JMEPEG (2010) 19:906–911
DOI: 10.1007/s11665-009-9556-x

1059-9495/$19.00

Effect of Milling Time on Electrical Breakdown Behavior of Al_2O_3/Cu Composite

Xianhui Wang, Shuhua Liang, Ping Yang, and Zhikang Fan

(Submitted March 3, 2009; in revised form August 31, 2009)

In order to clarify the relationship between the microstructure and the arc erosion behavior of metal-matrix composite, Al_2O_3/Cu composites with different distributions of Al_2O_3 particles were prepared by high energy ball milling and powder metallurgy. The effect of milling time on microstructure, properties, and arc erosion behavior of Al_2O_3/Cu composite was investigated. The results show that the distribution of Al_2O_3 particles improves significantly with increase of milling time, but Al_2O_3 particles will be aggregated if milling time is too long. The optimal milling time is 24 h in the range of experiments. A uniform distribution of Al_2O_3 particles in copper matrix can improve the hardness, electrical conductivity, average breakdown strength, chopping level, and arc life. With improvement in the distribution of Al_2O_3 particles, the erosion area becomes larger, and the erosion pits become shallower and are dispersed more uniformly.

Keywords electron microscopy, metal matrix composite, powder metallurgy

1. Introduction

The arc behavior of a vacuum breaker is closely related with the microstructure and properties of contact materials. Contact materials should satisfy a number of stringent requirements, such as a large breaking current ability, high breakdown strength, low chopping current, reliable fusion welding resistance, low arc erosion rate, small thermoelectric emission effect, excellent electrical conductivity, and high mechanical strength (Ref 1-4). Up to now, much knowledge on vacuum arc and cathode spots has been obtained from extensive investigations of the surface roughness of cathode and the contents of impurity elements, but the effect of microstructure on vacuum arc and cathode spots is still obscure (Ref 5, 6). In order to gain a deep understanding of the electrical properties of contact materials, it is necessary to study the vacuum breakdown and the relation of cathode spots and microstructure. Currently, the Al_2O_3/Cu composite is regarded as an ideal substitute for high-conductivity copper alloys due to a good combination of high strength and excellent conductivity, and has wide potential applications as electrical contact and electrode materials (Ref 7, 8). Although a number of studies have been done on the high temperature properties of Al_2O_3/Cu composite, the effect of subsequent deformation on microstructure and properties of Al_2O_3/Cu composite (Ref 9-15) and new preparation technologies (Ref 16-21), scant information is available on the relationship between microstructure and the arc erosion behavior of Al_2O_3/Cu composite. In the investigation, the effect of milling time on microstructure, density, hardness, electrical conductivity, arc erosion characteristics, breakdown strength, chopping level, and arc life of Al_2O_3/Cu composite was studied. The purpose is to elucidate the relationship between the microstructure and the arc erosion of Al_2O_3/Cu composite and provide references for the design of oxide/metal contact materials and the arc control.

Xianhui Wang, Shuhua Liang, Ping Yang, and **Zhikang Fan**, School of Materials Science and Engineering, Xi'an University of Technology, Xi'an 710048, P.R. China. Contact e-mails: xhwang693@xaut.edu.cn and liangsh@xaut.edu.cn.

2. Experimental

Cu powders with a purity of 99 wt.% and particle size of 50 μm and Al_2O_3 powders with a purity of 99.5 wt.% and initial size of 30 nm were used to fabricate Al_2O_3/Cu composite. At the ball-to-powder mass ratio of 10:1 and rotation rate of 200 rpm, the powders with Cu/Al_2O_3 mass ratio of 99:1 were milled under argon gas for 4, 24, 36, 48, 60, and 90 h, respectively. The milled powders were hot-pressed and sintered at 950 °C in a XP-80B sintering furnace. For convenience, those samples prepared by Al_2O_3 powders with different milling time are designated as No. 1 to No. 6. The electrical conductivity and hardness test were performed on a 7501 eddy electrical conductivity meter and a HB-3000 Brinell hardness tester, respectively, and the mean values were the average of three measured results. The microstructure was analyzed using an OLYMPUS optical microscope. The vacuum breakdown was tested on a modified TDR240A single crystal furnace. After polishing, the sample as a cathode was put in a sample holder, which could move vertically in the vacuum chamber. Above the cathode there was a pure tungsten rod with a radius of 5 mm and a tip radius of about 1 mm as an anode. When the chamber was evacuated to 5.0×10^{-3} Pa and the capacitor of 120 μF was charged to the voltage (U) of 9 kV, the lower cathode moved upward at a velocity of 0.2 mm/min until the gap was broken down. The gap (D) between the cathode and the anode, i.e., the breakdown distance, was measured. The breakdown voltage was calculated as U/D. The mean values of breakdown voltage and chopping current were calculated from the average of 100 measured data. The breakdown voltage, current and chopping current can be collected from the discharged waveform recorded by a Tektronix TDS-2014 dual channel digital memory oscilloscope (200MH_Z). The microstructures of the Cu/Al_2O_3 composite after electric breakdown were examined by an OXFORD JSM-6700F field emission scanning electron microscope (SEM).

