Research Article

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Hot deformation behavior of nano- Al_2O_3 -dispersion-strengthened Cu20W composite

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Abstract: Nano- Al_2O_3 dispersion-strengthened Cu20W composite was fabricated by vacuum hot-pressing sintering process. The electrical conductivity, relative density, and Brinell hardness were tested, respectively. The gleeble-1500D thermomechanical simulator was used to conduct isothermal compression with strain rates ranging from 0.001 to 10 s^{-1} and the temperatures ranging from 650 to 950°C . The microstructure of the Cu- Al_2O_3 /20W composite was observed using an optical microscope and a transmission electron microscope, and the true stress-strain curves were analyzed. In addition, the influence of the nano- Al_2O_3 and tungsten on the thermal deformation process of the composite was analyzed. The relationship and interaction among work hardening, dynamic recovery, and dynamic recrystallization were illustrated. The results show that nano- Al_2O_3 particles pin dislocations and inhibit dynamic recovery and dynamic recrystallization. Consequently, the Cu- Al_2O_3 /20W composite has typical dynamic recovery characteristics. Hence, the Cu- Al_2O_3 /20W composite possesses outstanding high-temperature performance. The optimal processing domain of the Cu- Al_2O_3 /20W composite ranged from 760 to 950°C with strain rates ranging from 0.01 to 0.1 s^{-1} . Furthermore, the constitutive equation of the Cu- Al_2O_3 /20W composite is established, and the activation energy is $155.069\text{ kJ mol}^{-1}$.

Keywords: vacuum hot-pressing sintering, hot deformation, dynamic recrystallization, constitutive equation

1 Introduction

Switches are widely used in power transmission, industrial production, aerospace, and other fields. As the core component of various switchgear, electrical contacts play a significant role [1,2]. With the development of industry and technological progress, the requirement for electrical contacts is increasing. In the process of making or breaking the circuit, temperature elevation, welding, wear, and arc erosion take place during initiating, maintaining, and terminating electrical contacts [3,4]. Therefore, electrical contacts must possess good mechanical properties and arc erosion resistance. Copper is widely used in electrical contact materials because of its good electrical and thermal conductivity [5]. Considering the arc erosion resistance of electrical contacts and mechanical properties, such as hardness, copper-based composite materials with composite strengthening as the preparation mechanism are ideal electrical contact materials [6]. Dispersion-strengthened copper alloys are often used in harsh environments because of their good electrical and thermal conductivity and high-temperature stability. Talekar et al. [7] added 1 wt% of nano- Al_2O_3 to the W-Ni alloy, which shows good oxidation resistance at a high temperature of $1,400^\circ\text{C}$. Vladimirova and Shalunov [8] prepared Al_2O_3 dispersion-strengthened copper alloy by powder metallurgy. It was found that the nano-alumina is $\gamma\text{-Al}_2\text{O}_3$ with a particle size of 39 nm , and the recrystallization temperature reached about $1,000^\circ\text{C}$. Lee et al. [9] used the internal oxidation method to prepare a large number of alumina particles with a diameter of about 10 nm dispersed in the copper matrix, and the relationship between mechanical properties and matrix was explained by the Orowan mechanism. In addition to dispersing copper as the matrix, in order to improve the voltage strength and welding resistance of the electrical contacts, W with a high melting point and high hardness is often added to it. Zhang et al. [10] revealed that the tungsten in the copper-tungsten

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electrical contacts gradually accumulates into a needle-like skeleton under the high temperature of the arc. This skeleton-like structure can effectively reduce the flow of low-melting copper liquid. The research of Li et al. [11] showed that the welding probability of Cu–W is lower than that of Cu, and the addition of W can improve the welding resistance of the vacuum switch contacts.

In recent years, most of the research on nano- Al_2O_3 -reinforced dispersed copper and Cu–W composites has focused on the preparation methods and arc erosion resistance. However, studies on the thermal deformation behavior of copper-based composites are rarely reported. Therefore, the deformation behavior and microstructure evolution of Cu–W composites during high-temperature compression have not been understood. Li [12] studied the hot deformation behavior of Al_2O_3 -dispersion-strengthened copper alloys through hot deformation tests and verified that the best deformation zone temperature range is $750\text{--}850^\circ\text{C}$, and the strain rate ranges from 1 to 10 s^{-1} . Hiraoka et al. [13] proposed that the internal grains of the W(80)/Cu composite material exhibited a fibrous structure as the amount of deformation increased during the compression deformation process. Furthermore, although Zhang et al. [14] investigated the flow stress changes of Al_2O_3 -Cu/35W5Cr composites at high temperatures and the evolution of the microstructure during thermal deformation, they did not establish a constitutive equation or related thermal processing diagrams.

