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Thermal Compatibility Between Perfluoroisobutyronitrile-CO₂ Gas Mixture With Copper and Aluminum Switchgear

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ABSTRACT Perfluoroisobutyronitrile (C₄F₇N) has received extensive attention over the past three years as an environmental friendly gas insulating medium. At present, there are few studies on the compatibility of C₄F₇N with metal materials used in the electrical equipment. In this paper, we constructed a gas–solid interfacial reaction platform and carried out aging tests for C₄F₇N-CO₂ gas mixture at different temperatures. The composition of the gas mixture and the surface morphology and the elemental composition of copper, aluminum were explored. The interaction mechanism between C₄F₇N-CO₂ gas mixture and metal surface was also analyzed. It was found that C₄F₇N-CO₂ gas mixture has better compatibility with aluminum than copper. The reaction between C₄F₇N-CO₂ gas mixture and heated copper at 220 °C produces the gaseous by-product C₃F₆. The surface color of copper deepens and the corrosive degree increases with temperature, while the interaction between C₄F₇N and heated aluminum does not cause gas decomposition and metal corrosion. In general, the thermal stability of C₄F₇N-CO₂ gas mixture associated with heated copper and aluminum is inferior to that of SF₆.

INDEX TERMS C₄F₇N-CO₂, thermal compatibility, copper, aluminum, SF₆ alternative gas.

I. INTRODUCTION

Sulphur hexafluoride (SF₆) is widely used in the electrical industry as insulating and switching medium for medium-voltage (MV) and high-voltage (HV) gas insulated switchgear (GIS) due to its excellent properties of high dielectric strength, nontoxicity, chemical inertness, broad operating temperatures range and exceptional arc quenching properties [1]–[3]. However, SF₆ has the global warming potential (GWP) of 23500 CO₂ equivalents and an atmospheric life span about 3200 years, making it the most potent greenhouse gas (GHG) [4], [5]. It is reported that the current equilibrium warming due to SF₆ is 0.004 °C, with a clear tendency to increase [6]. Moreover, electrical industry is the main consumer and emitter of SF₆ and the total mass of banked SF₆ worldwide in electrical equipment is in the order of magnitude of 10⁵ tons [6]. Thus, much effort has been made across the industry over the last decades to find a viable alternative gas to SF₆.

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Recently, C₄F₇N (2,3,3,3-tetrafluoro-2-(trifluoromethyl)-2-propanenitrile, Perfluoroisobutyronitrile) has been listed as a potential substitute gas for SF₆. C₄F₇N has an exceptionally low GWP of 2090, the zero ozone depletion potential (ODP) and a dielectric withstand roughly twice as high as that of SF₆ [7], [8]. While its boiling point reaches to −4.7°C at normal pressure that can only be used as additive to a background gas such as CO₂, N₂ or technical air. Experimental and theoretical studies have been conducted on the dielectric strength [9]–[11], decomposition characteristics [12]–[15], switching behavior [16], [17] of C₄F₇N gas mixture over the past few years, confirming that C₄F₇N has potential using in HV gas insulated equipment (GIE).

In addition, the stability of alternative gases can be a key factor for the application in high voltage GIE which is typically designed for a life-time of 50 years and more with very few maintenance activities [18]. Therefore reliable data about the chemical long-term stability of gas insulating medium is required. Moreover, gas mixture is contacting with different materials in the GIE, such as metals, polymers and desiccants. Thus the interaction between the gas mixture and materials

also need to be investigated and understand. And the aging principles on gas mixture as well as the materials in contact with it should be known to get a base for prediction of the lifetime of the equipment.

At present, there are few reports on the compatibility between C₄F₇N gas mixture and materials using in the equipment. Kessler *et al.* [19] investigated the gas-material interaction for C₄F₇N gas mixture and small quantities of decomposition products such as hexafluoro propene and heptafluoro propane were found when C₄F₇N contacted with desiccant for several weeks. Our team explored the interaction between C₄F₇N and Cu (1 1 1), Ag (1 1 1), Al (1 1 1) based on the density functional theory (DFT). We found that the adsorption of C₄F₇N on Cu (1 1 1) has higher adsorption energy and total charge transfer value than that of Ag (1 1 1) and Al (1 1 1) surface, indicating that the interaction between C₄F₇N and Cu (1 1 1) is stronger [20], [21].