3. Results and Discussions

3.1 Microstructure of Al_2O_3/Cu Composites

Figure 1 is the microstructure of samples No. 1-6 prepared at different milling time. It can be seen from Fig. 1(a) that the distribution of Al_2O_3 particles in copper matrix is quite non-uniform in sample No. 1. With increase of milling time, the spatial distribution of Al_2O_3 particles is improved significantly and no obvious aggregation is observed (see Fig. 1b, c). However, it can be clearly seen from Fig. 1(d), (e), and (f) that with increasing milling time, the spatial distribution of Al_2O_3 particles becomes worse, and Al_2O_3 particles aggregate, especially for sample No. 6. It suggests that too long a milling time cannot improve the spatial distribution of Al_2O_3 particles in the Cu matrix. In the range of experiments, the optimum dispersion of Al_2O_3 particles can be obtained at 24 h.

3.2 Physical Properties of Al_2O_3/Cu Composites

The hardness and electrical conductivity of samples No. 1-6 are shown in Fig. 2. As seen from Fig. 2, the milling time has a significant effect on the physical properties of Al_2O_3/Cu composites. This can be explained as follows. Milling time has a remarkable effect on the spatial distribution of Al_2O_3 particles in copper matrix, as discussed in Section 3.1. Poor spatial distribution of Al_2O_3 particles cannot block the motion of dislocation effectively, thus decreasing the hardness. Since the aggregation of Al_2O_3 particles aggravates the electron scattering, the electrical conductivity of Al_2O_3/Cu composites decreases.

