Improvement of interfacial bonding and mechanical properties of Cu-Al₂O₃ composite by Cr-nanoparticle-induced interfacial modification

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ABSTRACT: The strength and ductility of copper matrix composites are usually limited due to the interfacial debonding between the reinforcement and matrix. Interfacial debonding is mainly caused by the interfacial stress induced by the differences in elastic modulus and thermal expansion coefficient (CTE) between the reinforcement and matrix. Cu-Cr-Al₂O₃ composite with Cr nanoparticles precipitated at the interface of Cu and Al₂O₃ was fabricated. It was found that the hardness of Cu-1 wt% Cr-4 wt% Al₂O₃ composite is higher than that of Cu-5 wt% Al₂O₃ composite over the temperature range from 25 °C to 1000 °C. Notably, the hardness increment reached 133% at 1000 °C. In addition, the compressibility increased 36% for Cu-1 wt% Cr-4 wt% Al₂O₃ composite compared with Cu-5 wt% Al₂O₃. The finite element simulation results confirmed that the interfacial residual stress in Cu-Al₂O₃ composite decreased by adding Cr, resulting in the improvement in mechanical properties.

Keywords: Mechanical property, Interfacial bonding, Residual stress, Composite

Introduction

Cu-Al₂O₃ composite has attracted much attention for the combination of high strength and high electrical conductivity. Al₂O₃ particles distributed in copper matrix work as hard obstacles to pin dislocation, improve the strength of Cu-Al₂O₃ composite. Numerous approaches have been optimized for further improving the strength of Cu-Al₂O₃ composite [1-4]. However, the Al₂O₃ content in the commercial Cu-Al₂O₃ composite is usually limited to not more than 1 wt%. The reason is that the strength of Cu-Al₂O₃ composite was found to decrease with higher volume fraction of

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Al₂O₃ [5, 6], which was attributed to the interfacial debonding between Cu matrix and Al₂O₃ particles [7-9] due to the large misfit in lattice parameters, CTE and elastic modulus [10, 11]. Micro-cracks are consequently inclined to form along the debonded interface when external stress is applied. Meanwhile, substantial decrease in ductility was observed with the increase of Al₂O₃ content, which further results in the poor workability of Cu-Al₂O₃ composite [12, 13].

A variety of methods are employed to improve the interfacial bonding of Cu-Al₂O₃ composite. One approach is to try to form coherent interface between the reinforcement and matrix. Ren et al. [14] reported that coherent interfaces formed between the nano-sized Al₂O₃ particles and copper matrix. The composite were fabricated by the internal oxidation of Cu-0.16 wt% Al alloy and subsequently hot extrusion processes. But Cu-Al₂O₃ composite with higher Al₂O₃ content is difficult to fabricate by internal oxidation because the long duration of internal oxidation and the difficulty in completing the oxidation of Al. Another approach is to improve the wettability between the reinforcement and matrix. Electroplating [15, 16], electroless plating [17, 18] and spraying [19, 20] methods are employed to coat Al₂O₃ nanoparticles with copper film to improve the interfacial bonding. However, the nature of large misfit between copper matrix and Al₂O₃ nanoparticles is not changed, and interfacial debonding still took place while external higher stress applied.

The interfacial debonding in metal matrix composites is considered mainly caused by the severe interfacial stress [21-24]. Interfacial stress in Cu-Al₂O₃ composite is caused by the difference in CTE and elastic modulus between copper and Al₂O₃. In this paper, Cu-Cr-Al₂O₃ composite was fabricated and the effect of precipitated Cr at the interface of Cu and Al₂O₃ and the mechanical properties were evaluated. Pure copper and Cu-Al₂O₃ composite without Cr addition were also investigated for comparison.