Because the electrical contact has a high arc temperature at the moment of making and breaking, it has to withstand the combined action of various forces during the making and breaking process. Hence, the hot deformation behavior of the Cu- Al_2O_3 /20W composite is very crucial to the service of the electrical contacts. In addition, hot deformation can be used to precisely extrude the contact blanks, which saves materials and reduces costs. On the other hand, hot deformation is also a kind of densification process, which can improve the performance of the contact material, and the final hot processing can also refine the structure and further improve the performance. In order to analyze the deformation behavior and the microstructure evolution of the different reinforcing phases and copper matrix at high temperature, the Cu- Al_2O_3 /20W composite was systematically studied by the Gleeble-1500D thermo-mechanical simulator. Conducting the analysis of the as-sintered composite material and the microstructure after thermal compression, the deformation mechanism of the nano- Al_2O_3 and W are determined. Moreover, the constitutive equation is constructed.

2 Experimental procedure

W powder and Cu-0.4 wt% Al powder with an average particle diameter of $2\text{--}5\text{ }\mu\text{m}$ were used. The Cu_2O powder provides oxygen atoms with sizes ranging from 2 to $5\text{ }\mu\text{m}$. Figure 1 shows the nominal composition ratio of the Al_2O_3 -Cu/20W composite. These powders were adequately mixed in a YH-10 mixer for 2 h, and the ball-to-powder weight ratio was 5:1. Copper balls served as grinding media. After the powders were mixed, the sample was sintered in a ZT-120-22Y vacuum hot-pressing sintering furnace. During the sintering process, the vacuum degree was kept at 0.06 Pa and the heating rate was $10^\circ\text{C min}^{-1}$. The uniaxial contact pressure was applied as 30 MPa when the temperature reached 650°C , and held for 1 h, and continued by heating to 950°C . After 1 hour, the temperature was decreased to room temperature prior to sample removal. The cylindrical sample size was $\Phi 50\text{ mm} \times 15\text{ mm}$.

After the surface of the sintered cylindrical sample was polished, the electrical conductivity was measured with a Sigma2008B1 digital conductivity meter. Then, the Brinell hardness test was conducted using the HB-3000B Brinell hardness tester according to GB/T231.1-2009 standard. Moreover, the relative density was measured and calculated using the Archimedes drainage method. Finally, the microstructure of the sintered sample was determined using a JSM-7800F field emission scanning electron microscope. The sintered composite material was cut into a cylinder of $\Phi 8\text{ mm} \times 12\text{ mm}$ and then the hot deformation test was performed on a Gleeble-1500D thermal simulation test machine. The hot deformation temperatures were set to 650 , 750 , 850 , and 950°C , and the deformation rates were 0.001 , 0.01 , 0.1 , 1 , and 10 s^{-1} . At the beginning of the test, the temperature was increased to the deformation temperature at a temperature increase rate of 10°C s^{-1} , and then the compression was started after holding for 3 min. At the end of the compression process,

Figure 1: Composition ratio of the Cu- Al_2O_3 /20W composite, wt%.

the sample was taken out and immediately cooled in water. There was an interval of 3–5 s between unloading and quenching. The test process is shown in Figure 2.

3 Results

3.1 Microstructure and comprehensive properties

Figure 3(a) shows the SEM image of the Cu–0.4 wt% Al/20W composite powders after mixing. The various powders are uniformly mixed without obvious agglomeration. The SEM image of the sintered sample is shown in Figure 3(b). After sintering, the microstructure is compact, and there are no holes or cracks on the surface of the sample. Moreover, the distribution of W particles is relatively uniform. Figure 3(c) is the EDS energy spectrum analysis data of the sintered sample, and the distribution of various elements coincided with the theoretical value in Figure 1. In addition, the line scan results of W particles show that the curve at the Cu–Al₂O₃/W interface is steep and they are completely incompatible.

Figure 4 shows the comprehensive performance of the Cu–Al₂O₃/20W composite. After vacuum hot-pressing sintering, the relative density of the as-sintered composite reaches 98.3%. Compared with Cu–Al₂O₃ prepared by the same material and the same process, the Brinell hardness increases from 51.1 to 67.2 HBW with the addition of W. However, the electrical conductivity decreased from 90.3 to 78.6% IACS. The W particles possessing high hardness are relatively evenly distributed on the matrix and hinder dislocation movement, resulting in the increase of the hardness. Nevertheless, poor electrical conductivity

and interface bonding lead to the decrease of the electrical conductivity of the as-sintered composite.

