Microstructural Characteristics and Mechanical Behavior of Spark Plasma-Sintered Cu–Cr–rGO Copper Matrix Composites

Xin-Jiang Zhang¹ · Zhong-Kui Dai¹ · Xue-Ran Liu¹ · Wen-Chao Yang² · Meng He¹ · Zi-Run Yang¹

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Abstract

Copper matrix composites were prepared through spark plasma sintering (SPS) process, mixing fixed amount of reduced graphene oxide (rGO) with the different amounts of Cr. In the sintered bulk composites, the layered rGO network and uniform Cr particles distributed in the Cu matrix. Both of mechanical blending and freeze-drying stages of the wet-mixing process obtained the Cu/Cr/rGO mixture powders, and then SPS solid-phase sintering realized the faster densification of these mixture powders. The hardness and compressive yield strength of the Cu–Cr–rGO composites depicted the higher values than those of pure Cu and single rGO-added composite, and they were gradually increased with increasing Cr. The rGO/Cr hybrid second-phases are believed to be beneficial to strengthening Cu matrix. The relevant formation and strengthening mechanisms involved in Cu–Cr–rGO composites were discussed.

Keywords Metal matrix composites (MMC) · Powder metallurgy · Sintering · Mechanical properties

1 Introduction

Copper matrix composites (CMCs) are considered as potential materials in industrial application due to their high strength, high electrical conductivity and excellent self-lubricating performance [1, 2]. As the attractive fillers, various carbon materials, e.g., graphite [3, 4], carbon fiber [5] and carbon nanotube [6], have been extensively used for developing high-performance CMCs. These reinforcing fillers achieved the significant enhancements of hardness, strength and electrical conductivity of Cu matrix [3–6]. It is worth to note that the Cr incorporation has often necessary in the graphite-reinforced [7, 8], carbon nanotubes-reinforced [9] or carbon fibers-reinforced [5] CMCs. Their hybrid additions have developed as one of the efficient strategies to improve properties of Cu matrix, such as hardness, strength, wear resistance and thermal

conductivity [5, 7–10]. For instance, the Cr-incorporated Cu–carbon nanotube composites by the milling and hot-pressed sintering exhibited the improved hardness, strength and interfacial bonding [9]. The Cr-added Cu–graphite composites possessed a lower wear rate than pure Cu and Cu–Cr alloys [7]. The solid-state sintered Cu–Cr composites made the strong interaction at Cu/Cr interface and dissolved Cr atoms to strengthen the composites [11].

Recently, the graphene has demonstrated its excellent properties, such as super charge-carrier mobility and strengths [12, 13]. It has been attempted as the reinforcement filler of metal matrix composites, and provided better enhancement of mechanical and tribological properties toward metal matrix [13, 14]. The graphene-added CMCs were found to maintain high electrical conductivity without any strength loss [14]. Interestingly, the graphene/Cu composites exhibited the excellent interfacial bonding and reinforcement effect [14]. However, a large number of studies focused on the single rGO-incorporated Cu composites. Few efforts have made to optimize the graphene–copper composites by using alloying component. As the two-dimensional layered carbon atoms, the graphene shows the large structural difference with graphite, carbon nanotubes and carbon fibers. It is essential to evaluate the effectiveness of the alloying component for the performance modification of Cu–graphene composites.

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✉ Xin-Jiang Zhang
zhangxj1983@yahoo.com

¹ School of Materials Science and Engineering, Yancheng Institute of Technology, Yancheng 224002, China

² College of Materials Science and Engineering, Guangxi University, Nanning 530004, China

In the present study, the novel CMCs containing a fixed amount of rGO and different Cr contents were synthesized by SPS. Microstructural characteristic and mechanical behavior of the as-prepared Cu–Cr–rGO composites were investigated in comparison with those of single rGO-added composite. Also, microstructure and strengthening mechanism of such composite were analyzed and discussed.