In this paper, we carried out accelerated aging tests on C₄F₇N-CO₂ gas mixture interacting with copper, aluminum using in GIS as current-carrying conductors [22]. The composition of chemical elements and morphology of metal surface after aging tests were tested using the optical microscope, scanning electron microscope (SEM) and X-ray photoelectron spectroscopy (XPS). The gas composition of C₄F₇N gas mixture after tests was also detected and analyzed by gas chromatography-mass spectrometer (GC-MS). Relevant results not only revealed the thermal compatibility between C₄F₇N and copper, aluminum comprehensively, but also provide references for evaluating the lifetime of the equipment with C₄F₇N-CO₂ gas mixture.

II. METHOD

A. EXPERIMENTAL METHODS

In order to explore the interaction between C₄F₇N-CO₂ gas mixture and copper, aluminum, the aging test platform shown in Figure 1 was used. The test platform is mainly consist of the gas chamber, the temperature control system and the gas-solid interfacial reaction module. The gas chamber which can withstand the pressure of about 0.6MPa is made of 304L stainless steel. The pressure and temperature of the gas mixture in the chamber is monitored using the barometer and gas temperature sensor. We used the K-type temperature sensor to test the temperature of the heating element. The heating and temperature control system mainly includes the heating element, the switching power supply, the solid state relay, the proportion integration differentiation (PID) controller and interface temperature sensor components. The PID controller receives the signal of the interface temperature sensor and controls the switch power supply through solid state relay, so as to control on-off of the heating element and keep its temperature at a stable value. The gas-solid interfacial reaction module is made of copper or aluminum, which is used to simulate the current-carrying bus in the real electrical equipment.

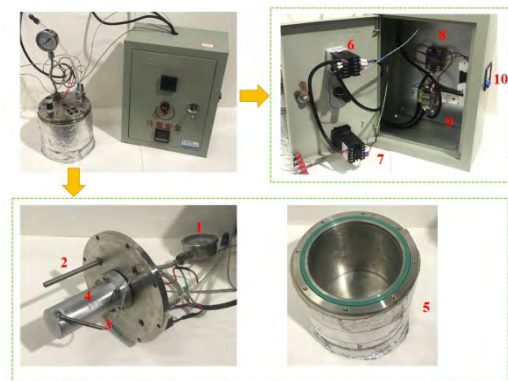


FIGURE 1. Structure of test platform: 1- barometer, 2- gas temperature sensor, 3- interface temperature sensor, 4- gas-solid interfacial reaction module, 5- gas chamber, 6,7 - temperature control and display module, 8,9 - solid state relay, 10- switch.

The inner wall of the gas chamber as well as the metal sheet and the gas-solid interfacial reaction module were cleaned before the test using the absolute alcohol and distilled water to remove the impurities. Then the gas-solid interfacial reaction module and the metal sheet with the size of 0.2*5*100mm are installed. The metal sheet fixed on the gas-solid interfacial reaction module can be used for sample characterization. The gas chamber was then pumped and filled with pure CO₂ gas (99.999%) to 0.3MPa, then pumped again to remove the gas impurities. This process is repeated for three times. Finally, the 10%C₄F₇N-90%CO₂ gas mixture was filled to the gas chamber to 0.3MPa to carry out relevant thermal aging tests at different temperatures.

Considering the requirement of pressure and liquefaction temperature for gas insulating medium in the equipment, the content of C₄F₇N in C₄F₇N-CO₂ gas mixture is generally less than 10% [23]. Therefore, we chooses 10%C₄F₇N-90%CO₂ gas mixture to carry out relevant tests. Moreover, the temperature of the current-carrying metal in the equipment under rated working condition is generally about 115-120°C. And the local overheating faults caused by poor contact of the breaker may lead to the temperature higher than the normal conditions. Thus we carried out relevant aging testes at 120°C, 170°C and 220°C to simulate the actual state of the current-carrying metals in the equipment under normal and overheating fault conditions. We carried out aging tests for C₄F₇N-CO₂ and copper at 220°C for several different period and found that the color of metal surface changed obviously after 40 hours interaction, indicating that this period is enough for reactions between gas mixture and copper to occur. Thus the test period was set to 40hours.

B. SAMPLE CHARACTERIZATION

The characterization of test samples includes gas composition analysis and metal characteristic analysis.

The GC-MS is used to detect components of C₄F₇N-CO₂ gas mixture before and after the test to determine whether some by-products are generated during the aging process.

