Effects of oxidation layer and roughness on the fretting wear behavior of copper under electrical contact

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Abstract

Bulk pure copper samples with varying roughness were prepared, heated and oxidized at different temperatures in air by using an ordinary box-type heat treatment sintering furnace to form different oxide layers. Electrical contact fretting wear tests were conducted on pure and treated copper in contact with a brass ball to investigate the effects of surface roughness and oxidation layers on electrical contact fretting wear properties. Test results showed that the roughness of the samples increased after oxidation and became prominent after oxidation at 400 °C. The friction coefficient and contact resistance of the samples oxidized at 200 °C were lower than those of the samples oxidized at room temperature and 400 °C. The adhesion and transfer in the wear process decreased with the increase in oxidation temperature, whereas the oxidation of copper increased with the increase in temperature. A competitive relationship was observed between adhesion and oxidation, and this relation increased the electrical contact resistance. A low oxidation treatment temperature corresponded to serious adhesion. A high the oxidation treatment temperature resulted in intensified oxidation and reduced adhesion. Adhesion accumulation and oxidation increase the contact resistance of the samples. The samples oxidized at 200 °C, 400 °C and RT had lowest, moderate, and worst contact resistance, respectively.

1. Introduction

Electrical contact is a state produced by live contact between two conductive components, and its reliability is indispensable for all power electronic circuits. However, in practice, people pay attention to electrical contact only when its failure leads to serious or catastrophic accidents. From a microscopic perspective, any machined surface is rough and uneven. Only a few protruding points or small surfaces between two conductors are in contact with one another. These contact points or small cross-sections are effective, when the current flows from one conductor to another. They facilitate current contraction and generate contraction resistance. In addition, the oxide layer or other contaminants generate thin film resistance on the surface of actual conductor [1, 2]. Contraction and film resistance constitute contact resistance [3]. Under stress disturbance, such as external mechanical vibration and high- and low-temperature environments, a small relative motion called fretting occurs between electrical contact points, fretting leads to fretting wear, fretting corrosion and fretting fatigue between metal contacts [4, 5]. Fretting wear may produce a large amount of debris, which accumulates on the electrical contact surface. Accumulation of debris reduces the number of effective contact spots, and gradually increases the resistance between contacts. An increase in contact resistance leads to an increment in the temperature rise of the contact surface, thus accelerating the oxidation of the metal contact surface. Most oxidation products are semiconductors or insulators. Their contact resistance increases intermittently from milliohms to several or tens
of ohms, and this increase eventually triggers intermittent non-recurrence failure [6], such as circuit opening, circuit breaking and interruption.

The physical and chemical processes in electrical contacts are extremely complex because of the different types and sizes of loads, contact forms, environmental conditions and materials. The factors that affect fretting wear behavior in electrical contact systems are as follows: (a) electrical contact materials, (b) test conditions (current, displacement amplitude, load, number of cycles, etc), (c) test environment (temperature, humidity and atmospheric environment). The properties of electrical contact materials directly affect contact resistance under certain contact and environment conditions. The basic requirements of electrical contact materials are: good thermal conductivity, low contact resistance and temperature rise, anti-fusion welding and anti-environmental media pollution [7]. Noble metals, such as gold and silver, are widely used as electrical contact materials because of their excellent electrical conductivity, oxidation resistance and ductility. However, their application in the industrial field is limited because of their high costs. Robert [8] found that Gr electrical contact performs well in terms of electrical and thermal performance and suitable for use in future electrical components, however Gr is expensive and difficult to produce. Copper is second only to silver in terms of conductivity and is widely used as an electrical contact material because of its low cost. However, copper easily oxidizes at room temperature (RT). The oxide film of copper is one of the main reasons for delamination and cracks in reflow soldering of plastic packaging. Copper increases contact resistance when oxidized at high temperatures, and such an increment in contact resistance decreases the working efficiency of components and even leads to component failure [9]. The fretting wear characteristics of copper-based contact materials were comprehensively investigated by Park [10, 11]. Romanov [12] used a CuO-Ag system to create contacts of switches for high-power electrical networks and found that the fretting wear characteristics of the contact materials are affected mainly by fretting amplitude, fretting frequency, positive pressure, temperature, humidity and atmospheric environment. The complexity and particularity of application environments make electrical contact wear problems have strong system dependence, non-portability and time-varying process characteristics. The wear failure mechanisms are also different. The influence mechanism of load ($F_0$), displacement amplitude ($D$), hardness, coating thickness, current ($I$) and coefficient of friction (COF) indicates that a small displacement amplitude corresponds to a low failure probability [13]. Fretting amplitude is one of the main factors that determine the fretting failure of copper-plated gold contacts. The amplitude of complete slip of contacts can make the gold plating layer fall off. Therefore, this factor is the precondition for intermittent contact resistance [14]. The electrical conductivity of contacts under large loads is better than that under small loads. Debris generation is proportional to displacement amplitude when the load is constant. The contact resistance at high currents is lower than that at low currents when the load magnitude and relative displacement are constant [15]. The temperature rise in the contact area can accelerate the frictional oxidation of the contact interface. The contact resistance in anaerobic environments is lower than that in oxygen-rich environments. In a certain period, high relative humidity can reduce the contact resistance [16, 17]. The change in interface and surface products (debris, corrosive substances, delamination, etc) in the contact area are the main factors that affect the fluctuation and increase in contact resistance [15].

