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Reliability Test and Analysis of Silicon and Nitrile Butadiene Rubber Seals Under Extremely Low Air Temperature

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Abstract

Rubber O-rings are widely used in various engineering equipment. Low in-service temperature atmospheric conditions will degrade the sealing performance of rubber O-ring or even cause the sealing failures, resulting in equipment failure and even safety accidents. This paper aims at solving the problem of sealing failures analysis of rubber O-ring in equipment under extremely low air temperature. We first analyse the sealing principle and failure criteria of rubber O-ring. The general failure criteria for rubber O-ring are maximum contact stress criterion and maximum shear stress criterion. When the maximum contact stress is less than the medium pressure or the maximum shear stress is greater than the shear strength of the sealing ring, rubber O-rings will fail. The main performance parameters of the O-rings are determined to be hardness and elasticity. Then low air temperature test is carried out for silicon rubber and nitrile butadiene rubber O-ring. The failure mechanism of rubber O-ring under low air temperature is revealed. Research ressults show that the low temperature resistance of rubber O-ring is mainly affected by glass transition and crystallization process. The relationship between performance parameters, such as compression set, hardness, and low temperature is explored. With the decrease of temperature, the thermal movement of the molecular chain of the rubber O-ring material is gradually weakened. The side groups, links and segments of the molecular chain are gradually frozen resulting in the gradual decrease of elasticity and the increase of hardness. The low temperature sealing performance of silicon rubber sealing ring is good, which is mainly affected by the crystallization process of rubber material. At the same time, attention should be paid to the effect of low temperature test time to ensure the accuracy of the test results. The nitrile rubber O-ring has poor sealing performance at low temperature, so it is not suitable to be selected as the sealing part in low temperature environment.

Keywords: rubber seal, reliability, low air temperature, failure mechanism

1. Introduction

Rubber O-ring is an elastic sealing unit with circular cross-section. Rubber O-ring is widely used in pneumatic, hydraulic and power systems of equipment because of its characters of simple structure, convenient assembly, and low cost. The air temperature in some areas, such as Scandinavia Peninsula in Nordic Europe, Northeast China and Siberia Plain in Russia, is extremely low. Low in-service temperature will degrade the sealing performance of rubber O-ring or even cause the sealing failures, resulting in the leakage of sealed media, equipment failure, environmental pollution and even safety accidents. Environmental adaptability refers to the capability of materiel, subsystem, or component to perform its full array of intended functions in intended environments. The low air temperature environmental adaptability of rubber O-rings is of great significance to ensure the reliability and safety of equipment.

Research on the failure of rubber O-rings mostly focuses on the thermal oxidation, design of sealing structure, material selection of units, processing technology, improper assembly and so on (Lu et al., 2017; Sun et al., 2017; Qin et al., 2019; Chandrasekaran, 2010; Wang et al., 2019; Bhaumik et al., 2016; Zhou et al., 2019). However, rubber O-rings are more likely to fail in low temperature environment. Troufflard et al studied the temperature-dependent model of hydrogenated nitrile-butadiene rubber (HNBR) sealed O-ring above and below the glass transition temperature, and predicted the variation of its mechanical behaviour with temperature. However, the model needs relaxation test data, and it agrees well with the experimental results only at 20% compression rate

(Troufflard et al., 2018). Douglas et al studied the test methods, procedures and specifications for accurately measuring the low-temperature operating limit of elastic seals to provide reliable guidance for users on lowtemperature sealing capability of their compounds (Douglas et al., 2016; Warren, 2008). The performance of rubber materials during low temperature is generally characterized by Gehmann test modulus and compression set rate (Spetz, 1990; Mostafa et al., 2009). Weise et al studied fluorine rubber, silicon rubber, and ethylene propylene diene monomer (EPDM) rubber O-rings at low temperature by measuring the gas leakage rate and sealing force during the thermal cycle between +20°C and -70°C (Weise et al., 1992). Jaunich et al proposed a new method for evaluating the low temperature properties of rubber materials by using a considerably short-time test to measure the standardized compression set of sealing materials, and compared the low temperature properties of several common rubber sealing materials. It is found that compression set test can be used as an important supplement to the thermal analysis method to accurately analyse the mechanical behaviour of rubber materials at low temperature (Matthias et al., 2010, 2012). Grelle designed a test device to apply a dynamic load to the seal ring in a period of less than 1 second (Grelle et al., 2015, 2017). And the leakage rate of the elastomer seal under low temperature and dynamic load was studied. Akulichev studied the mechanism of interfacial leakage of HNBR O-ring seals at low temperature (Akulichev et al., 2018). The main cause for the cold seal failures was the detachment of the elastomer seals from their mating sealing parts due to the elastomer thermal contraction and the negligible recovery of the HNBR in cold environment.

