



TECHNICAL ARTICLE

# Tribological Properties and Electrical Conductivity of Carbon Nanotube-Reinforced Copper Matrix Composites

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Submitted: 5 August 2021 / Revised: 20 October 2021 / Accepted: 5 January 2022 / Published online: 3 March 2022

**Carbon nanotube-reinforced copper matrix (CNT/Cu) composites were prepared by a method involving solution-aging treatment, in situ chemical vapor deposition (CVD), and spark plasma sintering (SPS). The tribological properties of the CNT/Cu composites were greatly improved, and the friction stability was better than that of Cu. These performance improvements were attributed to the uniform distribution of the CNTs without agglomeration and the strong interfacial bonding between the Cu matrix and CNTs. The coefficient of friction was 0.2, which was lower than that of pure copper (0.55), and the wear rate of the CNT/Cu composites was 2 times lower than that of pure copper. The wear mechanism was also discussed from the perspective of the wear morphology and interface structure. Furthermore, the electrical conductivity remained at a high level. The preparation technique is simple and easy to be controlled and can be used to synthesize CNT/Cu composites with excellent tribological properties and electrical conductivity.**

**Keywords** carbon nanotubes, chemical vapor deposition, copper matrix composites, solution-aging treatment, spark plasma sintering, tribological properties

## 1. Introduction

Copper-based composites play a crucial role in railway, military vehicles, electrical components, electrical contact materials, special conductors and other fields due to their outstanding electrical and thermal conductivity and good wear resistance (Ref 1-7). However, with the rapid development of the railway and automobile industries, there is a high demand for new materials with improved tribological properties that maintain their good electrical conductivity. Examples of such materials include pantograph sliders for high-speed trains, electric brushes, and other sliding electrical contact materials. CNTs are an ideal enhancement phase for improving the properties of copper alloys due to their excellent comprehensive properties, such as low density, high strength, and high Young's modulus (Ref 8-11). CNT-reinforced metal matrix composites are self-lubricating materials due to their outstanding friction properties (Ref 12). Thus, they can improve the wear resistance and reduce the coefficient of friction, while also eliminating the need for external lubricants to avoid jamming, which makes them potential structural materials in the aerospace and automotive fields (Ref

13-15). The tribological properties of CNT/Cu composites have been investigated in recent years (Ref 16-19). For example, Dong et al. (Ref 20) prepared a Cu-matrix composite reinforced by CNTs using powder metallurgy, which displayed a lower coefficient of friction and reduced weight loss. Kim et al. (Ref 21) prepared Cu/CNT composites by a molecular-level technique and SPS, and showed that the friction properties of the composites were three times higher than that of pure copper. Xu et al. (Ref 22) mixed copper powders and CNTs by ball milling and prepared CNT-Cu composites by powder metallurgy. The wear mechanism of the CNT-Cu composites was mainly adhesive wear and plastic flow deformation. However, due to the randomness of the ball milling process (Ref 23), it was difficult to obtain a homogeneous dispersion while maintaining the integrity of the CNTs, which greatly weakened the self-lubrication property of the CNTs. In addition, some impurities may be introduced during ball milling. Guiderdoni et al. (Ref 24) prepared CNT/Cu composites by SPS, which had a hardness that was 1.5 times that of pure copper, and a coefficient of friction and wear rate that were, respectively, 3-4 times and 10-20 times lower than that of pure copper. Huang et al. (Ref 25) prepared aligned CNT-reinforced copper-based composites by electroless plating, ultrasonic mechanical stirring, sintering, forging and die drawing. The coefficient of friction and wear rate were decreased by 68 and 90.2% compared with pure copper, respectively.

Although the composites prepared by the above processes showed improved friction properties, the CNTs required acidification, cleaning, drying, and other relatively complicated processes that made it difficult to ensure a repeatable preparation process. In addition, surface modification can damage CNTs, causing them easy to agglomerate due to strong van der Waals force among them. Therefore, it is difficult to ensure good dispersion of CNTs in the copper matrix and strong interfacial bonding between Cu matrix and CNTs, which prevents the full utilization of the excellent properties of CNTs (Ref 26). Hence, there is a need for a method to prepare CNTs

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with complete structure, and ensure that they are uniformly dispersed in the Cu matrix. Moreover, the preparation technology should be simple, repeatable and easy to operate.

The in situ chemical vapor deposition (CVD) method can ensure the good growth of CNTs, and SPS has the advantages of fast heating rate, short sintering time and low sintering temperature (Ref 27-29), which greatly improve the production efficiency and save much energy. Therefore, CNT/Cu composites were prepared by a new process that involved solution-aging treatment, in situ CVD and SPS.

