TECHNICAL PAPER

The limits of low temperature sealing tests for elastomeric materials

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There are a wide range of industries and applications where the low temperature sealing performance of elastomer components is of central importance. From Arctic oil and gas exploration (*Fig.1*) and production through to specialist food and pharmaceutical applications, thousands of operators around the world depend on the ability of their elastomer seals to perform reliably in aggressive media and under high pressure down to very low temperatures.

Figure 1 — Arctic oil and gas exploration

The failure of a seal can be incredibly costly, both financially to the operators in lost production and revenue, but also potentially the human cost if something were to go catastrophically wrong in environments as critical as oil and gas, food and pharmaceutical production or aerospace. As a result, it is vital to continue building an understanding of the elastomer materials suitable for use in low temperature sealing, and how these materials might be further refined and developed in future.

Many well-established tests already exist upon which seal compatibility is founded. To equipment manufacturers or operators, the results of these tests can give a strong indication as to a material's suitability for low temperature sealing. However, there are some difficulties when it comes to testing at extreme low temperatures. Tests can discern a material's physical properties in extreme low temperatures, such as brittleness testing or torsion modulus. However, it is more difficult to make accurate predictions on how that material will perform as a seal in these conditions. Proprietary sealing tests for low temperature sealing all require that the seal is energized by the pressure of the test media before being subjected to low temperatures. This doesn't reflect the reality of low temperature sealing applications – and indeed, if the seal is cooled to low temperatures prior to exposure to pressurising media, the seal may be too stiff to energize and therefore be unable to form a reliable seal.

It is an ongoing challenge for scientists to improve on current test methodologies for evaluating the efficacy of sealing materials in low temperature applications. Enhanced testing is key to giving operators a greater degree of accuracy and reliability pertaining to the low temperature operating limits of their elastomeric materials. Let us investigate why low temperatures are problematic for elastomers, explore the current test options available, and look at what the future might hold for low temperature testing of elastomeric seals.

Why do seals fail in low temperatures?

All elastomers are made up of long chain molecules, chemically cross-linked to form a three dimensional structure (*Fig.2*). These molecules are typically free to move at temperatures within their normal operating range. However, when energy is reduced in the system through a decrease in temperature, molecular movement is also reduced. The molecules which make up the elastomer material move closer together at these lower temperatures, becoming gradually stiffer and less elastic. The molecules eventually reach what is known as a glass transition temperature (T_g).

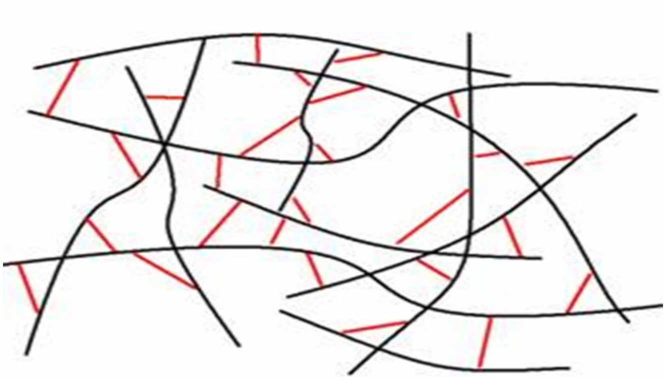

Figure 2 — Polymer chains (black) joined by chemical cross-links (red)

At this point the elastomer has undergone a phase change, becoming glassy and brittle. Some residual elasticity may still remain at this point, although it can be challenging to create or maintain a sealing effect. Additionally, the elastomer will contract as the material cools, as per the material's co-efficient of thermal expansion (CTE). This is compounded by the fact that stresses which are induced at higher temperatures may be unable to relax out at lower temperatures, a process known as 'cold set'. Contraction, cold set and reduction in chain mobility are the three key contributing factors to elastomeric seal failure and leakage in extreme cold conditions.

The temperature at which the polymer becomes brittle is largely due to its chemical structure. The introduction of monomers into the molecular chains can increase chain mobility, improving flexibility and elasticity at low temperatures. The molecular structure of a rubber polymer has the most significant influence on the subsequent low temperature flexibility of the final elastomer seal. Other factors include compound hardness, modulus, or the presence or absence of additional plasticizers.

The anatomy of an elastomer seal

For most high pressure oil and gas applications, static seals are the most common elastomer seal type. The most common static seal is the O-ring. O-rings are squeezed into rectangular housings, forming a seal through initial compression. The sealing force applied by this initial squeeze is increased to system pressure by the reaction of the seal. The seal is formed by this balance of forces. Whilst the seal is energized by the system pressure, the residual stress within the elastomer is critical to maintain a sealing force above the pressure being contained. This sealing force can reduce over time due to stress relaxation brought about by physical and chemical changes to the seal material, and at low temperatures the residual sealing force can also reduce to a point where the system will fail.

Testing elastomers at low temperatures

There are two main types of testing for determining the low temperature operation limits for elastomers. There are test methods which determine changes in elastomer material properties and then set arbitrary limits to the changes to derive an operating limit (material testing). Secondly, there are methods which are more relevant to the actual mechanism of sealing but are still not representative of the usual mode of sealing within low temperature environments (existing low temperature sealing tests). Let us examine some of these testing methods in more detail.

