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ORIGINAL RESEARCH

Effect of sintering temperature on the preparation of Cu–Ti₃SiC₂ metal matrix composite

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KEYWORDS

Warm compaction; Spark plasma sintering; Electrical conductivity; Mechanical property; Impurity **Abstract** Ti₃SiC₂ has the potential to replace graphite as reinforcing particles in Cu matrix composites for applications in brush, electrical contacts and electrode materials. In this paper the fabrication of Cu–Ti₃SiC₂ metal matrix composites prepared by warm compaction powder metallurgy forming and spark plasma sintering (SPS) was studied. The stability of Ti₃SiC₂ at different sintering temperatures was also studied. The present experimental results indicate that the reinforcing particles in Cu–Ti₃SiC₂ composites are not stable at and above 800 °C. The decomposition of Ti₃SiC₂ will lead to the formation of TiC and/or other carbides and TiSi₂. If purity is the major concern, the processing and servicing temperatures of the Cu–Ti₃SiC₂ composite should be limited to 750 °C or lower. The composites prepared by warm compaction forming and SPS sintering at 750 °C have lower density when compared with the composites prepared by SPS sintering at 950 °C, but their electrical resistivity values are very close to each other and even lower.

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1. Introduction

Pure copper has excellent electrical and thermal conductivity; however, its poor wear resistance and low mechanical properties

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limit its application. It is well known that adding any alloying element to copper will decrease its conductivity significantly. Ag exerts the least negative effect on Cu's conductivity, but Ag is expensive. In order to avoid the adverse effect of solid solution strengthening that is introduced by alloying elements, the use of reinforcing particles (dispersion strengthening) to improve the mechanical properties and wear resistance of Cu-base materials is a feasible choice. Since C has very limited solid solubility in Cu and graphite is a layered material with very low friction, Cu– graphite metal matrix composites (MMC) are widely used as electro-friction material for current collectors and commutating brushes in motors. However, as advanced progress in electricmechanical industry is made, traditionally used Cu–graphite

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composites cannot fulfill the modern requirement for high sliding speed with high current density, for instance, in the application of overhead catenary current collection system of the modern electrified railway [1,2].

Ti₃SiC₂ is an advanced MAX phase material ($M_{n+1}AX_n$, where M is an early transition metal, A is an A-group element, and X is either carbon or nitrogen) which possesses a combination of both metallic and ceramic characteristics. For instance, its electrical conductivity and thermal conductivity are better than Ti metal at room temperature, and compared to regular ceramics, Ti₃SiC₂ is relatively "soft" and easy to machine. It is also not sensitive to thermal shock and shows plastic behavior at elevated temperature. As a ceramic, it possesses excellent oxidation resistance and thermal stability at high temperatures. Similar to graphite, Ti₃SiC₂ is a good anti-friction material due to its layered structure, and its electrical conductivity is roughly two orders of magnitude higher than that of graphite [3–5]. Thus, the use of Ti_3SiC_2 to replace graphite should produce a new class of self-lubricating materials for electrical sliding contacts. In recent years, many researches [6-27] concerning Cu-Ti₃SiC₂ composites have been performed and most of the literature was published by Chinese researchers. In addition to these, Cu-Ti₃SiC₂ composites with a second reinforcing element such as TiC, graphite, carbon fibers, etc., were also reported [28-36]. Almost all of the researchers adopted the costly hot press sintering process. Although hot press can provide high density materials easily, due to the equipment restriction, the hot pressed products are limited both in size and shape. Among all these studies, the stability of Ti₃SiC₂ during high temperature sintering was generally neglected; only Zhou's research group studied the chemical reaction and stability of Ti₃SiC₂ in Cu during high-temperature processing of Cu-Ti₃SiC₂ composites [17]. The stability of Ti₃SiC₂ is crucial, since the decomposition of Ti₃SiC₂ will cause the composite to lose its lubrication characteristic, which is vital for self-lubricating electrical sliding contact material.

In this paper, the fabrication and characterization of Cu– Ti_3SiC_2 MMC prepared by warm compaction powder metallurgy forming and spark plasma sintering (SPS) were studied. Different from previous researches, lower sintering temperatures were used and the stability of Ti_3SiC_2 at different sintering temperatures was studied. Lastly, literatures concerning Cu– Ti_3SiC_2 composites were reviewed and the results obtained in this study were compared with the literature data.