3.2 True stress–strain curves

Figure 5 shows the true stress–strain curves of the Cu–Al₂O₃/20W composite. The flow stress under different deformation conditions decreases with the increase of the deformation temperature. However, it increases with the increase of the strain rate. In addition, the Cu–Al₂O₃/20W composite has typical continuous dynamic recrystallization characteristics.

As shown in Figure 5(a and b), the flow stress of the Cu–Al₂O₃/20W composite increases to a peak value rapidly at the beginning of hot deformation, which is consistent with the characteristics of work hardening. This is because in the initial stage of hot deformation, as the strain increases, dislocations are first generated at the stress concentration of the sample, and dislocations entangled after rapid multiplication, resulting in work hardening. As the deformation continues, the slope of the curve decreases, and the flow stress increases slowly until the peak stress appears. This is because when the second stage of hardening begins, a large number of jogs are produced, which causes the flow stress to increase continuously until a peak occurs. However, the resistance caused by the formation of jogs is sensitive to temperature and the increase of atomic thermal motion can reduce the resistance of jogs. Later, the flow stress dropped again and finally go to a stable state, which is a characteristic of dynamic recrystallization. The softening behavior of materials during high-temperature deformation can be divided into dynamic recovery and dynamic recrystallization [15,16]. During the dynamic recovery process, the cross-slip and climbing of dislocations play a vital role. Then, the defect density is reduced, the opposite sign dislocations are canceled, and the dislocations are rearranged. Furthermore, dynamic recrystallization occurs through the nucleation of the crystal grain. The way to eliminate dislocations and sub-grain boundaries in the deformed matrix is mainly through the migration of large-angle grain boundaries. In the process of high-temperature deformation, the stress–strain behavior depends on the balance between work hardening and dynamic softening [17,18]. It is worth noting that the curve drops sharply at a strain rate of 10 s^{−1} when the strain is greater than 0.6 under the deformation temperatures of 650 and 850°C, which are shown in Figure 5(c) and (d). This is because, at high strain rates, dynamic softening is too late to balance work hardening, resulting in cracking of the specimen.

Figure 2: Schematic of the hot compression process.

Figure 3: SEM images of the composite powders and the Cu–Al₂O₃/20W composite.

Figure 4: Comprehensive properties of Al₂O₃–Cu/(W, Cr) composites.

3.3 The constitutive equation of the Cu–Al₂O₃/20W composite

Through the above true stress–strain curve, it can be found that in the hot simulation test of copper-based composites, the change of the flow stress is mainly affected by two factors, temperature, and the strain rate. Therefore,

discussing the relationship between the deformation temperature and strain rate on flow stress can provide a theoretical basis for the hot working process of the copper alloys. Researchers commonly use constitutive equations to describe the hot deformation behavior of metallic materials [19]. It is usually described in the following three forms:

$$\dot{\epsilon} = A_1 \sigma^{n_1} \exp\left[-\frac{Q}{RT}\right], \quad \alpha\sigma < 0.8, \quad (1)$$

$$\dot{\epsilon} = A_2 \exp(\beta\sigma) \exp\left[-\frac{Q}{RT}\right], \quad \alpha\sigma > 1.2, \quad (2)$$

$$\dot{\epsilon} = A[\sinh(\alpha\sigma)]^n \exp\left[-\frac{Q}{RT}\right], \quad (\text{for all}). \quad (3)$$

As mentioned above, $\dot{\epsilon}$ is the strain rate (s^{-1}), σ is the peak flow stress (MPa), Q represents the hot activation energy (kJ mol^{-1}), and A , A_1 , A_2 , α , β , n_1 , and n are constants, which are influenced by the materials. T is the deformation temperature (K) and R is the universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$). The Zener–Hollomon parameters (Z parameters) proposed by Zener and Hollomon for the first time [20,21] is used to express the influence of temperature and the deformation rate on the deformation behavior:

Figure 5: True stress–true strain curves of Cu–Al₂O₃/20W electrical contacts at different temperature and strain rates: (a) 0.01 s^{−1}; (b) 1 s^{−1}; (c) 650 °C; and (d) 850 °C.

$$Z = [\sinh(\alpha\sigma)]^n = \dot{\epsilon} \exp\left[-\frac{Q}{RT}\right]. \quad (4)$$