Material Testing

Temperature Retraction

(ISO 2921/ASTM D1329/BS ISO2921)

Often referred to as the TR10 test, this test involves the immersion of a stretched, standard test piece in an alcohol bath cooled with solid carbon dioxide to -70°C until the test piece becomes rigid. The sample is then allowed to retract freely while the temperature is raised at 1°C per minute. The temperature at which the test piece has retracted 10% of the original stretch is referred to as its 'TR10'. A value appended to TR10 defines the initial stretch, for example TR10/50, so test will stretch the sample by 50%. It is worth noting that the elastic modulus of an elastomer may influence the results independently of its low temperature properties.

Glass Transition Temperature and Differential Scanning Calorimetry (DSC)

(ISO 22768/ASTM D7426)

The glass transition of an elastomer is, as previously touched on, the temperature below which the elastomer changes from a rubbery state and behaves like a glassy solid. Differential Scanning Calorimetry (DSC) measures the heat flow associated with transitions in materials as a function of time and temperature, measuring heat flow into or out of a sample as it is heated, cooled or held at a set temperature. This technique can provide a wide range of data including T_g.

Gehman Torsional Modulus
(ISO 1432/ASTM 1053/BS 903 A13)

The Gehman test measures the torsional modulus of a standard test piece at a range of temperatures. The relative modulus values at the measured temperatures are then determined. The temperature at which the relative modulus is 10 is reported as T10, or 5 as T5. The absolute torsional modulus of a given material may to some extent influence the result. A minimum operating temperature can also be estimated by finding at which point a given ratio of torsional moduli between room temperature and a lower temperature is reached.

Figure 3 — Low temperature retraction test apparatus

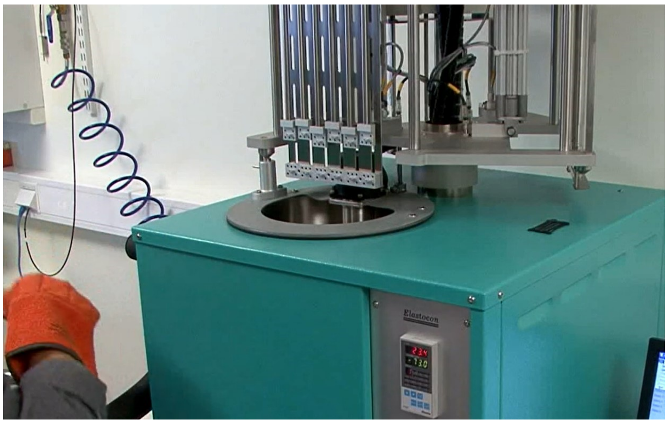

Figure 4 — Equipment used in a Gehman torsional modulus test

Bend Brittle Test
(e.g. DTD 458)

This test involves an elastomer sample placed between two jaws and a screw. After conditioning at the specified temperature in a cooled alcohol bath, the jaws are screwed together, and the subsequently flexed sample is then examined for splits or cracks. This test only measures brittleness at a given temperature, with no measurement on elasticity.

Brittleness Temperature by Impact
(ASTM D746, ISO 812)

This test evaluates the brittleness of rubber materials when exposed to low temperature flex with an impact under specified conditions of striker speed. The tests performed are used to determine the lowest temperature at which rubber compounds will not show fractures when exposed to specified impact conditions.

Dynamic Mechanical Thermal Analysis (DMTA)

A sample of elastomer is flexed and properties such as modulus and damping are measured over a range of temperatures at fixed frequencies. Elastic and viscous moduli are recorded along with a ratio of these (Tanδ). An assessment of T_g and Brittle Onset Temperature can be made from the data.

Existing Low Temperature Sealing Tests

This sealing test involves a setup of O-rings with a fixed amount of compression (typically 10%), using nitrogen at a particular pressure and temperature. In this test, the temperature of an energized seal is reduced until failure occurs. Results obtained from the testing were typically significantly below T_g (between 10 and 15°C). Most testing involved fluoroelastomer (FKM) sealing materials, and the results were applicable to real life applications where seals of the same size are compressed by the same ratio, with the same lubrication, and pressurized to 200 psi before cooling down. In reality, very few seals would be pressurized before the temperature was reduced.

A test method with greater validity?

There is scope for the development of a new testing method for elastomer materials in low temperature sealing applications, which would enable analyses of seals in conditions more representative of those faced in situ. The European Sealing Agency (ESA), with the involvement of Precision Polymer Engineering scientists, is currently involved in trials to define a test method which seeks to make these improvements.

If the methodology and results are valid, then for the first time there is the possibility of an industry -agreed specification implementable by all reputable elastomer seal suppliers. This would give end users a more reliable guide as to the low temperature operating limits of their components, and would result in improvements across sealing performance, operational safety and profitability.

While these experimental conclusions are awaited, end users looking for expert support with elastomeric sealing in critical low temperature applications should consult with a specialist sealing engineer. These experts can make an informed recommendation on the correct material type and grade for a sealing system, in line with the pressure, temperature and chemical resistance needed.

The decision as to which elastomer material should be used for the seal has several facets. These variables would depend on the unique demands of each application or industry, but to take our example of an oil and gas application above, a seal material would be specified on the basis of variables including chemical resistance data, mechanical properties, operating temperature range, Rapid Gas Decompression (RGD) resistance, and resistance to the corrosive effects of sour gas (H_2S). Datasheets and reference literature can help to navigate these factors, but with the additional concerns of data limitations at minimum operating temperatures, there is an extra factor of complexity to be considered. It may typically be assumed that a quoted minimum temperature represents the lowest temperature at which seals will be used in all situations. This assumption is frequently flawed, and so additional care must be taken when specifying for low temperature applications.

Figure 5 — O-ring molded from specialist low temperature FFKM (Perlast® ICE)

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