Influence of contact layer on the sulphur corrosion of copper conductors in power transformers

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Abstract
Owing to the corrosive sulphides in the mineral insulating oil such as dibenzyl disulphide (DBDS), copper windings in oil-immersed transformers may suffer sulphur corrosion. This study investigates the influence of different contact conditions on the corrosion of copper conductors in transformers. Oil-immersed accelerated corrosion experiment was conducted on copper conductors with various contact materials and different layers of insulating paper. The surface micromorphology, deposition component and the potentiodynamic polarization characteristics of copper conductors were explored. The results show that copper with different contact materials suffer sulphur corrosion; conversely, bare copper rarely does. It was also verified that the existence of a particular gap between copper and contact layer is a significant factor in sulphur corrosion by the formation of a concentration cell on the copper surface. The insulating paper wrapped around copper conductors does not increase the corrosion tendency of copper; however, it contributes to the formation of a region concentrated with DBDS, Cu2+, H+ by adsorption and impediment, which plays a key role in facilitating the sulphur corrosion of copper conductors. Moreover, sulphur corrosion can also occur on other metal parts in transformers when there is a particular gap on surfaces other than copper.

1 | INTRODUCTION

The mechanism of sulphur corrosion of copper conductors in oil-immersed power transformers and shunt reactors has been intensively studied in the recent years. Copper conductors react with corrosive sulphur in mineral insulating oil, particularly the dibenzyl disulphide (DBDS), to form conductive copper sulphide (Cu₂S) on the surface of the copper and the insulating paper. Cu₂S deposited on windings weaken the strength of the oil-paper insulation and even lead to serious insulation failures [1–4]. Figure 1 shows an inter-turn breakdown case in a 500 kV transformer caused by sulphur corrosion of the copper conductor, whose insulation strength was weakened by the corrosion deposition on the insulating paper. Current investigations reveal that sulphur corrosion of copper conductors is influenced by various factors, such as temperature, atmosphere, DBDS content, Cu²⁺, acids and other additives in the insulating oil [5–11]. Previous studies show that the electric field applied to copper windings accelerates sulphur corrosion by the electrolytic cell effect with obvious polar effects [12–14].

Insulating oil must undergo a non-corrosive test to reduce the risk of sulphur corrosion in the power equipment. To date, several testing standards have been proposed including DIN 51353, ISO 5662, ASTM D1275A, ASTM D1275B and IEC...
62535-2008. The corrosiveness of an insulating oil is graded according to the test method ASTM D 130/TP 154 by an accelerated corrosion test. However, different standards may lead to diverse judgements on the same oil sample, and the result of IEC 62535-2008 tends to be more rigorous than the others [15]. Using a copper conductor wrapped in insulating paper is one of the major features that differentiates IEC 62535-2008 from other standards. Investigations have found that a particular contact between the insulating paper and the copper conductor contributes to the deposition of Cu₂S on the surface of the insulating paper [16]. Previous studies have also shown that the packaging of the insulating paper facilitates the corrosion of the copper conductors in oil-immersed transformers by accelerating the formation of acids and preventing the diffusion of Cu²⁺ into the oil, thus resulting in more Cu₂S deposition on both the copper and the insulating paper [17–19].

However, current studies neither consider the effect of different contact materials on copper corrosion, nor explain whether the existence of corrosion deposition or insulating paper essentially changes the corrosion tendency of copper in the oil. This paper studies the influence of different contact materials and the layer of insulating paper on the corrosion degree of copper, and the absorption of DBDS and Cu²⁺ in the oil by insulating paper. Moreover, the influence of the Cu₂S deposition and the insulating paper on the corrosion tendency and the corrosion current of copper are investigated to further explore the mechanism of copper corrosion. This work is a complement to the current study of sulphur corrosion mechanism in oil-immersed power transformers.

