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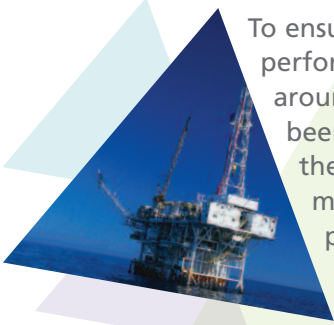
VICTREX® PEEK Meets Norsok M710 Compliance and Continues to Aggressively Test its Polymers in Support of Industry-Leading Test Standards

Geoff Small, Victrex Technical Program Manager

The use of elastomeric seals and thermoplastic back up rings is commonplace in the oil and gas industry with ever increasing levels of ingenuity and sophistication in their design and application. At the core of these critical components is the quality of the materials from which they are made: frequently using high performance elastomers to fulfil the sealing function and rigid thermoplastics of varying performance levels as back up rings to provide resistance to extrusion of the seal from its intended location under conditions of high-temperature and pressure.

deal with property degradation after aging in representative environments (chemical and temperature) and also after rapid decompression events.

Standards such as API 6 "Specification for Wellhead and Christmas Tree Equipment" and the Norsok M710 "Qualification of Non-Metallic Sealing Materials and Manufacturers" propose specific environments which may be single or multi-phase and which consist of a number of 'typical' gases or gas mixtures and aqueous and hydrocarbon fluids. As oil reserves have become increasingly sour, these standards indicate the addition of hydrogen sulfide (H₂S) to the gas mixture to account for this.



To ensure safe and consistent performance across the industry and around the globe, standards have been developed which go beyond the information supplied in manufacturer data sheets to provide some assurance that these materials have the right balance of mechanical and physical properties to fulfil their function for the requisite lifetime. These standards have been developed to show not only that the fundamental mechanical properties are sufficient to function correctly, but moreover that such properties can be adequately maintained throughout the service life thereby ensuring the same level of seal assurance at the end of life as at the beginning.

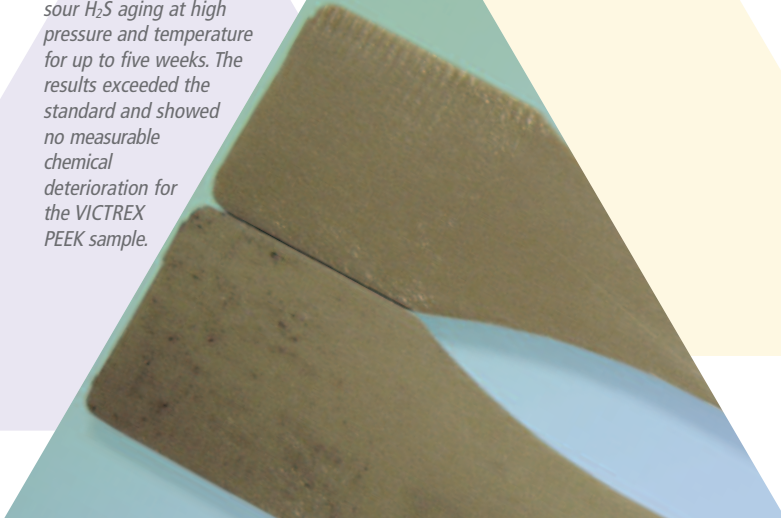
To that end, the internationally recognized standards have specific sections which



The Norsok M710 Rev 2 (October 2001) standard is intended to "assure that the non-metallic sealing and experience with the applicable materials to provide them with acceptable performance in the specified a successful qualification of a manufacturer and a specific material shall be valid for a majority of future development projects and for different operators."

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In response to standardization requirements of the oil and gas industry, Victrex requested an evaluation of the extent to which VICTREX PEEK complies with the Norsok M-710 standard for sour H₂S aging at high pressure and temperature for up to five weeks. The results exceeded the standard and showed no measurable chemical deterioration for the VICTREX PEEK sample.



In addition, using the data on the effect of aging on tensile strength, the standard requires that the results are used to predict the service life (i.e. time to 50% of properties) at the service temperature using an Arrhenius approach. The test report from the chemical aging must contain details of the chemical environment, (which may be different to the standard Norsok oil by agreement) the test temperatures, pressure and test duration, and, if possible, the trends and predictions obtained from the Arrhenius plot.

