Low-temperature characteristics of rubbers and performance tests of type 120 emergency valve diaphragms

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Abstract

Purpose – The type 120 emergency valve is an essential braking component of railway freight trains, but corresponding diaphragms consisting of natural rubber (NR) and chloroprene rubber (CR) exhibit insufficient aging resistance and low-temperature resistance, respectively. In order to develop type 120 emergency valve rubber diaphragms with long-life and high-performance, low-temperatureresistant CR and NR were processed. Design/methodology/approach – The physical properties of the low-temperature-resistant CR and NR were tested by low-temperature stretching, dynamic mechanical analysis, differential scanning calorimetry and thermogravimetric analysis. Single-valve and single-vehicle tests of type 120 emergency valves were carried out for emergency diaphragms consisting of NR and CR.

Findings – The low-temperature-resistant CR and NR exhibited excellent physical properties. The elasticity and low-temperature resistance of NR were superior to those of CR, whereas the mechanical properties of the two rubbers were similar in the temperature range of $0^{\circ}C$ –150 °C. The NR and CR emergency diaphragms met the requirements of the single-valve test. In the low-temperature single-vehicle test, only the low-temperature sensitivity test of the NR emergency diaphragm met the requirements.

Originality/value – The innovation of this study is that it provides valuable data and experience for future development of type 120 valve rubber diaphragms.

Keywords Natural rubber, Chloroprene rubber, Low-temperature characteristic, 120 emergency valve, Diaphragm

Paper type Research paper

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Type 120 emergency valve diaphragms

47

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1. Introduction RS

3,1

48

The type 120 air control valve (hereafter the 120 valve) is an important braking component of goods wagons, and is usually composed of four parts – including the intermediate, main, semi-automatic relief and emergency valves. The internal components of the valve body perform specific actions at various pressures, which are generated mainly due to the sealing and regulating effects of the rubber diaphragms inside the valves. Thus, the performance of the rubber diaphragms has a substantial impact on the safety, work sensitivity, durability and environmental resistance of 120 valves (Wu *et al.*[, 2017;](#page-11-0) [Wei, Zhang, Zhang, & Zhao, 2023](#page-11-1); [Xu, Zhang, Zhang, Li, & Zhu, 2021;](#page-11-2) [Zhang, Wei, Liu, Zhang, & Zhu, 2023\)](#page-11-3). Especially upon rapidly braking railway locomotives and vehicles, the 120 emergency valves must prompt the train tube to exhaust quickly, generating a large pressure difference, ensuring the reliability and sensitivity of the emergency braking, thereby increasing the emergency braking wave speed. In addition, the emergency brake exhaust should not be activated during common braking. Thus, the 120 emergency valve has a direct impact on the safety of railway rolling stock operation [\(Yang, 2009](#page-11-4); [Zhao, 2008](#page-11-5)). Therefore, the rubber diaphragms of 120 emergency valves are generally required to have a good low-temperature resistance, flexor flexibility and strong mechanical properties, and they must meet the defined sensitivity, gas tightness and service life under cryogenic conditions.

Good low-temperature resistance is particularly important for 120 emergency valve diaphragms. The low-temperature resistance of rubber usually refers to the ability of rubber to maintain elasticity and product properties at low temperatures. Especially in dynamic conditions, low temperatures often lead to a substantial reduction in the properties of rubber products, which is attributable to the fact that the increased hardness of rubber diaphragms at low temperature can produce a decrease in braking sensitivity. As a result, the majority of rubbers mismatch the low-temperature requirements for railroad rolling stock brake valves in China. Although some rubbers such as silicone rubber and butadiene rubber exhibit excellent low-temperature resistance, their mechanical properties and fatigue resistance usually mismatch the requirements of practical applications.