2 | EXPERIMENT

2.1 | Material preparation

The copper windings were purchased from the transformer factory Fuling, in Chongqing, China. The copper conductor was 12 × 3 mm wrapped by 12 layers of 0.08 mm insulating paper and was cut into segments of 70 mm in length. As shown in Table 1, four types of samples with different contact materials were prepared to investigate the influence of contact materials on the sulphur corrosion of copper conductors.

Sample S1 was prepared to simulate the corrosion of the paper-wrapped copper conductors. To investigate the influence of different layers of insulating paper on corrosion, samples S1-1, S1-2, S1-3, and S1-4 with 2, 4, 8, and 12 layers of insulating paper, respectively, were prepared. Sample S2 consisted of four pieces of laminated copper conductors of 70 × 12 × 3 mm and was to simulate the direct contact of copper conductors of the outlet terminal; the conductors were piled up and surrounded by insulating paper so as to maintain physical contact with each other. Sample S3 consisted of a bare copper conductor set on a glass sheet of 72 × 25 × 1 mm and was used to simulate the condition that occurred in a laboratory experiment. For example, when performing the corrosion tests of ASTM D1275 A, a contact is formed between bare copper and the bottom of glass container. Moreover, a bare copper conductor was set as a blank sample labelled S0.
Fresh KI-25X naphthenic transformer oil and the samples listed in Table 1 were first put in a vacuum oven at 90°C/50 Pa for 24 h. Next, the samples were immersed in the oil and kept in the vacuum oven at 60°C/50 Pa for another 48 h to reduce the moisture and gas in the oil. Corrosive oil was prepared by dissolving a specific amount of DBDS in the insulating oil.

### 2.2 Experimental procedure

To investigate the influence of different contact materials and the insulating paper layer on sulphur corrosion of copper, an accelerated corrosion experiment was performed in sealed glass containers under the conditions listed in Table 2. After the experiment, all samples were retrieved with tweezers, washed carefully with n-hexane to remove residual oil, and air-dried for 60 s for characterisation.

To investigate the adsorption of the insulating paper on the DBDS and Cu\(^{2+}\) content in the oil, a thermal ageing experiment was performed in sealed glass containers under the conditions listed in Table 3. After the experiment, the DBDS content in the oil of E1, E2, and E3 were tested, as well as the Cu\(^{2+}\) content in the oil of E4 and E5.

Furthermore, to verify the influence of Cu\(_2\)S deposition and the insulating paper on the corrosion tendency and corrosion current of copper, the potentiodynamic polarization curves, the corrosion potential and corrosion current of the copper strips of different corrosion degrees and those wrapped with different layers of insulating paper were tested.

#### 2.3 Characterisation

The corrosiveness of the insulating oil was graded from level 1 to level 4 by the discolouration of the copper conductors according to the ASTM D130/TP 154 method. Levels 1a and 1b represent slight tarnish, 2a, 2b, 2c, 2d, and 2e represent moderate tarnish, 3a and 3b represent dark tarnish, while 4a, 4b, and 4c represent corrosion. The micromorphology and element content on the surface of the copper conductors were tested by the scanning electron microscope and the energy dispersive X-ray spectroscopy (SEM/EDS). The components of depositions on the surface of the copper conductors wrapped in insulating paper were measured by X-ray fluorescence spectroscopy (XRF). The DBDS content in the oil was tested using the Agilent 7890A Gas Chromatography instrument and Electron Capture Detector (ECD) according to IEC 62697-1. The CH1660E C1631 electrochemical workstation was used to test the potentiodynamic polarization curve of the copper conductors. As is shown in Figure 2, a laminated three-electrode device was used for copper strips of varying corrosion degrees, and the electrode area was 1.13 sq.cm. An immersed three-electrode device was used for the paper-wrapped copper strips, and the electrode area is 4.4 sq.cm. For all tests, saturated calomel was the reference electrode, a platinum electrode was the counter electrode, and the electrolyte solution was 0.1 mol/L Na\(_2\)SO\(_4\).

