Compatibility and Interaction Mechanism between EPDM Rubber and a SF₆ Alternative Gas—C₄F₇N/CO₂/O₂

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ABSTRACT: Among the numerous novel eco-friendly insulating gases, C₄F₇N has attracted much attention due to its excellent electrical performance. However, except for the electrical performance, the compatibility between the gas medium and the sealing materials is equally important for gas-insulated equipment. At present, studies about the compatibility between C₄F₇N and EPDM, a widely used sealing material in power systems, are available in some previous works, but few focused on the compatibility comparison between C₄F₇N gas mixtures and EPDM with different third monomers. In this paper, we carried out the thermal aging test on ENB-EPDM, DCPD-EPDM, and C₄F₇N gas mixture to perfect the compatibility mechanism between EPDM and C₄F₇N. It was found that both of the EPDM reacted with the gas mixture and led to the property changes in the solid samples and the decomposition of C₄F₇N. On the other hand, by coating silicone grease, the contact between gas and rubber was effectively blocked and the concentration of the decomposition product was significantly reduced. The performance comparison indicates that ENB-EPDM is more suitable for sealing the C₄F₇N gas mixture, which is due to the superior thermal stability of ENB.

1. INTRODUCTION

SF₆, as a gas with excellent insulation and arc extinguishing performance, is widely used in gas insulation equipment of power systems. However, the Global Warming Potential (GWP) of SF₆ is 23,500 times higher than that of CO₂, making it one of the six major greenhouse gases in the Kyoto Protocol. According to statistics, the SF₆ usage in the power industry accounts for more than 80% of its annual output. For building a green and low-carbon sustainable energy system, seeking an environmentally friendly gas insulating medium to gradually reduce the use of SF₆ has become a hot issue in recent years.

Perfluorosbutyronitrile (C₄F₇N) is an eco-friendly gas with great application potential. Its GWP is only 2090, Ozone Depletion Potential (ODP) is 0, atmospheric life is 22 years and its insulation capacity reaches 2.2 times that of SF₆. Due to its high liquefaction temperature (−4.7 °C), C₄F₇N needs to be mixed with CO₂, N₂, or O₂ to meet the requirements of the lowest temperature in engineering applications. The published works show that a C₄F₇N/CO₂ mixture containing 18—20% C₄F₇N possesses insulation performance comparable to SF₆. Besides, in consideration that C₄F₇N is easy to decompose and produces a solid substance under the influence of high-energy arc, a certain amount of O₂ is used to suppress the generation of solid decomposition products in the practical application of GIS.

For the new eco-friendly insulating gas, not only its environmental friendliness and outstanding electrical performance but its compatibility with the internal sealing materials of the equipment should also be taken into account. As is designed, the maintenance cycle of GIS is long. Once the insulating medium is incompatible with the sealing material, it may cause the corrosion of the sealing material and leads to worsening of its sealing performance. The other part of the result is the decomposition of the insulating medium, which will lead to the decline of the internal insulation capacity of the equipment and safety risks. Currently, the sealing materials used in gas insulating equipment mainly include ethylene propylene diene monomer (EPDM) rubber, neoprene (CR), nitrile butadiene rubber (NBR), etc. Because of the good chemical stability of SF₆, little attention has been paid to the compatibility between insulating gas and rubber sealing materials in academic research. For C₄F₇N and its mixture, General Electric and Siemens conducted compatibility experiments of C₄F₇N/CO₂ with materials contained in high-voltage equipment and GIS. The results indicated that a certain extent reaction happened between the gas and rubber materials, which was judged by the purity of gas. Scholars in
Wuhan University carried out an experiment of compatibility between C₄F₇N and ENB-EPDM rubber and found that the internal cross-linker of ENB-EPDM would appear on the surface and react with C₄F₇N under a long-term thermal aging test.10