Currently, the natural rubber (NR) used by 120 emergency valve rubber diaphragms exhibits favorable low-temperature resistance and mechanical strength. In addition, the NR tends to be in a semi-crystalline state at low temperature or under an external force, which imparts high tensile strength, high tear resistance, low heat production and other positive mechanical properties. However, NR exhibits unsatisfactory oxygen aging, heat resistance and oil resistance properties due to its nonpolar structure and large number of unsaturated double bonds, which greatly limits its service life (He *et al.*[, 2015;](#page-10-0) [Huang](#page-10-1) *et al.*, 2018). Chloroprene rubber (CR) has good mechanical properties as well as excellent oil resistance and thermal stability, which are particularly in line with the performance requirements of 120 emergency valve diaphragms, but its polarity is high and its crystallinity is strong, which leads to a decrease in its low-temperature resistance. Mixture of CR and NR can theoretically combine the advantages of the two rubbers to compensate for each other's shortcomings, but the two are not compatible [\(Zhao, Xiang, Wu, & Gao, 2022\)](#page-11-6). Low temperature has a substantial impact on the viscoelasticity of rubber, and studying the low-temperature characteristics of NR as well as CR is crucial for 120 valve diaphragms' product design and correctly evaluating their performance. With ongoing reforms of the railway freight car repair system in China, there are increasing requirements for the use performance and life of the 120 valve. At present, a large number of studies have been reported on improving the performance and service life of the 120 valve and its metal parts [\(Hou, 2017;](#page-10-2) Lü, Zhu, Yi, & Guo, 2006; [Liu & Tang, 2011;](#page-10-4) [Zhang, 2014;](#page-11-7) [Shen, Liu,](#page-10-5) [& An, 2014\)](#page-10-5), whereas research based on 120 valve rubber diaphragms have rarely been reported. This is the gap in the literature that was addressed in this study. Specifically, the present authors hypothesize that low-temperature-resistant CR meets the technical requirements for 120 emergency valve diaphragms.

In this paper, to determine the relationship between the low-temperature characteristics of rubbers and the braking performances of the 120 emergency valve rubber diaphragms, a lowtemperature-resistant CR is developed. The differences and changes in the performances of NR as well as CR at the room temperature and low temperature as well as the braking performances of the corresponding 120 emergency valve rubber diaphragms at the room temperature and low temperature $(-40 \degree C)$, one of the minimum temperatures specified in Chinese railway industry standards), are evaluated. Thus, this study presents a technical basis for using diaphragms at low temperature.

2. Materials and methods

2.1 Materials

All of the chemicals used in the present experiments were purchased from suppliers and used directly without further treatment. NR (RSS1) was purchased from Indonesia. CR (M-40) was purchased from Japan Denka. Carbon black (N330) was purchased from Shanghai Cabot Chemical.

2.2 Preparation of samples

Typically, 100 parts of massed NR or CR was placed into a mixer and plasticized for 1 min. Afterward, six parts of zinc oxide, two parts of stearic acid, 20 parts of carbon black, ten parts of cold resistant plasticizer and five parts of antioxidant were sequentially added and mixed for ca. 1 min. Subsequently, the remaining 20 parts of carbon black was added and mixed for 6 min, and the rubber was drained at 115 \degree C. The mixed rubber was cooled and refined on a kneader, followed by adding two parts of sulfur and two parts of accelerator. After the rubber was evenly mixed, it was thinly passed $5\times$, rolled $3\times$ and flaked. Finally, after the rubber compounds were placed at the room temperature for 16 h, the samples were vulcanized with a vacuum flat curing machine at (10 ± 0.2) MPa and (160 ± 2) °C for (15 ± 2) min.

2.3 Measurements

The hardness, tensile strength, compressive cold resistance coefficient, brittleness temperature and flexural cracking were evaluated in accordance with GB/T 531.1-2008, GB/T 528-2009, HG/T 3866-2008, GB/T 1682-2014 and GB/T 13934-2006, respectively. The performance tests of the 120 emergency valve diaphragms were in accordance with the relevant requirements of TB/T 2206-2018, TB/T 1492-2017 and TB/T 2951.2-2018.

2.3.1 Low-temperature tensile strength. The low-temperature tensile strength of the samples was investigated with an electronic universal pull test machine (UTM5305, Shenzhen Sansi Vertical and Horizontal Technology). The samples were Type 1 [\(Figure 1a\)](#page-3-0) at a tensile rate of 100 mm/min and subjected to low-temperature testing at -40 °C for 30 min.

