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The effect of hot pressing sintering temperature and holding time on the properties of graphene/Cu layered composites

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The effect of hot pressing sintering temperature and holding time on the properties of graphene/Cu layered composites

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Abstract: Graphene is a promising reinforcement in metal matrix composites, which has been developing rapidly in recent years. The aim of this paper is to investigate the effect of sintering temperature and holding time on the properties of graphene nanoplatelets (GNPs)/Cu composites prepared by hot pressing. Sintering temperature and time were in the range of 550–750 ℃ for 0–60 min. A correlation is established between the mechanical properties of GNPs/Cu composites and sintering parameters. The results show that the variations in tensile strength of GNPs/Cu composites are dependent on both sintering temperature and time. With increase of sintering temperature, the tensile strength increases first and then decreases. When sintered at 650 ℃ for 30 min, the maximum tensile strength of 303 MPa is achieved. The tensile strength of GNPs/Cu composite sintered 30 min at 650 ℃ is also higher than that of 0 and 60 min.

1. Introduction
Metal matrix composites have been developed in recent years, among which copper matrix composites have found various applications in the industry due to their good physical and mechanical properties. It has been well established that the addition of carbon nanomaterial to copper matrix improves its strength[1], electrical [2] and thermal conductivity[3]. Graphene possesses a unique structure and properties that have attracted significant interest worldwide[4]. Its ultrahigh strength and modulus are exploited to make stronger metal matrix composites[5][6].

Powder metallurgy (PM) is one of the most common preparation methods for graphene reinforced metal composites at present[7]. PM is economical, productive and easily to control the microstructure with good distribution of graphene in compacts. One of the factors affecting microstructure and mechanical properties of as-sintered composites is diffusion bonding between powders during sintering. Diffusion itself is mainly dependent on sintering temperature and time. However, little has been reported about the effect of hot pressing sintering parameters on microstructure and properties of graphene/Cu composites.

In this study, graphene nanoplatelets (GNPs)/Cu layered composites were prepared by hot pressing based on the self-assembly of flake powder. The effect of hot pressing sintering temperature and holding time on mechanical properties of GNPs/Cu composites was investigated.

2. Experimental

Flake Cu powder and GNPs (purchased from JCNANO Tech Co., Ltd, China) were used as raw materials in this work. 99% purity flake Cu powder was with an average thickness of 100 nm and a lateral size of ~10 μm. 99.7% purity GNPs were with an average thickness of 2 nm and a lateral size of ~6 μm. Firstly, Cu and GNPs were mixed together in ethanol under ultrasonication for 2 h to form a homogeneous suspension. Then the suspension was filtered and dried. The mixture was further mixed thoroughly by ball milling under a rate of 160 rpm for 5 h applying sample/ball weight ratio 1:8. After that, the composite powder was loaded into a graphite mold and vibrated in a loose state to promote self-assembly. Finally, the composite powder was sintered by hot pressing under vacuum at 550, 600, 650, 700 and 750 °C for 0, 30 and 60 min under a pressure of 35 MPa to prepare GNPs(1vol.%)/Cu composite. Figure 1 shows the technology route of the hot pressing sintering.

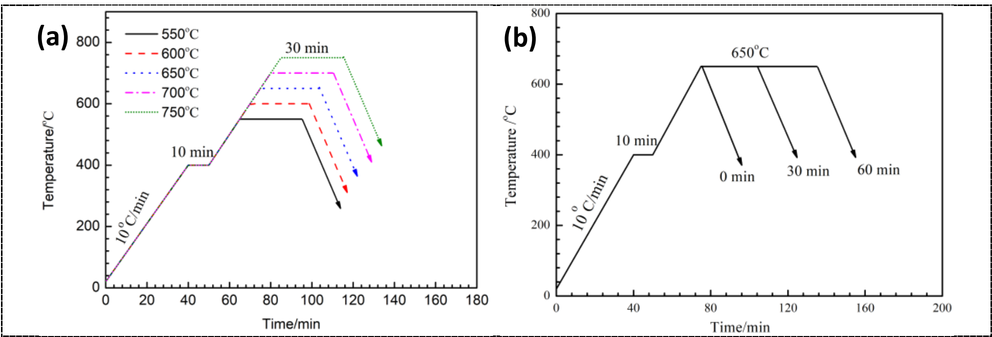

Figure 1. Sintering process of graphene/Cu composites with different sintering temperature (a) and different holding time (b).

