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# Fabrication of RGO/Cu composites based on electrostatic adsorption

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**Abstract:** To address the issues of reduced graphene oxide (RGO) dispersion in copper (Cu) matrix and interface bonding between RGO and Cu, an electrostatic adsorption method with interface transition phase design was employed to prepare the RGO/Cu based composites. Cu–Ti alloy powder was employed to improve the combination by forming carbides at the RGO–Cu interface. It was noted that the mechanical property of 0.3wt.%RGO/Cu–Ti composite was increased by 60% compared with that of the matrix. Strengthening mechanism analysis suggested that the enhancement of the mechanical property was ascribed to the load transfer and second phase strengthening which were from the improved dispersion of RGO and the in-situ formed titanium carbide phase.

Key words: graphene oxide (GO); electrostatic adsorption; Cu–Ti; in-situ  $Ti_xC_y$ ; mechanical property

## **1** Introduction

Applications of graphene oxide (GO) or reduced graphene oxide (RGO) in metal matrix composites (MMCs), which offer excellent mechanical properties and comprehensive performance, have received considerable concern in recent years [1,2]. Conventional preparation methods have certain limitations to address the problems faced in preparing RGO/MMCs, which include uniform dispersion of graphene (GR) reinforcement in metal matrix without agglomeration and strong interface bonding between carbon strengthening phase and the metal matrix [1,3].

Uniform dispersion and distribution of GR in metal matrix are essential to improve the properties

of MMCs [4,5]. Electrostatic adsorption, a one-step method without the problem of introducing additional impurities, can be employed to disperse the GR in the copper matrix. This novelty method is built on the zeta potential difference between Cu and GR particles. A huge amount of chemical functional groups on the surface of GO give them hydrophilic properties and make them have a negative zeta potential in hydrochloric acid solution. At the same conditions, however, Cu powder has a positive zeta potential because of the weaker electronegativity Cu atom than that of chlorine atom. Hence, by adjusting and controlling the pH value of the solution where the two kinds of powders soaked in can attract them together with a well-dispersed and distributed GO [6].

However, GO has poor wettability with the Cu matrix, which is not beneficial to transfer the

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Jian-hong YI, Tel: +86-13808794121, E-mail: yijianhong@kmust.edu.cn DOI: 10.1016/S1003-6326(20)65270-0 load efficiently. To improve the interface bonding, the third element atoms are usually added to form carbide at the interface. For example, graphite/ Cu–Cr composites can generate chromium carbide to make the interface bond stronger [7,8]. However, limited attention is paid to the in-situ generated titanium–carbide reinforced copper based composites.

In this research, the electrostatic adsorption method for preparing composite powder was used. Then the interface bonding between Cu and RGO was investigated by using Cu-Ti alloy powder to obtain an in-situ generated titanium-carbon transition phase. This experimental method had not been reported before. Due to the important influence of these compounds on the mechanical properties of RGO/Cu-Ti composites. the microstructure and strengthening mechanism of RGO/Cu-Ti composite were discussed underlying titanium-carbon compounds formation. Moreover, this research could effectively balance the integrity and dispersion of the composite enhanced phase (nanometer carbide phases and RGO phase) in the preparation, and achieve a good improvement in the mechanical performance of composite materials.

## 2 Experimental

When GO (or RGO) is introduced into the Cu powders, the tendency of agglomeration between their powders should be conquered. Hence, we produced RGO/Cu composite powders by a simple electrostatic adsorption technique, which can enhance the dispersion of RGO in the Cu matrix.