## 2. Experimental

CuCr (0.6 wt.% Cr,  $\leq 200$  mesh, Changsha Tianjiu metal material Co., Ltd, China) alloy powders were annealed at 550 °C for 30 min to remove residual oxides on the surface. Then, the CuCr alloy powders were solution-treated at 850 °C for 1 h and aging treated at 450 °C for 2 h under a mixed atmosphere with a flow rate of 1650 mL/min (Ar) and 1350 mL/min ( $H_2$ ) to uniformly disperse the Cr particles in the copper matrix. Cr was used to catalyze the growth of CNTs without reducing the electrical conductivity of the copper matrix due to its low solid solubility in Cu. Then, CNT/Cu composite powders were synthesized by in situ CVD at 800 °C for 60 min, with a  $C_2H_4 : Ar + H_2O : H_2$  gas ratio of 100: 1200: 2450 (mL/min). All the gases were provided by Shanghai Jiangnan Gas Co., Ltd. The heat treatment was performed in a tubular furnace with a diameter of 80 mm (OTF-1200X, Hefei Kejing material technology Co., Ltd, China). The size of the CNT/Cu composite samples was  $\varnothing 30$  mm  $\times$  3 mm (height). Five samples were studied for each test, and the average was reported. Finally, we prepared CNT/Cu composites by SPS (750 °C, 10 min, 45 MPa). The CNTs were uniformly distributed in the Cu matrix without agglomeration, and the graphite lattice diffused to the Cu lattice at the interface, so the interfacial bonding between Cu and CNTs was strong. Thus, the tribological properties of the CNT/Cu composites significantly improved compared with Cu under keeping the electrical conductivity at a high level. In addition, the volume fraction of CNTs in the CNT/Cu composites is 0.5%. Cu samples were used for comparison. The preparation process flow is shown in Fig. 1.

The morphologies of the CNT/Cu composites were observed by scanning electron microscope (SEM, Quanta FEG 450 FEI USA). Transmission electron microscope (TEM, Tecnai G2 F30 FEI USA) was utilized to characterize the CNT/Cu composite interfaces. The friction-wear properties were analyzed using a reciprocating friction-wear tester (HSR-2M, Lanzhou Zhongke Kaihua Technology development Co., Ltd China). Three-dimensional optical microscope (Contour GT-K Bruker USA) was used to investigate the morphologies and three-dimensional roughness of the wear scars. A digital metal conductivity tester (D60K, Xiamen Xinbot Technology Co., Ltd, China) was used to measure the electrical conductivity of Cu and CNT/Cu composites.

## 3. Results

### 3.1 Characterization of CNT/Cu Composite Powders

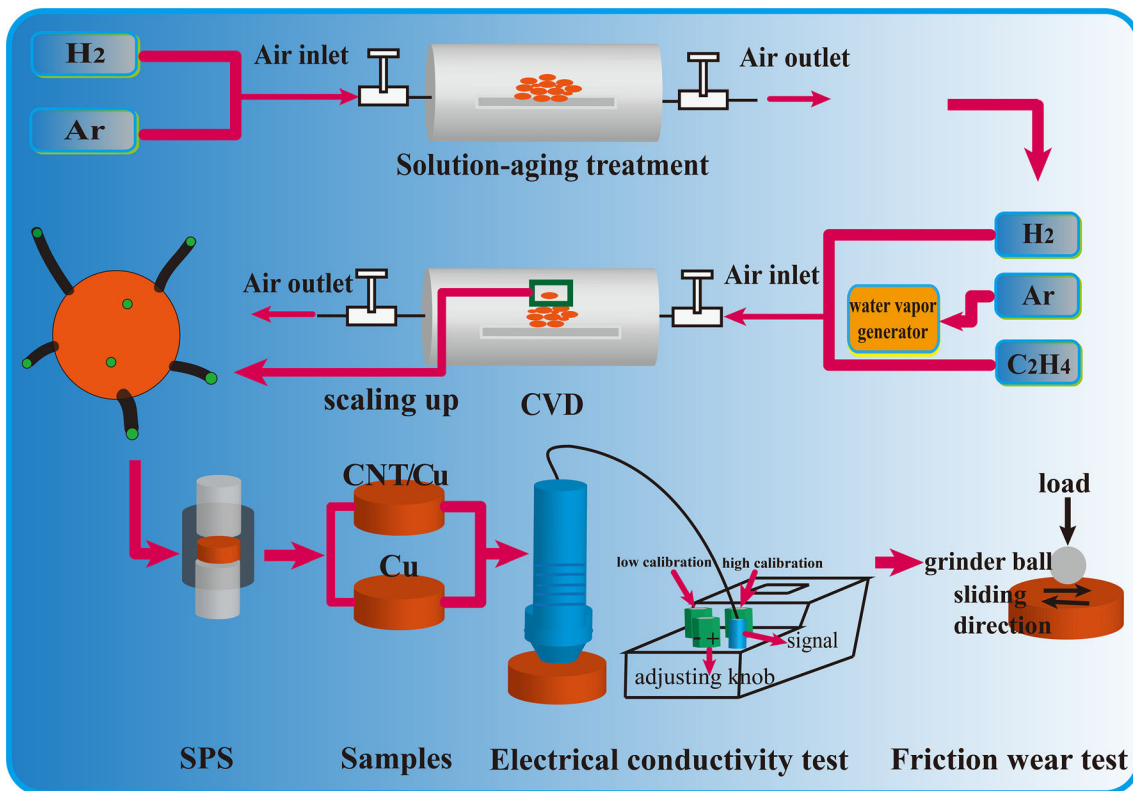
Figure 2(a) shows the SEM images of the CuCr alloy powders. The uniform distribution of Cr particles in the copper