As the most used sealing material in power systems, EPDM can be divided into ENB-EPDM, DCPD-EPDM, and HD-EPDM according to the type of third monomer. At present, there is no relevant research focusing on the compatibility of C₄F₇N gas mixtures and EPDM with different third monomers. A comprehensive research about the basic reason of incompatibility between EPDM and C₄F₇N can help in improving the machining process of EPDM or selecting more suitable sealing materials. Moreover, coating silicone grease on the sealing materials is an effective method for enhancing the gas tightness of an equipment.11 Adding silicone grease could be used as a measure to improve the gas tightness of an equipment.12

In this paper, ENB-EPDM and DCPD-EPDM were chosen to perform the compatibility experiment with the C₄F₇N gas mixture. The compatibility of rubbers and the 15% C₄F₇N-85% CO₂ gas mixture and 15%C₄F₇N-79%CO₂-6%O₂ gas mixture were first tested to find out the influence of O₂ during the experiment. Then the rubbers were coated with silicone grease and tested under the same conditions to make it clear whether silicone grease could be used as a measure to improve the compatibility. Finally, the corresponding improvement scheme was proposed based on the analysis of test results. This study has clarified the interaction mechanism between C₄F₇N and EPDM; at the same time, it can also provide important references for the selection and development of sealing materials for C₄F₇N gas insulation equipment.

2. METHODS

2.1. Materials. The types of EPDM rubber samples used in the experiment were PG807 and 3-2-72, with the respective third monomers of dicyclopentadiene (DCPD) and ethyl-enobornene (ENB), and both were provided by State Grid PingGao Group Co., Ltd. The structural formulas of the two EPDM samples are shown in Figure 1 a,b, and the size parameters of the two rubber samples are shown in Figure 1 c,d. The thickness of the square sheet sample was 0.8 mm and the side length was 4 mm, which was used for morphology characterization and element characterization. The cylindrical sample with a diameter of 29 mm and a thickness of 12.5 mm was used for the compression modulus of elasticity test and compression set test, whose size was selected from China National Standard (CNS) GB/T 7759.1-2015.12 The silicone grease used in this experiment was high-vacuum sealing silicone grease with good insulation capacity and chemical stability in the temperature range from −40 to +230 °C.

2.2. Test Conditions. A previous study13 has shown that the C₄F₇N/CO₂ gas mixture, with 15% C₄F₇N under a pressure of 0.14 MPa, can reach a minimum operating temperature of −25 °C and achieve the same insulation strength of pure SF₆ under a pressure of 0.12 MPa, which is the commonly used pressure in SF₆ switchgear.14 Besides, the breakdown test result indicates that the insulation performance and stability of the C₄F₇N/CO₂/O₂ gas mixture are best when O₂ accounts for 6%.15 Based on these studies, the proportion of the C₄F₇N/CO₂/O₂ gas mixture in this paper was 15%-79%-6%. The heat resistance of EPDM equips it with a long-term working temperature of over 100 °C16 and according to the IEC 62271-203,17 the operating temperature of GIS is below 50 °C. Combined with the recommended aging temperature and time18 given in CNS GB/T 2941-2006, we conducted the thermal aging test with temperatures of 85 and 100 °C for 28 days to explore the interaction between rubbers and the gas mixture.

2.3. Steps and Devices. The sealing device and compression device are shown in Figure 2, in which the volume and the maximum working pressure were 0.3 L and 0.6 MPa, respectively. The height H of the compression device used for the compression set test was 9.375 mm, ensuring a 25% compression of the cylindrical rubber.

Before the experiment, all the devices and rubber samples were wiped with absolute alcohol. After drying for 12 h at room temperature, the samples were divided into two groups. The first group was directly put into the sealing device, and silicone grease was applied to the other group before sealing.

When the thermal aging test was finished, the gas mixture was extracted and injected into a gas chromatograph-mass spectrometer (GC-MS) to analyze its gas composition. Then the device was vacuumed again and left for 16 h according to the requirement of CNS GB/T 3512-201419 to restore the stability of the rubber properties. Finally, the samples were taken out for the test of physical properties and chemical properties so as to comprehensively assess the compatibility of the C₄F₇N gas mixture and EPDM.