2.3.2 Dynamic mechanical analysis. The dynamic mechanical properties were performed by dynamic mechanical analysis (DMA; DMA1, Mettler-Toledo). The specimens were cut to be 30-mm long, 4-mm wide, and 1.4-mm thick [\(Figure 1b](#page-3-0)). Tensile testing jigs were used, and the tests were conducted between -130 °C and $+150$ °C at a heating rate of 5 °C/min, 1-Hz frequency and 10-μm amplitude.

2.3.3 Differential scanning calorimetry. Differential scanning calorimetry (DSC) curves were obtained with a Netzsch DSC 204 F1 under nitrogen. The scanning temperature range was -75 °C to $+20$ °C and the scanning speed was 10 °C/min. The data were acquired after thermal history elimination.

2.3.4 Thermal gravimetric analysis. Thermogravimetric analysis (TGA) was carried out with a Netzsch TG 209 F1 under nitrogen from 50 °C to 650 °C, at a heating rate of 10 °C/min.

Type 120 emergency valve diaphragms

3. Results RS

3,1

50

3.1 Physical properties of NR and CR

Table 1 shows the physical properties of NR and CR. NR and CR have excellent mechanical properties because they have substantial self-complementary properties as crystalline rubbers. The tensile strength and elongation at break of NR were slightly higher than those of CR, whereas the hardness and tensile stress at given elongations of NR were lower than those of CR, especially at low temperatures. Compared with CR, NR has a flexible molecular chain, small polarity, low cohesion energy and good low-temperature resistance. Therefore, the compression cold-resistance coefficient of NR was greater than that of CR [\(Zou, Kang, Yang,](#page-11-8) [& Fang, 2019;](#page-11-8) Zhao et al.[, 2022](#page-11-6)). The number of flexions of CR was ca. $3.6\times$ that of NR. NR is an unsaturated rubber and exhibits poor heat as well as oxygen aging properties. In contrast, the chlorine atoms connected on the double bonds of the CR molecular chain limit the reactivity of the double bonds and chlorine atoms ([Wang & Qin, 2022](#page-11-9); Zhao *et al.*[, 2022](#page-11-6)), which leads to the outstanding aging resistance of CR.

Figure 1. Specimens for the tests of low-temperature stretching (a) and dynamic mechanical analysis (b) of the rubbers. CR, chloroprene rubber; NR, natural rubber

Source(s): Authors own work

	Test items			Test results NR. CR.	
	Shore A hardness	RT	45	47	
		-40 °C	58	80	
	Tensile strength/MPa	RT	16.41 ± 0.42	14.67 ± 0.35	
		-40 °C	19.32 ± 0.47	$15.07 + 0.36$	
	Tensile stress at 100% elongation/MPa	RT	0.58 ± 0.04	0.94 ± 0.06	
		-40 °C	$0.84 + 0.06$	$1.76 + 0.07$	
	Tensile stress at 200% elongation/MPa	RT	$0.84 + 0.05$	$1.53 + 0.09$	
		-40 °C	2.15 ± 0.12	4.83 ± 0.16	
	Tensile stress at 300% elongation/MPa	RT	$1.12 + 0.07$	$2.75 + 0.14$	
		-40 °C	$4.43 + 0.15$	$9.63 + 0.20$	
	Elongation at break	RT	$1146\% \pm 52\%$	$823\% \pm 40\%$	
		-40 °C	$593\% \pm 29\%$	$449\% \pm 21\%$	
	Coefficient of cold resistance under compression $(-40 °C,$ compression rate 20%)		0.66	0.56	
	Low-temperature brittleness		-40 °C undamaged	-40 °C undamaged	
Table 1.	Flex cracking and crack growth/times		14×10^5	5.0×10^5	
Physical properties of NR and CR^a	Note(s): ^a CR, chloroprene rubber; NR, natural rubber Source(s): Authors' own work				

3.2 Low-temperature characteristics of NR and CR

The relaxation of rubber chain segments can be quickly decreased at the low temperature, which corresponds to internal friction of the rubber molecules and increases the elastic modulus as well as hardness, thus substantially reducing the elasticity of the rubber. In the present experiments, low-temperature stretching, DMA and DSC were carried out to investigate the low-temperature properties of NR and CR.