Some other studies include low temperature performance of compound rubber, PPR (polypropylene random copolymer) composites and modified asphalt (Bao et al., 2023; Ma et al., 2022; Wang et al., 2023; Xue et al., 2023; Yu et al., 2022).

This paper focuses on reliability and the environmental adaptability of rubber O-rings at low air temperature. Low air temperature failure mechanism of rubber O-rings is studied by theoretical analysis, low air temperature environmental tests. The rest of this article is organized as follows. Section 2 analyzes the main failure modes of O-ring rubber seals. Section 3 briefly describes the working principle and failure criteria of rubber O-rings. Section 4 implements low-temperature environmental adaptability test for rubber O-rings. Finally, a brief conclusion is offered in Section 5.

1. Failure mode analysis of rubber O-ring

In its production, storage, installation and use stages, rubber O-ring will be affected by various factors, resulting in performance degradation and even damage. In this section, the main failure modes of rubber O-ring are analyzed, and the failure causes and manifestations of different failure modes are clarified, so as to lay a foundation for subsequent experimental research.

1.1. Aging failure

The aging failure of the rubber O-ring seal refers to the fracture and cross-linking reaction of the molecular chain of the sealing component material, which changes the molecular structure and composition of the material. The aging of the O-ring rubber seal is mainly caused by ultraviolet rays, high temperature, oxygen and ozone. And in the storage and use of the O-ring rubber seal, it will be hardened, cracked, sticky and other aging phenomena due to the action of these environmental factors. If the O-ring rubber seal is subjected to repeated mechanical stress during the aging process, the aging process of the sealing parts will be accelerated. And the compression deformation generated by the sealing ring will be fixed due to the aging reaction under the static load, that is, the compression set will be permanently generated, which will affect the working performance of the O-ring rubber seal.

1.2. Failure under low temperature

In the low temperature environment, the molecular thermal movement of the O-ring rubber sealing ring is weakened and the molecular chain is frozen, resulting in the decrease of resilience, the increase of hardness and brittleness of the sealing parts. And the sealing performance of the sealing parts is significantly reduced, and it is easy to be damaged due to external force. The O-ring rubber seal in the storage stage does not bear the action of compressive force. And the mechanical properties of the sealing parts decrease in the low temperature environment (Jaunich et al., 2013). But when the temperature returns to normal temperature, the performance of the sealing parts will also be restored. The O-ring rubber sealing ring in the working stage bears the action of pre-compression stress and the pressure of the working medium. And the decrease in the resilience of the sealing parts in the low temperature environment will lead to the decrease of the rebound pressure, the increase of the hardness and

brittleness of the sealing parts. And the extrusion fracture is easy to occur under the action of the pressure of the working medium.

1.3. Other failure modes

In addition to the failure modes caused by the above environmental factors, there are also sealing failures caused by insufficient machining accuracy, improper human operation, and unreasonable dimensional design, such as: the roughness of the sealing surface is too high, the distortion and deformation of the sealing parts during installation, and the unreasonable design of the sealing gap and the size of the sealing groove. Therefore, it is necessary to ensure that the machining accuracy of the sealing structure, the cross-sectional diameter of the sealing ring and the pre-compression ratio meet the requirements, and scientific installation methods should be adopted to avoid the sealing failure caused by human-controllable factors as much as possible.