By taking the logarithm of the above formula,

$$\ln \dot{\epsilon} = n_1 \ln \sigma + \ln A_1 - \frac{Q}{RT}, \quad (5)$$

$$\ln \dot{\epsilon} = \beta \sigma + \ln A_2 - \frac{Q}{RT}, \quad (6)$$

$$\ln \dot{\epsilon} = n \ln[\sinh(\alpha\sigma)] - \frac{Q}{RT} + \ln A. \quad (7)$$

By taking the partial derivative of $1/T$, we obtain the thermal deformation activation energy formula of the composite material in equation (3):

$$Q = R \left[\frac{\partial \ln \dot{\epsilon}}{\partial \ln[\sinh(\alpha\sigma)]} \right]_T \left[\frac{\partial \ln[\sinh(\alpha\sigma)]}{\partial (1/T)} \right] = RnS. \quad (8)$$

Table 1 lists the peak stress of the Cu–Al₂O₃/20W composite. Substituting the peak stress value into the above equations, one can obtain the relationship curve between the peak stress, strain rate, and the deformation

temperature, which are shown in Figure 6. n_1 , β , n , and S are the average values of the slopes of the curves in Figure 6(a)–(d), respectively. According to calculations, $n_1 = 5.652$, $\beta = 0.116$, and then $\alpha = \beta/n_1 = 0.021$, $n = 4.06$, and $S = 4.594$. From equation (8), the intercept of the straight line $\ln Z - \ln[\sinh(\alpha\sigma)]$ in Figure 6(e) is $\ln A$. After linear fitting from Figure 6(e), $\ln A = 13.805$, so $A = e^{13.805}$ can be obtained. According to equation (8), the hot deformation activation energy, $Q = RnS = 8.314 \times 4.06 \times$

Table 1: Peak flow stress of the Cu–Al₂O₃/20W composite at various conditions (MPa)

Strain rate (s ^{−1})	Peak stress (MPa)			
	650 °C	750 °C	850 °C	950 °C
0.001	32.09	24.14	17.35	12.94
0.01	56.12	40.60	29.62	21.56
0.1	84.07	64.74	50.36	35.91
1	113.23	89.19	70.17	53.83
10	136.96	112.47	91.82	70.16

Figure 6: Functional relationship among peak flow stress, strain rate, and deformation temperature of the Cu–Al₂O₃/20W composite: (a) $\ln \dot{\epsilon} - \ln \sigma$; (b) $\ln \dot{\epsilon} - \sigma$; (c) $\ln \dot{\epsilon} - \ln [\sinh(\alpha\sigma)]$; (d) $\ln [\sinh(\alpha\sigma)] - 1,000/T$; and (e) $\ln Z - \ln [\sinh(\alpha\sigma)]$.

$4.594 = 155.069 \text{ kJ mol}^{-1}$, can be obtained. Therefore, the constitutive equation of the Cu–Al₂O₃/20W composite can be expressed as follows:

$$\dot{\epsilon} = e^{13.805} [\sinh(0.021\sigma)] \exp\left(-\frac{155069}{8.314T}\right). \quad (9)$$

3.4 Microstructure evolution of the Cu–Al₂O₃/20W composite

Figure 7 shows the microstructure of the Cu–Al₂O₃/20W composite after deformation at 850°C and 0.01 s^{−1}. It can

Figure 7: TEM micrographs of the Cu-Al₂O₃/20W composite deformed at 850°C and 0.01 s⁻¹. (a, c, e, and f) Bright field images and (b and d) selected area electron diffraction pattern and indexing.

be seen from Figure 7(a) that a large number of nanoparticles are dispersed on the matrix of the composite, pinning and hindering the movement of dislocations, which is shown in Figure 7(e). The diffraction spots were calibrated and the nanoparticles were γ -Al₂O₃. In addition, although tungsten has high hardness, some researchers have studied the deformation behavior of tungsten alloys. Woodward et al. [22] revealed the strain rate effects on the flow stress of the heavy tungsten alloys by the hot deformation experiment. Karl [23] proposed that the tungsten-copper alloy undergo ductile-brittle transition at 400°C. It can be seen from Figure 7(c) that a slight deformation has occurred at the interface between the W particles and the

copper matrix. The reason for this phenomenon is that the movement of a large number of dislocations at the interface is hindered by W particles and the dislocation accumulation generates stress. After further analysis of the W particles, it is found that a large number of dislocations have sprouted inside the W particles, as shown in Figure 7(c).