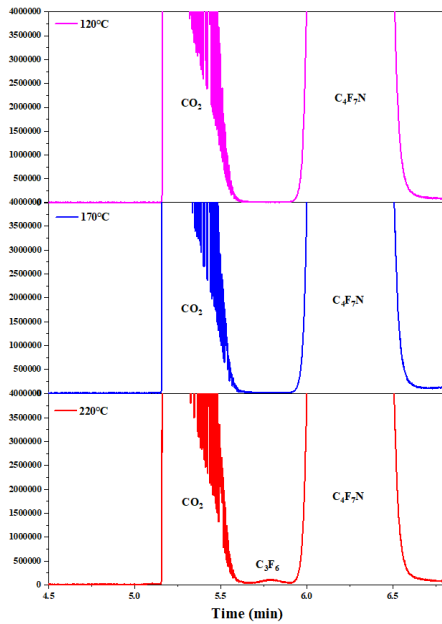


FIGURE 2. GC-MS spectra of C₄F₇N-CO₂ gas mixture after ageing tests.

The model of GC-MS and chromatographic column is Shimadzu Ultra 2010plus and CP-Sil 5CB (60m*8um*0.32mm), respectively. The working conditions of GC-MS are listed as follows: the temperature of the inlet and ion source is 200 °C, the split ratio is set to 10. The oven temperature is kept at 32°C for 10 minutes, and then heated up to 150°C with the increasing rate at 60°C/min.

In addition, the optical microscopy and field emission scanning electron microscopy (FESEM, Zeiss SIGMA) are used to characterize the morphology of the metal sheets for each group to reveal their morphological changes before and after the ageing tests. The elemental composition of the metal surface is also investigated using the X-ray photoelectron spectroscopy (XPS).

III. RESULTS AND DISCUSSION

A. COMPATIBILITY BETWEEN C₄F₇N-CO₂ AND COPPER

1) GAS COMPONENTS

Figure 2 gives the gas chromatograms of C₄F₇N-CO₂ gas mixture after aging for 40 hours at different temperatures.

For the copper surface heated at 120°C and 170°C (The temperature of gas mixture at 69°C and 121 °C, respectively), there is no new characteristic peak appears in the gas chromatogram after the test, which indicates that the interaction process does not cause the decomposition of C₄F₇N-CO₂ gas mixture and the thermal stability of the gas mixture is great. When the temperature of gas-solid interface reached to 220 °C (The temperature of gas mixture reach 168°C), one decomposition by-product C₃F₆ can be found. Actually, it is reported that the thermal decomposition of C₄F₇N-CO₂ gas mixture starts at 650 and the first detected decomposition component is CO, while the other products such

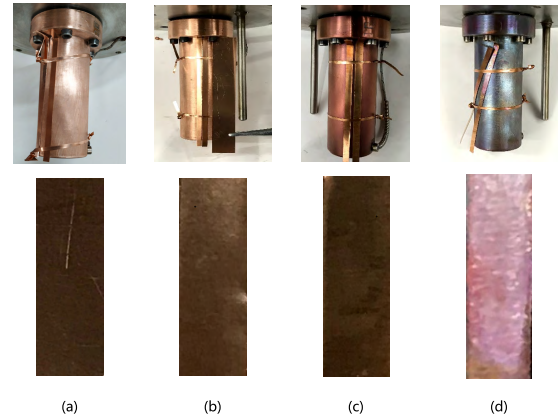


FIGURE 3. Photo images of copper surfaces before and after exposed to 10% C₄F₇N- 90%CO₂ environment for 40 hours: (a) Unheated, (b) 120°C, (c) 170°C, (d) 220°C.

as COF₂, CF₃CN and C₂F₅CN are founded at 800°C [8]. While the temperature of gas mixture is lower than 200°C during the aging tests, which means the C₄F₇N-CO₂ gas mixture could not decomposition at this temperature. Therefore, C₄F₇N-CO₂ gas mixture is strongly interacted with copper at 220°C, causing the decomposition of C₄F₇N to generate C₃F₆.

On the whole, considering the temperature rise effect under normal working condition, C₄F₇N-CO₂ gas mixture exhibits excellent thermal stability with copper and the long-term interaction process do not cause the gas decomposition. While the high temperature (above 220°C) caused by the overheating faults will result in the decomposition of C₄F₇N-CO₂ gas mixture.

2) SURFACE MORPHOLOGY CHARACTERIZATION OF COPPER

Figure3 gives the photo images of copper surface before and after exposed to 10% C₄F₇N-90%CO₂ environment for 40 hours. It can be seen that the surface color of copper after 40 hours' interaction with C₄F₇N-CO₂ at 120°C is evenly distributed and has no significant difference with the untreated one. With the increase of the gas-solid interface temperature, the surface color of the copper exists some variations. The surface color changes from pale orange to dark orange after aging for 40 hours at 170°C, indicating that the copper has been corroded to a certain extent. When the temperature reaches to 220°C, the copper sheet becomes to fuchsia (purple) after interaction and the color distribution is uneven, which confirms that the copper has been seriously corroded at this condition.