A schematic diagram of the fabrication process is given in Fig. 1. First, the pretreated GOs (0.5-5  $\mu$ m in lateral size and 0.8–1.2 nm in thickness) were dispersed in deionized water by ultrasonic for 15-20 min. At the same time, electrolytic Cu powder (99.9% purity, ~44 µm in average particle size) was ball milled into the flaky shape and scattered in the deionized water under mechanical stirring. Next, the two suspensions were mixed together under the condition of continuous stirring, when the pH of the Cu powder suspension was adjusted to ~4.0 by adding HCl solution. After filtering and drying, micro-sized GO/Cu<sub>2</sub>O composite powder was prepared. As-prepared micro-sized GO/Cu<sub>2</sub>O composite powders with different contents of GO were mixed with Cu-1.0wt.%Ti alloy powders using a low-energy ball milling. RGO/Cu-Ti composite powder was obtained by thermal reduction at 553 K for 6 h with  $N_2+H_2$ . According to the nominal mass fraction of RGO in the mixed powder, the samples were marked as 0, 0.3%, 0.6% and 0.9%, respectively. Finally, the powders were sintered and densified by spark plasma sintering (SPS, LABOX-650F furnace). SPS was operated at 1023 K under a vacuum atmosphere. Heating rate was 373 K/min and held at 1023 K for 10 min, it should be noted that it was necessary to maintain a pressure of 50 MPa from the start to the end of the sintering.

The functional group of the GO was characterized by Fourier transform infrared spectroscopy (FTIR; Nicolet IS10). The morphology and microstructure of RGO/Cu–Ti were obtained by scanning electron microscopy (SEM, Nova



Fig. 1 Schematic of fabrication process of RGO/Cu-Ti nanocomposite

Namo-450, FEI), transmission electron microscopy (TEM, Tecnai G2-TF30, S-Twin) and X-ray diffraction (XRD, RINT, Rigaku, Japan) with Cu K<sub>a</sub> radiation. Structural defects in the GO and RGO/Cu-Ti composite powders were investigated using Raman spectroscopy (Labram HR Evolution, HORIBA JOBIN YVON Corp, French). The density of composites was evaluated according to the principle of Archimedes. Vickers microhardness of samples was measured by MC010 microhardness analysis system (Shanghai Microcre Light-Machine Tech Co., Ltd.) with a load of 0.98 N applied for 15 s. Tensile test of RGO/Cu-Ti composites was conducted on SHIMADZU equipment (AG-X plus, Japan) at ambient temperature with a tensile rate of 0.2 mm/min. Each strength value represents the average result of four measurements.

### **3** Result and discussion

## 3.1 Starting materials

Figures 2(a)–(c) illustrate that the spherical Cu–1.0wt.%Ti alloy powder has a correspondingly wide size distribution of  $1-50 \mu m$ , and EDS mapping images show the uniformly distributed Ti element in the Cu–Ti alloy powders. TEM image of GO (Fig. 2(d)) reveals a typical pleated sheet structure of GO. The functional groups of GO are

identified according to the FTIR spectrum presence demonstrating the (Fig. 2(e)), of C-O (~1118 cm<sup>-1</sup>), C-H (~2924 cm<sup>-1</sup>), O-H  $(\sim 3450 \text{ cm}^{-1})$  and C=C  $(\sim 1667 \text{ cm}^{-1})$  on the GO. These groups provide GO hydrophilic properties and show a negative zeta potential under HCl conditions. On the contrary, Cu powder has a positive zeta potential because the Cu atom has weaker electronegativity than the chlorine atom in the same environment. This zeta potential difference indicates the feasibility of electrostatic adsorption between Cu and GO in theory [9,10].

#### **3.2 Composite powders**

To increase the specific surface area, the spherical Cu powders were transformed into flake powders by the ball-milling process, which was beneficial to the adsorption of Cu powder with GO (Fig. 3(a)). Morphology of the GO/Cu<sub>2</sub>O composite powders prepared by electrostatic adsorption shows that all powders are turned into small block-shape particles during the electrostatic adsorption and no flaked powders are observed, even though the process only lasts ~10 min (Fig. 3(b)). Meanwhile, some Cu<sub>2</sub>O powders form on both sides of the GO, suggesting a well-dispersed GO in the composite powders. Our previous studies have found that these cubic powders are cuprous oxide



**Fig. 2** Morphology of Cu–Ti alloy powder (a) and corresponding EDS mapping images of Cu (b) and Ti (c), TEM image of GO (d) and FTIR spectrum of GO (e)



**Fig. 3** SEM images of flaked Cu powders (a), GO/Cu<sub>2</sub>O composite powders (b) and RGO/Cu–Ti composite powders (c), XRD patterns (d), and Raman spectra (e)

and the basic reaction principles and mechanism were discussed elsewhere [6]. And compared with Refs. [11–13], it is found that by the first synthesizing the GO/Cu<sub>2</sub>O composite powder instead using the GO directly, by forming cube-shaped Cu<sub>2</sub>O, the GO is dispersed more uniformly, and the GO and the Cu<sub>2</sub>O are more tightly combined, which makes a good foundation for subsequent experiments and performance.