Experimental

2.1. Materials

Elemental powders of commercially pure Cu (99.5 %, <75 μm), Cr (99.5 %, <75 μm) and Al₂O₃ (99.9 %, <100 nm) were mixed according to the desired nominal compositions. The milling of the mixtures was carried out in a Simoloyer miller with hardened alloy vial and balls under argon atmosphere. In the first step, pre-milling of Al₂O₃ and Cr was carried out to form finely dispersed reinforcement hybrids. Then the reinforcement hybrids were further milled with copper, and the milling parameters

are listed in Table 1. The milled powders were compacted into $\phi 35 \times 10$ mm billets under the pressure about 200 MPa and sintered at 950 °C for 3 h, and then hot-pressed at 850 °C for 2 h under 30 MPa. In addition, the aging treatments were carried out at 500 °C for 100 min. For comparison, a pure copper sample was also prepared in the same process.

2.2. Morphology and microstructure characterization

The phase identification of composite powders were evaluated by XRD-7000S diffractometer (Shimadzu, Ltd., Kyoto, Japan) using Cu Ka radiation ($\lambda = 1.54060 \text{ \AA}$). The microstructure was evaluated by JEOL JSM-7000F scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectroscopy (EDS) system. Details on the interfacial microstructure were studied by JEOL JEM-2100 transmission electron microscopy (TEM) equipment.

2.3. Hardness test at elevated temperatures

The hardness measurements were implemented on the polished surface of copper matrix samples using a high temperature Vickers Hardness Tester (HTV-PHS30, Archimedes, Ltd., Beijing, China). The test was carried out under argon atmosphere, in the temperature range between room temperature (25 °C) and 1000 °C within a 50 °C interval. The applied load was 500 g and the loading time was 5 s. Hardness was measured randomly at 5 different points, the maximum and the minimum results were disregarded, and the mean hardness value was calculated using the remaining three values.

2.4. Compressibility and electrical conductivity

Compressibility of the copper matrix specimen was evaluated by cold rolling at room temperature. Samples with diameter of $\phi 20$ mm and thickness of 4 mm cut from the copper matrix composites were rolled at room temperature with a twin-roller (diameter, 80 mm) apparatus. The rolling speed was controlled to be around 1.0-1.5 cm-s. The final thickness t was measured when these samples were fractured after repeated rolling process. The total degree of deformation, $\varepsilon = (t_0 - t)/t$, where t_0 denote initial thickness of the samples. The electrical conductivity was tested by eddy current method on a D60K-1201 digital conductivity meter.

2.5. Simulation of interfacial residual stresses

Interfacial residual stresses generated during the Vickers Hardness Testing were simulated using Finite element model (ANSYS 15.0). A three-dimensional model with axisymmetric axis (X, Y and Z) was built according to the morphology of three

phases in bulk composites. The size of Al_2O_3 and Cr added to copper matrix is defined 100 nm and 60 nm, respectively. A 3-D mechanical element Solid 186 was employed in a mechanical finite element solution, which has twenty nodes and a maximum of three degrees of freedom per node. All materials are considered homogeneous and isotropic.

Tab.1 Parameters of high energy ball milling process

Results and discussion

Fig.1 shows the SEM images of the pre-milled Cr- Al_2O_3 composite powders. As shown in Fig.1 (a), Cr and Al_2O_3 particles were uniformly dispersed in composite powders after high energy milling. The particle size of Cr (light colored phase) indicated from Fig.1 (b) was refined to several microns as a consequence of repeated fracturing during milling process, which is much smaller than the initial size ($\sim 100 \mu\text{m}$) as shown in the inset image in Fig.1 (b).

Fig. 1 SEM images of (a) Pre-milled Cr- Al_2O_3 particles; (b) magnified image, inset is raw Cr particles.