Combined with the optical microscope images shown in Figure4, we can confirm that the copper begins to be corroded by 10%C₄F₇N-90%CO₂ at 170°C. Some surface area becomes to faint red at 170°C and the copper is deeply discolored when the temperature reaches to 220°C. The surface color turns into golden or fuchsia at this condition. Above all, the corrosion degree of copper is intensified with the

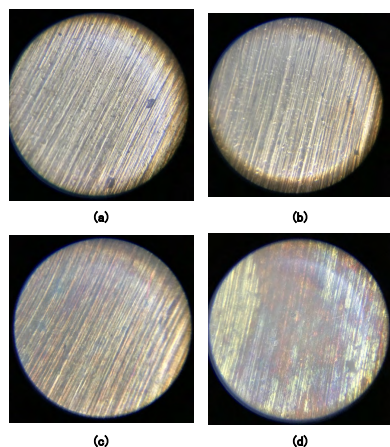


FIGURE 4. Optical microscope images of copper surfaces before and after exposed to heated 10% C_4F_7N -90% CO_2 environment for 40 hours (Magnification: 200X): (a) Unheated, (b) 120°C, (c) 170°C, (d) 220°C.

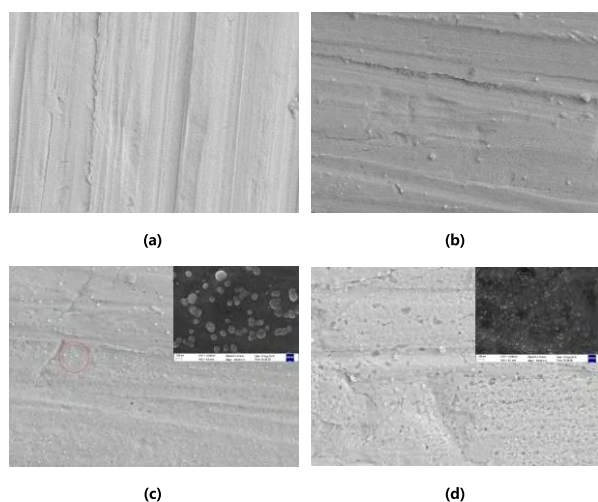


FIGURE 5. SEM images of copper surfaces before and after exposed to 10% C_4F_7N -90% CO_2 environment for 40 hours (Magnification: 5K): (a) Unheated, (b) 120°C, (c) 170°C, (d) 220°C.

increase of interface temperature. The main characteristics of corrosion includes the deepening and uneven distributing of the surface color. Thus there exists incompatibility between C_4F_7N - CO_2 gas mixture and copper to a certain extent.

In order to further analyze the morphological modifications of the copper surface after the aging tests, all the samples were tested using the FESEM (as shown in Figure 5). We can find that the surface structure of untreated copper is flat and the cutting cross-section (in the form of thin texture) is clearly visible. A small amount of corrosion points which is randomly distributed can be found on the surface at 120°C. The structure of copper surface does not change significantly as a whole, indicating that the interaction between C_4F_7N - CO_2 gas mixture and copper does not destroy the microstructure of the metal surface. The distribution area and density of the corrosion points is increased with the interface temperature. Spherical or cube-like crystal particles can be found when the corrosion area is enlarged. The copper surface is basically covered by cube-like crystal particles when the temperature

increases to 170°C, indicating that the microstructures of surface has been changed significantly. When the interface temperature reaches to 220°C, a large number of crystal particles produced are densely distributed in all areas of the copper surface. High magnification image shows that the particles are layered and the microstructure of copper surface is seriously damaged.

3) XPS ANALYSIS

Figure 6 gives the XPS spectra of characteristics elements in copper surface before and after exposed to 10% C_4F_7N -90% CO_2 environment for 40 hours. All the XPS spectra were internally calibrated by adventitious carbon C1s binding energy at 284.8 eV and then fitted using the Gaussian method [24], [25]. Relevant chemical states analysis were finally carried out based on the National Institute of Standards and Technology (NIST) XPS database.

According to the high-resolution spectra of Cu2p shown in Figure 6 (a), there exists four main peaks locate at 932.46eV, 934.75eV, 952.33eV and 954.72eV, corresponding to the Cu 2p_{3/2}, CuO 2p_{3/2}, CuO2p_{1/2} and Cu 2p_{1/2}, respectively. The O1s spectra of the samples after aging tests can be described as two peaks by Gaussian distribution locating at 530.42eV and 531.84eV, which belongs to the C=O and CuO components. Therefore, the copper surface was oxidized after interacting with C_4F_7N - CO_2 gas mixture at 120°C-220°C for 40 hours.