Figure 3(c) shows the microstructure of the RGO/Cu–Ti composite powders after ball milling and reduction, all cubic powders are turned into nearly spherical particles with a reduced size, and the wrinkled structure of RGO can be observed, indicating a good dispersity and topographic integrity of RGO. XRD investigation only shows the presence of characteristic peaks of fcc-Cu in the patterns, which may be attributed to the low content of RGO (Fig. 3(d)).

Raman spectroscopy shows the structural change of GO in each fabrication process by analyzing the intensity ratio of D (~1350 cm<sup>-1</sup>) to G (~1600 cm<sup>-1</sup>) band (Fig. 3(e)). The  $I_D:I_G$  value of the starting GO is ~0.83, indicating a certain quantity of defects, which can provide nucleation sites for Cu<sub>2</sub>O particles. The ratio is raised to 0.94 for 0.3wt.%RGO/Cu–Ti powder, inferring the increased defects of RGO during the ball milling

process, which are still retained even after H<sub>2</sub> reduction. In addition, the G band of 0.3wt.%RGO/ Cu–Ti composite powder is shifted to the high wavenumber by ~10 cm<sup>-1</sup> compared with that of the GO due to the strong interaction of electrons between Cu<sub>2</sub>O and GO, and the powerful electron coupling contributes to the adsorption of Cu<sub>2</sub>O by GO [14]. However, the  $I_D:I_G$  value is reduced to 0.68 in the composite, suggesting that barely any new defects are introduced, and the D band vibration is suppressed [15].

## 3.3 Microstructural characterization of 0.3wt.% RGO/Cu-Ti composite

At the interface of Cu and RGO (Fig. 4(a)), the areas labeled by the red arrows indicate the location of the RGO under a bright field. Further magnification of the interface between RGO and Cu in Fig. 4(b) shows a transition interface instead of a clear and intuitive interface. The fast Fourier transform (FFT) images (Fig. 4(c)) of the marked *c* reveal the characteristic (220) and (200) diffraction spots of TiC phase. Based on the noise-filtered inverse fast Fourier transform (IFFT) image, the lattice inter-planar spacing is measured to be ~0.222 nm, which is perfectly matched to the *d*-spacing of (200) planes of TiC. Interestingly, different from the green box area, a titanium-



**Fig. 4** Morphology of 0.3wt.%RGO/Cu-Ti composite: (a) TEM image of RGO position in matrix; (b) HRTEM image of interface; (c, d) FFT, IFFT and corresponding lattice spacing measurement recorded at *c* and *d* regions respectively in (b); (e) Schematics of possible carbide formation mechanism in RGO/Cu-Ti composite

carbon compound is found in the blue box which is closer to the matrix (Fig. 4(d)). Based on the FFT and IFFT patterns of the marked areas, the inter-planar spacing of these lattice fringes are measured to be  $\sim 0.162$  nm and  $\sim 0.261$  nm,

corresponding to the *d*-spacing of (009) and (021) planes of cubic  $Ti_8C_5$  phase, respectively. It is well known that the interfacial reaction between carbon and Cu binary systems cannot occur [16]. Therefore, the interface bonding between the reinforcement of

RGO and the Cu–Ti matrix can be improved by these formed transition carbides. The appearance of the transition phase indicates that Ti atoms have been segregated from the lattice of Cu–1.0Ti alloy, and reacted with the higher activity C atoms at the interface.

