, this lifetime will exceed 200 years. It should be noted that for the original joint formulation this prediction, albeit conservative, indicates a lifetime of less than 5 years.

4.4 Correlation with service experience

The previous sections have shown that the long term sealing behavior of nitrile O-rings can be estimated by accelerated laboratory tests. However, it must also be shown that the results from these tests correspond to the response of joints used on the DSV. Two additional series of tests were therefore performed. The first involved four test fixtures similar to the one shown in Figure 4, which were placed on the outer titanium structure of the spherical hull of the submersible. These were subjected to the same pressure and temperature cycles as the equatorial joint over a 5 year period. Figure 14 shows how the compression set values of these joints, measured at different intervals, compare to the 25°C laboratory aging results.

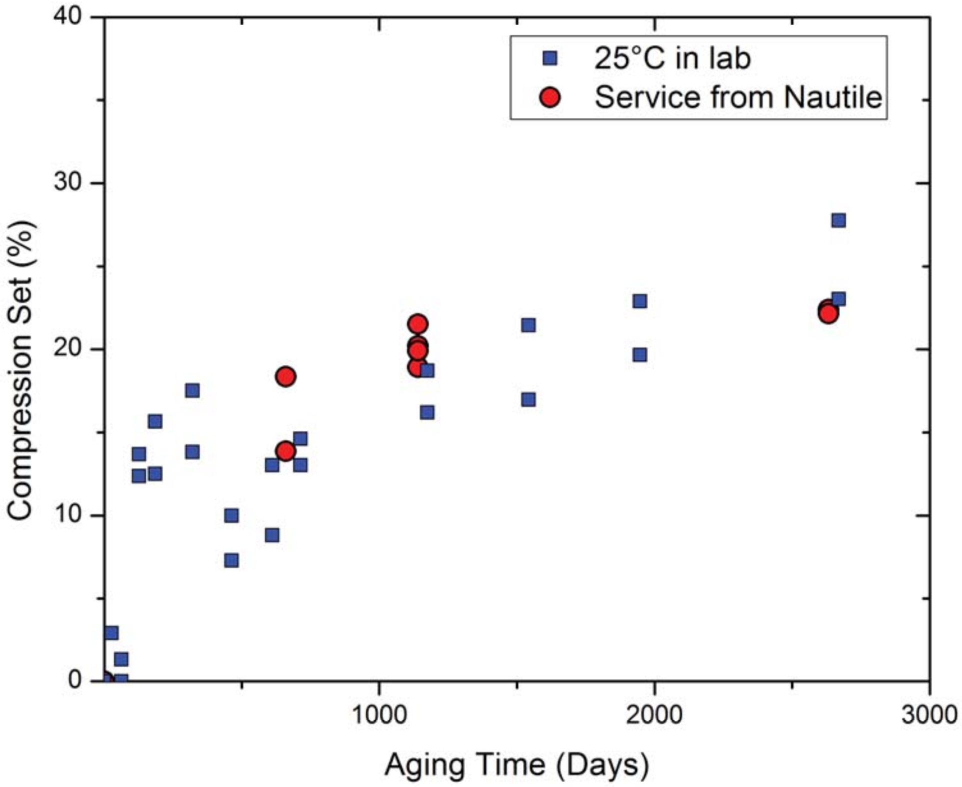


Figure 14. Correlation between compression set values for laboratory aged samples (25°C water) and for compressed joints placed on the exterior of the submersible sphere.

The measurements made on samples placed on the submersible during service are similar to, and within the scatter of, test values for samples after 25°C immersion in the laboratory.

A second verification was performed directly on an equatorial joint removed from the submersible after 10 years in service. The equatorial joint thickness was measured at 16 points around the joint, and a mean compression set of 19% was determined. Taking account of thickness variations the range of compression set values varied from 14 to 24%. The values measured in the laboratory were slightly higher, indicating that these measurements are conservative. This may indicate that the small laboratory samples, with a much smaller radius of curvature than the equatorial joint, are more severely loaded, or that the temperatures were lower in service.