2. Operating principle and failure criterion of rubber O-ring

In this section, the operating principle of rubber O-ring is analysed, and its failure criteria are determined. The characteristic quantities of low-temperature performance are extracted. Research results can lay a foundation for the experimental study of failure mechanism of rubber O-ring in the low-air-temperature environment.

2.1. Operating principle of rubber O-ring

The rubber O-ring is elastically deformed by the extrusion of the internal sealing structure, thereby generating an initial contact pressure of P_0 on the sealing contact surface. The sealing ring only relies on its own elastic force to achieve the sealing action, as shown in Figure 1(a). When the sealing system is operating, the sealing ring is subjected to the operating pressure of P generated by the sealed medium. It will move to the low-pressure side of the sealing structure. Hence, the extrusion deformation of the sealing ring is increased. The sealing ring is squeezed into the gap of the sealing structure. The contact pressure on the contact surface will increase to P_1 , as shown in Figure 1(b). When the contact pressure P_1 is greater than the operating pressure P of the sealing medium, the sealing system does not leak, and the rubber O-ring achieves its sealing effect (Douglas et al. 2016).



Fig. 1. Schematic diagram of the sealing principle of rubber O-ring: (a) No medium pressure; (b) With medium pressure.

2.2. Failure criteria of rubber O-ring

When the maximum contact stress between rubber O-ring and the seal structure is lower than the operating pressure of the sealed medium, the medium will leak. When the maximum shear stress of the O-ring at the seal groove is greater than the shear strength of the seal material, the rubber O-ring will break. The maximum contact stress and shear stress of a rubber O-ring are called characteristic stresses. Therefore, the general failure criteria for rubber O-ring can be determined as follows.

(1) Maximum contact stress criterion

Maximum contact stress criterion can be expressed by

 $\sigma_{\max} \ge P$

(1)

where σ_{max} is the maximum contact stress and *P* is the medium pressure. Equation (1) must hold to ensure normal operation of rubber O-ring. Under normal operating conditions, the maximum contact stress mainly correlates to the pre-compression stress of the sealing ring and the medium pressure. When the rubber O-ring operates in low temperature environment, the elasticity and hardness of the sealing ring will change, which will affect the maximum contact stress value and change the sealing performance of the rubber O-ring.

(2) Maximum shear stress criterion

Maximum shear stress criterion can be expressed by

 $\sigma_{xy} < \tau_b$

where σ_{xy} is the maximum shear stress and τ_b is the shear strength of the sealing ring. The maximum shear stress of rubber O-ring often occurs at the corner of sealing structure and near the sealing gap. Under normal operating conditions, the maximum shear stress mainly correlates to the size of sealing structure. When the sealing ring operates in low temperature environment, the elasticity and hardness of the sealing ring will change, thus affecting the maximum shear stress value, which may lead to the failure of the rubber O-ring.

In summary, low air temperature environment will affect the elasticity and hardness of rubber O-rings, thus changing the maximum contact stress and shear stress and affecting the sealing performance, which may lead to failure of sealing structure. Ultimately, the low temperature environmental adaptability of rubber O-rings is affected. The resilience of rubber O-ring at low temperature can be expressed by compression set (Jaunich et al., 2010). The smaller the compression set, the better the resilience and sealing performance of rubber O-ring. At the same time, hardness is another important index of rubber O-ring. Therefore, compression set and hardness are selected as indexes to characterize the sealing performance of rubber O-rings at low temperature.

3. Reliability test of rubber O-ring under low air temperature

Silicon rubber O-rings and nitrile rubber O-rings are taken as test units. Test devices and fixtures of low temperature test are specified. The test and measurement plan are designed. Then low temperature environmental adaptability test of rubber O-rings is carried out to study the performance parameters of test units under low temperature environment. Finally, the test results are analysed. The low temperature failure mechanism of rubber O-ring is concluded from a macroscopic point of view.