The XPS spectra of C1s after aging tests at 120 °C is made up of two individual peaks at binding energies near 284.8 and 288.56eV, which are attributed to the C1s and COO components. As to the samples treated at 170°C and 220°C, the C1s peak at 293.53 eV can be found, which is associated with the C-F component. Moreover, the high-resolution spectra of F shows that the CF_x component located at 688.98eV can be seen for all groups after interaction. And the peaks centered around 684.69eV can be ascribed to CuF_2 . Thus the interaction between copper and C_4F_7N - CO_2 gas mixture brings fluorine to the copper surface and generates CuF_2 at 170°C and 220°C.

As a whole, C_4F_7N - CO_2 gas mixture could react with copper at high temperature. The composition of the gas mixture did not change significantly at 120°C and 170°C, while the interaction between C_4F_7N - CO_2 gas mixture and copper at 220°C resulted in decomposition of gas mixture to produce some by-products such as C_3F_6 . As the interface temperature increases, the color of the copper surface gradually deepens and its distribution becomes uneven. SEM results shows that a large number of crystalline particles are generated, and the corrosion of the surface is further aggravated. XPS results also show that the C_4F_7N - CO_2 gas mixture reacted with copper could produce CuO, CuF_2 and cause fluorine accumulation on the metal surface. Although the temperature rise effect under normal condition will not corrode copper or cause gas decomposition, the high temperature generated under overheating fault conditions will promote the interaction between C_4F_7N - CO_2 gas mixture and current-carrying

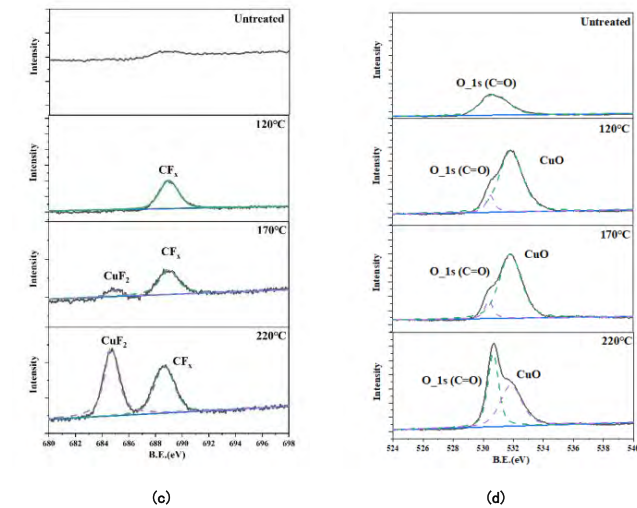
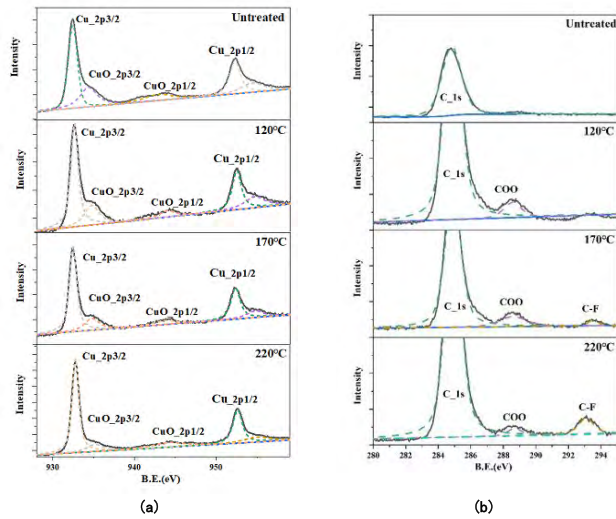


FIGURE 6. XPS spectra of characteristics elements in copper surfaces before and after exposed to 10% C_4F_7N -90% CO_2 environment for 40 hours: (a) Cu, (b) C, (c) F, (d) O.

copper, which will pose a certain threat on the service life of the equipment.