matrix provided a foundation for the homogenous growth of CNTs. Figure 2(b), (c) and (d) illustrates the CNTs prepared by *in situ* CVD. The CNTs grew uniformly (Fig. 2b) and independently of each other without agglomeration (Fig. 2c). By reducing the CVD reaction time, the number of CNTs was reduced and their length was shortened. The blue circle in Fig. 2(d) shows that one end of the CNTs was completely embedded in the copper matrix. This can limit the CNT's movement and reduce their agglomeration, thereby ensuring their homogeneous dispersion and improving the wettability between Cu and CNTs. The TEM images of the CNTs in Fig. 2(e) and the inset ( $e_1$ ) show that the CNTs are hollow multi-walled tubes with uniform diameters of about 20.16 nm. Furthermore, the walls are smooth and clean with few defects. Figure 2(f) shows the Raman spectrum of the CNT/Cu composite powders. The  $I_D/I_G$  is about 0.909, which indicates that the degree of graphitization of CNTs was good, and the defect density was small. Thus, it can be concluded that high-quality CNT/Cu composite powders were prepared by this process, which laid the foundation for the preparation of CNT/Cu composites with good friction performance and electrical conductivity.

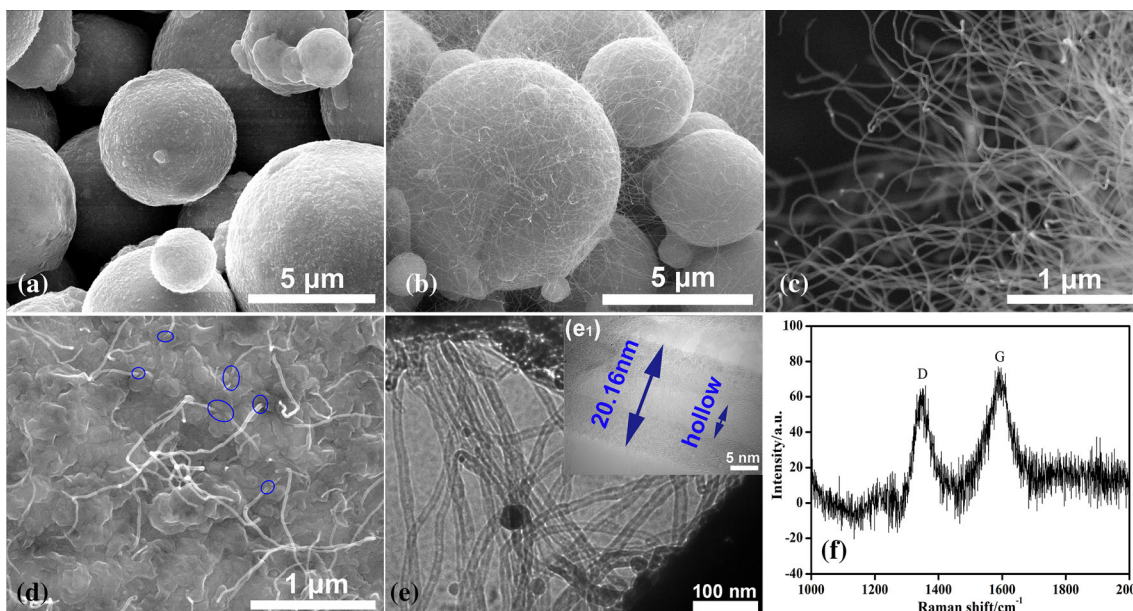
### 3.2 Electrical Conductivity and Friction Properties of CNT/Cu Composites