2 Experimental Procedure

Figure 1 shows the schematic representation of fabrication process of CMCs corresponding to Cu–Cr–rGO system. GO was firstly prepared by a modified Hummer's method, using purified natural graphite as the starting materials [15]. The micrographs of starting Cu and Cr powders are shown in Fig. 2. Under the mechanical stirring, Cu powders (particle size: ~ 500 mesh, 99.9% purity), Cr powders (particle size: ~ 400 mesh, 99.9% purity) and 80 wt % $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ were added into GO aqueous dispersion, forming a mixed powder slurry. After the mixed slurry was mechanically stirred for 2 h, it was frozen at -60°C . The frozen mixtures were maintained under vacuum (1 Pa) to remove the water by sublimation in a LGJ-12 freeze-dryer, obtaining the freeze-dried Cu/Cr/rGO powder mixtures. These mixtures were compacted at a pressure of 450 MPa to form a cylindrical-shaped green compact. Finally, the compact powders were sintered by SPS (SPS-211Lx) at 750°C for 8 min under vacuum, and the heating rate was $50^\circ\text{C}/\text{min}$. A constant pressure of 45 MPa was applied for the entire sintering process. In this work, the samples with nominal composition of Cu– x Cr–2.5 wt% rGO (x : 0, 0.5, 2.0 and 4.0 wt%), which respectively termed as 0Cr, 0.5Cr, 2Cr and 4Cr samples, were prepared under the same experimental conditions. For

meaningful comparison, the as-sintered pure copper was also fabricated using the same SPS processing.

The phase constitutions were characterized by X-ray diffraction (XRD) via Rigaku D/max-2200 diffractometer. In order to detect rGO and/or Cr, the concentrated nitric acid was used to entirely remove Cu matrix of the sintered composites, and then the solution was filtered by suction filtration. Microstructural observations were carried out using a scanning electron microscope (SEM, FEI Quanta 200F) equipped with backscattered electron detector (BSE) and energy-dispersive X-ray analysis (EDX). A Tecnai G2 F30 field emission gun transmission electron microscope (TEM) was used for observations. The hardness was tested using a Brinell hardness tester (HBE-3000A) at a load of 1226 N with a holding time of 15 s. The eight indentations of each sample were measured. Compression test was performed using an Instron 8801 testing machine under an initial strain rate of $3.70 \times 10^{-4} \text{ s}^{-1}$ at room temperature. The test sample was a cylinder shape with 4 mm in diameter and 8 mm in length. Each bulk composite was evaluated by at least three compressive specimens.

3 Results

3.1 Phase Identification

XRD patterns of the dried Cu/Cr/rGO powder mixtures are shown in Fig. 3a. It can be found that the diffraction peaks of face-centered cubic (fcc) Cu phase appeared in all powder mixtures. After SPS, the diffraction peaks of fcc-structured Cu were also identified in each as-sintered specimen (Fig. 3b). However, it was noted that the characteristic peaks of rGO and/or Cr phases were not detected,

Fig. 1 Schematic representation of fabrication process of CMCs corresponding to Cu–Cr–rGO system

Fig. 2 SEM micrographs of Cu **a**, Cr **b** powders used as the raw materials

Fig. 3 XRD patterns of the dried powder mixtures **a**, the sintered composites **b**

possibly because their peaks were relatively very weak compared with the strong peaks of Cu.

For further detected possible rGO and/or Cr phases, the starting GO and the as-sintered bulk composites removed from Cu matrix were investigated and shown in Fig. 4. A strong diffraction peak appeared at $2\theta = 9.8^\circ$ for GO (Fig. 4a), being also confirmed by the previous work [16]. It was noted that the reflection peak at $2\theta = 9.8^\circ$ disappeared completely in each as-sintered composite, but an abroad peak of rGO at $2\theta = 27.1^\circ$ appeared (Fig. 4b–e). From Ref. [17], a broad reflection peak of graphene obtained by $\text{N}_2\text{H}_4\text{-H}_2\text{O}$ reduced GO presented at $2\theta = 27.1^\circ$. Thus, it is believed that the GO is effectively reduced to form rGO in the composites. Meanwhile, the characteristic peaks ascribed to body-centered cubic (bcc) Cr phase were also found in each as-sintered composite with Cr addition, as shown Fig. 4c–e. These results further confirmed the existence of rGO and/or Cr phases in the sintered composites.