The cross-section SEM images of polished copper matrix composite powders, which were aging treated at 500°C for 100 min after mechanical milling, are shown in Fig.2. The composite powders of C5A were refined to several microns during high energy milling as shown in Fig.2 (a). As shown in the magnified image in Fig.2 (b), Al_2O_3 nanoparticles were uniformly dispersed in copper matrix without Al_2O_3 aggregations. The SEM image of C1C4A composite powders is shown in Fig.2 (c), the Cr- Al_2O_3 hybrids were homogeneously distributed in copper matrix. As shown in the magnified image inset in Fig.2 (d), the corresponding EDS spectrum indicates that the Cr nanoparticles (green dotted circles) were dispersed around Al_2O_3 nanoparticle. It is the large numbers of interfaces in copper matrix introduced by Al_2O_3 nanoparticles that act as heterogeneous nucleation sites for Cr to precipitate on during aging treatment [25, 26]. Precipitated Cr nanoparticles tend to preferentially nucleate on the heterogeneous nucleation sites [27].

The copper matrix composite powders were further analyzed by X-ray diffraction (XRD). The XRD patterns of the non-milled, as-milled and aging treated C1C4A composite powders are shown in Fig.3. The diffraction peaks of Cr and Al_2O_3 are invisible due to their low weight percentage. The broadened diffraction peaks with decreased peak intensity indicate a crystallite refinement for as-milled copper

compared with the non-milled copper [28]. The slight shift of copper peaks to lower angle after milling suggests the lattice parameter of copper increases slightly. This shift is in agreement with reference [29] and could be mainly explained as the solution of Cr in Cu during the high energy milling process. The peak intensity of copper increased and a shift to higher angle after aging treatment. This is considered due to that Cr solute atoms were driven out from the solid solution and preferentially precipitated on the heterogeneous nucleation sites during aging treatment.

Fig. 2 SEM images of (a), (b) C5A composite powders; (c), (d) and C1C4A composite powders.

Fig. 3 XRD patterns of non-milled, as-milled, and aging treated C1C4A composite powders.

Fig. 4 shows the SEM images of copper matrix composites. High densification was achieved for C5A composite as shown in Fig.4 (a). Al_2O_3 nanoparticles were uniformly distributed in Cu matrix with an average particle spacing of 300 nm, and no Al_2O_3 aggregations are observed in Fig.4 (b). Fig.4 (c) shows the macroscopic morphology of C1C4A composite, the Al_2O_3 -Cr hybrids were homogeneously dispersed in copper matrix. It is notable that the precipitated Cr nanoparticles were distributed on or near Al_2O_3 nanoparticle forming a “discontinuous nano-sized Cr transition zone” as shown in the magnified image inset in Fig.4 (d). The formation of this discontinuous nano-sized Cr transition zone is attributed to the nucleation of precipitated Cr on the heterogeneous nucleation sites in copper matrix. As is generally accepted in most precipitation processes, heterogeneous nucleation of solute Cr occurs at those preferential nucleation sites such as grain boundary, dislocation, and second phase [25]. As a matter of fact, the addition of Al_2O_3 nanoparticles in copper matrix increases the volume fraction of interface. And Al_2O_3 nanoparticle itself acts as second phases to provide heterogeneous nucleation sites. Interfaces of Cu- Al_2O_3 as well as Al_2O_3 nanoparticles are potentially ideal heterogeneous nucleation sites for solute Cr to precipitate on.

Fig.5 shows the TEM image of interfacial microstructures in C1C4A composite. The bright field image (BF) and dark field image (DF) of C1C4A composite are shown in Fig. 5 (a) and (b), respectively. The corresponding selected area diffraction pattern (SAD) inset in Fig. 5 (b) identifies the particle in DF image to be copper with crystallographic plane of (111). The corresponding SAD patterns also reveal the