The CNTs with complete structure are important to the properties of CNT/Cu composite. Therefore, the SEM on the CNT structural intactness in the final bulk CNT/Cu composites are displayed in Fig. 3(a). It is shown in Fig. 3(a) that the CNTs in the bulk CNT/Cu composite had a complete structure without fracture or breakage. Figure 3(b) compares the friction coefficients of Cu and CNT/Cu composites and the loading time. Both friction coefficients greatly fluctuated during the initial stage of the test because of the polishing process during the wear test period, i.e., the surface of the wear track was smoothed by grinding away the uneven surface (Ref 30). We also found that the friction stability of the CNT/Cu composites was better than that of Cu, which is one of the important characteristics of friction materials (Ref 31, 32). After 30 s, the coefficient of friction of both the Cu and CNT/Cu composites is stabilized at 0.55 and 0.2, respectively. Due to the good self-lubricity of the CNTs (Ref 20), they can form a protective film on the surface of CNT/Cu composites. During friction and wear processes, the CNTs gradually bent and deformed until they were destroyed at longer friction times. The fragments formed by the broken CNTs accumulated and diffused between the wear surface and grinding ball to form a self-lubricating film that slowed damage to the CNT/Cu composites. The interfacial bonding between the CNTs and Cu matrix was very good, giving the CNT/Cu composites high densification, which prevented the interfacial scattering from affecting the electrical conductivity. Therefore, the CNT/Cu composites displayed an excellent electrical conductivity of 92.9% IACS, and the relative density and electrical conductivity as shown in Fig. 3(c). The Vickers hardness of the CNT/Cu composites (102 HV) was higher than that of Cu (82 HV) (Fig. 3c) (Ref 33). All of the above factors show that the friction properties of the CNT/Cu composites were better than pure copper, which can reduce the damage to CNT/Cu composites.

Figure 4(a) and (b) shows the SEM images of the friction-wear morphologies of Cu and CNT/Cu composites. It can be seen that the wear width of Cu and CNT/Cu composites was



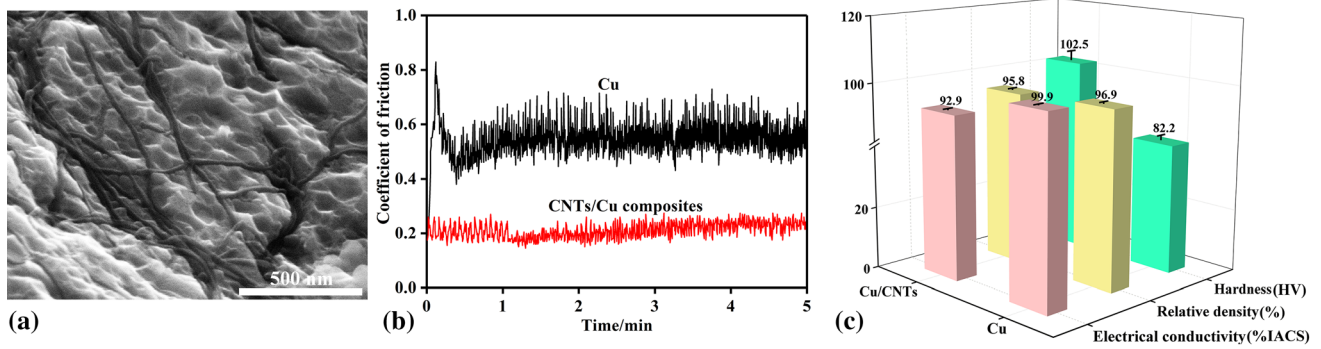
**Fig. 1** Schematic diagram of the preparation process of CNT/Cu composites