### RESULTS AND DISCUSSION

#### 3.1 Influence of contact material on sulphur corrosion of copper

Table 4 shows the surface morphology and corrosion degree of the copper conductors with different contact materials. No discolouration nor any trace of corrosion was observed on the S0 surface after the accelerated corrosion experiment, and the degree of corrosion of the copper conductor was 1a (non-corroded). However, obvious traces of corrosion were verified on the surfaces of S1-1, S2, and S3, graded from 2d (moderate tarnish) to 3b (dark tarnish). The phenomena allow one to infer that the contact of other materials is a considerable factor in the increase of the sulphur corrosion of copper conductors.

Figure 3 shows the micromorphology magnified 3000 times and the surface components of the copper strips after the accelerated corrosion experiment. No obvious corrosion or sulphur element was detected on S0 surface, meaning that there was no direct reaction between DBDS and copper. Depositions were found on the copper conductor of S1 and 3 wt% sulphur content was recognized, indicating the occurrence of sulphur corrosion. The copper conductor of S2 was deposited with relatively dense substance and 12.5 wt% S
content was found on the copper surface, indicating the occurrence of a severe sulphur corrosion. On the copper conductor of S3, dense depositions and several agglomerates were observed, and the sulphur content was as high as 14.7 wt%.

The sulphur content represents the amount of Cu₂S deposition, and one can infer that the corrosion rate of the copper conductors is closely related to the contact materials based on the test result.
3.2 Influence of insulating paper on sulphur corrosion of copper

Table 5 shows the morphology of the copper conductors and the innermost insulating paper from S1-1 to S1-4. For the original winding, the surface of copper was fresh and there was no deposition on the insulating paper. As for the windings after the corrosion experiment, the corrosion degrees of the copper conductors rose from 3a to 4c with the increase of the insulating paper from 2 to 12 layers. Meanwhile, more depositions were observed on the surface of the innermost insulating paper, which means that extra Cu²⁺ formed and migrated to the insulating paper.

The relative sulphur content on the surface of the copper conductors was measured by XRF and the result is shown in Figure 4. No sulphur was detected on the original copper conductor, while the sulphur content rose with the increase of the layers of insulating paper wrapped around the copper conductors from 0.0192 wt% to 0.1987 wt%. The sulphur content of S1-4 was approximately 10 times that of S1-1, indicating that increasing the layers of insulating paper significantly augments the sulphur corrosion of copper conductors.

DBDS and Cu²⁺ content play a key role in the formation of Cu₂S. Figure 5 substantiates the DBDS content in the insulating oil. No DBDS was detected in the oil of E3, indicating that neither the fresh oil nor the insulating paper contains DBDS itself. Both the DBDS content of E1 and E2 show a declining trend during the accelerated corrosion process. The DBDS content of E1 decreased from 498 mg/kg to 480 mg/kg at an average rate of 0.03 ppm/h due to the mass loss of DBDS at 130°C. In contrast, the DBDS content of E2 decreased by 76 mg/kg at an average rate of 0.127 ppm/h, which should be a result of both thermal mass loss and adsorption by insulating paper. On deducting a loss of 18 mg/kg due to the thermal mass loss, one can infer that the adsorption rate of DBDS by the insulating paper was approximately 0.0031 mg/kg per gram of the insulating paper per hour. Hence, the DBDS content in the oil near copper conductors was relatively high due to the adsorption by the insulating paper, thus leading to an acceleration of sulphur corrosion [5,20]. Moreover, the adsorption of DBDS rose with increased layers of insulating paper wrapped around the copper conductors, which results in more serious corrosion.