Figure 1. Structural formulas of the rubber samples: (a) DCPD-EPDM and (b) ENB-EPDM. Rubber sample size parameters: (c) square sample and (d) cylindrical sample.

Figure 2. Experimental device: (a) sealing tank and (b) fixture.
3. RESULTS AND DISCUSSION

For the convenience of reading, gas mixture A in the following text refers to the C$_4$F$_7$N/CO$_2$ gas mixture and gas mixture B refers to the C$_4$F$_7$N/CO$_2$/O$_2$ gas mixture. Rubbers A and B, respectively, refer to ENB-EPDM and DCPD-EPDM.

3.1. Mechanical Properties. Compression performance is one of the most important parameters for sealing rubber materials. The smaller the compression set (CS) and the greater the compression elastic modulus (CEM), the better the sealing performance of rubber. The CEMs of untreated rubber A and rubber B are 10.156 and 11.226 MPa, respectively. The compression performances after the test are listed in Table 1.

| Table 1. Compression Properties of Rubber after the Experiment |
|---------------------------------------------------------------|
| rubber sample-gas mixture | CEM (MPa) | CS (%) |
|                            | 85 °C | 100 °C   | 85 °C | 100 °C   |
| A-A                         | 7.122 | 7.120     | 31.008 | 38.688   |
| B-A                         | 10.056 | 9.651     | 14.848 | 16.480   |
| A-B                         | 7.325 | 7.258     | 31.808 | 39.328   |
| B-B                         | 9.041 | 7.919     | 15.968 | 17.888   |

It can be seen from the table that the CEM decreased in both of the rubbers. The reduction range of CEMs in rubber A is 27.88%–29.89%. By comparing the results obtained under different conditions, it is found that temperature and O$_2$ have little effect on the CEM of rubber A. For rubber B, the reduction range of CEMs is 10.42%–15.25% (gas mixture A) and 9.99% (gas mixture B), respectively. On the other hand, the presence of O$_2$ reduced CEM by 9.04% (85 °C) and 15.25% (100 °C), suggesting that rubber B is significantly affected by experimental conditions and is more sensitive to O$_2$ than the aging temperature. In terms of the CS, the values of rubber A range from 31.01 to 39.33%, about twice the values of rubber B (14.85%–17.89%). This phenomenon may result from the different ethylene contents in the two rubbers since ethylene can improve the strength of rubber and reduce the value of CS.

From the obvious deterioration of compression properties, it can be inferred that there is a certain degree of chemical reaction that happened and the compression properties of rubber B are more vulnerable to O$_2$ than those of rubber A. To intuitively investigate the intensity of the reaction between rubber and gas, the surface morphologies of rubbers were characterized by field emission scanning electron microscopy (FESEM) and are given in the next section.

3.2. Surface Morphology Characterization. The surface morphology results at 500 times and higher magnification are shown in Figures 3–5. It can be recognized from the surfaces of rubber A and B that the intensity of reaction was quite different. Under the effect of gas mixture A, the surface of rubber A was no longer flat and bulges appeared, which turned to be more obvious as the temperature rose, and a large number of scale-like substances grew above the bulges when O$_2$ was added. For rubber B tested at 85 °C, some lamellar structures appeared on the surface in the sealing device with gas mixture A, and the lamellar structures transformed to finer spiked structures in the samples of the sealing device with gas mixture B. When temperature was increased to 100 °C, the lamellar structures caked in gas mixture A, and some cracks could be found on its surface when rubber B was placed in gas mixture B. These cracks somehow explain the sudden degradation of the compression properties when the temperature was increased from 85 to 100 °C in gas mixture B.