Friction, wear and contact resistance are known to be system properties rather than material properties which means that they result from the action of two surfaces in relative tribofilms formed on sliding surfaces [18]. Contact resistance partly consists of a constriction and film resistance. Tribofilms can influence contact resistance, although such films are commonly thought regarded as a thin oxide or moisture films. Surface roughness affects shrinkage resistance. Therefore, surface roughness and oxides directly influence contact resistance. Contact resistance is the most important and common feature of all electrical contacts. Although contact resistance is much smaller than circuit resistance, a change in it leads to major equipment failure. The total resistance caused by contraction and film resistance is called contact resistance. Surface morphology affects all contact characteristics and mechanical properties. Another important factor that influences contact performance is the various films or fretting wear products, such as oxides, pollutants, reaction products, debris, generated on the surface. Liu et al [19] investigated the effects of surface roughness on the fretting properties of pure copper. Surfaces with high roughness are likely to produce abrasive debris, which is difficult to discharge on the contact surface and leads to an increase in contact resistance. However, the mechanism and mode of the effect of copper oxide on electrical contact properties have not been reported. This study evaluated the effects of different roughness values on the electrical contact properties of pure copper after oxidation at different temperatures in air. The influence of roughness and the oxide layer on electrical contact fretting properties of copper were revealed.
2. Experimental and materials

2.1. Materials and equipment
Pure copper, a regular electrical contact material (wt%; Cu99.9, P0.0116, Fe0.016, Pb0.0019, S0.0047, Zn0.0216, Sn0.003, and other allowances) was selected as the experimental specimen. Brass balls (wt%; Cu60.5–63.5, Fe0.01, Pb0.08, P0.15, So0.005, Bi0.002, and Zn allowance) were used against materials. Their mechanical properties are shown in table 1.

Copper was manufactured into plate specimens with various roughness. First, the plate specimens were subjected to polishing by using various sand papers (60 cw, 120 cw, 400 cw, 800 cw, 1500 cw) and mirror polishing to obtain different surface roughness values. Second, the specimens were heated and oxidized in a sintering furnace box for heat treatment. The test samples were oxidized at different temperatures (RT, 200 °C, 400 °C) for 30 min to form different oxide layers on the surface of pure copper. A fretting wear test on electrical contact was conducted. A self-made acquisition system involving electrical contact fretting wear equipment was used to collect data on contact resistance and COF (figure 1). The experimental parameters used were as follows: \( f = 2 \text{ Hz}, \) sliding displacement \( D = 45 \mu\text{m}, \) \( F_n = 4 - 6 \text{ N}, J = 0.002 \text{ A}, \) cycles \( N = 10^4. \) After the test was completed, the surface roughness of worn scars were measured by with Nano Map500 DLS dual-mode profiler, 3D-profile is used to obtain the wear volume. An optical microscope (OM) (Zeiss AXIO Imager-M1) and a scanning electron microscope (SEM)(FEI-Quanta200) were used to analyze the morphology of the wear scars. Electron probe microanalysis (EPMA) and x-ray photoelectron spectroscopy (XPS) (Thermo Fisher ESCALAB 250Xi) were performed to measure the chemical composition of the wear scars.