The insulating paper also acted as a barrier against the diffusion of Cu²⁺ generated by the copper conductors into the insulating oil. As shown in Figure 6, the Cu²⁺ content in the insulating oil increased continuously both in E4 and E5. Compared with E4, the copper conductor in E5 should have released more Cu²⁺ due to the catalysis of acids generated from the degradation of the insulating paper. However, the

| Sample | Copper conductor | Copper conductor | Surface of the innermost paper | Corrosion degree of copper |
|--------|-----------------|-----------------|--------------------------------|---------------------------|
| Origin | ![Image](origin.jpg)  | ![Image](origin.jpg)  | ![Image](origin.jpg)  | /                         |
| S1-1   | ![Image](s1-1.jpg)  | ![Image](s1-1.jpg)  | ![Image](s1-1.jpg)  | 3a                        |
| S1-2   | ![Image](s1-2.jpg)  | ![Image](s1-2.jpg)  | ![Image](s1-2.jpg)  | 4a                        |
| S1-3   | ![Image](s1-3.jpg)  | ![Image](s1-3.jpg)  | ![Image](s1-3.jpg)  | 4b                        |
| S1-4   | ![Image](s1-4.jpg)  | ![Image](s1-4.jpg)  | ![Image](s1-4.jpg)  | 4c                        |
result shows that the content of copper ions of E5 is only 2.8 mg/kg, while the Cu²⁺ content of E4 is approximately 17.3 mg/kg at the end of the accelerated corrosion process, almost 6.2 times of E5. It is reasonable to assume that insulating paper prevents the release of copper ions by copper conductors from diffusing into the insulating oil, thus creating a region with a relatively high concentration of Cu²⁺ in the gap between the copper conductors and the insulating paper.

3.3 Influence of the insulating paper and deposition on the corrosion tendency and the current of copper

The possibility of whether the existence of insulating paper changes the corrosion tendency and corrosion current of copper and accelerates the corrosion process was investigated by an electrochemical test and the result is shown in Figure 7. Little difference was found between the corrosion potential of all samples with different layers of insulating paper, which means that the corrosion tendency of copper hardly changes with increased layers of the insulating paper. However, there was a marked decrease in the corrosion current with increasing layers of paper, which indicates that insulating paper reduces the corrosion rate of copper.

Cu₂S deposition shows similar influence on the corrosion of copper conductors and the test result is shown in Figure 8. Little difference was found between the corrosion potential of all samples with different amounts of deposition, which means that corrosion deposition hardly influences the corrosion tendency of copper. However, there was a marked decrease in the corrosion current with increasing deposition on the surface of copper, which indicates that the insulating paper reduces the corrosion rate of copper.

4 MECHANISM ANALYSIS

4.1 Mechanism of gap-induced concentration cell corrosion

Current research has revealed that the content of corrosive sulphide, atmosphere and temperature are the dominant factors in sulphur corrosion. However, these studies do not explain the phenomenon that, after 240 h of accelerated corrosion experiment at 130°C, no obvious corrosion occurs on bare copper, while copper conductors in contact with paper, copper or glass all suffer various degrees of corrosion. Therefore, one can infer that the contact condition may be another key factor in the sulphur corrosion of copper conductors.

The effect of contact layer on the sulphur corrosion of copper conductors can be explained by the crevice corrosion theory, which refers to the phenomenon that when there is a tiny gap on the surface of a metal immersed in a solution, the corrosion rate in the gap is usually aggravated markedly [21]. The gap must be sufficiently wide to contain the solution and sufficiently narrow to ensure that the solution in the gap is in a stagnant state, making it difficult for material migrations [22].
In general, the gap ranges from 0.025 to 0.2 mm wide, and the gaps between copper and other contact materials meet the requirement. When immersed in corrosive oil, a tiny gap forms between the copper conductor and other contact materials inevitably, and a stagnant layer of corrosive oil is created within the gap. During the accelerated corrosion experiment, H\(^+\), Cu\(^{2+}\), and H\(_2\)O were generated by oil-paper degradation [23]. These reactive substances have difficulty diffusing out of the gap, while the dissolved O\(_2\) consumed in the gap cannot be replenished. This results in an increasing difference in substance concentration and pH value between the inside and outside of the gap. The substance concentration gradient leads to a potential difference between the covered and uncovered surfaces of a copper conductor, which influences the electrode dynamics and results in the formation of electrochemical cells. An oxidation reaction (i.e. corrosion) occurs at the anode and a reduction reaction occurs at the cathode of the concentration cell. Thus, the corrosion rate of the covered part of copper is accelerated, while the corrosion rate of the uncovered part is relatively slow.