Furthermore, the element compositions on the surfaces of samples were detected by energy dispersive spectrometry (EDS), and the energy spectra of the initial samples are shown in Figure 6. The elements on the untreated surface are mainly C, O, Zn, Si, S, and Ca. The source of C is each monomer of rubber, Zn, Si, and Ca come from the activator ZnO, $^{21}$ reinforcing agent SiO$_2$, $^{22}$ and CaCO$_3$, $^{23}$ and S belongs to the curing agent in the vulcanization process. In order to accurately verify the valence of surface elements, the rubber was further characterized through XPS.

The XPS characterization of the sample before the experiment is shown in Figure 7, and the nuclear calibration reference element is C (1s) 284.80 eV. The characteristic
peaks of C mainly located at 286.11 and 288.77 eV, which belongs to the C−C, C−H, C−O−C and O−C−O, C=O, respectively. For the O 1s spectra, the characteristic peaks located at 531.77 and 533.62 eV are assigned to the C−O and C=O components, respectively. It cannot find F and N elements on the natural samples. On the other hand, after the test, as is shown in Figure 8, the element intensities of F and N elements significantly increased. The characteristic peaks located at 688 and 684.5 eV correspond to the CF<sub>x</sub>CH<sub>x</sub> and C−F groups, and the peak located at 399 eV corresponds to C−N and C=N. The occurrence of the F element confirms the adsorption of the CF<sub>x</sub>N molecule, and the intensities of F and N in each condition differ greatly. In the XPS characterization of rubber A, the peak intensity of the F element is much stronger than that of N under the same conditions, while the result of rubber B is opposite. If the N element comes from the adsorption of C<sub>4</sub>F<sub>7</sub>N, then the intensity of the F element is supposed to be higher than that of the N element. Therefore, combined with the seriously damaged surface of rubber B, it is speculated that most of the N element belongs to N-containing cross-linking agents in rubber, such as TAIC (C<sub>12</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub>) or TAC (C<sub>12</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub>).

To make clear the variation of the gas mixture, we list the analysis results of the gas mixture obtained from GC−MS in the next section.

### 3.3. Gas Component Analysis

The main components of the gas mixture after the test are depicted in Figure 9, and the specific quantitative results are listed in Table 2.

![Figure 6](image1)
**Figure 6.** Energy spectra of the initial samples: (a) rubber A and (b) rubber B.

![Figure 7](image2)
**Figure 7.** Elements on the surfaces of the rubber samples before the test: (a) rubber A and (b) rubber B.

![Figure 8](image3)
**Figure 8.** Elements on the surfaces of the rubber samples after the test: (a) F - rubber A, (b) N - rubber A, (c) F - rubber B, and (d) N - rubber B.

![Figure 9](image4)
**Figure 9.** Results of gas component analysis: gas mixture A, (a) rubber A and (b) rubber B, gas mixture B, (c) rubber A, and (d) rubber B.

| Table 2. Concentration of Each Gas after the Experiment |
|-----------------------------------------------|
| rubber sample-gas mixture | product | concentration (ppm) |
| A-A | CF<sub>4</sub> | 0.006 | 2.353 |
| CO | 88.162 | 97.117 |
| C,F<sub>4</sub> | 0.025 | 0.018 |
| C,F<sub>6</sub> | 0.001 | 0 |
| C,F<sub>8</sub> | 3.373 | 29.928 |
| B-A | CF<sub>4</sub> | 0.004 | 0.014 |
| CO | 24.726 | 114.419 |
| C,F<sub>4</sub> | 0.016 | 0.010 |
| C,F<sub>6</sub> | 0.002 | 0.009 |
| C,F<sub>8</sub> | 2.251 | 73.729 |
| A-B | CF<sub>4</sub> | 0.013 | 0.023 |
| CO | 15.631 | 83.645 |
| C,F<sub>4</sub> | 0.019 | 0.020 |
| C,F<sub>6</sub> | 0.004 | 0.008 |
| C,F<sub>8</sub> | 15.631 | 83.645 |
| B-B | CF<sub>4</sub> | 0.100 | 0.013 |
| CO | 23.167 | 216.648 |