Phase transformation reactions will occur in the Ti-C system due to the sufficiently high sintering temperature at ~1073 K [17]. Based on the HRTEM analysis, a possible formation mechanism of interfacial Ti<sub>x</sub>C<sub>v</sub> nanophase is put forward (Fig. 4(e)). The Ti-C compound will be formed due to the combination of active C atoms and Ti atoms precipitated from the copper matrix. The Gibbs free energy of Ti<sub>8</sub>C<sub>5</sub> is calculated as -280.58 kJ/mol at sintering temperature of 1073 K, whereas that of TiC is -172.23 kJ/mol. However, the diffusion rate of C atoms will be accelerated with the new phase formation. At this point, the previously generated Ti<sub>8</sub>C<sub>5</sub> will react with C to form TiC (the Gibbs free energy is -445.92 kJ/mol). In addition, a phase to will change from Ti<sub>8</sub>C<sub>5</sub> TiC occur  $(Ti_8C_5+3C=8TiC)$  due to the more stable facecentered structure of the TiC phase [18].

#### 3.4 Mechanical properties

The relative density and mechanical properties are summarized in Table 1 and Figs. 5(a, b). The measured hardness, yield strength (YS) and ultimate tensile strength (UTS) of the composite increase to HV 147.2, 222 MPa and 440 MPa, respectively, when the RGO content is 0.3 wt.% compared with those of the matrix material. And, all of them maintain high levels at HV 132.7, 215 MPa and 398 MPa, respectively, even when the RGO is increased to 0.6 wt.%. However, for the 0.9wt.%RGO/Cu–Ti sample, mechanical properties have been severely deteriorated, the relative density is only 97.3% and elongation is only 3.9%, which are much lower than those of the matrix material.

To better understand the evolution of the mechanical behavior of the composites, the

fractured surface was analyzed by SEM (Fig. (5)). All samples show the ductile fracture properties due to the dimples on the fracture surface. Compared with the fracture morphology of a Cu-Ti composite (Fig. 5(c)), pulled-out RGO can be observed on the fracture surface (Figs. 5(d, e)), suggesting the load transfer from the Cu matrix to the reinforcements during tensile tests. It is clear that detected RGO is adhered and embedded firmly into the metal matrix, which is inseparable from the strong interface bonding. However, relatively large, shallow and fewer dimples appear in 0.9wt.%RGO/Cu-Ti sample (Fig. 5(f)), showing poor plasticity because of the stress-concentration caused by RGO clusters. These clusters also reduce the relative density, which is consistent with the finding in Section 3.2.

#### **3.5 Strengthening mechanisms**

Strengthening mechanisms, normally, can be discussed as: load transfer strengthening, thermal mismatch strengthening, grain refinement and second phase strengthening. These mechanisms may occur simultaneously during the strengthening process.

It is worth noting that the thermal mismatch strengthening is possible and has already been reported in MMCs when the composite is cooled down at a rapid rate, such as by the water quenching [19]. In this research, due to the comparatively low cooling rate, the thermal mismatch effect is ignored. Based on the results of the microstructure analysis, relevant theoretical discussion is given, the relevant calculation formulas and specific assignments can be referred elsewhere [20].

Cu grain boundary migration is hindered by a sufficiently high content of RGO which is surrounded at the grain boundaries, resulting in grain refinement in Fig. 6. The better dispersed GO can improve the in-situ generation of  $\text{Ti}_x \text{C}_y$  which is beneficial for the refinement of grains. The strengthening effect of grain reinforcement ( $\Delta \sigma_{\text{GR}}$ )

Table 1 Relative density and mechanical properties of composites with different RGO contents

Sample	Relative density/%	Hardness (HV)	YS/MPa	UTS/MPa	Elongation/%
Cu-Ti	98.6	90.3±8	140±5	274±10	11.9±0.3
0.3wt.%RGO/Cu-Ti	98.8	147.2±3	222±3.5	440±7	10.3±0.4
0.6wt.%RGO/Cu-Ti	98.4	132.7±2	215±4	398±3	10.6±0.4
0.9wt.%RGO/Cu-Ti	97.3	80.6±2	130±6	241±8	3.9±0.3



**Fig. 5** Stress-strain curves (a) and obtained engineering stresses (b) of composites, fracture surface morphologies of composites with different contents of RGO: 0 wt.% (c), 0.3 wt.% (d), 0.6 wt.% (e) and 0.9 wt.% (f)

can be estimated as ~7.71 MPa from the Hall–Petch relationship [21]. RGO acts as a bridge, and the load in the matrix is effectively transferred according to the fracture surface feature. The strength increased by load transfer ( $\Delta \sigma_{LT}$ ) has been estimated by the shear lag model [22] and the measured value is 103.2 MPa.