To use such predictive approaches requires there to be some measurable and repeatable change in the properties of the material in question within the chosen timeframe and experimental conditions. Use of the Arrhenius approach also assumes that a single degradation mechanism is responsible for the reduction in properties: this may often be a significant over-simplification.

The standard is divided into two sections: one for elastomers which covers chemical ageing and rapid gas decompression, and one for thermoplastics which covers chemical ageing only. For both sections the Norsok standard requires submission of various mechanical properties, which indicate the general suitability of the material for the proposed loads and environments.

In addition, the thermoplastics section requires submission of a report on ageing characteristics of the thermoplastic material in the end use environment.

The thermoplastics section of Norsok M710 Rev 2 prescribes that ageing take place in either a sweet or sour Norsok oil environment at a minimum of three temperatures 'above the specified usage temperature'.

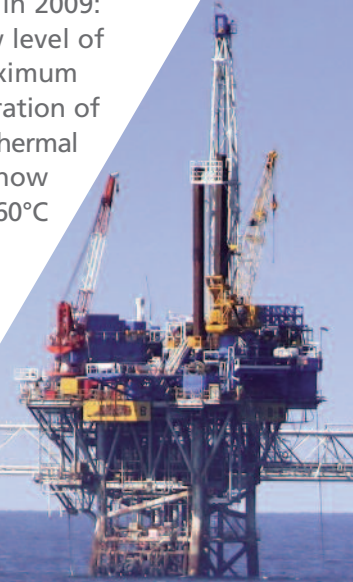
The standard requires that minimum acceptance criteria are established prior to the test but provides the following as a baseline:

- ▲ Swelling +5% / -1%
- ▲ Tensile strength, elongation and modulus +/- 50%
- ▲ Visual inspection (no dissolution, cracking blistering etc.)

More recently, a new ISO standard, ISO 23936-2 "Petroleum, petrochemical and natural gas industries - non-metallic materials in contact with media related to oil and gas production- Part 2: elastomers" and the current third revision of Norsok M710 (DRAFT), offer a wider scope of test environments and classifications for the temperature 'index' of the approval rating.

Both of these recent documents still rely on an Arrhenius approach and specifically have limits of testing temperature which end at 225°C (437°F). Although it is not a component manufacturer, Victrex undertook Norsok M710 Rev 2 approval of its VICTREX PEEK 450G polymer in 2009: unsurprisingly with a relatively low level of H₂S (2% of the gas phase) at a maximum temperature of 220°C (428°F), a duration of 800 hours and for a polymer where thermal aging tests according to UL 746B show that a duration of 50,000 hours at 260°C (500°F) is required to reduce the strength by 50%, no measurable change in properties was seen.

Strictly speaking, this means that the polymer meets the minimum requirements of the standard but no specific prediction of long term properties at lower temperatures can be



made because of the inability to measure sufficient change to permit Arrhenius type extrapolations.

This may fail to provide the level of information required by the user and is an issue not only restricted to PEEK polymer but also to other materials such as PTFE.

To this end, the later revision of NORSOK M710 (Rev 3 Draft Sept 2013) and ISO23936-2 standard both note that "...polymers which are very resistant to sour aging cannot be appraised using methods which rely on progressive property changes with time and temperature," and conclude that they are "...best described as being chemically stable under all the accelerated test conditions employed and therefore likely to have a very long useful life at lower temperatures" (i.e., service temperatures lower than aging temperatures).

To further investigate the prediction of long term high temperature and aggressive environment performance and provide useful information to its customers, Victrex is researching how its polymers will perform in even more aggressive environments. Victrex is also seeking and researching new predictive techniques to benefit its customers in the energy sector.

One specific area of concern is the level of H₂S in the sour environment under test: there is an accepted equivalence between the concentration of a gas in a mixture and its partial pressure. Dalton's law of partial pressures states that 'the partial pressure of an ideal gas in a mixture is equal to the pressure it would exert if it occupied the same volume alone at the same temperature.

This means that the total pressure of a mixture of gases (assumed ideal) is equal to the sum of the partial pressures of the individual gases in the mixture and that the ratio of partial pressures is the same as the ratio of the number of molecules. The mole fraction of an individual gas component in an ideal gas mixture may be expressed in terms of its partial pressure.