2. Experimental procedures

Commercially available pure (99.7 mass%) copper powder with a particle size of \leq 74 µm, and self-prepared Ti₃SiC₂ powder [37] with a particle size of \leq 50 µm were used in this study. The mixed powder was prepared by mixing Cu powder with 5, 10, 15, 20, 25, 30, 35 and 40 mass% of Ti₃SiC₂ powder in a V-type mixer for 24 h. In order to overcome the low density problem that was caused by the low sintering temperature, warm compaction (WC) was used for samples that sintered at lower temperature (750 °C). The mixed powder was heated to 150 °C and then warm compacted into disk shaped samples 20 mm in diameter and approximately 6 mm in thickness, under a pressure of 1000 MPa in a heated steel mold, which was maintained at 150 °C. Polytetrafluoroethylene (PTFE) suspension was used for die wall lubrication. The green compacts were spark plasma sintered (SPS) in a graphite mold under vacuum by using a Dr. Sinter Type SPS-825 apparatus. A pressure of 50 MPa and a heating rate of 100 K/ min were used to raise the temperature up to 700 °C and then the sample was slowly heated up to the sintering temperature of 750 °C. After being sintered for 10 min, the sample was then furnace cooled to ambient temperature by turning off the power. For samples sintered at 950 °C, no warm compaction forming was involved and regular SPS with a sintering procedure similar to the above described schedule was used. Regular SPS sintering at 750, 800, 850 °C for 10 min and hotpress sintering (HP) at 800 °C for 2 h, under a pressure of 50 MPa, were also carried out on a Cu–25 mass% Ti₃SiC₂ sample to study the stability of Ti₃SiC₂ during sintering.

Samples with a size of $16 \text{ mm} \times 6 \text{ mm} \times 6 \text{ mm}$ were cut from the sintered composites. A PANalytical X'Pert PRO xray diffractometer operating at 40 kV and 40 mA was used for x-ray diffraction (XRD) analysis. Cu K α radiation was used and the scanning rate was 0.02° /s. The microstructure was studied by an optical microscope and a LEO 1530 VP Model of scanning electron microscope (SEM). Density of the samples was measured by the Archimedes method. Electrical resistivity of the samples was measured by a digital DC resistance tester. Compression tests were carried out by a Shimadzu AG-X/100 kN universal testing machine. The compression speed was 0.5 mm/min and the sample size was $16 \text{ mm} \times 4 \text{ mm} \times 2 \text{ mm}$. Brinell hardness was also measured.

3. Results and discussion

Different from most of the reported literature, the Ti_3SiC_2 used in this study is very pure. It is important because impurity is harmful to the composite's self-lubrication behavior; besides, it facilitates the identification of new phase formation after sintering, if any. Fig. 1 shows the morphology and XRD pattern of the self-prepared pure Ti_3SiC_2 powder. The layered structure of Ti_3SiC_2 can be seen clearly in Fig. 1(a), and as shown in Fig. 1(b), no impurity was detected.

High density, even distribution of filler particles, and good binding between filler particles and matrix are essential elements in obtaining composites with good mechanical and electrical properties. Fig. 2 shows the microstructure of the SPS sintered composites (WC+SPS sintered at 750 °C) containing 10, 15, 20, 25 and 30 mass% Ti₃SiC₂. The dark gray areas in the figures are Ti₃SiC₂ particles which are embedded in the light colored Cu matrix. The black areas are voids. Fig. 2(a) and (b) shows that Ti₃SiC₂ particles were distributed quite evenly in the Cu matrix for samples containing 15 mass% Ti₃SiC₂ or less. As Ti₃SiC₂ concentration increased, agglomeration of the filler particles became a problem. Agglomeration of hard particles will lead to large voids and void concentration, which are harmful to the mechanical properties and electrical conductivity of the materials. The 30 mass% Ti₃SiC₂ sample contains a significant amount of localized voids, as shown in Fig. 2(e).

Fig. 3(a) and (b) shows the XRD patterns of the Cu–25 mass% Ti₃SiC₂ samples sintered at different temperatures by using SPS and HP sintering, respectively. From Fig. 3(a), it can be seen that there is no phase change after SPS sintering at 750 °C or lower, but significant amounts of SiC, TiC and TiSi₂ were formed when the sintering temperature reached 800 °C. These results are not consistent with those obtained by previous reports [17,38–39]. Zhou et al. studied the reactions between



Fig. 1 (a) Morphology and (b) XRD pattern of pure Ti₃SiC₂.



Fig. 2 SEM micrographs showing the microstructure of sintered composites (WC+SPS sintered at 750 $^{\circ}$ C) containing (a) 10; (b) 15; (c) 20; (d) 25 and (e) 30 mass%Ti₃SiC₂.