3.1. Test units

The main chain of silicon rubber is composed of Si-O bond. The bond energy is much higher than that of C-C bond of general rubber molecular main chain. So silicon rubber has excellent elasticity, high and low temperature resistance, and is widely used in high and low temperature and other harsh environments. Nitrile rubber has excellent oil resistance, good wear resistance and heat resistance, and is widely used in vehicle engine, oil exploitation and other oil seal fields.

Therefore, silicon rubber and nitrile rubber O-ring is taken as an example to carry out the low-temperature adaptability test study. The O-ring size is Φ 36×3.5mm (outer diameter × wire diameter). The shape of test units is shown in Figure 2.



Fig. 2. Test units of rubber O-ring: (a) Silicon rubber; (b) Nitrile rubber.

3.2. Test device

The low temperature adaptability test is carried out using a constant temperature test chamber, as shown in Figure 3. The chamber has a volume of $0.8m^3$ and a temperature range of $-70^{\circ}C \sim +150^{\circ}C$ with temperature stability of $\pm 1^{\circ}C$, which meets the test needs for low temperature conditions.



Fig. 3. The constant temperature test chamber used in this test.

Pre-compression stress is applied to the test units using a compression setter as shown in Figure 4.



Fig. 4. Compression set device.

Compression set is usually used to evaluate the recovery performance of elastic seals (as in ISO815-2 Rubber, vulcanized or thermoplastic - determination of compression set - part 2: At low temperatures, 2014). Therefore, a certain amount of compression is applied to the rubber O-ring during the test. After it is kept at the test temperature for a period of time, the fastener of the compression device is loosened. The axial section size of the rubber O-ring is measured after restoring 30 minutes. Based on the axial section size before and after low temperature test, the compression set of rubber O-ring in low temperature can be calculated as

$$k = \frac{d_1 - d_2}{d_1 - h_s} \tag{3}$$

where d_1 is the initial axial section size of O-ring, and d_2 is the axial section size after the O-ring is restored, and h_s is the thickness of the limiter in the compression set.

Hardness is another key performance index of rubber materials. Therefore, the hardness of test units of O-ring is also measured during the low temperature environmental adaptability test. For the hardness of the O-ring specimens with curvature surface cannot be measured by the existing instruments, the cylindrical sample of the same material of the sealing ring is selected. Its size is in accordance with the requirements of ISO 48-4 (Rubber, vulcanized or thermoplastic determination of hardness - part 4: Indentation hardness by durometer method (shore hardness), 2018). The hardness of silicon rubber and nitrile rubber samples at low temperature is measured by Shaw A rubber hardness tester.

3.3. Test design and performance measure methods

According to the temperature extreme value in cold region, temperature levels of -20° C, -30° C, -40° C, -50° C, and -60° C are selected to study the variation of characteristic performance parameters of rubber O-ring with temperature. The test time at each temperature level is 24 hours. After reaching the test time, the axial section size of the sealing ring specimens and the Shore A hardness of the cylindrical specimens are measured.

The temperature level of -50°C is selected as the test temperature to study the effect of operating time under low temperature on the performance of rubber O-ring. When the test time reaches 0.5, 1, 4, 8, 12, 24 and 48 hour, the characteristic performance parameters of rubber O-ring are measured to further explore the failure mechanism of rubber O-ring under low temperature condition.

Three specimens are selected for each group of tests considering the error of test data. The axial section size and the hardness of the specimens is measured every 120° of rotation. So each specimen is measured 3 times, and the average value is taken as the test data of the specimen. Finally, the measurement of the three specimens are averaged as the test data of the group.

3.4. Test results and analysis

(1) Effect of temperature on sealing performance

The compression set and increase of Shore A hardness of rubber O-rings of the two materials under different test temperature is shown in Figure 5, where "SR" and "NR" denote silicon rubber O-ring and nitrile rubber O-ring respectively.



Fig. 5. Compression set and increase of hardness with temperature.

It can be seen from Figure 5 that the compression set and hardness of silicon rubber O-ring and nitrile rubber O-ring gradually increase with the decrease of test temperature. The sealing performance of silicon rubber O-ring at low temperature is obviously better than that of nitrile rubber O-ring. The variation of compression set and hardness of silicon rubber O-ring are still small at -40°C, so the O-ring can maintain good operating performance. When the test temperature is -20°C, the compression set and hardness of nitrile rubber O-ring change significantly indicating poor environmental adaptability of the O-ring at low temperature.