Extending this concept to the original NORSOK testing carried out on VICTREX PEEK 450G [175°C, 195°C, and 210°C (347°F, 383°F, and 410°F)] and 1,450 psi (10 MPa) with 2% by volume of H₂S within the gas component), the results would be equivalent to the same

Figure 1: Retention of Tensile Strength After Aging in Standard NORSOK M710 Rev2 Sour Environment (samples immersed in hydrocarbon phase)

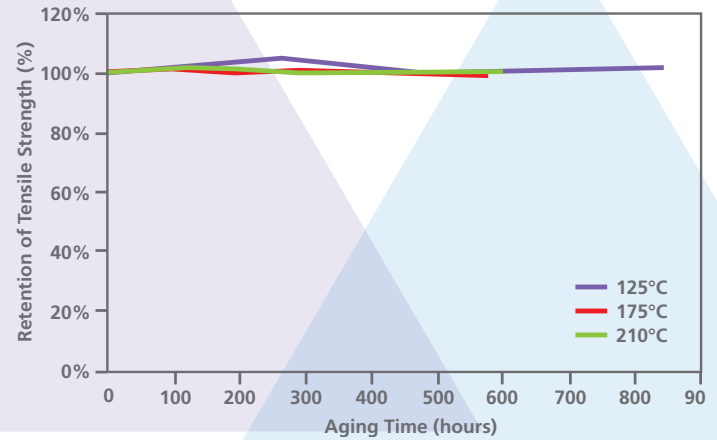
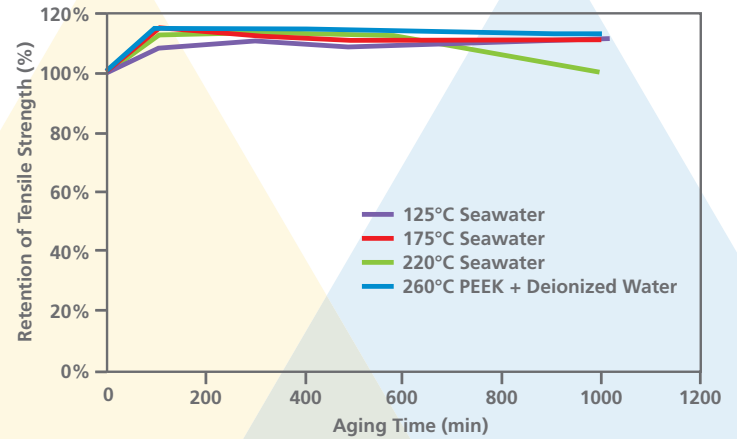
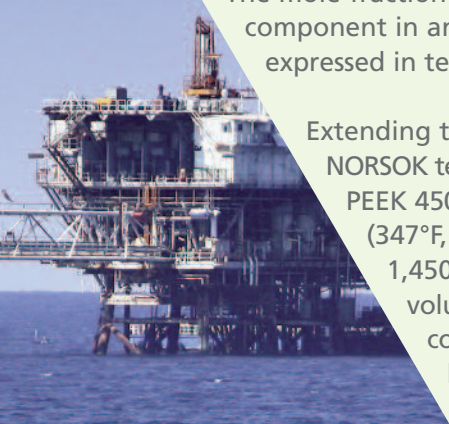
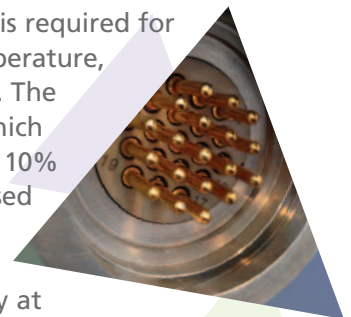


Figure 2: Retention of Tensile Strength After Aging in "Sour Seawater" (3-phase test with 100% H₂S gas phase) and Deionized Water



test at 10000 psi but with a H₂S concentration of only 0.29%, or 0.145% at 20,000 psi (138 MPa). The data generated may not go far enough to offer the level of confidence in performance that is required for environments with varying temperature, pressure and gas concentrations. The DRAFT revision 3 of NORSOK which proposes a higher H₂S content of 10% may be tested, but at the proposed pressure of 870 psi (6 MPa) corresponds to H₂S mol fractions of 0.87% and 0.44% respectively at the higher pressures.