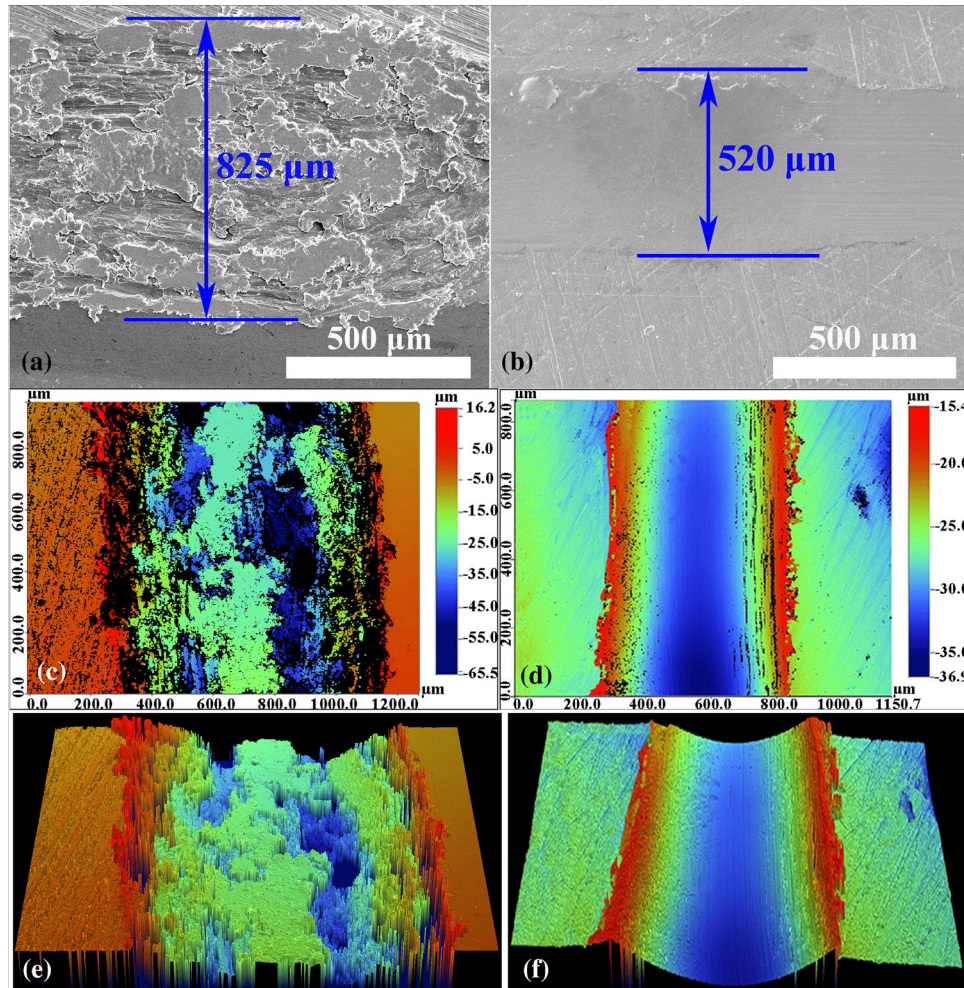
**Fig. 2** SEM images of (a) CuCr alloy powders, (b, c, d) CNT/Cu composite powders, (e, e1) TEM images, and (f) Raman spectrum of CNT/Cu composite powders

825 and 520  $\mu\text{m}$ , respectively, and there were many exfoliated fragments and grooves on the friction-wear surface of Cu (Ref 34). This showed that the main wear mechanism of Cu was serious adhesive wear (Ref 35, 36) because the contact stress caused plastic flow on the contact surface, accompanied by a large amount of heat, which melted the contact interface and underwent micro-welding. These factors made the wear areas

form peeling marks that eventually formed grooves and debris (Ref 37, 38). The self-protection of the CNT/Cu composites was enhanced because the uniformly-distributed CNTs provided good self-lubricity and superior load transfer between the Cu matrix and CNTs (Ref 39, 40). Hence, only an extremely weak lamellar phenomenon was observed at the edges, as shown in Fig. 4(b).



**Fig. 3** (a) SEM image of the CNTs in the final bulk CNT/Cu composites, Properties of Cu and CNT/Cu composites (b) coefficient of friction (c) electrical conductivity, relative density and Vickers hardness



**Fig. 4** SEM images of the friction-wear morphologies of Cu (a) and CNT/Cu composites (b), three-dimensional morphologies of the wear scars of Cu (c, e) and CNT/Cu composites (d, f)

Figure 4(c, e) and (d, f) shows the depth, width, and roughness of the three-dimensional morphologies of the wear scars of Cu and CNT/Cu composites. The wear surface of pure Cu was relatively rough and contained grooves, while the wear surface of the CNT/Cu composites was smoother, as shown in Fig. 4(d, f). The results were in agreement with SEM observations.

Figure 5 shows the 3D roughness characterization parameters ( $S_{ku}$ ,  $S_{sk}$ ,  $S_a$ ,  $S_q$ ,  $S_z$ ) of the surface pure Cu and CNT/Cu composites. The kurtosis of the surface height distribution  $S_{ku}$  were 1.778 and 1.121 for Cu and CNT/Cu composites, indicating a scattered distribution ( $S_{ku} < 3$ ). The skewness  $S_{sk}$  (-1.268 and -1.045) of both materials was less than 0, indicating that the height distribution was higher than that of the average plane. The arithmetic average deviation  $S_a$ , the root