The raw materials and as-prepared composites were characterized with regard to their microstructure by scanning electron microscopy (SEM), optical microscopy (OM) and transmission electron microscopy (TEM). Tensile tests in radial direction were conducted on a universal testing machine with the crosshead speed of 0.6 mm/min. The test samples with a gauge length of 10 mm and cross-section of 2 mm * 1.5 mm were machined from the sintered billets. Three samples were tested from each sintered billet to minimize the error.

3. Results and discussion

SEM micrographs of raw GNPs and Cu powder are shown in Figure 2. As shown in Figure 2(a), GNPs exhibit a typical stack-like morphology. Figure 2(b) shows the flake-like and irregular nature of the Cu particles.

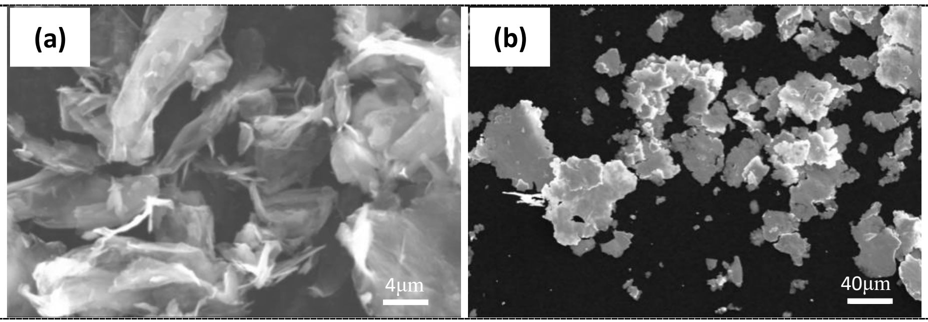

Figure 2. SEM micrographs of raw GNPs (a) and Cu powders (b).

The variation of tensile strength of GNPs/Cu composites with sintering temperature and holding time is shown in Figure 3. Figure 3(a) shows that tensile strength of the GNPs/Cu composites is improved with the increasing of sintering temperature from 550 °C to 650 °C. When the sintering temperature is 650 °C, the tensile strength of GNPs/Cu composite is 303±3 MPa, which is increased by ~14% compared with the composite sintered at 550 °C. The tensile strength begins to decrease when the sintering temperature continues to increase above 650 °C. At the sintering temperature of 750 °C, the tensile strength is reduced to 290±2 MPa. The main reason for this phenomenon may be that increasing the sintering temperature can significantly promote sintering diffusion and increase the density of the composite, thereby increasing the tensile strength. If the sintering temperature is too high, the grain may grow and the strength of the composite decrease.

As shown in Figure 3(b), the tensile strength of GNPs/Cu composite sintered at 650 °C without holding time is about 289 MPa. When the holding time is 30 min, the tensile strength is increased to 303±3 MPa. However, the tensile strength is slightly reduced when the holding time is 60 min. It indicates that the densification of the GNPs/Cu composites is basically completed within the heating process and first 30 min of the holding process. With the prolongation of the holding time, the sintering enters the later stage, in which the densification rate mainly depends on the isolated closed pores diffusing from the inside to the surface of the composites. Accordingly, the densification rate is very slow. The fact that the tensile strength of the composite sintered for 60 min is slightly decreased also indicates that excessively prolonging the sintering holding time has little significance for improving the property of the GNPs/Cu composites, and may even have adverse effects.