B. COMPATIBILITY BETWEEN C_4F_7N - CO_2 AND ALUMINUM

1) GAS COMPONENTS

Aging tests for aluminum and C_4F_7N - CO_2 gas mixture were also conducted at 120°C, 170°C and 220°C to investigate the compatibility between them. Figure 7 gives the GC-MS spectra of C_4F_7N - CO_2 gas mixture after ageing tests for 40hours. It can be seen that the C_4F_7N - CO_2 gas mixture does not generate any new characteristic peaks after interaction with the heated aluminum surfaces at 120°C, 170°C and 220°C (The temperature of gas mixture at 71°C, 122 °C, and 174°C, respectively), indicating that composition of the gas mixture does not change significantly, that is, the C_4F_7N - CO_2 gas mixture exhibits great compatibility with aluminum at 120°C-220°C.

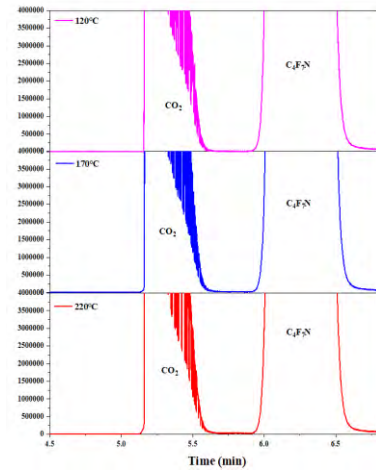


FIGURE 7. GC-MS spectra of C_4F_7N - CO_2 gas mixture after ageing tests.

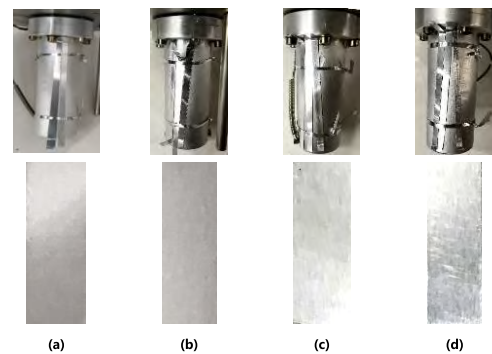


FIGURE 8. Photo images of aluminum surfaces before and after exposed to heated 10% C_4F_7N -90% CO_2 environment for 40 hours: (a) Unheated, (b) 120°C, (c) 170°C, (d) 220°C.

2) SURFACE MORPHOLOGY CHARACTERIZATION OF ALUMINUM

Figure 8 gives the photo images of aluminum surfaces before and after exposed to 10% C_4F_7N -90% CO_2 for 40 hours. We can find that the aluminum surface before the test has a uniform color distribution. And the surface color does not change significantly after interacting with C_4F_7N - CO_2 gas mixture at different temperatures. Combining with the optical microscopy results shown in Figure 9, it can be seen that the texture of the aluminum is clear, and the surface structure is not corroded after the tests. Therefore, the interaction between C_4F_7N - CO_2 gas mixture and aluminum will not lead to significant changes in the surface structure of aluminum.

Figure 10 shows the SEM results of aluminum after the aging tests. It can be seen that the untreated aluminum has clear texture and flat structure. After interacted with C_4F_7N - CO_2 gas mixture, a small amount of particles are generated and randomly distributed on the aluminum surface. With the increase of surface temperature, the content of the above particles increased slightly. On the whole, C_4F_7N - CO_2 gas mixture did not cause serious corrosion to the aluminum, thus the compatibility between them is great.

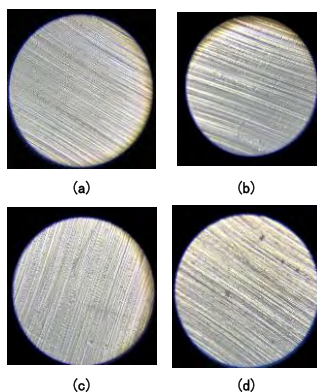


FIGURE 9. Optical microscope images of aluminum surfaces before and after exposed to heated 10% C_4F_7N -90% CO_2 environment for 40 hours (Magnification: 200X): (a) Unheated, (b) 120°C, (c) 170°C, (d) 220°C.

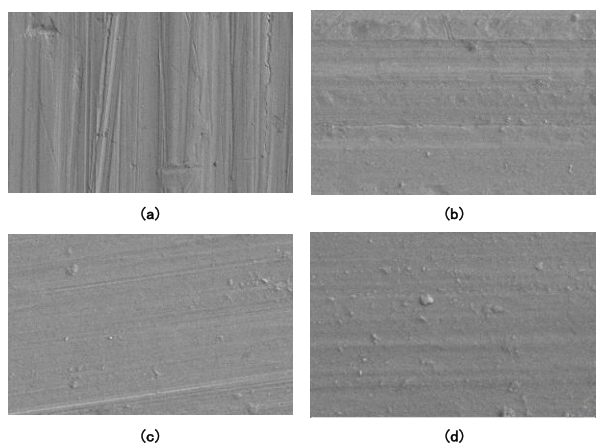


FIGURE 10. SEM images of aluminum surfaces before and after exposed to heated 10% C_4F_7N -90% CO_2 environment for 40 hours (Magnification:5K): (a) Unheated, (b) 120°C, (c) 170°C, (d) 220°C.