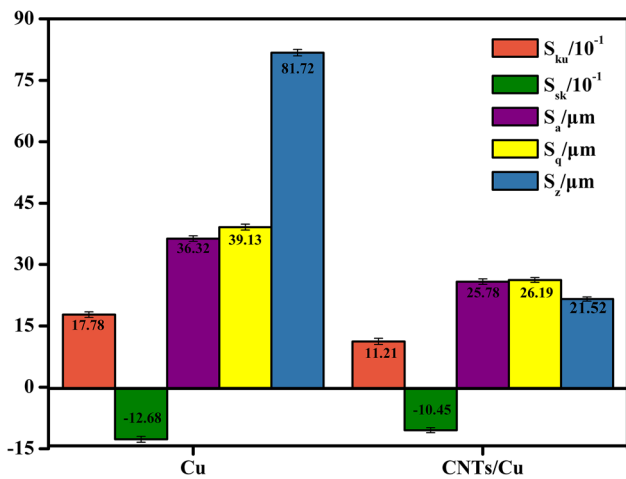


Fig. 5 3D roughness characterization parameters ( $S_{ku}$ ,  $S_{sk}$ ,  $S_a$ ,  $S_q$ ,  $S_z$ ) of the wear surface of pure copper and CNT/Cu composites

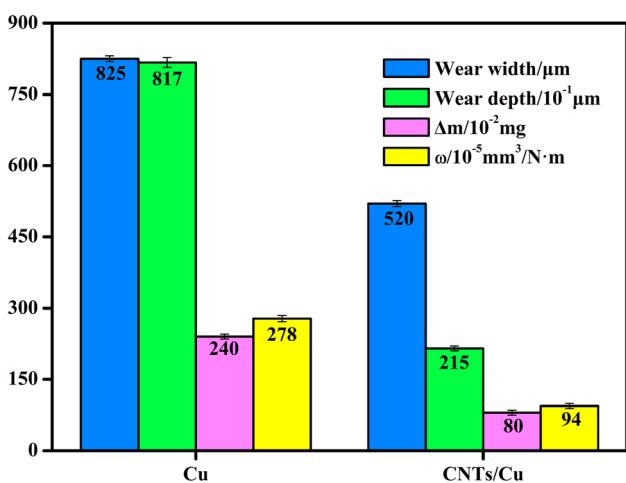


Fig. 6 Wear width, wear depth, wear mass and wear rate for Cu and CNT/Cu composites

mean square deviation  $S_q$ , and the ten-point height  $S_z$  of the CNT/Cu composites were smaller than those of pure Cu, demonstrating that the wear surface of the CNT/Cu composites was smoother. Therefore, the wear resistance of the CNT/Cu composites was much better than pure copper.

Wear rate is an important parameter for friction materials, so it is necessary to calculate the wear rate of Cu and CNT/Cu composite. The number of wear test repeats was five. The wear rate can be calculated by the following formula (Ref 41, 42):

$$\omega = \frac{\Delta V}{F \cdot d} = \frac{\Delta m}{\rho \cdot F \cdot d} \quad (\text{Eq 1})$$

Where  $\omega$  is the wear rate,  $\Delta V$  is the worn volume,  $F$  is the applied load (10 N),  $d$  is the sliding distance (10 m),  $\Delta m$  is the worn mass and  $\rho$  is the density of the materials ( $\rho_{\text{Cu}}=8.63\text{g}/\text{cm}^3$ ,  $\rho_{\text{CNT/Cu}}=8.51\text{g}/\text{cm}^3$ ). Figure 6 shows that the wear width and wear depth is less than copper, and the wear rate of the CNT/Cu composites was about 2 times lower than that of Cu. Hence, the tribological properties of CNT/Cu are significantly improved owing to the present of CNTs.