Figure 2 shows tensile stress–strain curves of NR and CR at the room temperature and low temperature. Specifically, the samples all exhibited noticeable rubber elastic characteristics and maintained high elastic deformation under low stress, exhibited a plateau of slow stress growth in the early stage and a rapid upward trend until specimen fracture (Ding *et al.*[, 2019](#page-10-6); [He, Zhang, Chen, & Dong, 2007\)](#page-10-7). NR and CR exhibited higher elongation at break at the room temperature than those at low temperature. In other words, at low temperature, higher stresses for NR and CR were all obtained than those at low temperature, at the same strain as room temperature. As crystalline rubber, some segments of NR and CR are prone to crystallization at low temperature, resulting in decreases in the locomotor activity of the segments and increases in the tensile modulus of the rubbers. As a result, there were substantial reductions in the strain or elongation at break for the rubbers at the same stress ([Bennacer, Liu, Yang, & Chen, 2022](#page-10-8); [Chen & Xu, 2012](#page-10-9); [Huang](#page-10-10) et al., 2018; [Plagge & Kl](#page-10-11)ü[ppel,](#page-10-11) [2018\)](#page-10-11). Moreover, NR at the room or low temperature exhibited greater strain compared with CR under the equivalent stress, which indicates that NR had higher elasticity than CR.

The fracture energy G_F was obtained by integrating the area of the aforementioned stress–strain curve [\(Figure 3\)](#page-5-0). The -40 °C low-temperature G_F values of NR and CR were respectively reduced by approximately 25.9% and 40.5%, respectively, compared with those at the room temperature. The decreases in the G_F values might be due to crystallization of some molecular chain segments at low temperature, which affects the mobility of the chain segments and the toughness of rubbers (Song *et al.*[, 2022\)](#page-11-10). Furthermore, the G_F values of NR were greater than those of CR at various temperatures, and NR had a lower decrease rate of G_F at -40 °C, implying that NR had stronger low-temperature resistance than CR, which is consistent with the aforementioned results.

To further evaluate the influence of the molecular structure of the rubbers on the mechanical properties at low temperatures, DMA tests were conducted on NR and CR in the range of -130 °C to $+150$ °C [\(Figure 4](#page-5-0)). NR (-130 °C to -60 °C) and CR (-130 °C to -45 °C) had a high tensile modulus E' in the glass state. With increasing temperature, the tensile modulus of NR and CR slowly decreased. Furthermore, their corresponding tensile moduli

Figure 2. Tensile stress–strain curves of natural rubber (NR) (a) and chloroprene rubber (CR) (b), at room temperature (RT) and -40 °C

Type 120 emergency valve diaphragms

exhibited a rapid downward trend in the glass transition region. The glass transition temperature measured by DMA is generally measured using tan δ (loss tangent) peak representation of the curve. δ is the phase difference between stress and strain, and also known as lag angle. Specifically, the glass transition regions of NR and CR were in the range of -60 °C to -20 °C, and -45 °C to 0 °C, respectively. Upon a further increase in temperature, some rubber crystals were gradually disrupted, and the locomotor activity of the crosslinking networks and molecular chain segments were also correspondingly enhanced. Accordingly, their loss moduli E'' first increased and then decreased [\(Kong, Dong, Chen, Xia,](#page-10-12) [& Pei, 2019](#page-10-12); [Wang, 2017](#page-11-11)). The glass transition regions of different rubbers differ because of the dissimilarity of the molecular structures, and the impacts of low temperature on rubbers were greatest in these areas. Specifically, the lowest temperature $(-60 \degree C)$ of NR in this region was lower than that of CR $(-45 \degree C)$, further demonstrating that the low-temperature resistance of NR was better than that of CR. As the temperature increased, NR as well as CR exhibited similar and almost constant moduli and loss angles in the rubber state (the high elasticity state) region, suggesting that the mechanical properties of these two types of rubbers were similar in the temperature range of 0 \degree C–150 \degree C.

