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ORIGINAL PAPER

Influence of Thermal Aging in Oil on the Friction and Wear Properties of Nitrile Butadiene Rubber

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Abstract

An experimental study on the influence of thermal aging on the friction and wear properties of a nitrile butadiene rubber (NBR)–steel pair was carried out. Rubber specimens were aged in hydraulic oil at 60 °C and 90 °C for 0–70 days. The Shore hardness, Young's modulus, rebound resilience, and other mechanical properties were measured. The friction coefficient and wear volume were tested to evaluate the tribology performance. To investigate the mechanism of NBR thermal aging, the molecular structure and crosslinking degree were analyzed. Crosslinking with rubber aging in oil led to differences in the physical and mechanical properties. The Shore A hardness decreased initially and then increased. The Young's modulus increased and the rebound resilience decreased. The friction coefficient increased but the wear volume decreased with aging time. These differences affected the friction and wear behavior of the NBR against the steel shafts and resulted in sealing-ring degradation and failure.

Keywords Nitrile butadiene rubber · Aging · Friction · Wear

1 Introduction

Nitrile butadiene rubber (NBR) is the most commonly used polymer material for oil seals and bearings owing to its good oil resistance, wide working-temperature range, and low cost [1, 2]. In general, oil seals are used in industry to retain lubrication and to exclude contamination in rotating shafts and bearing applications [3].

However, because the oil seals are immersed in hydraulic oil during operation, swelling and aging change the material composition and structure, which degrades the mechanical properties of the NBR [4], and results in a reduction in oil seal lifetime. Friction and wear that result from the rotation of the steel shaft also increase the leakage rate and weaken the sealing properties in dynamic seal applications [5]. Aging, friction, and wear have become a significant problem in the field of rubber and plastic dynamic seals.

Aging in oil of polymer materials, such as NBR, results from lubricant absorption by the elastomer, constituent leaching from the elastomer into the lubricant, and the chemical reaction between components in the lubricant and the elastomer [5], after which many mechanical properties (such as the hardness, elasticity modulus, and impact resilience) and the chemical composition of the NBR change [4, 6–8]. High temperatures accelerate elastomer aging, which is common in sealing applications. Erol et al. [6] found that thermal treatment and accelerated aging increase rubber specimen hardness. In their study, different types of rubber specimens, natural rubber (NR), nitrile butadiene rubber (NBR), fluoro rubber (FKM), chloroprene rubber (CR), styrene butadiene rubber (SBR), ethylene propylene diene elastomer (EPDM), and hydrogenated nitrile butadiene rubber (HNBR) were investigated, and NBR and FKM behaved best under different working conditions. However, Lv et al. [4] investigated the effect of cyclohexane on the swelling-induced aging of NBR, and pointed out that with an increase in aging time, the incremental NBR swelling increased, the hardness and shear strength decreased, and the surface damage worsened. Dong et al. [8] found that as the

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aging time and temperature increased, the NBR Shore hardness and tensile and tear strengths decreased.

During the thermal aging of rubber, two main processes occurred: crosslinking and oxidative degradation [9]. Zhao et al. [10] stated that during thermooxidative aging, the opposite processes were observed via a nuclear magnetic resonance (NMR) spectrometer. The network became denser and harder because of the continuous increase in crosslinking density. Network chains broke down after high oxidation. Changes in crosslinking and degradation will result in changes in the mechanical properties, including the tensile strength, tensile modulus, elongation at break, tear strength, compression set, and stress at a certain strain [11, 12]. Morrell et al. [13] investigated the accelerated thermal aging properties of NBR O-rings, and found that the main influence on rubber aging was oxidative crosslinking, which lead to the material becoming hard and brittle.

These changes in mechanical properties during thermal aging will influence the friction and wear properties of rubber for sealing, which is critical in the performance and lifetime of rubber and plastic dynamic seals, the energy-consumption losses of machinery and equipment and to prevent pollution [14, 15]. Friction between the elastomer and hard counterface materials comprises a hysteresis and adhesion component [16]. The hysteresis friction and adhesion friction are related to the Young's modulus of the elastomer [17, 18]. Differences in the surface morphology and components can also result in differences in the friction coefficient of rubber [1, 19, 20]. Previous studies focused mostly on the physical or chemical changes themselves [9–11, 21], not on the tribological performance, or friction tests under dry conditions [22, 23]. Aging and wear occur simultaneously while the sealing rings are working, but previous studies only investigated one of these, and few people have studied the impact of aging on wear.

The aims of this work were to investigate the aging performance and friction behavior of NBR, and the influence of thermal aging on the friction and wear properties of NBR in oil. The Shore hardness, Young's modulus, and rebound resilience at different temperatures during aging were determined. The surface morphology, molecular structure, and crosslinking degree were also investigated under conditions of high-temperature thermal aging. The results were used to explain the influence and mechanism of thermal aging on the friction and wear properties of NBR in oil.