2.2. Material preparation: polishing
The OM photographs and 3D morphologies of the samples polished with different sandpapers are shown in figure 2. The roughness values (Ra) were 3.09, 2.07, 0.53, 0.20, 0.09 \( \mu\text{m} \) after polishing with 60, 120, 400, 1500 CW sandpapers and mirror polishing, respectively.

| Material      | Size (mm\(^3\)) | Resistivity (10\(^{-8}\)Ω·m) | Density (g/cm\(^3\)) | Hardness Hv\(_{0.1}\) |
|---------------|-----------------|------------------------------|----------------------|----------------------|
| Pure copper   | 30 \times 10 \times 10 | 1.678                        | 8.96                 | 102                  |
| Brass ball    | \( \Phi 5 \) mm | 6.50                         | 8.50                 | 150                  |

Table 1. Properties of the tested materials.

Figure 1. Schematic of the electric contact fretting wear tester (a) Electrical contact resistance (ECR) fretting test system (1. Piezoelectric ceramic actuator 2. Upper fixture 3. Force transducer 4. Sample 5. Precision lead screw 6. Displacement sensor) (b) Operating principle of the equipment and resistance measurement mechanism (c) Resistance measurement principle.
2.3. Materials preparation: oxidation treatment

An oxide layer is formed on the surface when metals are heated, and this layer leads to change in surface structure. Figure 3 shows a comparison of the surface morphology of the polished samples after oxidation at different temperatures for 30 min. The surface of the samples oxidized at RT presented good smoothness. The surface of the samples oxidized at 200 °C had several protrusions, and the overall surface of these samples was not as smooth as that of the samples at RT. The surface of the samples oxidized at 400 °C was rough and uneven.

Figure 4 shows the change in the surface roughness of the six surface roughness samples after oxidation at RT, 200 °C and 400 °C. The surface roughness after oxidation at 200 °C was slightly higher than that after oxidation at RT. The surface roughness after oxidation at 400 °C increased considerably, indicating that the surface became rough and uneven, which may be related to the oxidations at different temperatures. The oxidation of pure copper is mainly caused by the chemical reaction between copper and oxygen, and the formation of copper oxides on its surface.

According to a relevant research [20], copper oxidizes when (∆G)\_\text{T,P} < 0, this means copper can spontaneous oxidation at RT. Oxidation is a spontaneous process. The Gibbs free energy of Cu\textsubscript{2}O is the small, and the positive trend of the corresponding oxidation reaction is large. The more spontaneous the reaction is, the easier to the reaction can proceed spontaneously. Cu\textsubscript{2}O is the most thermodynamically stable among all oxidation products, but when copper is oxidized in high temperatures in the atmosphere, the final stable oxidation is CuO [21].

The results of scanning electron microscopy (SEM) (figure 5) showed that the surfaces of the samples polished by sandpaper and oxidized at RT had different ravines (figure 5(a)). The surface of samples polished by sandpaper and oxidized at 200 °C still had traces of sandpaper polishing, but holes of different sizes were
observed in the surface layer (Figure 5(b)). After oxidation at 400 °C, the surface layer showed folds and a large area of delamination (Figure 5(c)). Many micro apertures were found and the binding degree of the surface and subsurface was poor. As shown in Figure 5(d), the change in the surface morphology of copper after oxidation was mainly due to the change in the structure and the formation of oxides at different oxidation temperatures.
The energy dispersive spectroscopy (EDX-EDAX-7760/68 ME) results showed a small amount of oxygen and a large amount of copper on the surface after oxidation at 200 °C. The percentage of oxygen and copper atoms on the surface after oxidation at 400 °C was the same, and the proportion of oxygen and copper atoms in the oxidized sub-layer was very low when the oxide layer was peeled off. The extremely low case indicated that surface oxidation prevented internal oxidation, and the oxidation products are different at varying oxidation treatment temperatures.