When the temperature was lower than the glass transition temperature (Tg), the rubber chain segment was frozen, exhibiting a hard solid glass state. As the temperature increased to

Tg, the chain segments began to thaw and the offset of the baseline on the DSC heating curve exhibited a step. Subsequently, the intersection point A was obtained by extending the two baselines in reverse, and Tg was its corresponding temperature ([Zheng, Wei, Luo, & Liao,](#page-11-12) [2019\)](#page-11-12). Figure 5 shows DSC curves of NR and CR, affording Tg of NR $(-62.6 \degree C)$ and CR (-49.7 °C), which indicates that CR was more prone to crystallization and hardening than NR at low temperature. Because CR mainly consisted of trans-1,4 structures, arranged in a regular linear manner with high regularity and contained polar groups such as chlorine atoms (increasing the intermolecular forces), it readily crystallized. Furthermore, CR was more easily affected by low temperature and NR had better low-temperature resistance than CR.

3.3 Thermal stability performance of NR and CR

Plasticizers could improve the low-temperature resistance of rubber but reduce its thermal stability. TGA was used to study the thermal stabilities of NR and CR. Figure 6 shows TGA and derivative thermogravimetry curves. Under nitrogen, the mass loss of NR was mainly divided into two stages. The temperature in the first stage was 200 °C–300 °C, and the mass loss rate was 8.10%, which might be caused by volatilization of small molecules such as oil.

Type 120 emergency valve diaphragms

53

The temperature in the second stage was 300° C–500 $^{\circ}$ C, and the mass loss rate was relatively high (reaching 72.81%), which might be caused by cracking reaction of NR. When the temperature reached 375 \degree C, the mass loss rate of NR (see [Figure 6b](#page-6-0)) reached its peak. The mass loss of CR was mainly divided into three stages. The first stage was at $\langle 300 \degree C$, when the volatile small molecules such as oil in CR first began to evaporate. The mass loss rate in this stage was 10.32%. The second stage was between 300 \degree C and 350 \degree C. As the temperature increased and the reaction progressed, the mass loss of the adhesive began to substantially increase at 307 \degree C. This is because the carbon–chlorine bond energy in CR is low and the bond-breaking and HCl removal reactions began at this temperature. When the temperature reached 317 \degree C, the mass loss rate [\(Figure 6b\)](#page-6-0) reached its peak, indicating that the CR HCl removal reaction had reached its peak. The mass loss rate of the second stage was 26.12%. The third stage was at 350 °C–500 °C, mainly due to the breakage of the CR main chain. When the temperature reached 428 \degree C, the mass loss rate ([Figure 6b\)](#page-6-0) reached its peak. In this stage, the mass loss rate of the CR was 14.51% . When the temperature was >500 °C, there was still a small extent of slow degradation of the main chain.

> Table 2 shows detailed data. The initial thermal decomposition temperature (T_{onset}) of NR was 16 \degree C higher than that of CR, possibly due to the presence of more oil in CR compared with NR. The temperature corresponding to the maximum thermal weight loss rate (T_{max}) of the main chain of NR was ca. 53 °C lower than that of CR, indicating that the thermal stability of the main chain molecule of CR was higher than that of NR. The residual mass fractions were NR 19.09% and CR 49.05%, which might consist of carbon black and ash.

3.4 Performance tests for the 120 emergency valve diaphragms of NR and CR

The 120 emergency valve utilized the pressure differences between the upper and lower sides of the emergency piston (i.e. emergency chamber and brake pipe) to control the actions of the pilot valve and vent valve, generating an emergency local pressure reduction ([Figure 7\)](#page-8-0). The emergency sensitivity (room and low temperature) and stability (room and low temperature) of the 120 emergency valve was a pair of conflicting technical indicators. When the emergency valve cover, emergency piston rod, stabilizer spring and other components were the same, the main factor affecting these two key performances was the 120 emergency valve rubber diaphragms [\(Han & Liu, 2010\)](#page-10-13).[Figure 8](#page-8-0) shows a schematic of 120 emergency valve diaphragms. The 120 emergency valve diaphragms consisting of NR and CR were subjected to single-valve tests at room temperature as well as single-vehicle tests at room temperature and -40° C.