2 Experimental Details

2.1 Specimens and Aging Conditions

When oil seals are in use, they are usually exposed to high-temperature oil, such as 80–90 °C in a hydraulic system and 60–70 °C for lip seals [6]. To investigate how thermal aging in oil affects the tribological performance of the NBR, a constant-temperature oil-bath aging test was used to simulate the aging behavior and to provide aged material samples. NBR specimens were supplied by Anhui Zhongding Co., Ltd. (China) in the form of 18 mm × 12 mm × 12 mm cuboids, and these were placed in an oil bath. The material composition is shown in Table 1. The aging oven was set at 60 °C and 90 °C to investigate the influence of different temperatures. A vessel that contained hydraulic oil #32 (SINOPEC, dynamic viscosity 0. 027 Pa.s at 40 °C and 0. 004 Pa.s at 100 °C) was placed into an aging oven (Type 401-A; Shanghai Exp. Company, China) for preheating, after which, all rubber specimens were inserted into the vessel. Table 2 summarizes the aging temperature, aging time, rubber specimens, and hydraulic oil used in this test.

Table 1 Concrete material compositions

Content	Weight percentage (wt%)
NBR (26 wt% acrylonitrile)	37.4
Carbon black	48.7
Vulcanization accelerator	2.5
Plasticizer	7.5
Other	3.9

Table 2 Specimens and aging conditions

Content	Details
Aging temperature (°C)	60, 90
Aging time (day)	0, 1, 2, 3, 5, 7, 10, 20, 30, 50, 70
Rubber specimens (mm)	18 × 12 × 12
Hydraulic oil	#32, SINOPEC

2.2 Friction and Wear Measurements

Friction tests were conducted using a standard block-on-ring testing machine (MR-5H; Jinan Shunmao Testing Machine Group, China) under the same hydraulic oil (SINOPEC #32)-lubricated conditions. A schematic diagram of the friction pair is shown in Fig. 1. The stationary block sample was NBR, and the rotational ring sample was GCr15 Steel. The surface of the ring was polished to $Ra = 0.4$ μm to remove the helical machining grooves. The normal loads applied to the samples were 100 N, 180 N, 250 N, and 300 N, which correspond to a maximum contact pressure of 1.11 MPa, 1.5 MPa, 1.76 MPa, and 1.93 MPa, respectively.

The rotational speeds were chosen as 200 rpm, 500 rpm, 1000 rpm, and 1500 rpm, which correspond to a linear velocity of 0.151 m/s, 1.289 m/s, 2.578 m/s, and 3.867 m/s, respectively. The above test conditions are in the typical working range of a dynamic oil seal [24–28]. To evaluate quantitatively the wear extent of NBR after aging as a vital indicator for determining the lifetime of dynamic oil seals, the wear volumes of original and aged NBR after 0, 5, 7, 20, 50, and 70 days at 90 °C in an oil bath after a 4-h friction test at 300 N and 1.289 m/s were obtained by comparing the surface profile before and after wear. For each test configuration, at least three tests were conducted to confirm the repeatability of the results.

For rubber materials, contact measurement creates additional pressure, which can cause deformation of the rubber surface and affect measurement results. Therefore, a non-contact optical measurement method is used in this study by a MicroXAM-3D confocal microscope.

2.3 Mechanical Properties Measurements

The tribological behaviors of polymer materials are affected by the surface and substrate properties, especially the hardness and Young's modulus [4, 29–31]. As a kind of sealing material, the high rebound resilience of the NBR is important to reduce the leakage value [3, 28]. To investigate the mechanism of aging and friction, the factors above were measured in this work. The NBR hardness was measured by using a Shore A durometer (HLX-AC; Handpi Instrument Co., Ltd; Leqing, China) with an accuracy of 0.1 Shore A. The Young's modulus was measured by using a uniaxial compression testing machine (WDW-3020; Weidu Instrument Co., Ltd; Wenzhou, China; load range 0–2 kN, displacement accuracy 0.01 mm). The rebound resilience was tested by using a rubber elastic resilience tester (FRD-6074; Furuda Instrument Co., Ltd; Dongguan, China, reading accuracy of ± 0.5%) according to GB/T 1681–2009. All tests were carried out at room temperature.

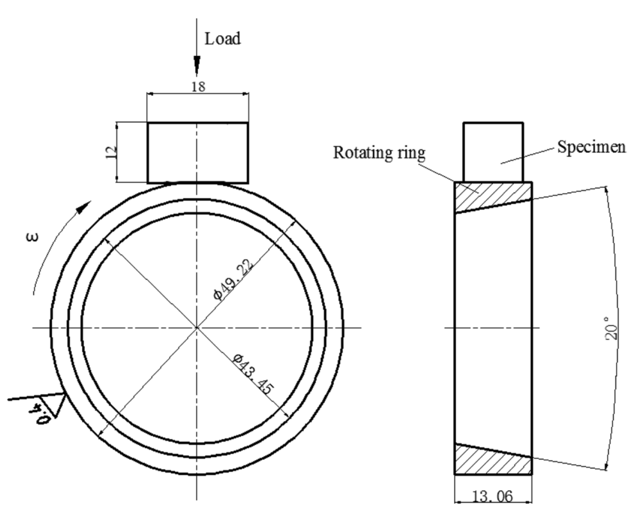

Fig. 1 Schematic diagram of frictional couple (mm)

2.4 NMR and Fourier-Transform Infrared (FTIR) Measurements

Crosslinking and oxidative degradation occur simultaneously with NBR aging, and the crosslinking density influences the mechanical and chemical properties of the NBR [10, 21]. To measure the crosslinking density and proportion of crosslinked chains and tail suspension chains of the NBR aged for different days, a MR-CDS3500 NMR spectrometer (IIC Co., Ltd., Germany) was used to characterize aged specimens. The NMR is very sensitive to changes in molecular motion, which can be used to detect the spin–spin relaxation time of chains and to gain useful structural information about the network, such as the distinction of crosslinked chains from free and dangling chains.

As the aging process evolves, the molecular structure of the NBR changes, which affects the network in the substrate and the mechanical properties. A Nicolet 6700 FTIR (Thermo Fisher Scientific Co., Ltd; USA) was used to establish the molecular structure of the NBR specimens that were aged for different times.

2.5 Surface Characterization Measurements

To calculate the wear volume, a MicroXAM-3D confocal microscope (Zygo; USA) was used to measure the surface morphology and profile of the aged NBR specimens. A Quanta 200 FEG (FEI; USA) scanning electron microscope was used to observe the aged specimen microstructure, and to detect the separation of internal additive components.

3 Results and Discussion

3.1 Mechanical Properties

The NBR sample was immersed in oil, and the increase in quality was caused by the immersion of the oil. The quality of the samples gradually increased within 0 to 10 days, indicating that the oil was immersed inside the sample at this stage, and the hardness of the sample was decreased. The quality of the aged samples at 90 °C increased more, indicating that more oil was immersed and therefore the hardness was lower. Thus, the more the oil that got into the rubber, the lower the hardness. After 10 days, the amount of oil immersion decreased, reached saturation, and the quality of the sample hardly changed (Fig. 2). The change in hardness thereafter was mainly affected by the crosslink process.

Figure 3 shows the variation in Shore A hardness with aging time at 60 °C and 90 °C. With an increase in aging time, the Shore A hardness at both temperatures decreased firstly from 0 to 10 days, increased gradually from 10 to 70 days, and reached a minimum at 10 days. In addition to the original value, the hardness of NBR aged at 90 °C was larger than that aged at 60 °C.

In the early stage, hydraulic oil penetration into the NBR network structure was dominant and softened the rubber. The first stage lasted for 10 days, and then reverted to another process. When the penetration process reached saturation, the oil could not get into the surface anymore, and thus the physical interaction had little to do with the specimen hardness. In the later stage of aging, the crosslinking process mainly affects the rubber hardness, and thus the Shore A hardness increased as the proportion of crosslinked chains increased for specimens aged for 10–70 days.

Figure 4 shows the variation in Young's modulus of NBR aged for different days. The Young's modulus increased rapidly for 0–30 days, and then changed slowly. The oil penetration stayed on the surface and did not affect the Young's modulus a lot. In general, the Young's modulus of specimens aged at 90 °C was larger than that of specimens aged at 60 °C.

Results of the rebound resilience (%) of NBR aged for different days are shown in Fig. 5. The rebound resilience properties of specimens at 60 °C and 90 °C decreased as the aging time increased, and that at 90 °C was lower than that at 60 °C, which meant that the resilience of NBR rubber was degraded during its operation in oil, and a high temperature accelerated this process. Molecular chains in the rubber cross-linked gradually and the network structure became tighter, and thus the elasticity and resilience were lost and the sealing rings failed.

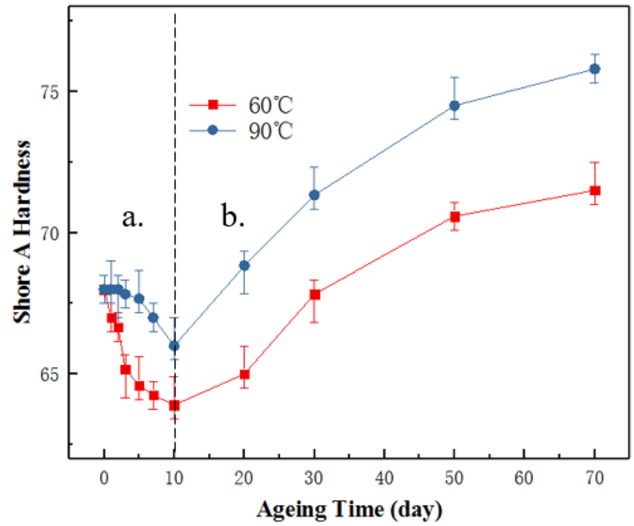

Fig. 3 Shore A hardness of NBR aging in hydraulic oil for 0–70 days

The surface microstructure and elemental distribution of the aged NBR are shown in Fig. 6. Elemental Mg, Ca, Na, S, Si, K, and Al were detected on the NBR specimen surface aged for 30–70 days. The element types and contents were the same as those of the internal NBR additives such as antioxidant and vulcanizator, which means that the internal additives separated out during aging. These additives were harder than the rubber, and thus the Shore A hardness increased with long-term aging. Similar results were found by Wang et al. [7], who reported the absorption of base oils by rubber, whereas aldehyde-, sulfur- and zinc-containing species were leached out by the base oils.

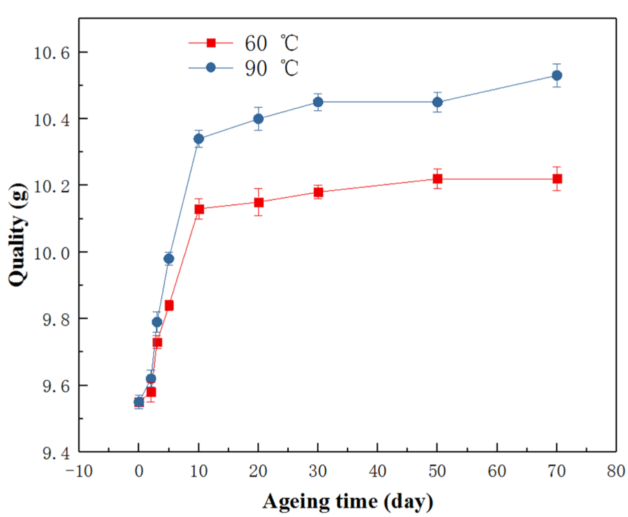

Fig. 2 Quality of NBR aging in hydraulic oil for 0–70 days

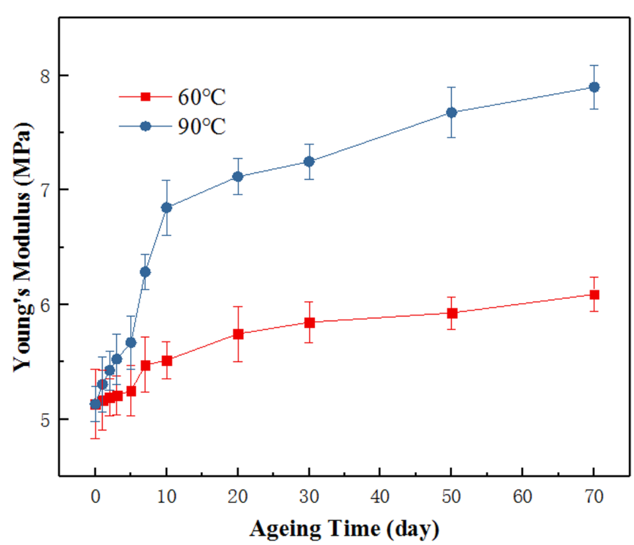

Fig. 4 Young's modulus of NBR aging in hydraulic oil for 0–70 days

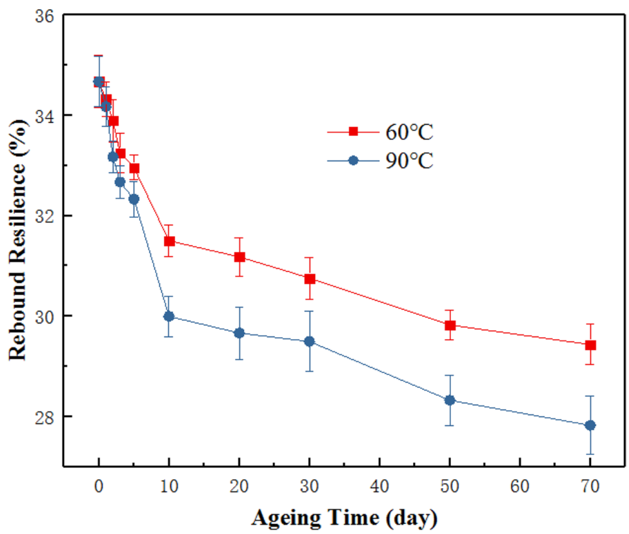

Fig. 5 Rebound resilience of NBR aging in hydraulic oil for 0–70 days

3.2 Friction and Wear Properties

The average friction coefficient between the aged NBR specimens and the GCr15 steel ring is shown in Fig. 7. The friction coefficient of the NBR specimens aged for 1 day was much lower than that of the original specimens. At this time, the hardness, Young's modulus, and rebound resilience changed only slightly, which meant that during the very early period of oil aging, the main process that affected the friction coefficient of the NBR was the permeation of oil into the substrate rubber. The oil moistened the superficial layer and made the surface smoother, and thus the friction coefficient was much lower. Generally speaking, the friction surface and the surface layer a few micrometers below the surface undergo plastic flow and transfer load during the friction process. The composition and structure of the surface layer are deviated from the original surface material due to the influence of the environmental medium. This area is called the superficial layer. The friction coefficient increased rapidly during the first 10 days of aging, and then increased slowly during the remaining period. Compared with the hardness curve and Young's modulus curve, in the first 10 days, the Shore A hardness decreased, which resulted in an increase in real contact area, and the Young's modulus increased, meaning that the elastic deformation was more difficult, which resulted in a decrease in the lubrication film thickness. Both factors contributed to an increase in friction coefficient. During the remaining period, the hardness increased, which counteracted the influence of the Young's modulus, and thus the friction coefficient increased slowly. It is worth mentioning that the friction coefficient of the NBR aged at 90 °C was larger than that of the NBR aged at 60 °C, which meant that a high temperature was an important factor that led to a deterioration in NBR friction properties.

The normal load and sliding speed can affect the lubrication and friction of the rubber–steel pair [19, 20]. Results of the variable load and sliding speed are shown in Fig. 8. As shown in Fig. 8a, the friction coefficient of the NBR specimens increased as the normal load increased, despite the oil aging length. NBR has a poor heat conductivity, and dissipates heat generated during friction with difficulty. When tested at a high pressure, this effect was more significant, and led to an increase in friction coefficient. Modern tribology studies have shown that normal loads can affect the actual contact area of the friction pair, and the shape and distribution of surface asperities [1]; thus, an increase in normal load resulted in an increase in friction coefficient. However, this effect was not significant under a lower load (less than 180 N); the influence of friction heat was slight and the increase in Young's modulus was most prominent, and thus the friction coefficient of the aged specimens was larger than that of the original specimens at 100 N. The changing range of aged specimens was narrower than that of the original specimens, which meant that oil that permeated in the rubber made it less sensitive to changes in load.

Figure 8b shows that NBR specimens aged for 0–2 days showed different trends from those aged for a long time (longer than 3 days). The minimum friction coefficient of the NBR specimens aged for 0–2 days appeared at 1.289 m/s, that of specimens aged for 3–30 days appeared at 2.578 m/s, whereas that of specimens aged for 50–70 days appeared at 3.867 m/s. In this work, the main factor that affected the lubrication was the contact between asperities on the NBR and steel surfaces. Because of the separation of internal additive while aging, the surface property was changed, and thus the lubrication condition was different between specimens aged for different times.

The wear volume results are shown in Fig. 9. The surface profiles of the NBR specimens before and after the wear test are shown in Fig. 9a, b. The profile data were processed as shown in Fig. 9c and the wear volume curve is shown in Fig. 9d. A significant volume difference resulted before and after the test. The wear volume curve in Fig. 9d shows that the wear volume decreased rapidly during the first 10 days of aging, and then decreased slowly during the remaining period. Because of the increase in proportion of crosslinked chains, the molecule network strengthened and the Young's modulus increased. The hardness should also have increased but oil permeation affected the measurement of hardness. Thus, in the wear test, the trend in wear volume matched the Young's modulus curve, but was slightly different from the hardness curve. In general, a strengthening of the substrate rubber made it more wear-resistant.

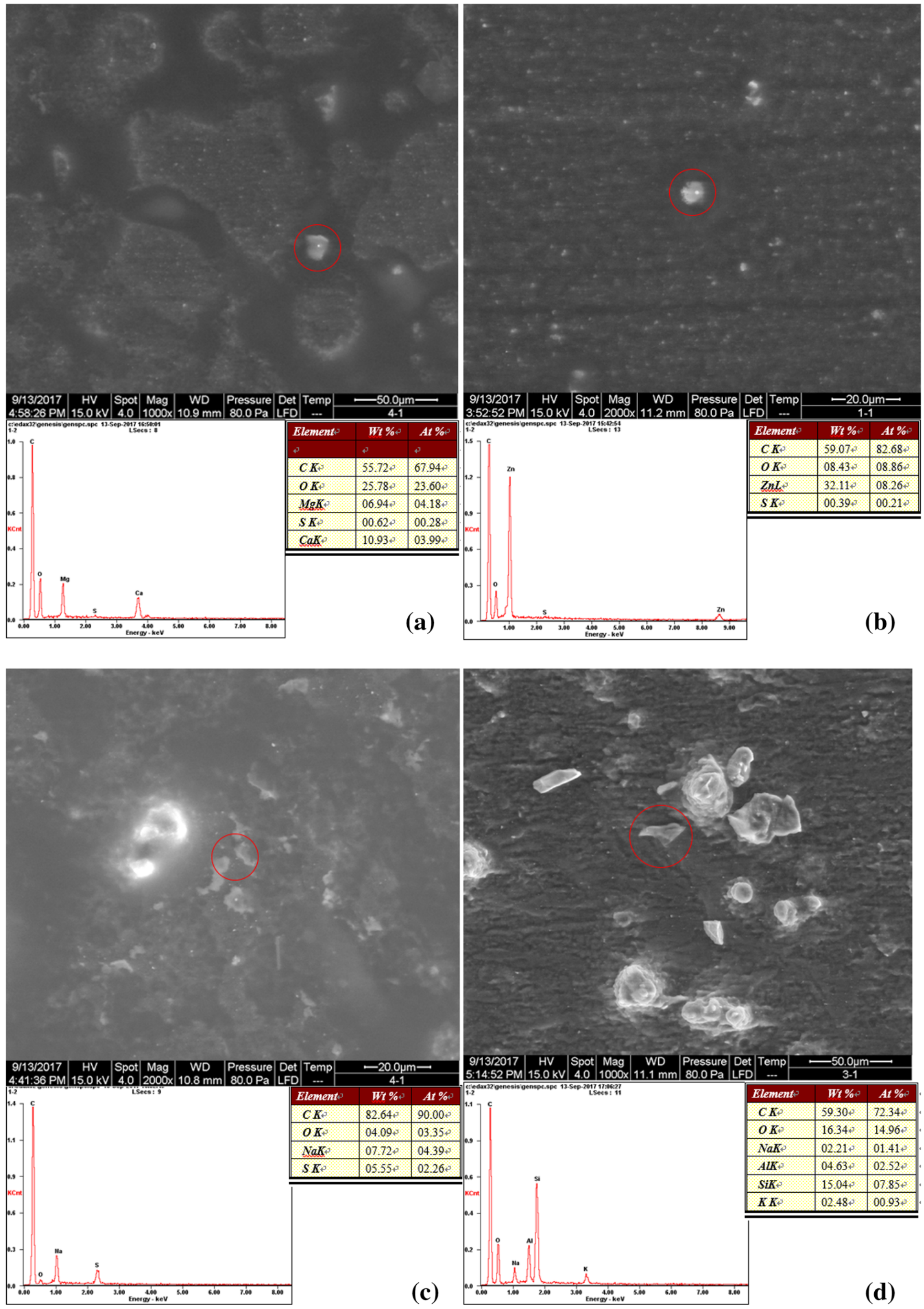

Fig. 6 Surface microstructure and elemental distribution of aged NBR

3.3 Aging Mechanism Analysis

As shown in Fig. 10, the proportion of crosslinked chains in the NBR specimens increased rapidly for 0–10 days, and increased slowly for 10–70 days. The network became denser and harder because of the continuous increase in crosslinking density. Network chains broke down after high oxidation. Because of the presence of unsaturated double bonds in butadiene units, the NBR becomes hard and brittle, and cracks often occur during the thermal process because of the predominant oxidation and crosslinking reactions [21]. The carbon atoms adjacent to double bonds are easily attacked because of electron shifts, and they form radicals to initiate thermal oxidation and crosslinking reactions during aging [32–34]. The number of acrylonitrile units and the curing agents, fillers, and other components affect the thermal stability of the NBR compound [12]. The proportion of crosslinked chains in the NBR specimens increased as the aging time increased, which meant that the crosslinking speed exceeded the oxidative degradation under the experimental conditions. Thus, the dominant

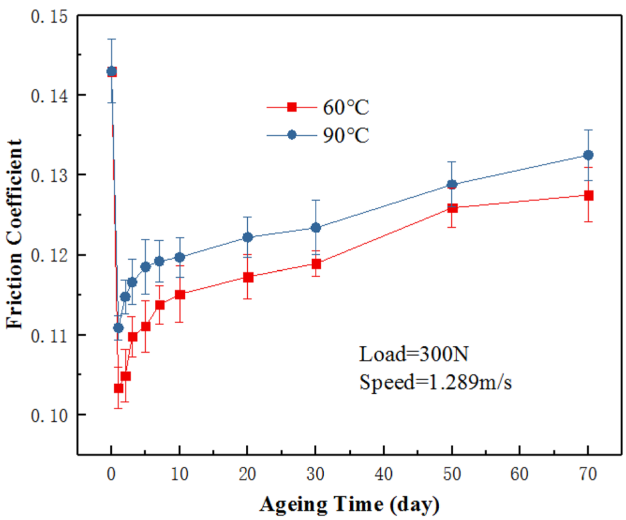

Fig. 7 Friction coefficient of GCr15 steel ring and NBR aging in hydraulic oil for 0–70 days

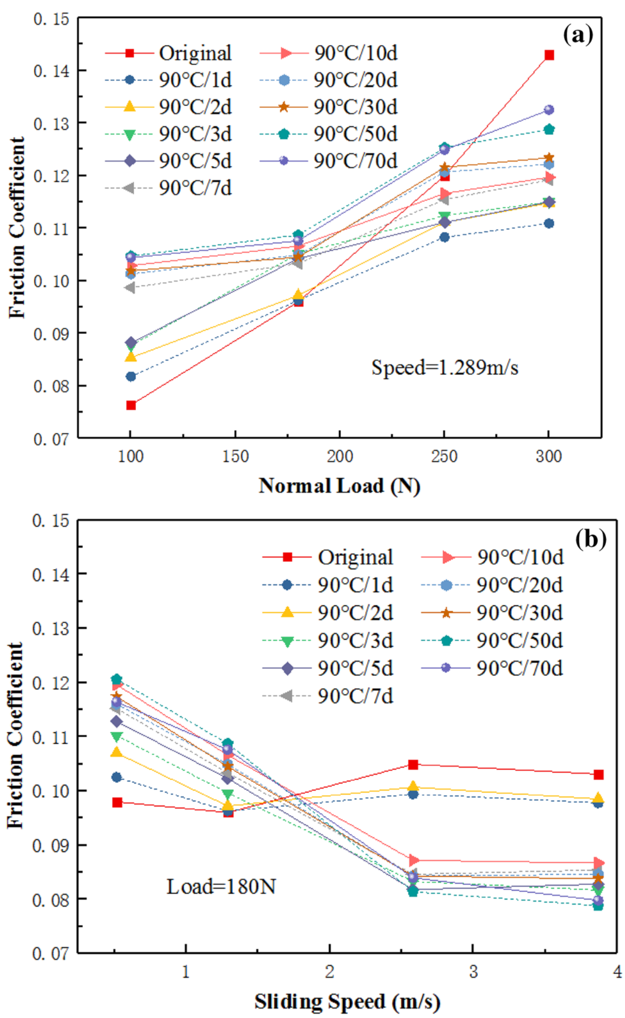

Fig. 8 Friction coefficient of GCr15 steel ring and aged NBR under variable loads and speeds

mechanism in this process was crosslinking. The proportion of crosslinked chains in the NBR aged at 90 °C was higher than that aged at 60 °C, which meant that the crosslinking process was accelerated by a high temperature, and thus, the Young's modulus and surface hardness were larger.

3.4 FTIR Analysis

Physicochemical modifications of NBR before and after aging are shown in Fig. 11. The aged specimens had weaker absorption peaks compared with the original specimens. The aged specimen baseline drifted, possibly because of the separation of carbon black and other internal additives. Rubber is a typical organic polymer material that is formed by crosslinking. The molecular chains that participate in the crosslinking usually have a high molecular weight. During aging in oil, the crosslinking and oxidative degradation reactions change the proportion of crosslinked chains, tail suspension chains and individual chains, and the bond chemistry. As shown in Fig. 11, compared with the spectra of the original specimens, the absorption peaks at 3356 cm^{-1} (N–H band), 3182 cm^{-1} (–OH group), 1630 cm^{-1} and 1659 cm^{-1} (C=C band), 1468 cm^{-1} and 1410 cm^{-1} (vibration bands of C–H band), and 700 cm^{-1} (=CH band) were weakened for the NBR specimens aged in oil for 2 to 5 days. These absorption peaks disappeared when specimens were aged for longer than 10 days. The absorption peaks at 2920 cm^{-1} and 2848 cm^{-1} are attributed to the stretching vibration bands of C–H that were weakened with an increase in aging time. The weakness of the absorption peaks meant that the individual chains decreased with aging, more crosslinked chains formed, and the substrate rubber hardened and strengthened.

The thermal aging of sealing rubber in oil after long-term immersion in hydraulic oil is inevitable during the lifetime of sealing rings. Because of lubricant immersion and chemical reaction, aging in the oil leads to changes in the physical and chemical properties of rubber. During aging, oil penetrated into the network structure of the NBR, which results in a rapid decrease in hardness and friction coefficient during the early period. A combination of crosslinking and oxidative degradation led to an increase in proportion of crosslinked chains in the NBR, which resulted in an increased hardness and Young's modulus, reduced rebound resilience, and increased friction coefficient, which had a deleterious effect on oil sealing. The wear volume decreased with rubber strengthening. The internal additive in NBR separated to the surface and affected the surface properties.

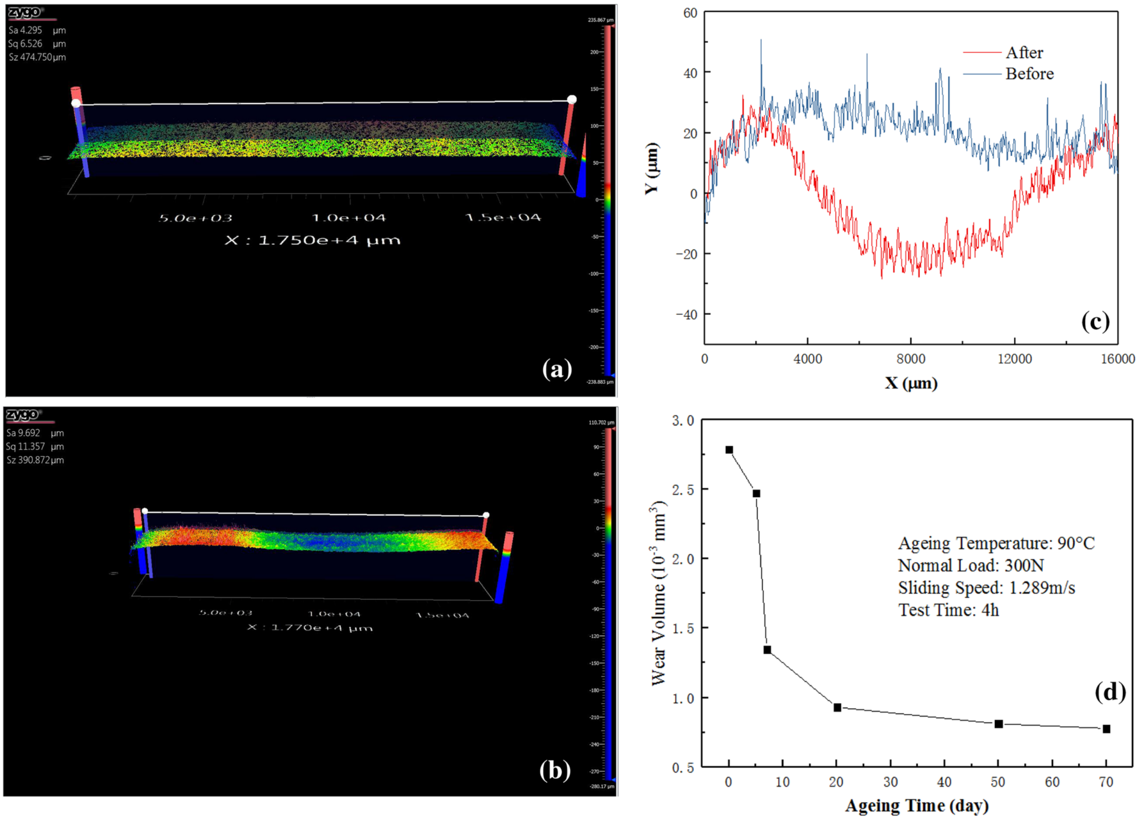

Fig. 9 Wear volume measurement of aged NBR

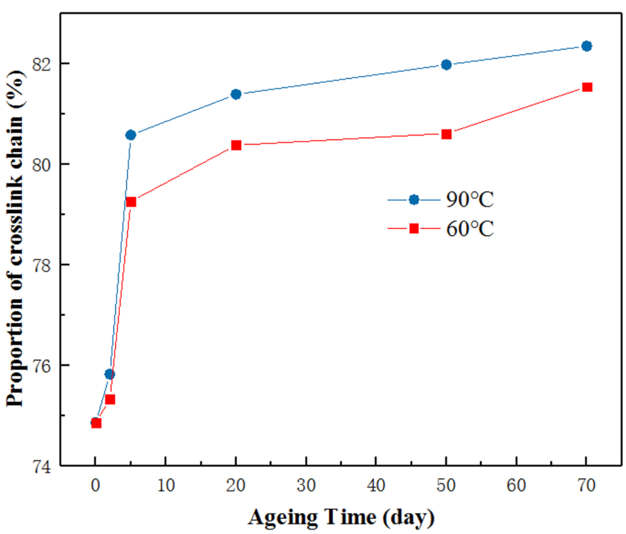

Fig. 10 Proportion of crosslinked chain in NBR specimens aged in hydraulic oil for 0–70 days

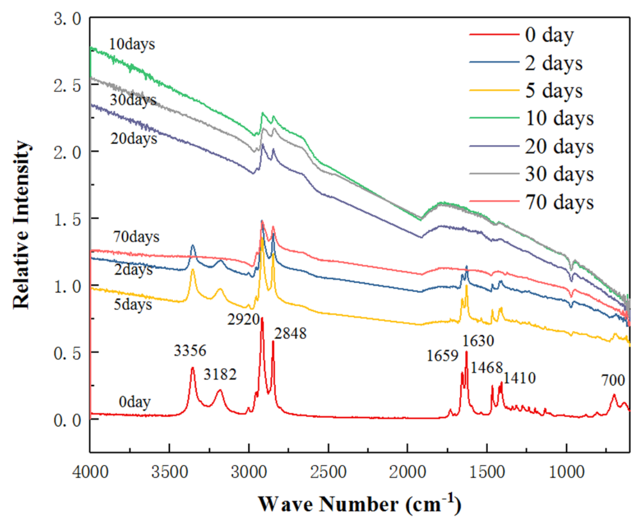

Fig. 11 FTIR spectra of NBR specimens aged in hydraulic oil for 0–70 days

4 Conclusions

The influence of thermal aging in oil in the friction and wear behavior of NBR was investigated. The following conclusions can be made:

1. During aging, crosslinking dominated in a combination of crosslinking and oxidative degradation. The number of individual chains decreased with aging, more

crosslinked chains formed, and the substrate rubber hardened and strengthened.
2. Because of oil permeation, the Shore A hardness of the NBR decreased initially and then increased. The Young's modulus increased and the rebound resilience decreased, which was harmful to the sealing rubber.
3. The friction coefficient increased but the wear volume decreased with aging time.
4. Internal additives in the NBR separated out with aging.
5. The NBR aging performance and mechanism for different aging temperatures (60 °C and 90 °C) were similar, but that at 90 °C differed significantly.

In general, crosslinking with rubber aging in oil led to differences in the physical and mechanical properties. These differences affected the friction and wear behavior of the NBR against steel shafts and resulted in the degradation and failure of sealing rings.

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