In our study, the strengthening of the second phase cannot be ignored due to the in-situ generated and uniformly dispersed  $\text{Ti}_x C_y$  nanophases. Adjacent particles accumulate to pin and form a dislocation loop, thereby hinder the movement of dislocations and result in reinforcing effect [23]. Therefore, from the analysis of the previous results, it can be known that the carbon source is provided by RGO, and the phase-reversed in-situ reaction with Ti precipitated in the matrix generates a hard and brittle nano-sized (non-deformable) Ti<sub>x</sub>C<sub>y</sub>, resulting in a second phase strengthening. The Orowan– Ashby model is used to estimate the second phase enhancement contribution ( $\Delta\sigma_{SP}$ ), which is ~52.31 MPa.

The predicted tensile strength of 0.3wt.%RGO/ Cu–Ti is 437.2 MPa by summing up the  $\Delta\sigma_{GR}$ ,  $\Delta\sigma_{LT}$ and  $\Delta\sigma_{SP}$  (Fig. 7(a)). This calculated value is close to the experimental value, confirming that the three



Fig. 6 TEM images (a, c) and distribution of grain size (b, d) of Cu-Ti (a, b) powder and 0.3wt.%RGO/Cu-Ti composite (c, d)



**Fig.** 7 Tensile strength (a) of matrix, experimental and calculated value (left part) and strengthening contributions (right part); (b) Reinforcing efficiency (R) of composite

enhancements analyzed above are valid. Obviously, load transfer is the major contributor to the strengthening effect. The high load transfer efficiency (R) of RGO/Cu-Ti composite is evidenced by the strong interfacial bonding. Figure 7(b) summarizes the R versus volume fraction of reinforcement ( $V_f$ ) obtained from the present RGO/Cu–Ti composite and those reported for related composites prepared by five typical methods including molecular-level mixing [24], in-situ growth of graphene on Cu powders [25,26], high-energy ball-milling [5], ball-milling and

high-ratio rolling [27] and CNT-GR hybrids reinforcement in Cu matrix composite [28]. In this study, the composites prepared by our strategy realize a good balance of R and  $V_{\rm f}$ , due to a good combination of the GO with the matrix material, and the firm interface bonding by the formation of the second phase. From comparison, it can be found that the R in our study is more accurate to the optimal R value achieved by the methods of molecular mixing and in-situ growth of GR on the surface of Cu powder. And, the process in this research method is much simpler and easier to operate. Therefore, this study is very meaningful and can provide a simple and effective preparation method for improving the mechanical properties of Cu matrix composites.

## **4** Conclusions

(1) A strategy of RGO/Cu-Ti composites preparation based on the electrostatic adsorption is achieved.

(2) The obtained composites have well dispersed and distributed RGO in the matrix.

(3) Interface bonding is improved by the in-situ formed carbide nanoparticles, which acts as load transfer bridges. Nucleation and growth of the carbides like  $Ti_8C_5$  and TiC occur from the precipitation and diffusion of Ti atoms in the carbon area.

(4) The as-fabricated RGO/Cu–Ti composite exhibits a significant enhancement in tensile strength and strengthening efficiency.

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## 静电吸附制备 RGO/Cu 复合材料

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摘 要:为了解决还原氧化石墨烯(RGO)在铜(Cu)基体中的分散问题以及还原氧化石墨烯与铜之间的界面结合问题,采用静电吸附方法,结合界面过渡相设计制备 RGO/Cu 复合材料。采用 Cu-Ti 合金粉通过生成过渡相来改善碳在金属基体中的润湿性。结果表明,与铜基体相比,制备的 0.3 wt.% RGO/Cu-Ti 的力学性能提高了 60%。强化机理分析表明,该复合材料的力学性能的提高是由于 RGO 在基体中分散性的改善,以及原位形成的第二相碳 钛化合物,因此载荷传递和第二相强化是主要的强化机理。

关键词:氧化石墨烯(GO);静电吸附;Cu-Ti;原位Ti<sub>x</sub>C<sub>y</sub>;力学性能

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