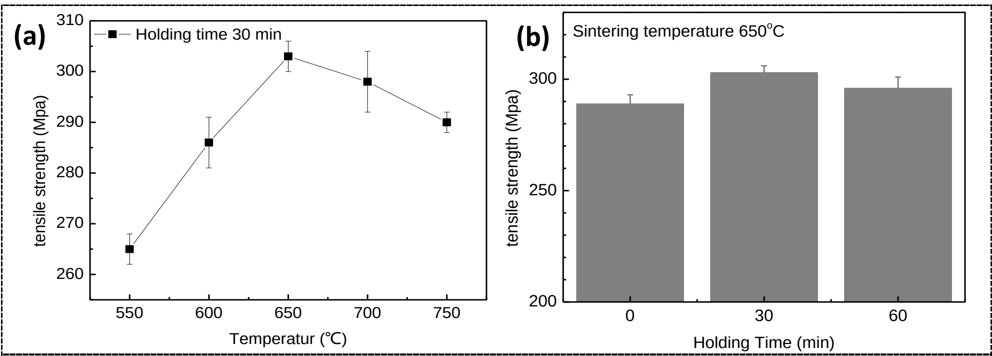

Figure 3. The effect of sintering temperature (a) and time (b) on the tensile strength of GNPs/Cu composites.

Figure 4(a) and 4(b) show the metallographs of GNPs/Cu composite sintered at 650 °C for 30 min. The metallographs show that the GNPs/Cu composite exhibits a typical layered structure, which can be attributed to the surface diffusion hindering effect of flake Cu powder by GNPs sheets. No coarse particles are observed, indicating that a relatively uniform dispersion of GNPs is obtained in the Cu matrix. The interface microstructure of GNPs/Cu composite was observed by TEM. As shown in Figure 4(c), GNPs distribute along the Cu grain boundary and maintain the layered structure. HRTEM image shown in Figure 4(d) indicate that good bonding without obvious holes or cracks forms at the interface between GNPs and Cu matrix, which is conducive to strength improvement.

4. Conclusions

In this study, 1vol.% GNPs/Cu layered composites were prepared by hot pressing under different sintering temperature and holding time. The maximum tensile strength of 303 MPa is achieved when sintered at 650 ℃ for 30 min. Exorbitant sintering temperature leads to a decrease of the tensile strength, while prolonging the sintering holding time has little significance for improving the tensile strength of the GNPs/Cu composites.

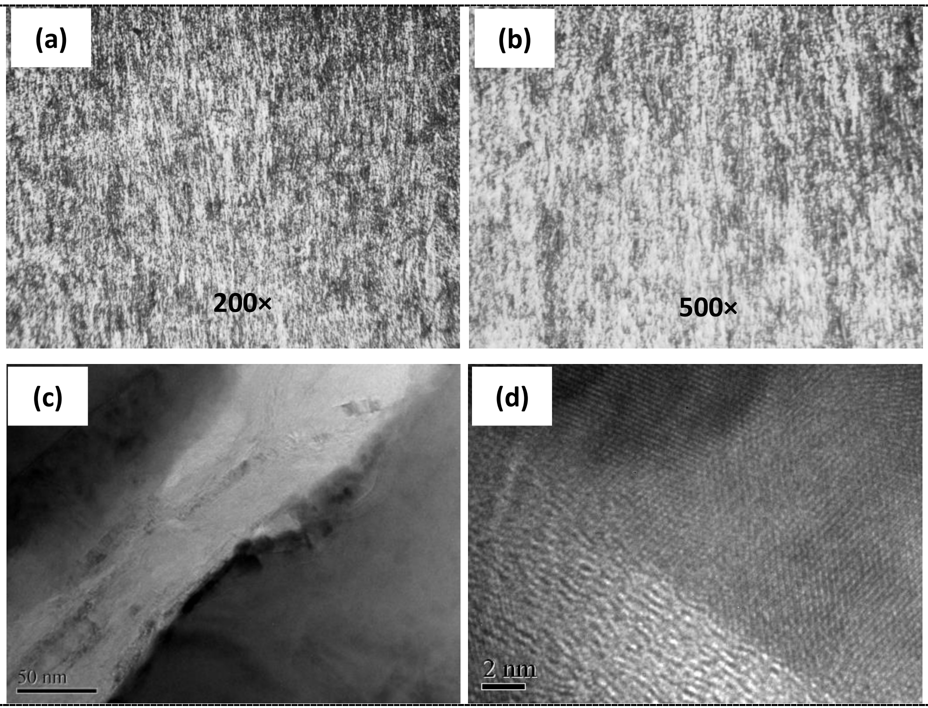

Figure 4. OM micrographs of pure GNPs/Cu composite (a, b), TEM (c) and HRTEM (d) images of GNPs/Cu composite.

Acknowledgments
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