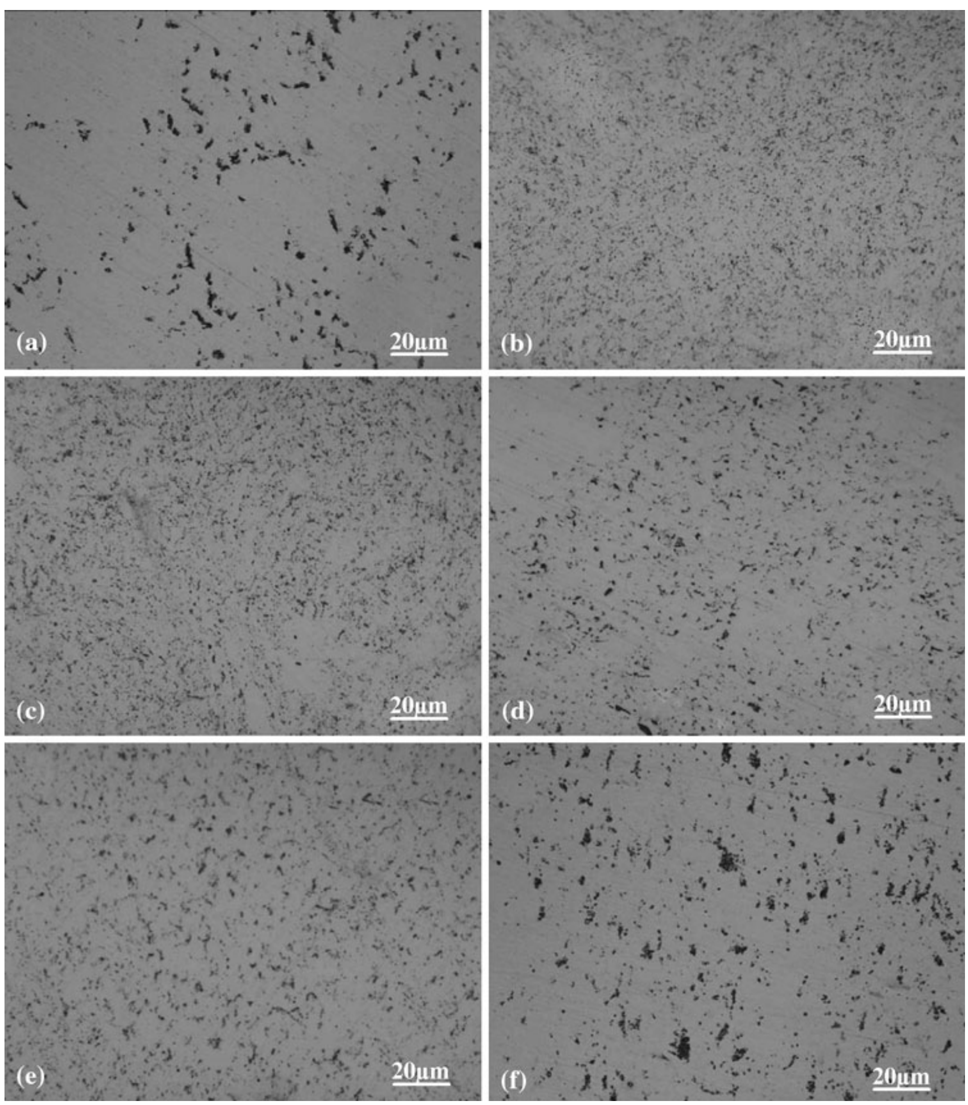

Fig. 1 Microstructures of Al_2O_3/Cu composites prepared with Al_2O_3/Cu powders milled for different milling time (a) 4 h, (b) 24 h, (c) 36 h, (d) 48 h, (e) 60 h, and (f) 90 h

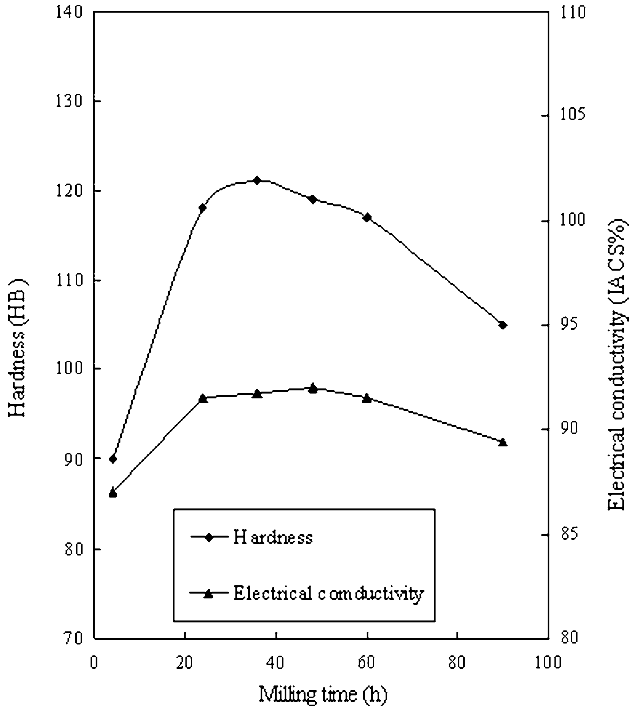

Fig. 2 Variation of electrical conductivity and hardness of Al_2O_3/Cu composite with milling time

3.3 Vacuum Breakdown Test and Analysis

3.3.1 The Effect of Milling Time on the Breakdown Strength, Chopping Current, and Arc Life. The vacuum breakdown experimental results of samples No. 1-6 are listed in Table 1. From Table 1, it can be seen that the breakdown strength, chopping current, and arc life have a parabolic relationship with milling time. Figure 3 illustrates the relation of breakdown strength and time of samples No. 1-3 and sample No. 6. It can be seen from Fig. 3 that at 4 and 90 h, there are a large dispersion and a decreased tendency for the breakdown strength, as shown in Fig. 3(a) and (d); whereas at 24 and 36 h, the breakdown strength is relatively stable (see Fig. 3b, c). However, for sample No. 2 prepared from the powders milled for 24 h, the distribution of breakdown strength is more concentrated.