presence of Cr and Al_2O_3 phases, which has crystallographic plane of (200) and (012), respectively. Fig. 5 (c) and (d) are the HRTEM images of the interface of Cu-Cr- Al_2O_3 and Cu- Al_2O_3 magnified from the dotted box 1 and 2 in Fig. 5(a), respectively. Cr nanoparticle precipitated on the surface of Al_2O_3 nanoparticle as shown in the irregular dotted green shape in Fig. 5 (c), Cr nanoparticle with (200) crystallographic plane was well bonded with Al_2O_3 on (012) crystallographic plane. A fuzzy interface was formed between Cu and Cr, which could be attributed to the solid solution reaction between Cu and Cr. The incoherent interface between Cu and Al_2O_3 were clearly shown in Fig. 5 (d). The interfacial bonding state varied from mechanical locking (Cu- Al_2O_3) to chemical bonding (Cu-Cr) and mechanical locking with higher load transfer and crack resistance (Cr- Al_2O_3) as Cr precipitated at the interface of Al_2O_3 nanoparticle and copper matrix.

Fig. 4 SEM images of C5A composite (a), (b); and C1C4A composite (c), (d).

Fig. 5 TEM image of C1C4A composite. BF (a), DF and corresponding SAD (b), HRTEM of the interface of Cu-Cr- Al_2O_3 (c) and Cu- Al_2O_3 (d).

Fig. 6 shows the hardness-temperature curves of copper matrix composites. Significant increment in hardness for C1C4A and C5A composite is observed compared with that of pure copper. While it is of great importance to notice that the hardness of C1C4A composite is higher than that of C5A composite during the whole elevated temperatures. The significant hardness increment ratio started from 700 °C indicates that the hardness retention is increasing with the increasing temperatures, and it can be as high as 133% at 1000 °C.

The compressibility and electrical conductivity of copper matrix composites are shown in Tab.2, both of which are 36% and 19% higher for C1C4A composite compared with C5A composite, respectively. The increment in compressibility and electrical conductivity is attributed to replacing 1wt% Al_2O_3 with 1wt% Cr for C1C4A composite compared with C5A composite. The decrease in compressibility for C5A composite compared with pure copper is mainly attributed to the dislocation pinning effect of Al_2O_3 nanoparticles. The dislocation sliding was usually inhibited by the incoherent interface formed between Al_2O_3 and copper, while the volume fraction of incoherent interface in C1C4A composite reduced by replacing 1 wt% Al_2O_3 with 1 wt% Cr. And the interfacial bonding is improved for the formation of chemical bonding between Cu and Cr, the compressibility is consequently improved for C1C4A

composite compared with C5A composite.

Fig.6 Hardness curves of copper matrix composites at elevated temperatures, and increment ratio curve (the ratio between the hardness increment of C1C4A composite compared with C5A composite and the hardness of C5A composite).

Tab.2. Compressibility and electrical conductivity of copper matrix composites

The corresponding hardness indentation of C5A composite at room temperature is shown in Fig.6 (a). Micro-cracks were formed at the interface between Al_2O_3 nanoparticle and copper matrix during the loading process as shown in the magnified indentation image in Fig.6 (b). The area of hardness indentation is smaller for C1C4A composite (Fig.6 (c)) compared with that of C5A composite (Fig.6 (a)) at room temperature, which is in agreement with the corresponding hardness values. And no crack was observed in the magnified image of hardness indentation of C1C4A composite in Fig.6 (d).

The increment in hardness and compressibility for C1C4A composite are related to the discontinuous nano-sized Cr transition zone between Al_2O_3 nanoparticle and copper matrix. Cr precipitates nucleated on the interface of Cu- Al_2O_3 is considered to improve the interfacial bonding between Cu and Al_2O_3 . A good chemical bonding was formed between Cu and Cr due to the solid solution reaction, which helps reduce the interfacial stress under external stress compared with the mechanical bonding between Al_2O_3 nanoparticle and copper matrix. From another point of view, micro-crack is considered to be the predominant cause for the failure of C5A composite, which is caused by the interfacial debonding induced by the severe interfacial stress between Al_2O_3 nanoparticle and copper matrix, finally results in the decreased hardness and compressibility for C5A composite. In addition, the obvious increase in hardness increment ratio started from 700 °C are mainly attributed to the much lower misfit in CTEs between Cr and Al_2O_3 compared with that between Cu and Al_2O_3 . Voids formed at the interface between copper matrix and Al_2O_3 nanoparticles as the CTE misfit increased with increased temperatures, which consequently resulted in interfacial debonding and further lead to interfacial micro-cracks with externally applied stress [30]. The strength of C5A composite hence decreased because of the interfacial micro-cracks.