The covered part of the conductor is the anode where copper loses electrons to form Cu\(^{2+}\), as in Equation (1), whereas the part outside the gap is the cathode where the reduction reaction occurs, as in Equation (2).

\[
\text{Anode: } \text{Cu} \rightarrow \text{Cu}^{2+} + 2e^- \quad (1)
\]

\[
\text{Cathode: } \text{O}_2 \rightarrow 4\text{OH}^- \quad (2)
\]

Meanwhile, as Cu\(^{2+}\) ions cluster around the anode, the possibility of a reaction between Cu\(^{2+}\) and DBDS increases, causing more serious corrosion. In addition, the H\(^+\) content in the gap increases due to the hydrolysis of Cu\(^{2+}\), conducing to a decrease of the pH value of insulating oil near the anode, which further facilitates the dissolution of copper as well as the sulphur corrosion in the gap. In addition to the reaction between Cu\(^{2+}\) and DBDS to form CuS, free ions inevitably accumulate in the gap, leading to a Cu\(^{2+}\) concentration gradient inside and outside the gap, thus forming a Cu\(^{2+}\) concentration cell and accelerating the corrosion by the edge of the gap.

Evidence shows the presence of some metal parts in the transformer other than copper windings suffering from sulphur corrosion due to the existence of a particular gap on the metal surface. Figure 9 shows the copper grounding wire at the top of the transformer, where the process of sulphur corrosion is accelerated by a relatively high temperature. The copper wire is enveloped in a black composite sleeve and corrosion is evident on the copper conductor. Massive depositions are observed and the sulphur content of the deposition is as high as 16.7 wt\%, which confirms the occurrence of sulphur corrosion. Figure 10 shows the copper wire of the grounding enclosure, which is used to make the enclosure and magnetic core equipotential. The copper wire is in direct contact with the aluminium foil and the insulating cardboard. There is evident discoloration and deposition on the surface of the copper wire. The sulphur content of the deposition is 1.4 wt\%, which is probably because of sulphur corrosion.

Parts of metals other than may also be vulnerable to sulphur corrosion. Figure 11 shows a magnetic core consisted of silicon steel sheets and aluminium sheets at the top of
the transformer. A tiny gap is formed between the silicon steel sheet and aluminium sheet, and depositions are observed on both. The sulphur content is 0.5 wt% on average on the surface of the silicon steel sheet, and the sulphur content of the granular substances reaches up to 5 wt% on silicon steel and aluminium, indicating the occurrence of sulphur corrosion.

The evidence above leads one to conclude that the corrosion caused by the contact layers occurs not only on copper conductors but also on other metal parts in oil-immersed power transformers.

4.2 Corrosion accelerated by insulating paper

Insulating paper has both negative and positive effects on the corrosion of copper. On one hand, the insulating paper restrained the corrosion current of copper by reducing the reaction active area, thus retarding the corrosion process. On the other hand, the existence of insulating paper contributed to the adsorption of DBDS in the oil and the prevention of Cu\(^{2+}\) from diffusion outside the winding, thus accelerating the corrosion process. Overall, the accelerating effect was considerably more evident than the inhibition effect, leading one to conclude that the corrosion of copper is promoted by the insulating paper.