As can be seen in Figure 9, the main products generated by the reaction of rubber and the gas mixture are CO and C<sub>3</sub>F<sub>6</sub>, and their concentrations increased in accompany with the temperature increase and the addition of O<sub>2</sub>. For the generation of CO, the largest concentration was 114 ppm in the results of samples in gas mixture A, indicating that the reaction is weak. However, for the samples with gas mixture B, the concentration of CO increased sharply to several times higher than the results in gas mixture A. It should be noted that the decomposition temperatures of CO<sub>2</sub> and C<sub>4</sub>F<sub>7</sub>N are far higher than the experimental temperature; hence, the source of CO should be the bond breaking of corresponding groups in...
rubber and the reaction between \( O_2 \) and the C element in rubber.

When it comes to the concentration difference of \( C_3F_6 \) obtained from the two types of rubbers, the main reason lies on the different heat resistances brought by different third monomers. The thermal stability in DCPD-EPDM is worse than that in ENB-EPDM,\(^8\) thus, the heat and gas can penetrate into rubber B easily and its molecular chain is prone to fracture reaction, causing the \( C_4F_7N \) molecule to interact with the dissociation group and decompose. The energy provided by the temperature increase directly promoted the reaction between the \( C_4F_7N \) molecule and rubber, while \( O_2 \) aggravated the corrosion of rubber, and both of the factors elevated the concentration of \( C_3F_6 \). Besides, other scholars\(^{29-31} \) showed that the metal and its compounds will react with the \(-CN\) group of the \( C_4F_7N \) molecule and even result in its decomposition. As is known, EPDM contains a certain amount of metal compounds, which can partly contribute to the generation of \( C_3F_6 \).

### 3.4. Effect of Silicone Grease on Compatibility.

From the results of the mechanical test, morphology characterization, XPS characterization, and gas composition analysis, it is undoubtedly that the composition of the gas mixture and the properties of rubber have been changed in varied degrees, and as time passes by, serious corrosion can happen on the rubber and \( C_4F_7N \) may decompose in large quantities. Therefore, to prevent the rubber from contacting with the gas mixture, we applied silicone grease to rubber and performed the thermal aging test; the mechanical properties and gas compositions after the test are listed in Tables 3 and 4, respectively.

#### Table 3. Compression Properties of Rubber after the Experiment

| rubber sample-gas mixture | CEM (MPa) | CS (%) |
|---------------------------|-----------|--------|
|                           | 85 °C     | 100 °C | 85 °C | 85 °C |
| A-A                       | 7.26      | 7.40   | 28.48 | 36.99 |
| B-A                       | 9.06      | 8.45   | 13.54 | 15.20 |
| A-B                       | 7.30      | 7.43   | 31.01 | 38.72 |
| B-B                       | 8.38      | 7.46   | 14.37 | 16.42 |

When rubber A was coated with silicone grease, its CEM changed little compared to the results without silicone grease, and the value of CS decreased slightly by 0.61–2.53%. The CEM of rubber B deteriorated to a certain extent as well as its CS. These variations in mechanical properties seem to be not so much caused by the protection of silicone grease as by random errors. However, at the same time, these results verified that silicone grease is harmless to rubber A.

In the results of gas composition analysis, the concentrations of CO and \( C_3F_6 \) generated in sealing devices with rubber A decreased significantly. For the concentration of CO, it has dropped to less than 30 and 40% compared with the results in Section 3.3 in gas mixtures A and B, respectively. On the other hand, the concentration of \( C_3F_6 \) in all test conditions dropped below 1 ppm. This phenomenon revealed that silicone grease can effectively cut off the contact between rubber A and the gas mixture, and by virtue of its excellent chemical stability and oxidation resistance, it weakens the reaction caused by the gas mixture and heat while preventing \( C_4F_7N \) from interacting with the active group on the surface of rubber and decomposing into \( C_3F_6 \). By contrast, the situation in rubber B was a far cry from rubber A. In the sealing devices with gas mixture A, the concentrations of CO and \( C_3F_6 \) reduced by several to tens of ppm under the protection of silicone grease. However, in sealing devices with gas mixture B, silicone grease seems to enhance the generation of CO and \( C_3F_6 \). By comparing the surfaces of the rubbers after the experiment, it can be found that the silicone grease layer of rubber A kept intact while a few bubbles occurred on the surface of rubber B, as is given in Figure 10. It is assumed that the silicone grease reacted with rubber B and generated some kind of gas that broke the silicone grease layer and made the rubber surface expose to the gas mixture again.