For the Al_2O_3/Cu composite with non-uniform distribution of Al_2O_3 particles, as the non-uniform sputtering causes a large roughness on the surface of Al_2O_3/Cu composite after vacuum breakdown, the next breakdown will occur at the same site, and the surface morphology will be deteriorated during the repeatable process. Subsequently, the distribution of the breakdown strength of the Al_2O_3/Cu composite with a poor spatial distribution is not uniform, and the average breakdown strength is low as well even if it is subjected to breakdown 100 times.

Due to the non-uniformity of Al_2O_3 particles, arcs cannot move rapidly. When metal vapor cannot sustain an arc, arc will be chopped sharply at a large current. Hence, there is a

Table 1 The electrical properties of Al_2O_3/Cu composites at different milling time

Sample No.	1	2	3	4	5	6
Milling time, h	4	24	36	48	60	90
Breakdown strength, $\times 10^7$ V/m	4.956	5.708	5.602	5.539	5.422	5.184
Chopping current, A	3.8203	3.6084	3.6095	3.5822	3.6149	3.7198
Arc life, ms	17.41	18.42	18.12	17.78	17.63	17.40

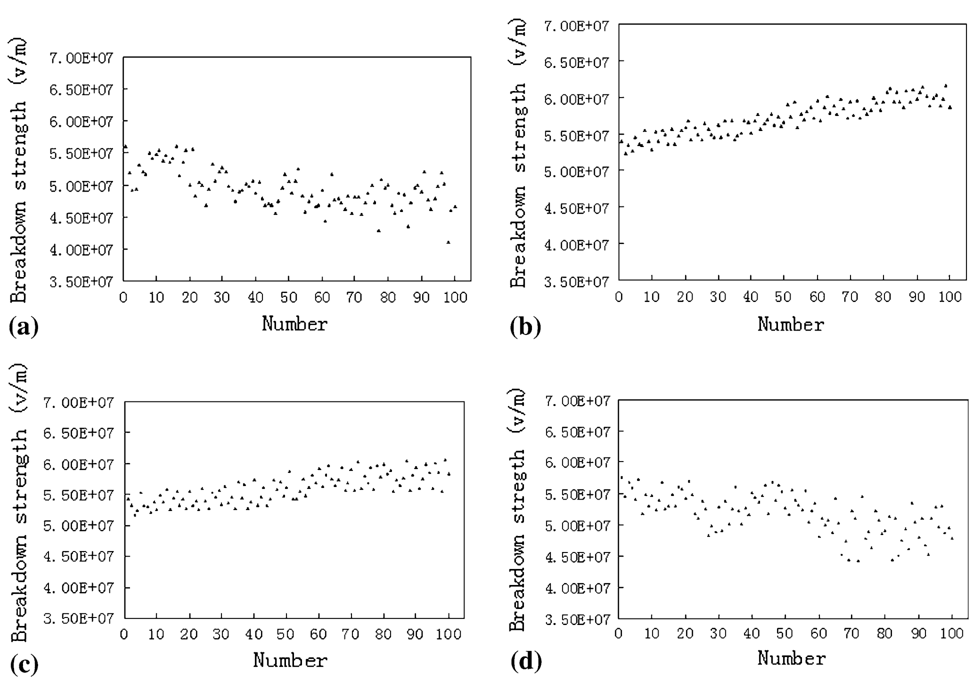

Fig. 3 Breakdown strength of samples No. 1-3 and sample No. 6. (a) Sample No. 1, (b) sample No. 2, (c) sample No. 3, and (d) sample No. 6