Fig. 4 XRD patterns of GO **a**, rGO collected from the sintered 0Cr composite **b**, rGO/Cr mixtures collected from the sintered 0.5Cr **c**, 2Cr **d**, 4Cr **e** composites

3.2 Microstructure

Figure 5 shows the SEM micrographs of the dried powder mixtures showing rGO distribution. In the powder mixtures from 0Cr to 4Cr sample, some layered rGO sheets containing some wrinkles were observed. Some previous reports about pure rGO and its composites also showed the layered structure of rGO sheets [18–20]. Moreover, some corrugated rGO sheets distributed clearly among the electrolytic Cu particles. The large hyaline rGO sheets covered on the Cu particles, and some rGO nanosheets embed into the hole of electrolytic Cu particles. Thus, these results indicated that the rGO sheets had been randomly distributed in the powder mixtures.

Figure 6 shows that SEM-BSE micrographs of the sintered bulk composites. Microscopic observation shows that the irregular rGOs distribute in the Cu matrix and form small agglomerations. The Cr particles with irregular shape were uniformly distributed in the whole microstructure, and more Cr particles were observed in the composites with higher Cr content. Figure 7 shows the EDX results from different phases in the bulk Cu–Cr–rGO composites. The strong Cu and Cr peaks were, respectively, detected in Cu matrix (Fig. 7b, e) and Cr particles (Fig. 7d, f), and the

stronger peak of carbon appeared in the rGO agglomerations (Fig. 7c). These results are consistent with XRD analysis (Figs. 3b, 4), indicating that Cu/Cr/rGO mixtures underwent a rapid sintering and could be maintained in the final bulk samples.

In order to further observe rGO and Cr, the metallographic sample of the sintered composites was corroded by the concentrated nitric acid to remove Cu matrix. The corresponding SEM observation results are shown in Fig. 8. Interestingly, the rGO sheets exhibit a three-dimensional network in the sintered composites, and Cr particles are irregular. The rGO coated on the Cr particles was also found. These results indicated that the sintered composite was mainly composed of Cu, rGO and Cr mixture, and the three-dimensional rGO network and irregular Cr particles were introduced successfully in the bulk composites.

3.3 Hardness and Compressive Properties

The sintered Cu–Cr–rGO composites had higher HB values (0.5Cr (HB47.4), 2Cr (HB55.5) and 4Cr (HB63.3)) than Cu–rGO composite (HB46.5) and pure Cu matrix (HB45.4), and HB value of them gradually increased with

Fig. 5 SEM micrographs of the dried powder mixtures showing rGO distribution: **a** 0Cr, **b** 0.5Cr, **c** 2Cr, **d** 4Cr

Fig. 6 SEM-BSE micrographs of the sintered bulk composites by SPS: **a** 0Cr, **b** 0.5Cr, **c** 2Cr, **d** 4Cr. The insets show the high-magnification micrographs of the rGO

the increase of Cr content. Here, the HB value for 4Cr composite was enhanced by about 36.1% compared with 0Cr composite. An increase in the hardness of Cu matrix composites of Cu–Cr or Cu–graphene systems was also confirmed [12, 21, 22]. It illustrates the substantial hardening of Cr/rGO hybrids to Cu matrix.