## 4. Discussion

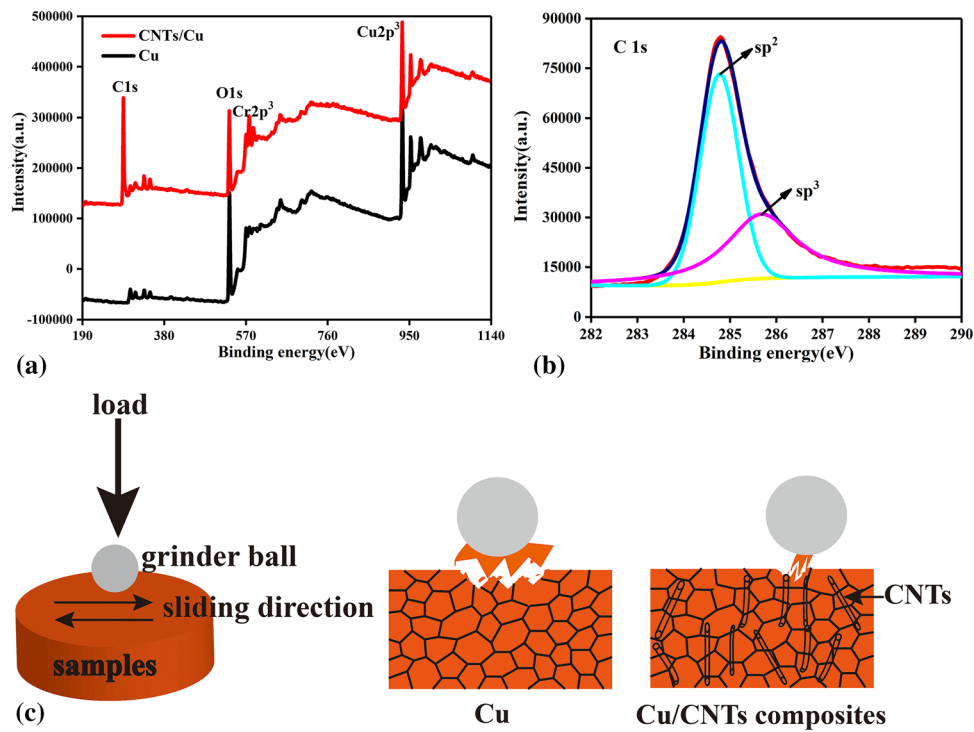
### 4.1 Wear Mechanism

It is crucial to investigate the chemical composition and bonding state of the wear surface to study the wear mechanism. Therefore, XPS analysis was performed on the wear surfaces of Cu and CNT/Cu composites. Figure 7(a) shows the full XPS spectra of the wear surface of Cu and CNT/Cu composites. The wear surface of pure copper contained only Cu and O peaks, while Cr and C peaks appeared on the wear surface of CNT/Cu composites. The intensity of the C peak was relatively large, indicating a high carbon content. Figure 7(b) shows that the main C 1s peak appeared at 284.5 eV, which was ascribed to  $sp^2$  hybridized carbon, and the minor peak at 285.7 eV was attributed to  $sp^3$  hybridized carbon (Ref 43). The results showed that the CNT/Cu composites contained a high CNTs content, which formed a self-lubricating film on the wear surface during the friction-wear process, which slowed damage to the CNT/Cu composites surface caused by the friction pair. This is in agreement with the SEM and three-dimensional morphology of the wear surface shown in Fig. 4. In addition, since Cr acted as a catalyst for CNTs growth, it was present throughout the whole process, and a Cr peak appeared in the XPS spectrum of the CNT/Cu wear surface.

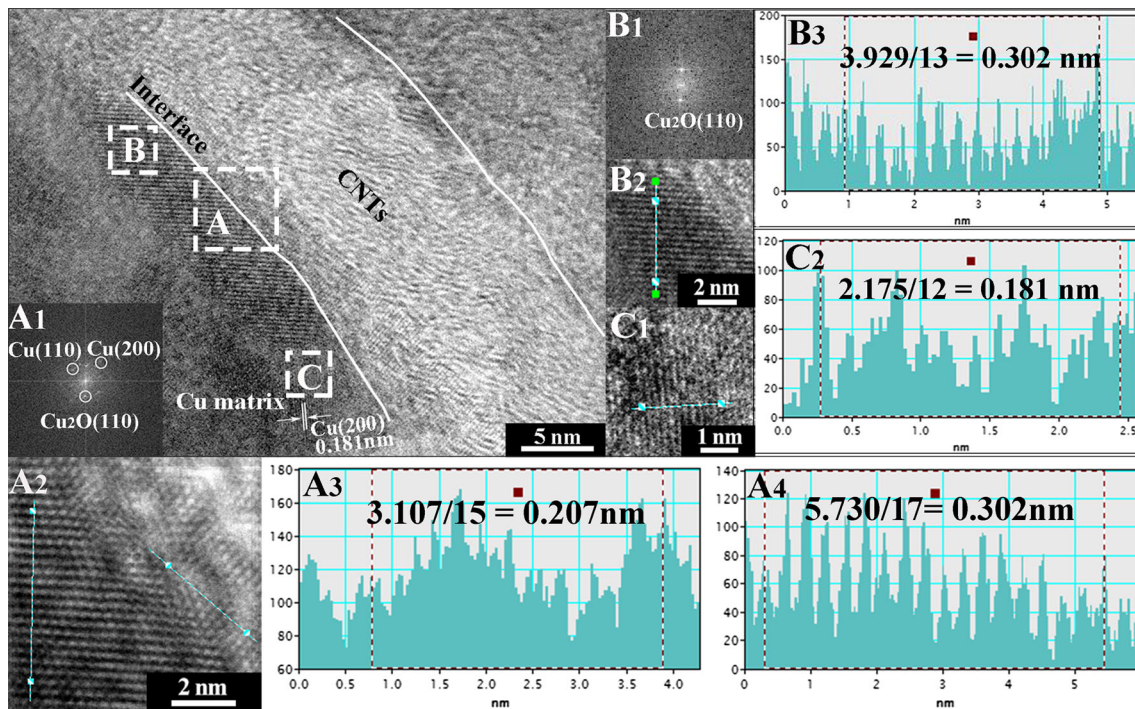
Figure 7(c) displays the wear mechanism of Cu and CNT/Cu composites. When the friction pair slides across the Cu surface, the shedding of abrasive debris accelerated the stripping of particles from the surface (Ref 21). In the CNT/Cu composites, the CNTs nailed the Cu particles due to strong interfacial bonding, making it difficult for the copper particles to be separated from the composites surface. Meanwhile, CNTs acted as a lubricant film during the friction and wear processes. All these served to reduce the coefficient of friction, so the tribological properties of the CNT/Cu composites were better than that of Cu.