Fig. 7 Hardness indentation of C5A composite (a), (b), and C1C4A composite (c), (d).

In order to take a deeper insight into the effect of the discontinuous nano-sized Cr transition zone on improving the interfacial stress between Al_2O_3 and Cu matrix, the interfacial residual stress generated during the Vickers Hardness testing was simulated using ANSYS finite element analysis code. As shown in Fig.8 (a), the schematic view of this 3-D model is made of the central Al_2O_3 nanoparticle surrounded by discontinuous transition zone of precipitated Cr nanoparticles, finally incorporated by the continuous Cu matrix. Due to the solid model entities, tetrahedron grid was selected for mesh construction. The second phases were finely meshed to enhance the sensitivity to the rigorous changes in stress distribution for this model. Due to axisymmetric configuration of the model, symmetric boundary conditions were defined as plane 2, 3, 4 and 5 as show in Fig.8 (b). The mesh model is built up in two representative forms, one is the precipitated Cr particles connected (on plane 3) with Al_2O_3 and the other unconnected (on plane 2). The model was subjected to a uniform compressive load of 5 N normal to plane1, and the displacement in X direction for plane 6 is constrained.

The distribution of residual stresses in copper matrix composites along the x-axis are shown in Fig.8 (c) and (d), the simulation results confirm that the maximum compressive residual stress σ_x is the interfacial residual stress, and the interfacial residual stress of Cu- Al_2O_3 (39.4 MPa) is higher compared with that of Cu-Cr (7.67 MPa) and Cr- Al_2O_3 (33.6 MPa) as shown in Fig.8 (c) and (d). The higher interfacial residual stress in C5A composite is responsible for the interfacial debonding, and hence results in the formation of interfacial micro-cracks. On the other hand, the C1C4A composite with decreased interfacial residual stress are capable of bearing higher external applied stress. Consequently, the discontinuous nano-sized Cr transition zone between copper matrix and Al_2O_3 nanoparticle reduced the interfacial residual stress, improved the interfacial bonding and finally resulted in the increase in hardness and compressibility for C1C4A composite.

Fig. 8 Schematic view of the 3-D numerical model (a); mesh model of the interfaces in copper matrix composites (b); contour of residual stresses field for interfaces in Cu- Al_2O_3 (c), and Cu-Cr- Al_2O_3 composite (d).

Conclusions

Cu-Cr- Al_2O_3 composite was fabricated by mechanical milling and vacuum hot pressing. The nano-sized Cr precipitates nucleating on the interface of Cu- Al_2O_3 formed discontinuous nano-sized transition zone around Al_2O_3 particle. Both the FEM

simulation results and the hardness indentation confirm that the interfacial residual stress decreased and the interfacial bonding improved with the precipitated Cr at the interface of Al_2O_3 and copper matrix. The hardness and compressibility are hence improved with the addition of Cr for Cu- Al_2O_3 composite.

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Tab.1 Parameters of high energy ball milling process

	Number	Nominal composition	Milling time, h	Milling speed, rmp	Ball/ powder ratio
Pre-milling		Cr-Al ₂ O ₃ (1:4 in weight)	2		
Milling	C5A	Cu-5.0 wt% Al ₂ O ₃	3	500	10:1
	C1C4A	Cu-1.0 wt% Cr-4.0 wt% Al ₂ O ₃			

Tab.2. Compressibility and electrical conductivity of copper matrix composites

	Pure copper	C5A	C1C4A
Compressibility (ϵ , %)	600	110	150
Electrical conductivity (IACS %)	100	52	62