In addition, dislocation wall and dislocation cell can be observed in Figure 7(e and f). In the process of hot deformation, due to work hardening, dislocations are produced at the grain boundary and other stress concentrations. When the stress increases to a certain level, a large amount of cross slip occurs and the dislocation

Figure 8: Hot processing maps and the microstructure of the Cu–Al₂O₃/20W composite: (a) hot processing maps and (b) the microstructure at 850°C and 0.01 s^{−1}.

density increases sharply. Furthermore, these cross-slips can react with dislocations to form dislocation walls. As the deformation increases, these dislocation walls form a cell structure as the center of dislocation entanglement [24]. Under the deformation conditions of 850°C and 0.01 s^{−1}, the dislocation wall gradually narrows due to dynamic recovery, as shown in Figure 7(f). The reason for this phenomenon is that a large number of nano-alumina particles are dispersed in the copper matrix, and the nano-particles pin the dislocations and hinder the movement of the dislocations, inhibiting dynamic recovery and dynamic recrystallization. Therefore, dispersed copper has a good high-temperature performance.

3.5 Cu–Al₂O₃/20W composite processing map

In order to determine the best processing area and reduce cracking, the hot processing map of the Cu–Al₂O₃/20W composite was drawn. The process parameters of the hot processing map can reflect the microstructure of the material during hot deformation. It is composed of a power dissipation map and instability map. The hot processing map was established by Prasad et al. [25–27] according to the dynamic materials model (DMM). The total power P in the process of the system deformation is composed of the plastic deformation power (G) and the microstructure evolution power (J). The relationship between them can be expressed by the following parameter:

$$P = \sigma \dot{\epsilon} = G + J = \int_0^{\dot{\epsilon}} \sigma d\dot{\epsilon} + \int_0^{\sigma} \dot{\epsilon} d\sigma. \quad (10)$$

According to the theory of the hot processing map, the power dissipation (η) can be expressed as

$$\eta = \frac{J}{J_{\max}} = \frac{2m}{m+1}, \quad (11)$$

where m is the sensitive parameter of the strain rate and is given by

$$m = \frac{\partial J}{\partial G} = \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}}. \quad (12)$$

According to the extreme value principle of irreversible thermodynamics, the flow instability criterion formula can be expressed as

$$\xi(\dot{\epsilon}) = \frac{\partial \ln \left(\frac{m}{m+1} \right)}{\partial \ln \dot{\epsilon}} + m < 0. \quad (13)$$

The flow instability parameter $\xi(\dot{\epsilon})$ represents the relationship among the deformation rate, the deformation temperature, and the strain rate during hot deformation. The hot processing map can be obtained by superposing the flow instability diagram and the power dissipation diagram.

Figure 8 shows the hot processing map of the Cu–Al₂O₃/20W composite at a true strain of 0.2. The values in the figure represent the power dissipation. With the increase of values, the microstructure evolution of the Cu–Al₂O₃/20W composite is stable and the hot workability of the composites is better. The results show that the optimal processing domain of the Cu–Al₂O₃/20W composite ranged from 760 to 950°C

with strain rates ranging from 0.01 to 0.1 s⁻¹. In order to better reveal the microstructure of the optimal processing domain, the microstructure of the Cu–Al₂O₃/20W composite at 850°C and 0.01 s⁻¹ was determined, as shown in Figure 8(b). It can be seen that the grains of the Cu–Al₂O₃/20W composite are mainly equiaxed grains. The existence of equiaxed grains makes the material have better plasticity and toughness and reduces the occurrence of cracking [28,29].

4 Conclusion

The Cu–Al₂O₃/20W composite was prepared by vacuum hot pressing and sintering. The as-sintered sample has a compact structure without obvious defects, and the relative density is greater than 98%. Because of the addition of W, the electrical conductivity of the composite decreases while the hardness increases significantly. The true stress–strain curves of the Cu–Al₂O₃/20W composite present the characteristics of dynamic recrystallization, and the flow stress increases with the increase of strain rate, while it decreases with the increase of temperature. The optimal processing domain of the Cu–Al₂O₃/20W composite ranged from 800 and 950°C with strain rates ranging from 0.01 to 0.1 s⁻¹, and the microstructure is equiaxed grains. The hot deformation activation energy of the Cu–Al₂O₃/20W composite is calculated to be 155.069 kJ mol⁻¹. The corresponding constitutive equation is

$$\dot{\epsilon} = e^{13.805} [\sinh 0.021\sigma] \exp\left(-\frac{155069}{8.314T}\right).$$

In the process of hot deformation, nano-Al₂O₃ particles pin the dislocation and hinder the movement of the dislocation. The dislocation wall changes from thick to narrow. The W particles can deform slightly and initiate dislocations on the surface.

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