Room-temperature compressive stress–strain curves of the as-sintered bulk composites are shown in Fig. 9. The compressive 0.2% yield strength ($\sigma_{0.2}$) is 109.4 MPa for 0Cr composite and 89.6 MPa for pure Cu matrix. Surprisingly, the higher $\sigma_{0.2}$ values are obtained for the composites from 0.5Cr to 4Cr, which are 114.3 MPa (0.5Cr), 122.5 MPa (2Cr) and 128.2 MPa (4Cr), respectively. More Cr addition results in the higher strength of composites. The 28.2 and 24.4% compressive strains were, respectively, obtained in the composites 2Cr and 4Cr, which were lower than the composites of 0Cr and 0.05Cr.

3.4 Deformed Microstructure and Compressive Fractography

To further investigate deformation evolution of the as-sintered bulk composites, the deformed microstructure of composite 4Cr was examined, as shown in Fig. 10. As seen

from Fig. 10a, b, Cu matrix was elongated along the direction perpendicular to the compressive loading, and some Cr particles and parallel rGO stripes were also found. Further observation (Fig. 10c) shows the bent rGO stripes on the upper and under leaves of the Cr particles. TEM observations of deformed microstructures of the composites are shown in Fig. 11. It can be seen that Cu matrix contained a high density of dislocations, and the blocking of rGO to dislocations containing in Cu matrix was also found. These results indicated that the Cu matrix underwent the large-scale plastic deformation during the compression process, and the three-dimensional rGO networks were extruded to form the banded rGO. The Cr particles acted as a crucial role to transfer the load during the deformation.

SEM images of compressive fractography of the sintered bulk composites are shown in Fig. 12. The observation from Fig. 12a–c shows some broken, pulled-out and warped rGO, indicating the load supporting during the compressive test. Interestingly, the broken Cr particles were also found on the fracture surfaces, and some tiny cracks existed on Cr particle/Cu matrix interfaces (Fig. 12d). These results indicated that the strength response of the as-sintered bulk composites was related to the Cu matrix, rGO sheets and Cr particles.

Fig. 7 High-magnification SEM-BSE image **a** and EDX results **b–d** of the sintered composite: **a** SEM-BSE image, **b** A point, **c** B point, **d** C point

Fig. 8 SEM micrographs of rGO and Cr in the sintered composites: **a** low-magnification image; **b** rGO; **c**, **d** Cr particle, where **d** is the higher-magnification image from the rectangle frame in **c**

Fig. 9 Room-temperature compressive stress–strain curves of the sintered bulk composites

4 Discussion

4.1 Microstructural Formation

The as-prepared Cu–Cr–rGO bulk composites were obtained from the sintering of powder mixtures. The

fabrication process of such composites involved the mechanical blending, freeze-drying and SPS rapid sintering of Cu/Cr/rGO powder mixtures. From the preparation, the $\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$ solution and Cu/Cr starting powders were added into the mechanically stirred GO aqueous dispersion. The $\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$ as a strong reductant could reduce effectively GO [23, 24]. Under the mechanical stirring, the Cu/rGO/Cr powders were intensively mixed in the slurry. Further, the frozen Cu/Cr/rGO mixtures slurry fixed the distribution state of rGO and Cr in the Cu matrix. The solidified water was removed by sublimation to retain the templated Cu/rGO/Cr mixture powders. Finally, the Cu/Cr/rGO mixture powders were sintered by SPS to obtain the bulk composites. After sintering, the rGO network and homogeneously distributed Cr particles were formed in the as-prepared composites (Figs. 6b–d, 8).

From Cu–C phase diagram [25], the solid and liquid Cu and C were always in a balanced state. No compound could be produced between Cu and C. In Cu–Cr phase diagram [26], it indicated a eutectic reaction of $\text{L} \leftrightarrow \text{Cu} + \text{Cr}$ at $\sim 1077^\circ\text{C}$, and no Cu–Cr compound was produced. Moreover, the maximum solubility of C or Cr in Cu was, respectively, ~ 0.03 and ~ 0.76 at.% [25, 26]. Fang et al. [11] and Liu et al. [27] used, respectively, the sintering and

Fig. 10 SEM-BSE micrographs of the room-temperature deformed 4Cr composite: **a** low-magnification image, **b** banded rGO agglomeration, **c** deformed rGO around Cr particles. The dark arrows indicate the compressive axis