3. Results and discussion

3.1. Electrical contact response

The contact resistance of different materials was investigated to determine the electrical contact response characteristics of materials oxidized at different temperatures. When the surface roughness was 3.09 μm (figure 6(a)), the contact resistance of the samples at the three oxidation treatment temperatures began to increase after 7000 cycles. The contact resistance of the RT samples increased considerably and reached 1400 mΩ. The contact resistance of the samples oxidized at 400 °C and 200 °C reached 1200 and 250 mΩ, respectively. When the surface roughness was 0.53 μm (figure 6(b)), the maximum resistance of the sample oxidized at 400 °C was approximately 1300 mΩ, and the maximum resistance of the samples oxidized at 200 °C and RT was 1000 mΩ. When the surface roughness was 0.20 μm (figure 6(c)), the contact resistance of the sample at RT increased sharply after 5000 cycles, and nearly reached 1400 mΩ at 6000 cycles. The contact resistance of the samples at 400 °C increased after 7000 cycles, and the maximum resistance was 1000 mΩ after 8500 cycles. The maximum contact resistance was reached after more than 9000 cycles in the samples oxidized at 200 °C and the maximum value was approximately 200 mΩ. When the surface of the sample was relatively smooth (Ra = 0.09 μm) (figure 6(d)), the contact resistance of the samples at RT gradually increased after 7500 cycles and reached the maximum contact resistance of 1400 mΩ after 8000 cycles. The contact resistance of the
samples at 400 °C increased after 9000 cycles and reached the peak of 900 mΩ. On the contrary, the contact resistance of the samples at 200 °C was low throughout the cycle. As shown in figure 6, under the four roughness values, the ECR of the samples oxidized at 200 °C was the lowest, and the ECR of the samples oxidized at 400 °C was the moderate. The ECR of the samples at RT was the highest, which had no direct relationship with the surface roughness of the samples. The electro-contact property of samples at different oxidation treatment temperature may be related to the different oxides formed on the contact surface.

Three important indicators, namely, life time, failure time and failure rate, are commonly used in the analysis of electrical contact reliability. Several criteria can be utilized to determine the lifetime of electrical contacts. The number of cycles of contact resistance was used as a lifetime indicator in previous studies because it has the strongest impact on the behavior of electrical contacts. The number of cycles that leads to a 300% increase in contact resistance is defined as lifetime [22]. Several researchers also used 100 and 300 mΩ as the lifetime limit [10, 22–25]. Ren et al used 100 mΩ as the critical threshold of contact resistance to investigate the effects of fretting frequency and fretting amplitude on the intermittent failure of electric contacts [26]. A contact resistance of 100 mΩ was set for finishing degradation in this study. As shown in figure 7, the service life of the samples oxidized at 200 °C was the longest under different surface roughness values, followed by that of the samples oxidized at 400 °C and RT (shortest). When Ra = 3.09 μm, the service life of the samples oxidized at RT, 200 °C and 400 °C was 6400, 8100 and 7300 respectively; when Ra = 0.53 μm, the respective lifetime values at the three temperatures were 6500, 5800 and 8200. The lifetime increased to different degrees at the three temperatures when the surface roughness decreased to 0.20 and 0.09 μm. Generally, the failure life of the entire electrical contact improved with the decrease of surface roughness.

3.2. Friction and wear behavior
The relationship between COF and the number of cycles at different temperatures is shown in figure 8. The temperature had little effect on COF when Ra = 3.09 μm (figure 8(a)). COF rapidly increased with the increase in the number of cycles at RT and entered a stable state when the surface became smooth (Ra = 0.53 μm) (figure 8(b)). The COF values at RT and 400 °C were significantly higher than that at 200 °C. The smooth surface (Ra = 0.20 and 0.09 μm (figures 8(c), (d)) showed a similar pattern. In the re-run phase, the COF value increased sharply at RT and became higher than that at 400 °C and 200 °C after entering the stabilization phase. Under the four roughness conditions, the COF of the samples oxidized at 200 °C was the lowest, followed by that of the samples oxidized at 400 °C and RT (the highest). COF changed with temperature after oxidation, which is consistent with the trend of ECR The average of the sum of instantaneous friction coefficients (after entering the steady state) was used as the average friction coefficient. The variation of the average friction coefficient is consistent with that of the instantaneous friction coefficient (figure 8(e)). Setting an appropriate oxidation treatment temperature helped to reduce the surface friction coefficient and improve the electrical contact performance.