3) XPS ANALYSIS

XPS measurement is also carried out to further analyze the composition of the elements and surface chemical states in the samples. Figure 11 shows the XPS spectra of characteristics elements in aluminum surface before and after exposed to 10% C_4F_7N -90% CO_2 environment for 40 hours.

It can be found that the binding energies for Al2p are mainly located at 71.89eV and 74.37eV, which are attributed to Al and Al₂O₃, respectively. According to the high-resolution spectra of O1s, the binding energy at 531.79eV can be ascribed to Al₂O₃. The other peaks at 533.27eV can be found for the aging samples at 220 °C, corresponding to the COO component.

Figure 11 (b) and (c) presents the high resolution XPS spectra of C1s and F1s. Two peaks centered at 288.8eV and 284.92eV belong to the C1s and COO components, respectively. And another two peaks located at 285.94eV and 292.83eV are attributed to CN group and CF_x component, indicating the aging process introduced CN and CF_x to the metal surface. The high resolution XPS spectra of F1s also confirms the generation of fluorine on the metal surface after

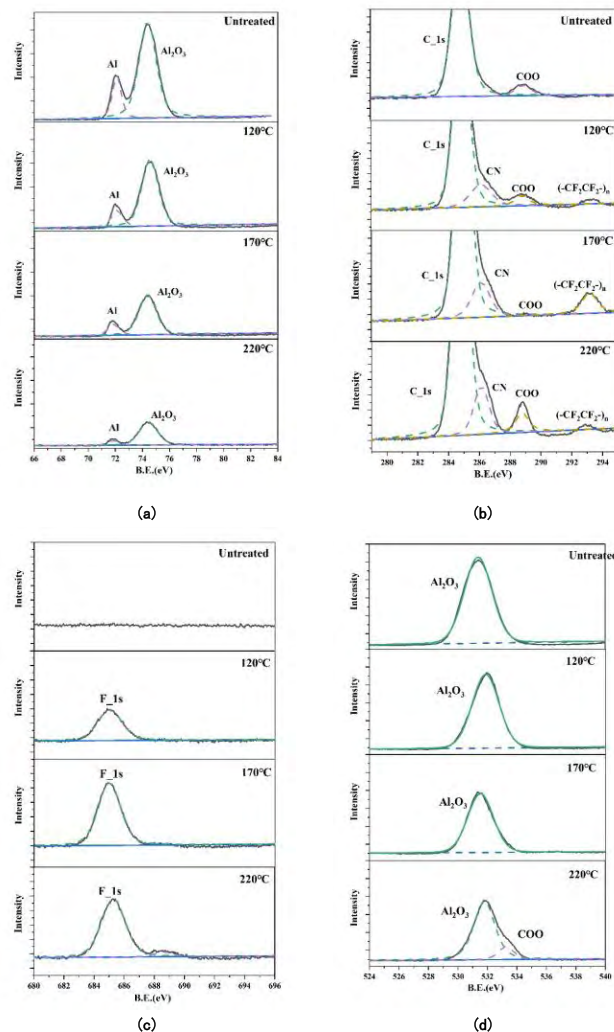


FIGURE 11. XPS spectra of characteristics elements in aluminum surfaces before and after exposed to 10% C_4F_7N -90% CO_2 environment for 40 hours: (a) Al, (b) C, (c) F, (d) O.

interaction and the peak located around 689.12eV at 220°C belongs to the CF_x components.

Therefore, the composition of the C_4F_7N - CO_2 gas mixture and the surface morphology of the aluminum does not change significantly after aging tests, but amount of fluorine accumulation occurs. On the whole, the compatibility of C_4F_7N - CO_2 gas mixture with aluminum is great. Considering several conditions such as temperature rise effect and potential overheating faults in real equipment, C_4F_7N - CO_2 gas mixture will not corrode the aluminum to a certain extent.