Fig. 11 TEM micrographs obtained from the deformed sample showing a high density of dislocations in Cu matrix. The inset shows the blocking of rGO to dislocations

melting methods to prepare Cu–Cr materials, further confirming that there was no compound formed between Cu and Cr. The sintered Cu–graphite and Cu–carbon nanotube composites showed both Cu and graphite-like C phases [28, 29]. The reported Cu–Cr/graphite system showed the formation of Cr_3C_2 or Cr_7C_3 phase at the high temperature

(> 1100 °C) [30, 31]. For instance, Devincent and Michal [30] confirmed that the 0.61–1.22 at.% Cr-added Cu–Cr/graphite system produced the stable Cr_3C_2 at 1130 °C for 1 h. Voitovitch et al. [31] determined the formation of Cr_7C_3 phase at the Cu–Cr/vitreous carbon interfaces after dwelling at 1100 °C for 5 min. However, Gautam et al. [7] suggested that only both Cr and graphite particles existed in the hot forged (700 °C) and annealed (650 °C for 1 h) Cu–Cr–graphite composites. In this work, the composites were sintered at 750 °C for 8 min. Cu–Cr–rGO mixture powders were rapidly heated at a low temperature in a short duration, and the reaction of Cr and rGO could not occur. SPS faster densification produced the Cu–Cr–rGO bulk composites.

4.2 Deformation mechanism

The as-sintered Cu–Cr–rGO composites exhibited the higher strength. For the composites with a fixed amount of rGO, more Cr particles led to a better strengthening effect (Fig. 9). The hybrid Cr/rGO contributed to the strengthening of bulk composites. When the metal matrix composites bore the load, the hard particles transferred the loading from the deformed matrix [32]. The hard Cr

Fig. 12 SEM images of compressive fractography of the sintered bulk composites showing **a** broken rGO, **b** pulled-out rGO, **c** warped rGO, **d** fractured Cr particles (white gridlines). The white arrows in **a–c** mark the rGO. The black arrows in **d** mark the tiny cracks

particles were uniformly distributed in the Cu matrix. The load transfer could occur from plastic Cu matrix to the irregular Cr to improve the strength (Fig. 10). The content of Cr particles increased to further enhance the capacity of load transferring. Higher Cr content led to higher pile-up dislocations. Consequently, the higher stress was provided to deform the composite to increase the strength. During the deformation, the fragmented and network rGO were forced by shear flow, resulting in a preferential banded structure of rGO along the elongated Cu grains. The rGO could also play a role in penning the dislocation motion (Fig. 11). The dislocation pile-up formed near the grain boundaries could result in the strengthening effect [12, 33]. Meanwhile, the Cr particles caused the bending of rGO stripes (Fig. 10). Such an aligned and bended rGO could also create the dislocations accumulation, contributing to the strength increment. Moreover, the large plastic deformation of Cu matrix occurred during the compression process. An increase in dislocation densities in the strain-hardened regions could be generated by large mismatch among the constituent phases [32]. The interaction among different dislocation areas also contributed to the strength increment of the composites [34]. The different constituent phases possessed the different deformation compatibility,

so that the stress easily concentrated near the interfaces to cause the micro-crack initiation during the compressive deformation. Thus, rGO was broken, pulled-out and warped, and Cr particles were broken (Fig. 12).

5 Conclusions

CMCs corresponding to Cu–Cr–rGO system were successfully prepared by SPS. The rGO incorporated with Cr was randomly mixed with Cu particles in the dried composite powders. As-sintered bulk composites contained the layered rGO network and uniformly distributed Cr particles. The room-temperature compressive yield strength and hardness of the as-sintered Cu–Cr–rGO composites were improved significantly, compared to pure Cu matrix and single rGO-added composite at the same SPS processing. More Cr addition resulted in the higher hardness and compressive yield strength of composites.

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