Figure 9(a) shows the surface wear 3D morphology of the samples at Ra = 0.20 μm after oxidation at different temperatures. The adhesion of the abrasive chips on the surface of the samples oxidized at RT and 200 °C was serious, and the stacking was high. Meanwhile the surface of the samples oxidized at 400 °C had less

![Figure 7: Electrical contact life under different surface roughness values. (Fn = 6 N, D = 45 μm).](image-url)
accumulation, which is consistent with the surface roughness test results. Figure 9(b) shows the surface wear profiles of the samples at \( Ra = 0.09 \) \( \mu m \) after oxidation at different temperatures. The adhesion of the grinding chips on the surface of the samples oxidized at RT and 200 °C was serious and exhibited certain accumulation, whereas the samples oxidized at 400 °C showed deep wear pit. Surface roughness could reflect serious wear and tear to a certain extent.

Figure 8. Friction coefficient and average friction coefficient under different surface roughness. \( (F_n = 6 N, D = 45 \mu m) \).
3.3. Wear mechanism

Three main wear mechanisms, namely: oxidation wear, adhesion wear and delamination, are present in the fretting wear test of copper.

Figures 10 (a), (c), (e) shows the SEM and EPMA results of the samples wear area after oxidation at three temperatures. As shown in the EPMA results in the figure 10(a), the O element in the wear area significantly increased compared with that in the unworn area, and the Cu element was lost, indicating that the wear area experienced obvious oxidation. Zn significantly increased, indicating serious transfer from the grinding ball. The EPMA in figure 10(c) had a similar pattern as that of in figure 10(a), except that the transferred Zn was smaller than that at RT. As shown in the EPMA in figure 10(e), Cu loss and Zn transfer were relatively less obvious, and the three SEM images showed that the wear zone in the samples oxidized at 400 °C was larger than that in the samples oxidized at the two other temperatures.

The 3D morphology revealed considerable, accumulation and adhesion on the wear area surface of the samples oxidized at RT and 200 °C. The EPMA test results in figures 10(a), (c), (e) indicate that a large amount of accumulated Zn was transferred from the grinding ball brass. The lower the oxidation treatment temperature was, the more serious the sample adhesion and accumulation were.

Figures 10(b), (d), (f) SEM shows that the wear zone had different degrees of delamination, crushing and oxidation of debris, which may be related to the oxide formed on the surface. The oxide layer on the surface of copper consists of CuO with fine grains on the outside and Cu₂O on the inside. A large grain size equates to a
loose structure. In figure 10(b), the thin oxide layer on the surface is visible. A thick crushing layer can be observed in figure 10(d). A relatively large and loose abrasive flaking layer can be seen in figure 10(f). Uneven folds were observed on the surface of the oxide layer from 300 °C to 550 °C, the folds increased with the increase in oxidation time and temperature [27].

According to the measurement results of loss volume in the wear scar area after wear (figure 11), the wear loss volume of the samples oxidized at RT was the smallest when the surface roughness was larger (Ra was no more than 0.53 μm), followed by the samples oxidized at 200 °C and 400 °C. When the surface roughness was small (Ra was less than or equal to 0.20 μm), the wear volume of the samples oxidized at RT was the largest, whereas that at 400 °C was the smallest. This result shows that the change in wear volume was not linear with the increase in temperature because of the effect of surface roughness and temperature.

The electrical contact performance varied because of the different oxidation treatment temperature, the different oxidation products and content. As shown in figure 12(a), the chemical composition of the contact pair surface at RT, 200 °C, 400 °C was (Cu + Cu2O) + CuO, (Cu + Cu2O) + CuO, and CuO, respectively.
This result indicates that high temperature provided considerable CuO content, and the CuO layer covered the entire surface at 400 °C. Copper oxidized in air and formed Cu₂O, and CuO was formed with the increase in oxidation. A schematic of copper oxidation in air is shown in figure 12(b). The CuO content decreased after wear compared with the XPS composition before and after wear (figure 12(c)). This result may be due to the crushing of the surface CuO during wear, resulting in the discharge of debris after peeling, which exposed the Cu₂O on the sub-layer. The conductivity of the sub-layer was better than that of the surface layer, and the electrical contact performance of the material was improved.