3.4.1 Single-valve tests of 120 emergency valve diaphragms of NR and CR. The 120 emergency valve must be tested on the 120 valve test bench before assembly or use, mainly to verify the emergency sensitivity and stability performances. Regarding the sensitivity, various control valve manufacturers generally require that the 120 emergency valve can produce emergency braking in a timely manner before the train pipe is depressurized from a value of 600 kPa to 500 kPa (more stringent than TB/T 2951.2-2018). A smaller corresponding

54

RS 3,1

emergency valve diaphragms

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pressure reduction corresponds to a higher sensitivity of the emergency valve. Table 3 shows the results for the NR and CR emergency valve diaphragms. The single-valve test emergency sensitivity of the CR emergency valve diaphragm is at $1.5\times$ the pressure of the NR emergency value diaphragm. Thus, the NR emergency valve diaphragm had better emergency sensitivity, which is consistent with the tensile performances and fracture energy test results.

The definition of emergency valve stability indicated that the emergency valve could not be activated during service braking or when there was a small extent of air leakage in the

train pipe but must be activated during emergency braking. At the same extent of pressure reduction, the emergency value experienced difficulties in undergoing emergency exhaust procedures, indicating an improved stability of the corresponding emergency valve ([Mao, Jin,](#page-10-14) [& Li, 2004\)](#page-10-14). The results of the NR and CR emergency valve diaphragms in the single-valve tests met the aforementioned requirements [\(Table 3](#page-8-0)). In summary, the sensitivity and stability tests of the 120 emergency valve single-valve tests were conducted at room temperature and the NR and CR emergency valve diaphragms met their technical specifications. This was consistent with the conclusion that the mechanical properties of NR and CR at room temperature are similar.

3.4.2 Single-vehicle tests of 120 emergency valve diaphragms of NR and CR. An air control valve to be installed on a railway truck should be tested by a single-vehicle test. The 120 emergency valve should be operated normally within the ambient temperature range of -50 °C to $+70$ °C. Table 4 shows experimental data of the NR and CR emergency valve diaphragms.

The NR and CR emergency valve diaphragms had a single-vehicle emergency sensitivity of 30 kPa at the room temperature, which is much lower than the requirement of 100 kPa. The experimental results of low-temperature emergency sensitivity on a single vehicle indicated that the result of the CR emergency valve diaphragm was $2.4\times$ that of the NR emergency valve diaphragm and only the NR emergency valve diaphragm met the indicator requirements, which is closely related to the corresponding low-temperature resistance performance. In addition, Table 4 also shows corresponding data on the emergency stability of a single vehicle at the room temperature and low temperature. The emergency exhaust effects of the NR and CR emergency valve diaphragms were not reactivated during common brake depressurization, confirming their positive emergency stabilization performance. In summary, when the sensitivity and stability tests of the 120 emergency valve single-vehicle test were conducted at room temperature and low temperature $(-40 \degree C)$, the single-vehicle cryogenic sensitivity value of the CR emergency valve diaphragm was unsatisfactory and the low-temperature resistance performance of NR was better than that of CR.

4. Conclusions

The processed NR and CR had excellent physical properties. The flex cracking and crack growth times of CR were ca. $3.6\times$ those of NR and the coefficient of cold resistance under compression of NR was greater than that of CR, indicating that NR had higher lowtemperature resistance and that CR had higher aging resistance. NR had a higher strain value than that of CR under the equivalent stress at room temperature or low temperature, indicating the higher elasticity of NR. Furthermore, the GF values of NR were greater than

56

RS 3,1

Table 4.

120 emergency

of NR and CR

those of CR at various temperatures, and NR had a lower decrease rate of GF at low temperature, implying that NR exhibited optimal low-temperature resistance. DMA indicated that the mechanical properties for these two types of rubbers were similar in the temperature range of 0 °C–150 °C, and the Tg of NR (–62.6 °C) was lower than that of CR (–49.7 °C), further indicating the superior low-temperature resistance of NR. The results of the 120 emergency valve single-valve test conducted at room temperature indicated that NR and CR emergency valve diaphragms met the requirements of corresponding sensitivity and stability specifications. More importantly, the sensitivity and stability tests of the 120 emergency valve single-vehicle tests conducted at the room temperature and low temperature $(-40 \degree C)$ indicated that only the CR emergency valve diaphragm did not meet the requirements. These findings provided data support and experimental methods for the future study of long-life type 120 valve rubber diaphragms.

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Type 120 emergency valve diaphragms

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RS 3,1