Condition	Compression set range, %
Laboratory aged 25°C immersion 10 years	25-28%
In service on Nautile DSV for 10 years	14-24%

Table 2. Comparison between permanent set values for laboratory aged and in-service O-rings.

Finally, Figure 15 shows a cross-section of an O-ring produced with the new joint formulation after 10 years in service on the submersible. There is some permanent deformation (around 20%) but significantly less than that noted for the original formulation after only 5 years (more than 50%, Figure 2).

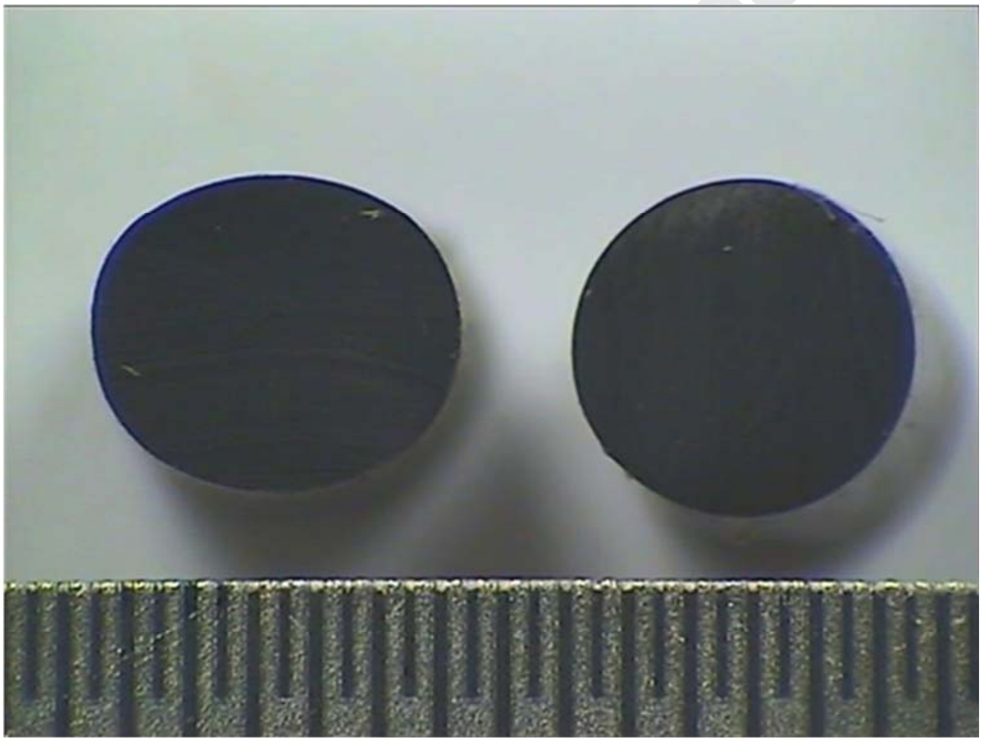


Figure 15. Sections through joints with new formulation. (Left) Joint removed from DSV after 10 years in service. (Right), unaged joint.

Although the complete formulations of the original and new O-rings are not known, the large reduction in compression set of the latter appears consistent with the conclusions of some previous studies, in which lower permanent set was correlated with lower filler levels [15] and higher carbon black content [19].

5 CONCLUSIONS

This paper shows results from a study of the aging of nitrile O-ring seals. Samples have been immersed under load in water in the laboratory for over 10 years, and results are compared with those from a joint which had spent 10 years under load in service on the Nautile DSV. The original joint formulation showed significant permanent deformation, while the new formulation employed today is much more elastic. A simplified extrapolation of the changes in properties with time, in combination with the measured changes after service, has been used to estimate lifetime. The results indicate that the lifetime of the joints is considerably longer than the current replacement time. Based on the results from this study the Safety Commission of the submersible agreed in 2014 to an extension of the O-ring replacement time, from 5 to 10 years.

ACKNOWLEDGEMENTS

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DATA AVAILABILITY

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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A laboratory prediction methodology for long term sealing behavior is described;

Relationship established between compression set and sealing force:

Leakage criterion proposed;

Laboratory test results are compared with results from an in-service underwater application and indicate a reasonable long-term correlation.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: