Lifetime Prediction and Aging Behaviors of Nitrile Butadiene Rubber under Operating Environment of Transformer

Yi-hua Qian[†], Hong-zhao Xiao**, Ming-hao Nie**, Yao-hong Zhao*, Yun-bai Luo** and Shu-ling Gong**

Abstract – Based on the actual operating environment of transformer, the aging tests of nitrile butadiene rubber (NBR) were conducted systematically under four conditions: in air, in transform oil, under compression in air and under compression in transform oil to studythe effect of high temperature, transform oil and compression stress simultaneously on the thermal aging behaviors of nitrile butadiene rubber and predict the lifetime. The effects of liquid media and compression stress simultaneously on the thermal aging behaviors of nitrile butadiene rubber were studied by using characterization methods such as IR spectrosc-opy, thermogravimetric measurements, Differential Scanning Calorimetry (DSC) measurements and mechanical property measurements. The changes in physical properties during the aging process were analyzed and compared. Different aging conditions vielded materials with different properties. Aging at 70°C under compression stress in oil, the change in elongation at break was lower than that aging in oil, but larger than that aging under compression in air. The compression set or elongation at break as evaluation indexes, 50% as critical value, the lifetime of NBR at 25°C was predicted and compared. When aging under compression in oil, the prediction lifetime was lower than in air and under compression in air, and in oil. It was clear that when predicting the service lifetime of NBR in oil sealing application, compression and media liquid should be involved simultaneously. Under compression in oil, compression set as the evaluation index, the prediction lifetime of NBR was shorter than that of elongation at break as the evaluation index. For the life prediction of NBR, we should take into account of the performance trends of NBR under actual operating conditions to select the appropriate evaluation index.

Keywords: Nitrile butadiene rubber, Aging; Lifetime prediction, Compression stress, Transform oil

1. Introduction

According to the summary of transformer work of the China southern power grid company in 2015, main transformer in entire network transformer faced serious problem of oil leakage, accounting for 15% of total defects. The main reason is due to seal rubber aging crack deformation. In addition, the oil leakage problem resulting from the rubber sealing material directly affects the safe and stable operation of the equipment. Thus, it is necessary for the research on lifetime prediction and aging behaviors of nitrile butadiene rubber under operating environment of transformer.

Nitrile rubber (NBR), the copolymer of butadiene and acrylonitrile, is an important material in many applications due to its good oil resistance and low gas permeability. It is often used in sealing applications, particularly in oil and gas distribution. Due to the unsaturated backbone of the butadiene part, nitrile butadiene rubber is, however, relatively sensitive to aging (Grassie and Heaney 1975; Datta et al. 2007; Kuczkowski 2011). After used for a period of time, it usually becomes hardened, and often loses its sealing capability. Evaluation of aging status and lifetime prediction can give useful information to the rubbers working properly.

The degradation and aging mechanisms of NBR have attracted a great deal of attention. Much work hasbeen done to study the aging behavior as well as the aging mechanism at elevated temperature, and interest still continues (Morrell et al. 2003; Hakkarainen et al. 2003; Kawashima and Ogawa 2005; Woo et al. 2010; Choi and Kim 2012; Bystritskaya et al. 2013; Zhao et al. 2015; Liu et al. 2016; Zhu et al. 2016). Besides temperature, UV light (Zhang and Chang 2015) and Ozone (Hara et al. 1998) aging also affected by applied load type, aggressive substances, and so on. The accelerating effect of applied mechanical loading on aging has also been observed. Xiong et al. studied the aging behavior of sulfured nitrilebutadiene rubber under the bending and tensile-bending stress, and calculated the storage life of NBR according to Arrhenius equation by using high temperature accelerated aging method and the criterion of elongation. It was found that stress accelerated the aging process and shortened the

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[†] Corresponding Author: Electric Power Research Institute of

Guangdong Power Grid Corporation, China. (qianyh@163.com) * Electric Power Research Institute of Guangdong Power Grid

Corporation, China. (kennyaoo@126.com) ** College of Chemistry and Molecular Science, Wuhan University, China.

^{({819966037, 734559262}@}qq.com, {ybai, gongsl}@ whu.edu.cn) Received: August 21, 2017; Accepted: December 27, 2017

storage life of NBR (2012). A research of the dependence of elongation at break of NBR on temperature and air pressure revealed the importance of air pressure as an accelerator in thermal degradation (Budrugeac 1995). Zhu et al. investigated the effect of different levels of uniaxial tensile strain on the aging of hydrogenated nitrile butadiene rubber by using fracture strain as the index of the material's performance. The results demonstrated that applied strain showed an equivalent accelerating effect on the aging of HNBR to that of elevated temperature (2015).

Many authors have also presented studies describing the aging behavior of various nitrile butadiene rubbers in chemical environments. Trakarnpruk et al. reported that NBR showed high increase in hardness, decrease in tensile strength and elongation at break after immersion in biodiesel (2008). Alcock et al. found that the HNBR compounds underwent a large increase in tensile stiffness and reduction in strain to failure when exposed to mixed hydrocarbon fluids (toluene, heptane and cyclohexane) at elevated temperatures and pressures (2015). Lv's research indicated that the swelling-induced aging of NBR samples accelerated its wear process (2015). Buckley et al. studied the influence of liquid media on lifetime predictions of nitrile butadiene rubber and it was determined that the activation energy for oxidative degradation of a particular NBR O-ring had a value, 82 kJ/mol, that was invariant to the immersion liquid (2014).

Currently, aggressive substances or applied load on the aging behavior of NBR is separately studied in most of the aging tests. However, NBR components experience complex mechanical loads in aggressive chemical environments in real applications, such as gaskets in transformer. It is well worth studying the effect of aggressive medium and applied load simultaneously on the properties of aged NBR. Historically, tensile and compressive stress-relaxation methods have been utilized to monitor and predict material lifetimes, but little work concerns the comparison of prediction lifetime by using fracture strain and compression set as the indexes of the material's performance. In this paper, the effect of liquid media and compression stress simultaneously on the thermal aging behaviors of nitrile butadiene rubber for oil seal applications was studied. The aging tests were conducted systematically under four conditions: in air, in transform oil, under compression in air and under compression in transform oil at a series of temperatures. Also, the prediction lifetime by using fracture strain or compression set as the index of the material's performance, was compared.

2. Experimental Section

2.1 Materials

Vulcanized NBR in sheet form with a thickness of 2 mm and cylindrical specimens with 12.5 mm thickness and 29



Fig. 1. Size of dumb-bell specimen

mm diameter were supplied by Hangzhou Dongqing Rubber Company Limited, China. The sheet was punched into dumb-bell specimens according to ISO 37-2011, as shown in Fig. 1. SINOPEC #25 transformer oil was supplied by China Petroleum & Chemical Corporation, China.

2.2 Accelerated aging tests

Thermooxidative aging tests were carried out in a convection oven. The NBR specimens were placed between stainless steel plates that were screwed together to compressing the samples to a constant deflection (25%), and then immersed fully in the SINOPEC #25 transformer oil contained in a covered vessel or put directly in the oven for aging. For comparison, other specimens, in a free state without compression, were also immersed in the oil bath or put directly in the oven for aging. The aging temperature was chosen as 70°C, 90°C, 110°C, 130°C.

2.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analyses were performed on a Fourier transform infrared (FTIR) spectrometer (Nicolet iS10, Thermo Scientific, USA) with a smart multi-bounce HATR accessory equipped with a germanium crystal. The measurements were taken in the range 4000-700 cm⁻¹ at room temperature with a resolution of 2 cm⁻¹ and accumulation of 32 scans.

2.4 Thermogravimetric (TG) measurements

Thermogravimetric measurements were performed on a Setsys Thermo Gravimetric Analyzer (Setaram, France). Specimens weighing $10\sim15$ mg were heated under a nitrogen atmosphere from ambient temperature up to 700° C at a heating rate of 10° C /min.

2.5 Differential Scanning Calorimetry (DSC) measurement

DSC measurements were performed on a DSC-204 instrument (Netzsch, Germany) under nitrogen atmosphere. Crucibles were of the aluminium type. The testing

temperature range was -80 to 20°C with the heating rate of $10^{\circ}C$ /min.

2.6 Mechanical properties

The tensile test was carried out with an AGS-X series electronic tensile testing machine (SHIMADZU Co., Ltd., Japan). Dumbbell shaped specimens were tested at a crosshead speed of 500 mm/min at room temperature following the requirement of ISO 37-2011.

The cylindrical shaped samples with 12.5 mm thickness and 29 mm diameter with smooth and uniform surface were used to measure the indentation hardness and compressions set of the samples. By applying a constant force without any disturbances for a specific time, hardness indentations were made in the various positions. Ten readings were taken from different areas of the testing samples and the average value was estimated. At least three specimens were tested.

The compression set (CS) was determined according to ISO 815 by compressing the cylindrical samples to a constant deflection (25%) and kept them in oven at evaluated temperature for a period of time. The height of un-aged sample was recorded as h0. Then the strained height of samples in the compression set jig was measured as hs. After removing samples from the oven and cooling down at room temperature for 30 min, the recovery height was measured immediately, signed as hr. Finally the percentage of compression set was calculated as (h0-hr)/ (h0-hs). Three samples were measured and the compression sets were averaged.

3. Results and Discussion

3.1 Analysis of FTIR

2913

3000

(a) (b)

(c)

(d)

(e)

3500

The infrared spectra of aged NBR samples at 70°C with testing time under four different conditions were measured.

2233

1536

1721

1500

1000



2500

There were great distances among curves from 0 day to 42 days under the same aging condition, and the curves of aged samples under four conditions were also separate from each other clearly, and their changes were shown in Fig. 2.

For the characteristic bands of NBR, the peaks at 2918 and 2845 cm⁻¹ were C-H stretching vibrations, while the peak at 1453 cm⁻¹ was CH2 scissor vibration. The characteristic band at 2233 cm⁻¹ was attributed to the nitrile group, while the band at 968 cm⁻¹ belonged to the carboncarbon double bond (1,4 trans-bonding). The peak at 1536 cm⁻¹ was C=N stretching vibration came from the additives in the rubber.

When NBR sample aged in air, the absorption peak at 963 cm-1 decreased with the heating time. In addition, the absorbance of hydroxyl groups at 3520-3100 cm⁻¹ and that of the carbonyl group at 1721 cm⁻¹ were observed and increased with the thermal treatment. The formation of hydroxyl groups represented the oxidation of the molecular chains. The formation of carbonyl groups was the result of further oxidation and chain scissions. The weakening of absorption peak at 1536 cm⁻¹ with time was attributed to the escaping of volatile additives from the rubber into the atmosphere.

When NBR sample aged in oil, the absorption peaks at 3000-2800 cm⁻¹ broadened and increased in intensity in the initial period of aging and then remained almost constant. On the other hand, the absorption peak at 1536 cm^{-1} decreased remarkably with aging time, after a treatment for 42 days, the abundance was only 1/10 of the original NBR sample. The absorbance of hydroxyl groups at 3520-3100 cm⁻¹ and that of the carbonyl group at 1721 cm⁻¹ were not apparent even aged for 42 day. These changes were mostly associated with oil into the rubber and the additive in the nitrile butadiene rubber dissolving in oil, while oil also prevented oxygen consumption.

When NBR sample aged under compression, no matter in air or in oil, the obvious differences of the spectra was that the absorption peak at 963 cm⁻¹ decreased rapidly with the aged time, and the decreasing rate was faster than that of only in air or in oil. The peak at 1536 cm⁻¹ decreased slowly with aging time because the compression set jig prevented the additive escaping from the rubber into air or oil. Aged under compression in air, the absorbance of carbonyl group at 1721 cm⁻¹ was observed, but weaker than that of aged in air. Aged under compression in oil, the absorbance of carbonyl group at 1721 cm⁻¹ was not clearly observed. Due to the compression set jig reducing oxygen permeability. The absorption peaks at 3000-2800 cm⁻¹ broadened slowly with the aging time owing to the jig's reducing oil into the rubber.

3.2 Thermogravimetric measurements

Thermogravimetric measurements of aged NBR samples at 70°C with testing time under four different conditions



Fig. 3 TG and DTG curves of nitrile butadiene rubber before aging

Table 1.	Weight	loss	and	the	temperat	ure	of	the	highest
	weight	loss	of	agec	l nitrile	bu	tadi	ene	rubber
(according to TG and DTG data)									

Aging		aged for	r 7 days		aged for 42 days			
condition	$T_1/^{\circ}C$	$w_1/\%_0$	$T_2/^{\circ}C$	$w_2/\%$	$T_1/^{\circ}C$	$w_1/\%_0$	$T_2/^{\circ}C$	$w_2/\%_0$
in air	290	7.24	462	45.67	288	6.31	461	43.66
in oil	246	10.37	462	45.32	241	10.40	461	44.57
under compression in air	288	9.67	463	45.88	286	9.14	463	44.56
under compression in oil	288	9.97	463	45.72	287	9.73	462	44.83

were investigated. Under a nitrogen atmosphere, the mass loss of NBR before aging occurred in two stages (Fig. 3). A mass loss of 9.98% in the temperature range of 150-330°C was observed in the first stage, and it was attributed to the volatile substances in the rubber. The main mass loss of 45.76% took place at the second degradation step corresponded to the thermal decomposition of rubber at about 330-550°C. The DTG curves had two clear maxima, the corresponding temperatures of these maxima were 290°C, 465°C, respectively.

The DTG curves of the aged NBR rubbers all had two clear maxima, showing the similar two-stage type thermal degradation. The weight loss in the first stage (w1), the weight loss in the second stage (w2), and the corresponding temperatures of the highest weight loss (T1, T2) were summarized in Table 1. It was clear that in the second degradation step, no matter aging for 7 days or aging for 42 days, under four aging conditions, the corresponding temperatures of the highest weight loss were $462\pm1^{\circ}$ C, and the total mass losses were about $44\pm2\%$, indicating that same thermal decomposition of aged rubber as that of the original NBR.

However, the mass losses at the first step were much different. For the NBR aged in air, compared to the NBR before aged, although the temperatures of the highest weight were almost the same (290°C, 288°C), the total mass losses in the first stage were smaller than that of the NBR before aged (9.98%), 7.24% for 7-day aging and



Fig. 4. DSC curves of nitrile butadiene rubber before aging and aged in air at 70°C

Table 2. Tg of aged NBR under four aging conditions at70°C for different aging time

	$T_{\rm g}$ /°C					
condition	before aging	aged for 28	aged for 42			
		days	days			
in air	-44	-41	-38			
in oil	-44	-34	-31			
under compression in air	-44	-40	-36			
under compression in oil	-44	-36	-34			

6.31% for 42-day aging; the changes increased with the heating time. The decrease mass loss indicated that the volatile additive of the rubber vapor into the atmosphere with time. NBR aged in oil was distinguished from the others. The temperatures of the highest weight loss decreased with the aging time, 246°C for 7-day aging and 241°C for 42-day aging; but the total mass losses in the first stage increased slightly, showing the additive escaping from the rubber and oil penetrating in rubber with time. When NBR sample aged under compression, no matter in air or in oil, there were very minor differences compared to the NBR before aging. The corresponding temperatures of the highest weight loss were 287±1°C, and the total mass losses were about 9-10%, indicating the compression set jig prevented the additive escaping from the rubber and oil into the rubber. These results were in accordance with the IR analyses.

3.3 Tg analysis

The effect of thermal aging on the polymer network would inevitably influence the transition behavior of rubber. DSC measurements were carried out to monitor the glass transition temperature (Tg) changes after aging. Fig. 4 showed DSC scans of the samples aged in air, and the Tg values of aged NBR under four aging conditions at 70°C for different aging time were showed in Table 2. The wide temperature range and shape of the glass transition were difficult in accurate temperature measurement. Nevertheless, it was possible to establish that the aged samples presented higher values and Tg increased with the aging time. The increase of Tg indicated that the segmental dynamics was hindered and the crosslinking reaction clearly took place. In the beginning of aging, in general, increased crosslink density of a sulfur-cured rubber is explained with the formations of new crosslinks by free curative residues, such as elemental sulfur, cure accelerator residues, and zinc complexes remained in the rubber. This proceeded until the majority of the free chains were crosslinked. For long-term aging, thermooxidative was dominant. The oxidation of NBR during aging gave rise to two results. First, oxidation induced extra crosslinking through the recombination of free radicals. Second, oxidation caused chain scissions in the network. There was a competition between further crosslinking and chain scissions during thermooxidative aging. Dominant crosslinking reaction had a restrain effect on the movement of molecular chain, resulting in the increasing of Tg.

The increase rate of Tg was different under four different conditions. Under the oil conditions, Tg increased rapidly, it increased to -31°C (in oil) and -34°C (compressed in oil), respectively for 42-day aging; and under oil free condition, it increased only to -38°C (in air) and -36°C (compressed in air), respectively. It can be explained that with the continuous aging, exudation of the polar additives from the rubber and oil permeation into the rubber leading the free space between the polymer chains was diverted to the oil molecules and the relative motion trend between molecular chains segments weakened; and oil also prevented oxygen consumption, chain scissions reaction decreased, which resulted in Tg larger.

In air aging, the change of Tg under compression was larger than that of the non-compression condition, because the applied compression stress squeeze out the "free volume" of NBR, which led to a larger Tg. But aging in the oil, Tg of compressed condition was lower than that of non-compressed condition, due to the compression jig reducing the oil permeation. Above mentioned compressive stress may cause Tg larger, but apparently oil affect Tg greater, resulting the Tg change under compression in oil less than that of in oil without compression.

3.4 Effect of compression stress and oil on the aging of NBR

Hardness, elongation at break and compression set are key mechanical properties for NBR rubbers. The mechanical properties of aged NBR samples at 70°C with testing time under four different conditions were measured. During thermal aging, elongation at break decreased with time, while hardness and compression set increased under four conditions. But the changes were different, as shown in Fig. 5, Fig. 6, and Fig. 7.

Fig. 5 showed the hardness change of NBR aged at 70°C under different conditions with the aging time. It can be seen that the curves of NBR aged in air and under



Fig. 5. Hardness versus aging time of NBR aged at 70°C under various aging conditions



Fig. 6. Elongation at break versus aging time of NBR aged at 70°C under various aging conditions



Fig. 7. Compression set versus aging time of NBR aged at 70°C

compression in air showed the similar trend. In the early few days of aging (0-4 days) the change of hardness was smaller, but from 4 days to 10 days the change increased notably, and then the increment became smaller. The hardness of NBR increased with aging time showed the aging of nitrile butadiene rubber was mainly dominated by crosslinking reaction. While, the hardness of NBR aged under compression in air was smaller than that of in air. The applied compressive stress greatly constrained the relaxation of molecular chain and reduced the molecular chain scission activation energy, the molecular chain scission more easily and chain degradation aggravated, which led NBR aged under compression had a relatively low hardness.

Meanwhile, in the whole aging stage the rubber hardness aged under oil condition was lower than the hardness under conditions of oil free. The curves of in oil and under compression in oil also had the similar trend. Hardness decreased rapidly in the initial aging (0-5 days) due to the oil swelling, after reaching the swelling equilibrium, the hardness of rubber increased gradually with aging time. But the hardness of aged in oil was smaller than that of under compression in oil. Compressive stress reduced the hardness of rubber and at the same time reduced the absorption of oil, so aged under compression in oil, the hardness decreased slowly.

Fig. 6 showed the elongation at break change of NBR aged at 70°C under different conditions with aging time. It was obvious that the elongation at break of NBR aged in the presence of oil was larger than that aged in oil free environment from 0 to 42 days; while after 42 days, on the contrary, it decreased rapidly and was smaller. This may be explained with that NBR soaked in transformer oil, in a certain extent, prevented air into the rubber which inhibited the rubber thermal oxidative aging chain scission reactions in the early aging period, so the elongation at break in oil condition was generally higher than that in the oil free conditions. But after 42 days, too much cross-linking led the elongation at break decreased dramatically.

The elongation at break of NBR aged under compression was also smaller than that aged in absence of compression at the same air or oil environment. The applied compressive stress made the molecular chain orientation, constrained the relaxation behaviour of NBR, and increased chain scission reactions. So in the whole aging stage, the elongation at break under compression condition was lower than that in the condition of no compression.

Fig. 7 showed the compression set change of NBR aged at 70°C under compression in oil or in air with the aging time. The curves showed the similar trend. In the initial aging (0-10 days) compression set increased rapidly (by about 25%, 20%, respectively), then gradually and slowly increased with heating time. The compression set of NBR aged in oil was larger than that in air in general. Reason of the difference was that the oil swelling effect reduced the compactness of the network structure of NBR and increased the freedom of molecular chains, led to the ability of returning to original state after the compression force disappearing decreases.

The overall change trend of rubber properties showed that the aging of NBR was dominated by crosslinking reaction. Both compression stress and oil media affected the aging process of NBR. The influence of stress and oil



Fig. 8. Change of the elongation at break: (a) in air; (b) under compression in air; (c) in oil; (d) under compression in oil; ε, ε0 is elongation at break after

aging and before aging

simultaneously on the performance of rubber is complex, different from that of stress and oil separately.

3.5 Lifetime prediction

Elongation at break (ϵ) and compression set (CS) are always chosen as the failure property for lifetime predictions, as both chain scission and crosslinking reactions add up to decrease elongation at break and increase CS, in contrast to e.g. hardness, which is influenced in opposite directions by each reaction type. The aging tests of nitrile butadiene rubber were conducted systematically at 70°C, 90°C, 110°C, 130°C under four conditions. Fig. 8 and Fig. 9 showed the change of compression set and elongation at break with aging time. In all cases, compression set increased and elongation at break decreased with time of exposure, and with increasing temperature.

The lifetime of NBR can be predicted by using the



Fig. 9. Change of the compression set: (a) under compression in oil; (b) under compression in air



Fig. 10. Arrhenius plots for elongation at break: (a) in air; (b) under compression in air; (c) in oil; (d) under compression in oil



Fig. 11. Arrhenius plots for compression set: (a) under compression in oil; (b) under compression in air

Table 3. Useful lifetime prediction of the NBR at 25°C

Failure criteria	Condition	Change of property	Lifetime at 25°C /year	
CS	Compression in air		11.30	
CS .	Compression in oil		8.11	
	In air	500/	31.26	
Elongation at break	Compression in air	30%	21.00	
	In oil		29.53	
	Compression in oil		19.93	

Arrhenius life prediction method. When appropriate, this method predicts a liner relation between the logarithm of time (t) to a specified amount of material property change (e.g. 50% in our work), and the reciprocal of the absolute temperature (T). The plot of lnt versus 1/T was fitted by the linear equation as shown in Fig. 10 and Fig. 11. Table 3 shows the useful lifetime for NBR based on the two failure criteria under different conditions at 25° C.

The activation energy was obtained by multiplying the slope of the fitting line by R, the gas constant. The lifetime at 25 °C was estimated by extrapolation of the fitting line. For the elongation at break as evaluation index, the activation energy was, in turn, ~81.4 kJ/mol, ~74.8 kJ/mol, ~77.6 kJ/mol, and~74.0 kJ/mol, respectively; while the lifetime was 31.26 years, 21.00 years, 29.53 years and

19.93 years, respectively for NBR aged in air, under compression in air, in oil, and under compression in oil. The lifetime of NBR aged in air was the longest, and that under compression in oil is the shortest, shorter than that affected by oil or stress separately. So oil and compression stress both accelerated the aging of rubber, aging accelerated faster when compression stress and oil both act on NBR.

For compression set as evaluation index, the activation energy was calculated to be \sim 63.7 kJ/mol under compression in air and \sim 62.2 kJ/mol under compression in oil, respectively. The lifetimes at 25°C were 11.30 years and 8.11 years, respectively. The lifetimes using compression set as evaluation index were much shorter than those for elongation at break as evaluation index.

Using different evaluation index, the predicted lifetime was different. The further study concerning that the predicted lifetime choosing which property as evaluation index can better fitting the actual lifetime is in progress.

4. Conclusions

Accelerated thermal aging was investigated under four different conditions at different temperatures of 70°C, 90°C, 110°C, 130°C, which resulted in a decrease in the tensile strength, elongation at break, and compression set, and an increase in the hardness. The overall change trend of rubber properties shows that the aging of NBR is dominated by cross-linking reaction. The percentage variation of elongation at break and compression set of rubber upon thermal aging were chosen as a key property parameter to evaluate the lifetime. It was predicted that the CS of the rubber would take approximately 11.30 years and 8.11 years to decrease by 50% at a temperature of 25°C, respectively for air compression and oil compression. Elongation at break values of approx. 50% would be reached after 31.26 years, 21.00 years, 29.53 years and 19.93 years, respectively for air, air compression, oil and oil compression. Different aging conditions yielded materials with different properties. The prediction lifetime using different evaluation index was different greatly. For the life prediction of NBR, we should take into account of the performance trends and performance requirements of NBR under actual operating conditions to select the appropriate evaluation index.

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Yi-Hua Qian She received M.S degree and works in the Electric Power Research Institute of GuangdongPower Grid Corporation now. Her research interests are electric oil testing and insulation materials study.



Hong-Zhao Xiao She studied in College of Chemistry and Molecular Science, Wuhan University from 2014 to 2016. Her research interests are polymer preparation and performances.



Ming-Hao Nie He studied in College of Chemistry and Molecular Science, Wuhan University from 2014 to 2016. He researches on aging diagnosis and life prediction of rubber sealing materials for electric power equipment.



Yao-Hong Zhao He received Sc.D degree and works in the Electric Power Research Institute of Guangdong Power Grid Corporation now. His research interests are electric oil testing and insulation materials study.



Yun-Bai Luo He received Sc.D degree in chemical engineering from Tianjin university. His research interests are chemical separation, environmental chemistry, application of organicchemistry and fine chemicals.



Shu-Ling Gong She received Sc.D degree in chemitry from Wuhan University. Her research interests are functional polymer, waterborne polymer, coatings and adhesives, and polyurethane chemistry and so on.