The corrosion processes of the copper conductors wrapped in insulating paper and other materials are different. As is shown in Table 4, the corrosion area of S1 is relatively uniform, whereas the corrosion of S2 and S3 are limited to certain areas. Meanwhile, the copper of S1 suffers less corrosion than the other two. The different corrosion results of S1, S2, and S3 can be explained as follows. The insulating oil permeates the gap outside the insulating paper because of the capillary effect of cellulose. Accordingly, the stagnant region between the insulating paper and the copper conductor is relatively uniform, resulting in a large anode of the oxygen concentration cell. Conversely, the insulating oil cannot penetrate glass or copper, and corrosion occurs only on the surface of copper where the gap is of a particular size (i.e. 0.025 to 0.2 mm), thus ending up with a relatively small anode. The corrosion rate of a metal is proportional to the current density, and a small anode leads to a large current density. Consequently, the corrosion rates of S2 and S3 are much faster than S1. In addition, the corrosion products of S2 and S3 have difficulty migrating to the contact materials, thus leaving dense Cu\(_2\)S deposition on the surface of copper.

The corrosion process of copper conductors with insulating paper is explained in Figure 12. There is an enrichment of DBDS within the insulating paper due to the adsorption of cellulose, which facilitates the reversible reaction between DBDS and mercaptan (R-SH) as shown in Equation (3) and accelerates the process of sulphur corrosion. Meanwhile, owing to the impediment effect of insulating paper, a region with a high concentration of Cu\(^{2+}\) and small molecular acids generated by the degradation of the insulating paper such as methane acid and acetic acid [24] forms on the surface of copper conductors. One should note that small molecular acid plays a key role in sulphur corrosion. Those factors enhance considerably the effect of concentration cell, leaving more Cu\(_2\)S on the surface of the copper conductors and the insulating paper. Moreover, O\(_2\) is consumed in the conversion from mercaptan to DBDS, which further reduces the O\(_2\) content in the gap and increases the potential difference between the cathode and anode of the oxygen concentration cell. Similarly, the enrichment of acids and Cu\(^{2+}\) in the gap further increases the potential difference between the cathode and anode of the Cu\(^{2+}\) concentration cell.

DBDS in the insulating oil decomposes into mercaptan, and mercaptan reacts with dissolved Cu\(^{2+}\) under the condition of heat and acid to generate Cu\(_2\)S [25]. The process of sulphur corrosion is shown in Equations (3) and (4).

\[
R-S-S - R^\text{Heat} + H^+ \xrightarrow{O_2} 2R - SH
\]  (3)
where R is benzyl, R-S-S-R is DBDS, R-SH is mercaptan and R-CH₃ is ethylbenzene.

Some of the small molecular acids (H⁺) originating from the degradation of the insulating paper will diffuse into insulating oil gradually. However, most of the acids tend to accumulate within the insulating paper due to the strong adsorption by cellulose. In addition, the acids generated by the inner layers of the insulating paper are blocked by the outer layers when diffusing outwards, leading to a relatively high concentration of H⁺ on the surface of copper conductors. The study by [26] showed that, when oil immersed paper-wrapped copper windings have undergone a thermal ageing process, the acid values of the innermost and outermost insulating paper are 5 mg KOH/g and 3.5 mg KOH/g, respectively, whereas the maximum acid value of the insulating oil is only 0.11 mg KOH/g, which confirms the adsorption of acids by insulating paper. The accumulation of acids in the gap accelerates Equations (3) and (4). These factors contribute remarkably to accelerated sulphur corrosion.

5 | CONCLUSION

This study investigated the influence of contact conditions on sulphur corrosion of copper conductors and identified the probable mechanism of corrosion. The main conclusions are as follows:

1. The existence of a gap of a particular size between the surface of copper conductors and the contact material is one of the key factors leading to sulphur corrosion. Copper-copper, copper-paper, and copper-glass interfaces lead to gap-induced corrosion caused by an oxygen concentration cell and a Cu²⁺ concentration cell.
2. Insulating paper forms not only a significant gap on the surface of copper conductors, but also a region with a high concentration of DBDS, Cu²⁺, and H⁺ because of the adsorption and barrier effect, thus accelerating the corrosion process without changing the tendency of copper corrosion.
3. Sulphur corrosion caused by contact layers occurs on other metal parts other than copper conductors in oil-immersed power transformers, and its mechanism requires further study.

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