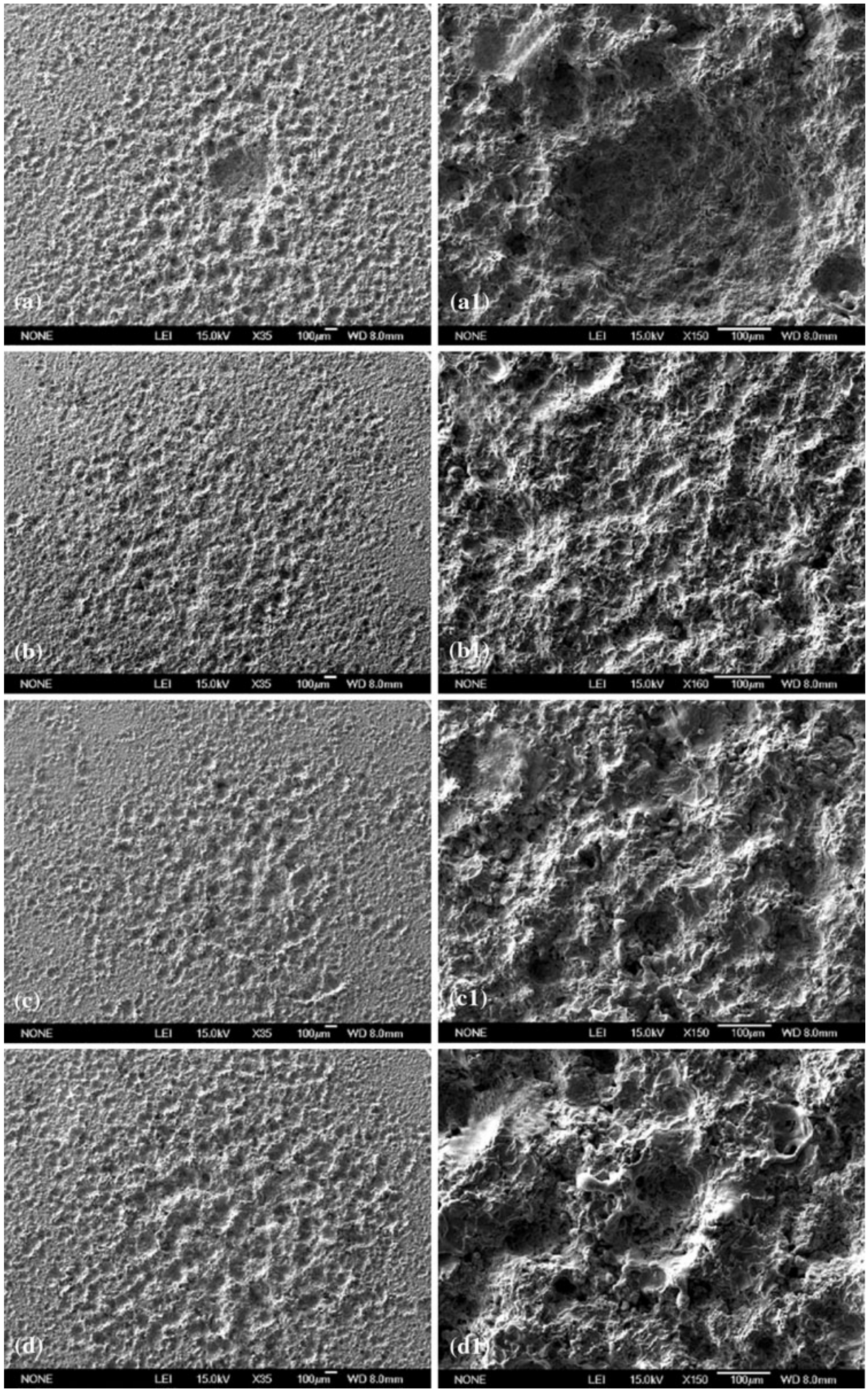

Fig. 4 SEM micrographs of Al_2O_3/Cu composite after breakdown 100 times. (a) Sample No. 1, (b) sample No. 2, (c) sample No. 3, and (d) sample No. 6. a1 to d1 are the magnifying images of (a) to (d)

larger chopping current for the Al_2O_3/Cu composite with a poor distribution of Al_2O_3 particles. Similarly, due to the instability of arc burning, arc lasting time becomes shorter as well.