It appears unclear which combinations of temperature, pressure and sour gas concentration lie ahead for the oil industry. In order to prepare for future challenges, Victrex has extended its three phase testing to encompass an



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Figure 3: Retention of Tensile Strength After Aging in 100% H₂S Phase of 3 Phase Aging Environment (seawater: 10%, gas: 30%, aromatic Norsok oil 60%)

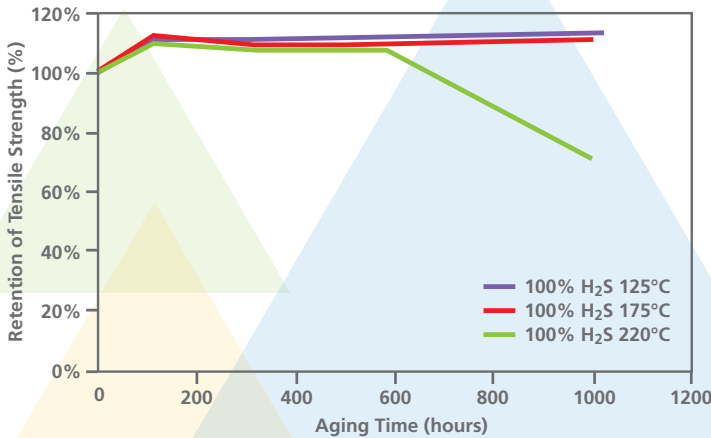
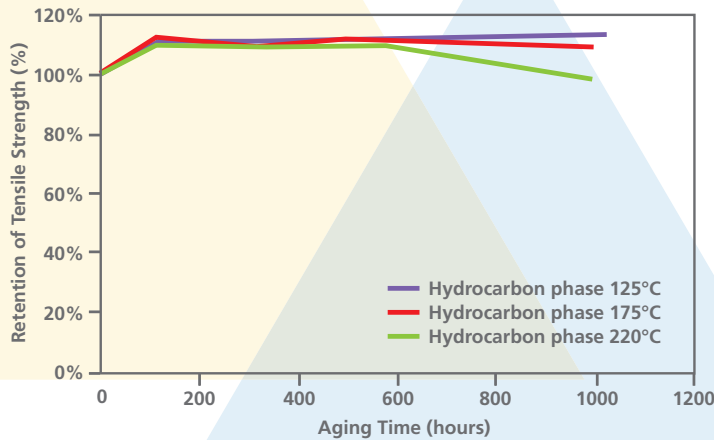


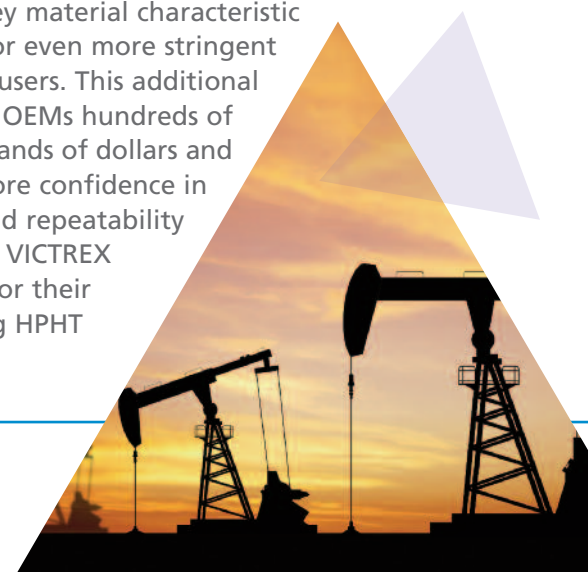
Figure 4: Retention of Tensile Strength After Aging in Hydrocarbon Phase of 3 Phase Aging Environment (seawater: 10%, gas: 30%, aromatic Norsok oil 60%)



environment (at test pressure) of 100% H₂S which corresponds to more than 1% at 30,000 psi (207 MPa) and at temperatures of 220°C (428°F). Based on testing under such conditions, mechanical properties are fully retained in the aqueous and hydrocarbon phases and more than 70% of strength retained in the highly aggressive gas phase.

Recognizing that all polymers will eventually be degraded by such harsh environments, the hope is that this data may begin to show more clearly the safe 'limits' of time, concentration and temperature for which components manufactured from such materials may be used.

Victrex continues to invest in the provision of meaningful and useable data for all users. To this end, the company continues to expand its oil and gas chemical resistance knowledge to cover a wide range of drilling, intervention and completion fluids and associated predictive methods for long-service applications where chemical resistance is a key material characteristic and will strive for even more stringent test data for all users. This additional testing can save OEMs hundreds of hours and thousands of dollars and provide even more confidence in the reliability and repeatability when specifying VICTREX PEEK polymers for their most demanding HPHT applications.



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