#### Table 4. Concentration of Each Gas after the Experiment

| rubber sample-gas mixture | product | concentration (ppm) |
|---------------------------|---------|---------------------|
|                           | 85 °C   | 100 °C              |
| A-A                       | CF\(_4\) | 0.029               | 0.021               |
|                            | CO      | 21.153              | 30.005              |
|                            | \( C_3F_6 \) | 0.020               | 0.029               |
|                            | \( C_2F_6 \) | 0.006               | 0              |
|                            | \( C_2F_4 \) | 0.299               | 0.844              |
| A-B                       | CF\(_4\) | 0.029               | 0.002               |
|                            | CO      | 40.536              | 106.078             |
|                            | \( C_3F_6 \) | 0.111               | 0.029               |
|                            | \( C_2F_6 \) | 0.002               | 0              |
|                            | \( C_2F_4 \) | 5.221               | 59.683              |
| B-B                       | CF\(_4\) | 0.018               | 0.040               |
|                            | CO      | 153.640             | 275.380             |
|                            | \( C_3F_6 \) | 0.038               | 0.029               |
|                            | \( C_2F_6 \) | 0.016               | 0.005               |
|                            | \( C_2F_4 \) | 0.406               | 3.563              |
|                            | CO      | 554.999             | 1272.283            |
|                            | \( C_3F_6 \) | 0.003               | 0.010               |
|                            | \( C_2F_6 \) | 0.013               | 0              |
|                            | \( C_2F_4 \) | 83.256              | 207.094             |

Figure 10. Silicone grease on the surface of (a) rubber A and (b) rubber B after the test.

4. CONCLUSIONS

In this paper, we carried out a thermal aging test to investigate the compatibility between the \( C_4F_7N \) gas mixture and EPDM with different third monomers. The rubber samples and the gas mixture after the test were characterized based on FESEM, EDS, XPS, and GC–MS, and thus, the interaction mechanism was unfolded through analyzing these results. The derived conclusions are as follows

1. The reaction between the gas mixture and rubbers took place in varying degrees. Compared with ENB-EPDM, the surface of DCPD-EPDM was damaged more seriously, and even cracks appeared at 100 °C when it was exposed to \( C_4F_7N/CO_2/O_2 \). In terms of the mechanical properties, both of the rubbers came across
a drop in their compression sets and compression elastic moduli, and the addition of 6% O₂ brought a greater influence on DCPD-EMPD than that on ENB-EPDM. In the results of gas composition, the concentrations of CO and C₄F₇N generated by the reaction between the gas mixture and DCPD-EMPD are much higher than those generated by ENB-EPDM. The different performances in various aspects mainly came from the difference in thermal stabilities of ENB and DCPD. ENB can provide high heat resistance and keeps the rubber maintain its properties under the effect of high temperature and gas mixture. Therefore, ENB-EPDM shows better performance than DCPD-EPDM.

(2) After coating silicone grease on the rubber surface, it was found that the concentrations of C₄F₇N and CO generated by the reaction of ENB-EPDM and gas mixture were sharply reduced. This fact signifies that silicone grease can effectively block the contact between the gas mixture and rubber. Moreover, the chemical stability and oxidation resistance of silicone grease are stronger than those of rubber, so the reaction triggered by the C₄F₇N gas mixture will be significantly weakened. However, for DCPD-EPDM, silicone grease will react with it and generate some kind of gas, leading to the breaking of the silicone grease layer and re-exposing the surface to the gas mixture and reacting again.

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Notes
The authors declare no competing financial interest.

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