3.3.2 The Effect of Milling Time on the Arc Erosion of Al_2O_3/Cu Composite. The arc erosion morphologies of samples No. 1-3 and sample No. 6 after vacuum breakdown 100 times are shown in Fig. 4(a)-(d), respectively. Figure 4(a1)-

(d1) is the central arc erosion morphologies of the corresponding samples. From Fig. 4(a) and (a1), it can be seen that when the milling time is 4 h, there is a large erosion pit in the center, which is surrounded by a large number of small and deep erosion pits. It is also noted that there is no metallic luster in these erosion regions, which may be caused by the flotation of large amounts of Al_2O_3 particles into the surface. At 24 h, it is shown that the erosion pits become shallower and are distributed more dispersedly and the boundaries between the erosion pits become obscure as well (see Fig. 4b, b1). With increasing the milling time, the concentrated erosion pits appear again (see Fig. 4c, c1, d, and d1). In comparison with the erosion pits shown in Fig. 4(a), there are no large erosion pits, and the boundaries between the erosion pits are still visible. Based on the above results, it can be concluded that an appropriate milling time can improve the uniform distribution of Al_2O_3 particles, and decrease the arc erosion of Al_2O_3/Cu composite as well. This can be explained as follows. According to the theory of arc motion characteristics along with physics knowledge, it is believed from the morphology of arc erosion that when the microstructure on the surface of Al_2O_3/Cu composite is uniform, the breakdown strength is equal in each site. At this time, arc will be generated simultaneously at these preferred breakdown micro-areas, i.e., the tiny regions surrounding Al_2O_3 particles, which are called as "preferred breakdown areas" (Ref 22). If Al_2O_3 particles are uniformly distributed in the Al_2O_3/Cu composite, the spacing between the preferred breakdown areas is decreased. Subsequently, arc will not be concentrated in a fixed site and will generate in the electrode materials simultaneously. Since those tiny arcs will repel each other under their own magnetic fields and spread rapidly, the arc behaves in a quick motion. As a consequence, the erosion pits become shallower and are dispersed uniformly. If Al_2O_3 particles are not distributed uniformly in the Al_2O_3/Cu composite, the spacing between the preferred breakdown areas is increased, those tiny arcs will gather into a large one and dwell on a fixed site until no sufficient metal vapor can sustain continuous burning, thus causing a serious local ablation.

4. Conclusions

The effect of milling time on the microstructure, properties, and arc erosion behavior of Al_2O_3/Cu composite was studied, and the following conclusions can be drawn from the investigation.

1. With increase of milling time, the spatial distribution of Al_2O_3 particles is improved significantly. But prolong milling time is not beneficial to the improvement of spatial distribution. In the range of experiments, the optimum milling time is 24 h.
2. The improvement of spatial distribution is helpful to the increase of the hardness and electrical conductivity of Al_2O_3/Cu composite.
3. Well dispersed Al_2O_3 in the Al_2O_3/Cu composite will narrow the distribution of breakdown strength, and improve the stability of breakdown properties. With increasing uniformity of Al_2O_3/Cu composite, the average breakdown strength, chopping current, and arc life increase.
4. With increase of spatial distribution of Al_2O_3/Cu composite, arc erosion area becomes larger, erosion pits become shallower, and the erosion pits are dispersed more uniformly.

Acknowledgments

The research was supported by Specialized Research Fund for the Doctoral Program of Higher Education (SRFDP20060700011), Program for New Century Excellent Talents in University (NCET-05-0873), and the National Natural Science Foundation of China (Project 50574075).