(2) Effect of test time on sealing performance

The compression set and increase of Shore A hardness of rubber O-rings of the two materials at different test time under -50°C is shown in Figure 6, where "SR" and "NR" denote silicon rubber O-ring and nitrile rubber O-ring respectively.



Fig. 6. Compression set and increase of hardness with test time.

It can be seen from Figure 6 that at -50°C, the performance parameters of the silicon rubber O-ring gradually increase in the initial test stage, and gradually tend to stabilize after 12 hours. But the performance parameters of nitrile rubber O-ring can reach a stable state in a short time, and there is almost no stage of gradual change.

silicon rubber is a kind of crystalline rubber, and its properties in low temperature environment are mainly affected by the crystallization process. The crystallinity index of silicon rubber increases with time at low temperature. When the crystallinity index reaches a certain stable value, it almost no longer changes with time. Therefore, only in a certain period of time, the test time will affect the performance parameters of silicon rubber O-ring. However, nitrile rubber is an amorphous rubber, and its properties in low temperature environment are mainly affected by glass transition process. At low temperature, the molecular chain and segment of the chain can reach equilibrium in a short time. Therefore, the test time has little effect on the performance parameters of nitrile rubber O-ring.

(3) Failure mechanism analysis of rubber O-ring at low temperature

The low temperature resistance of rubber O-ring is mainly affected by glass transition and crystallization process (Jaunich et al., 2011). Glass transition temperature refers to the transition temperature from movement to freezing of rubber molecular chain. The level of low temperature resistance of amorphous rubber can generally be expressed by T_g . The crystallization temperature of crystalline rubber is generally higher than the glass transition temperature. For example, the glass transition temperature T_g of dimethyl silicon rubber is about -125°C. However, dimethyl vulcanized rubber will lose its elasticity at -50°C due to strong crystallization, that is, the low temperature properties of crystalline rubber are related to test temperature and duration. So attention should be paid to the effect of time when studying the low temperature properties of crystalline rubber.

The rubber O-ring is in the high elastic state with excellent performance at room temperature. And the rubber O-ring has good elasticity, moderate hardness and brittleness, and can meet the expected sealing requirements. With the decrease of temperature, the thermal movement of the molecular chain of the rubber O-ring material is gradually weakened. The side groups, links and segments of the molecular chain are gradually frozen resulting in the gradual decrease of elasticity and the increase of hardness. When the temperature drops to the glass transition temperature or crystallization temperature, the rubber O-ring will change from the high elastic state to the glass state. The elasticity of rubber O-ring will be lost almost completely (Akulichev et al., 2017; Ilseng et al., 2016). The compression set of the rubber O-ring can be close to or even up to 100%, resulting in the loss of contact of the sealing medium, so the medium leakage is easy to occur. And the rubber O-ring material becomes hard and brittle and may occur cracking due to the pressure of the sealing medium.

4. Conclusions

Operating principle and failure criterion of rubber O-ring are analysed. The general failure criteria for rubber O-ring are maximum contact stress criterion and maximum shear stress criterion. When the maximum contact stress is less than the medium pressure or the maximum shear stress is greater than the shear strength of the sealing ring, rubber O-rings will fail. The main performance parameters of rubber O-ring are determined as hardness and

elasticity. The laboratory low air temperature tests of silicon rubber and nitrile rubber are carried out. The failure mechanism of rubber O-ring under low air temperature is revealed that the low temperature resistance of rubber O-ring is mainly affected by glass transition and crystallization process. The test results show that the low temperature sealing performance of silicon rubber sealing ring is good, which is mainly affected by the crystallization process of rubber material. At the same time, we should pay attention to the effect of low temperature test time to ensure the accuracy of the test results. The nitrile rubber O-ring has poor sealing performance at low temperature, so it is not suitable to be selected as the sealing part in low temperature environment.

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