4. Discussion

4.1. Contact resistance and roughness
Liu [15] investigated studied the effects of surface roughness on the fretting wear properties of pure copper under electrical contact without oxidation. After polishing, the contact resistance of the contact pair remained low, the contact resistance of the rough contact pair became highly unstable, and the electrical contact performance was poor. Smooth surfaces provide many contact points, and worn debris can be removed from the contact zone. Comparatively, rough surfaces lead to small contact area, and wear debris is difficult to
transfer. Semiconductor-like debris accumulated at the contact interface increased the contact resistance. The effects of oxidation on the fretting wear properties of pure copper with different surface roughness was investigated in this study. The surface roughness of samples changed greatly after oxidation at different temperatures. As shown in figure 13 (average contact resistance [28, 29] under different surface roughness during 10,000 cycles), the electrical contact performance of the samples was not related to roughness. The oxide layer

Figure 13. Relationship between average contact resistance and surface roughness.

(a) Average (b) Maximum instantaneous resistance

(c) Relationship between wear mechanism and contact resistance

Figure 14. Relationship between contact resistance and oxidation treatment temperature.
surface was removed during the wear process, and the adhesion phenomenon was not obvious because of the combined effect of large surface roughness and oxide layer. With the progress of wear, surface roughness decreased, and the surface became smooth. Adhesion and transfer mainly occur when the surface roughness was low. Surface roughness increased after wear, and the electrical contact performance had no linear relationship with the surface roughness.

4.2. Contact resistance and oxide temperature
The average and maximum contact resistance at different temperatures are shown in figure 14(a) (between $6 \times 10^2$–$10^4$ cycles, instantaneous contact resistance climb period) and figure 14(b). The contact resistance of the samples under the four roughness conditions initially decreased and increased. Three wear mechanisms, namely, oxidation, adhesion and delamination, were found in the wear process of copper. Oxidation and adhesion wear could increase the contact resistance. The oxidation products were semiconductors or insulators with high resistivity. The adhesion and exfoliation delamination of abrasive debris increased surface roughness and reduced the effective contact spot of contact pairs, thus causing contact resistance to increase or even the circuit to break. Therefore, oxidation and adhesion were a pair of competitive mechanisms. With the increase in treatment temperature, the effects of oxidation increased and those of adhesion decreased. At 200 °C, the contact resistance was small value, resulting in the coupling of the two factors (figure 14(c)).

Romanov [12] investigated the Structure and electrical erosion resistance of CuO–Ag coating, the coating had good electrical conductivity and wear resistance, but the method is complicated and difficult to control. Grandin [30] found that the mating couple with the highest amount of oxide in the tribofilm also had the lowest contact resistance. Hence, it is concluded that oxides are not necessarily detrimental for the contact resistance as long as there is unoxidized copper available.

This study provided a simple, low-cost, portable method to improve the electrical contact performance of pure copper. The method provides a theoretical basis for the application of copper materials in electrical contact.

5. Conclusion
(a) Elevated treatment temperature caused an increase in surface roughness. After oxidation at RT and 200 °C, surface roughness increased slightly, and the change was not obvious. Surface roughness generally increased after oxidation at 400 °C.

(b) Under various roughness, the COF of the samples oxidized at 200 °C was the lowest, the samples oxidized at 400 °C had a median value, and the samples oxidized at RT had the highest COF.

(c) The samples oxidized at 200 °C had low and stable ECR Two factors, namely, effective contact area and surface film resistance, affected contact resistance. The effective contact area and contact resistance decreased when the adhesion phenomenon on the contact surface was serious. The lower the oxidation treatment temperature was, the more serious the adhesion phenomenon was. The resistance of the surface film was mainly composed of the resistance of surface oxide.

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