C. DISCUSSION

According to the test results, the compatibility of C_4F_7N - CO_2 gas mixture with copper is inferior to that of aluminum. The reaction between C_4F_7N - CO_2 gas mixture and copper at high temperature results in the decomposition of C_4F_7N - CO_2 gas mixture and corrosion of metal surface. At the same time, XPS results show that copper surface generates CuO, CuF₂ and other substances after interaction, while the interaction

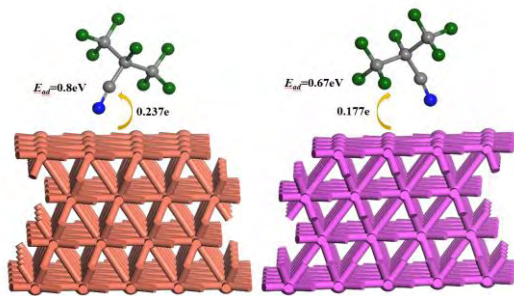


FIGURE 12. Interaction between C₄F₇N and Cu (1 1 1), Al (1 1 1).

between C₄F₇N-CO₂ gas mixture and aluminum does not lead to gas decomposition or metal corrosion.

Actually, our team has explored the interaction mechanism between C₄F₇N with copper and aluminum based on the DFT [21]. We optimized various initial interaction structures and found that the CN group in the C₄F₇N molecule had strong reactivity. The maximum adsorption energy of C₄F₇N absorbed on the Cu (1 1 1) surface reaches to 0.8eV, and the interaction process is accompanied by total charge transfer of 0.237e. While the maximum adsorption energy of C₄F₇N absorbed on the Al (1 1 1) surface is 0.67eV accompanied by charge transfer of 0.177e (As shown in Figure 12). Relevant theoretical calculation results show that the interaction between C₄F₇N and Cu (1 1 1) is stronger than that of Al (1 1 1), which is consistent with the experimental results in this paper.

The reaction mechanism of C₄F₇N with copper and aluminum surface is summarized as follows:



Moreover, literature [22] tested the thermal stability of SF₆ associated with heated copper and aluminum. It is pointed out that the interaction between SF₆ and copper at 300°C would not change the color and composition of copper surface. When the temperature reached to 330-385°C, the color of copper surface changed obviously, and some sulfur elements is accumulated. Thermal aging test results for SF₆ and aluminum shows that aluminum does not react with SF₆ gas for applied heating mantle temperatures up to 400°C. In fact, although aluminum is more active than copper, it is easy to oxidize and forming the dense Al₂O₃ film on the surface, preventing further corrosion of the aluminum.

Generally, the stability of C₄F₇N-CO₂ gas mixture associated with heated copper and aluminum is inferior to that of SF₆. C₄F₇N-CO₂ gas mixture could react with heated copper, which causes the decomposition of C₄F₇N and formation of some gaseous by-products, as well as copper corrosion. The above problems will have a negative impact on the service life of the equipment under long-term conditions. Therefore, the copper current-carrying conductor for electrical

equipment using C₄F₇N-CO₂ gas mixture gas mixture as the insulating medium should do anti-corrosion treatment to ensure the safety and lifetime of the equipment.

IV. CONCLUSION

In this paper, the gas-solid interfacial reaction platform was constructed and the thermal aging tests of C₄F₇N-CO₂ gas mixture with copper, aluminum at different interface temperatures were carried out. The composition of C₄F₇N-CO₂ gas mixture as well as the surface structure and element composition of metal materials are explored and analyzed. The gas-solid interfacial mechanism is discussed and the compatibility of C₄F₇N-CO₂ gas mixture with copper and aluminum are evaluated. Several conclusions can be obtained as follows:

- 1) C₄F₇N-CO₂ gas mixture could react with copper at 220°C, resulting in the generation of C₃F₆ and strong corrosion on the metal surface. With the increase of interface temperature, the color of copper surface gradually deepens, and a large number of crystalline particles are produced. The generation of CuO, CuF₂ and fluorine accumulation on the metal surface can be found.
- 2) The interaction between C₄F₇N-CO₂ gas mixture and aluminum at high temperature will not lead to the decomposition of gas insulating medium and strong corrosion of aluminum surface. The morphology of aluminum does not change significantly and the compatibility between them is great.
- 3) The compatibility of C₄F₇N-CO₂ gas mixture with copper is weaker than that of aluminum, and the high temperature generated under overheating fault conditions will promote the interaction between gas mixture and copper, threatening the service life and safety of the equipment.
- 4) Anti-corrosion treatment is needed to prevent the corrosion of copper in subsequent engineering applications. Temperature monitoring is also suggested to be strengthened to avoid strong corrosion caused by overheating faults.

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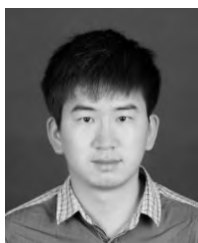
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