### 4.2 Interface Characterization

The interface diagram of the CNT/Cu composites in Fig. 8 shows that the interface between CNTs and Cu matrix was well bonded and that the graphite lattice of the CNTs diffused into the Cu lattice (Ref 44). This indicates that CNTs directly nucleated in the Cu matrix during the in situ synthesis of CNT/Cu composite powders. Therefore, carbon and copper atoms can undergo mutual diffusion, and the composites displayed Cu-Cu, C-C, and Cu-C bonds at the interface. The inverse fast Fourier transform (IFFT)  $C_1$  and the interplanar spacing  $C_2$  for area C in Fig. 8 showed that the matrix was Cu. The Fourier transformation  $A_1$  and inverse fast Fourier transform  $A_2$  of area A showed the presence of Cu and  $\text{Cu}_2\text{O}$  from the calculation interplanar spacing of 0.207 nm (Fig. A3) and 0.302 nm (Fig. A4). The diffraction spot calibration in Fig. B1 and the interplanar spacing 0.302 nm calculated from  $B_2$  and  $B_3$  verified the presence of the  $\text{Cu}_2\text{O}$  (110) crystal plane, which can improve the wettability between Cu and CNTs, leading to strong interfacial bonding. Thus, the CNT/Cu matrix interface remained flat and straight, with no accumulated dislocations and no obvious lattice distortion. Therefore, the interfacial scattering resistance, which is the main factor affecting the electrical conductivity, did not significantly change after the introduction of CNTs. Additionally, the CNTs have excellent electrical conductivity. We can see that the atoms of the CNTs



**Fig. 7** (a) XPS full spectra, (b) curve-fitted XPS spectra of C 1s, (c) schematic illustration of the wear mechanism of Cu and CNT/Cu composites



**Fig. 8** The interfacial structure of CNT/Cu composites. A<sub>1</sub> (A<sub>2</sub>) and B<sub>1</sub> (B<sub>2</sub>) are FFT (IFFT) images of the region A and B. C<sub>1</sub> is IFFT image for region C. A<sub>3</sub>, A<sub>4</sub>, B<sub>3</sub> and C<sub>2</sub> are the calculated interplanar spacings

in Fig. 8 were neatly arranged without an obvious bamboo-like structure or spiral or distortion caused by lattice misalignment. Although these CNTs are not completely metallic, they still

improved the overall electrical conductivity of the composites. Therefore, the wear resistance of the CNT/Cu composites was improved and the electrical conductivity was maintained.

## 5. Conclusion

Cr particles were evenly distributed in the Cu matrix after CuCr alloy powders were treated by a solution-aging treatment to obtain a homogeneous CNTs dispersion without agglomeration in the Cu matrix. Meanwhile, strong interfacial bonding was observed between the CNTs and Cu in the CNT/Cu composites, which was responsible for the improved tribological properties and friction stability compared with Cu. Furthermore, the electrical conductivity of CNT/Cu remained high at 92.9% IACS. The precursor powders could be obtained by a solution-aging treatment, providing a process that is simple and easy to control. Thus, this study provides guidance for synthesizing CNT/Cu composites.

## Acknowledgments

This work was supported by the National Key R&D Program of China (Grant No. 2017YFB0306405), the State Key Laboratory of Advanced Optical Communication Systems and Networks (Grant No. 2018GZKF03007), and the National Natural Science Foundation of China (Grant No. 512011107).

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