References

1. J.P. Zhang, J.C. Chen, and X.L. Zhou, 影响CuCr系触头材料性能的因素 (Effect on Performance of CuCr Contact Materials), *Electr. Eng. Mater.*, 2003, **1**, p 3–8 (in Chinese)
2. Z.B. Li and Y.C. Zhang, 真空触头材料的性能分析比较 (Comparison of Properties of Vacuum Contact Materials), *Low Voltage Apparatus*, 1998, **3**(9), p 15–18, 25 (in Chinese)
3. W.G. Chen and C.Q. Gu, 电触头材料的制造应用与研究进展 (Manufacturing, Application and Research Progress of Electrical Contact Materials), *Shanghai Electr. Apparatus Technol.*, 1997, **2**, p 12–17 (in Chinese)
4. J.X. Shi, 真空开关用触头材料 (Contact Materials for Vacuum Switchgear), *Shanghai Nonferrous Met.*, 2000, **21**(4), p 199–207 (in Chinese)
5. C.P. Lei, J.G. Chen, and Y.H. Xia, 新型钨铜复合材料的制备和性能研究的新进展 (Latest Developments in Preparation and Property Study of W-Cu Composites), *Metal. Funct. Mater.*, 2003, **10**(4), p 24–27 (in Chinese)
6. C.Y. Zhang, 纳晶及非晶Cu基电极表面的真空电弧研究 ("Investigation of Vacuum Arc on the Surface of Nanocrystal and Amorphous Copper Based Electrode"), Ph.D. Thesis, Xi'an Jiaotong University, Xi'an, China, 2004
7. J.S. Kiang, Y. Ni, and W.T. Wu, Critical Al Content to Form External-alumina Scales on Cu-Al alloys, *Intermetallics*, 2007, **15**, p 635–638
8. M.X. Guo, M.P. Wang, L.F. Cao, and R.S. Lei, Work Softening Characterization of Alumina Dispersion Strengthened Copper Alloys, *Mater. Charact.*, 2007, **58**(10), p 928–935
9. B.H. Tian, P. Liu, K.X. Song, L. Yan, Y. Liu, F.Z. Ren, and J.H. Su, Microstructure and Properties at Elevated Temperature of a Nano-Al_2O_3 Particles Dispersion strengthened Copper Base Composite, *Mater. Sci. Eng. A*, 2006, **435–436**, p 705–710
10. M.T. Marques, J.B. Correia, and J.M. Criado, High-Temperature Stability of a Nanostructured Cu-Al_2O_3 Alloy, *Key Eng. Mater.*, 2002, **230–232**, p 652–655
11. S.H. Kim and D.N. Lee, Recrystallization of Alumina Dispersion Strengthened Copper Strips, *Mater. Sci. Eng. A*, 2001, **313**, p 24–33
12. S.H. Kim and D.N. Lee, The Effect of Rolling Conditions on the Strength and Microstructure of Dispersion Strengthened Copper Strips, *Mater. Sci. Eng. A*, 2001, **319–321**, p 471–474
13. K. Kuchařová, S.J. Zhu, and J. Čadek, Creep in Copper Dispersion Strengthened with Alumina Particles (ODS Copper), *Mater. Sci. Eng. A*, 2003, **348**, p 170–179
14. K. Kuchařová, S.J. Zhu, and J. Čadek, Creep in ODS Copper Reinforced with Alumina Short Fibres-DRS Copper, *Mater. Sci. Eng. A*, 2003, **355**, p 267–276
15. R.K. Islamgaliev, W. Buchgraber, Y.R. Kolobov, N.M. Amirkhanov A.V. Sergueeva, K.V. Ivanov, and G.P. Grabovetskaya, Deformation Behavior of Cu-based Nanocomposite Processed by Severe Plastic Deformation, *Mater. Sci. Eng. A*, 2001, **319–321**, p 872–876
16. S.T. Oh, J.S. Lee, and T. Sekino, Fabrication of Cu Dispersed Al_2O_3 Nanocomposites Using Al_2O_3/CuO and Al_2O_3/Cu-nitrate Mixtures, *Scripta Mater.*, 2001, **44**, p 2117–2120

17. M.S. Motta, P.K. Jena, E.A. Brocchi, and I.G. Solórzano, Characterization of Cu-Al_2O_3 Nano-scale Composites Synthesized by In situ Reduction, *Mater. Sci. Eng. C*, 2001, **15**(1–2), p 175–177
18. P.K. Jena, E.A. Brocchi, and M.S. Motta, In situ Formation of Cu-Al_2O_3 Nano-scale Composites by Chemical Routes and Studies on Their Microstructures, *Mater. Sci. Eng. A*, 2001, **313**, p 180–186
19. D.W. Lee, G.H. Ha, and B.K. Kim, Synthesis of Cu-Al_2O_3 Nano Composite Powder, *Scripta Mater.*, 2001, **44**, p 2137–2140
20. S.J. Hwang, D. Wexler, and A. Calka, Mechanochemical Synthesis of Nanocrystalline Al_2O_3 Dispersed Copper, *J. Mater. Sci.*, 2004, **39**, p 4659–4662
21. J.F. Perez and D.G. Morris, Copper-Al_2O_3 Composites Prepared by Reactive Spray Deposition, *Scripta Metall.*, 1994, **31**, p 231–235
22. X. Wang, S. Liang, P. Yang, and Z. Fan, Effect of Al_2O_3 Particle Size on Vacuum Breakdown Behavior of Al_2O_3/Cu Composite, *Vacuum*, 2009, **83**(12), p 1475–1480