Ti₃SiC₂ and Cu during the processing of Cu-Ti₃SiC₂ composites. They found that there was no apparent reaction between Ti₃SiC₂ and Cu at temperatures below 900 °C. However, at higher temperatures (900-1070 °C), the deintercalation of Si from Ti₃SiC₂ leads to the formation of Cu(Si) solid solution or Cu-Si intermetallic compounds. At low Ti₃SiC₂ content or temperatures below 1000 °C, Cu(Si) solid solution and TiC_x were formed, whereas at high temperature and high Ti₃SiC₂ content, TiC_x and Cu–Si intermetallic compounds were observed. The reaction products depend on the temperature and the relative ratio of Cu and Ti₃SiC₂ [17]. By magnetron-sputtering deposition of Cu and subsequent annealing in a temperature range of 900-1100 °C for 30-60 min, Guo et al. improved the surface hardness and wear resistance of Ti₃SiC₂ by forming TiC_x and Cu₃Si [38]. Li et al. used Cu film to prepare a Ti₃SiC₂/Cu/Ti₃SiC₂ joint by subsequent heat treatment at 950 and 1100 °C for 120 min in an argon atmosphere, and they found that the bonding mechanism is attributed to the silicon atoms that diffused from the Ti₃SiC₂ substrate into the Cu film and reacted with the film to form Cu₃Si and TiC at the joint interface [39]. In the present study, Ti₃SiC₂ lost its stability at 800 °C, which is 100 °C lower than the reported value of Zhou et al. The difference may be attributed to the spark liberation and the uneven temperature distribution in the sample during SPS sintering (caused by the difference in local electrical conductivity of the composite). However, results obtained from HP sintering at 800 °C for 2 h on another Cu–25 mass% Ti₃SiC₂ sample shows no sign of Cu–Si intermetallic compound and significant amounts of TiC and TiSi₂ were formed, as shown in Fig. 3(b). Therefore, it can be concluded that if SPS or HP is employed, the sintering temperature should be limited to 750 °C or less if obtaining pure Cu–Ti₃SiC₂ composite is the objective.

As mentioned in the preceding paragraph, the reaction products obtained by previous researches [17,38,39] at 900 °C or above were either TiC_x and Cu–Si intermetallic compounds, or Cu(Si) solid solution and TiC_x . However, in this



Fig. 3 XRD patterns of Cu-25 mass% Ti₃SiC₂ samples sintered by (a) SPS and (b) HP.

study no Cu-Si intermetallic compounds can be found, and the reaction products that formed in SPS and HP sintering at 800 °C were SiC, TiC, TiSi₂ and TiC, TiSi₂, respectively. Since lower sintering temperatures were used in the present case, the Si that diffused into the Cu matrix is not enough to form Cu-Si compounds, and consequently Ti₃SiC₂ lost its stability and decomposed into SiC, TiC and TiSi2. The absence of Cu-Si compounds is in agreement with the finding in Zhou et al. that at low Ti₃SiC₂ content or low temperatures (below 1000 °C), Cu(Si) solid solution and TiC_x were formed from the destabilized Ti₃SiC₂ [17]. The heating elements of SPS are different from the conventionally heated HP furnace. In addition to Joule heating caused by the electric current passing through the sample itself and also through the graphite mold, there may be spark liberation that will generate a high local temperature in the sample, so the temperature distribution in the SPS sample is not as even as those in the HP samples. Moreover, SPS is a fast sintering technique; it takes minutes (10 min in this case) instead of hours (2 h in this case) to finish the sintering process. Therefore, the reaction products obtained through SPS sintering and HP sintering are different.

Fig. 4 depicts the density and relative density (RD=sample density divided by its theoretical density) of the green and sintered compacts. Fig. 4 shows that, as Ti₃SiC₂ concentration increases, the large density difference between Cu (8.9 g/cm^3) and Ti₃SiC₂ (4.53 g/cm³) leads to a sharp decrease in both green and sintered densities. Even though warm compaction was used to enhance the green density, the composites sintered at 750 °C have a tangible disadvantage of lower density when compared with those sintered at 950 °C. The sintered density is always higher than the warm compacted green density, but the increment decreases with increasing Ti3SiC2 content. For instance, when Ti₃SiC₂ concentration is 10 mass%, its green and sintered densities are 7.69 g/cm³ and 8.09 g/cm³ respectively, and there is a difference of 0.4 g/cm^3 , while for a 30 mass% Ti₃SiC₂ sample, its green and sintered densities are 6.28 g/cm³ and 6.48 g/cm³ respectively, and there is only a difference of 0.2 g/cm³. Extrapolation of the linear fits of the green density and sintered density data showed that they intersect approximately at 50 mass% Ti₃SiC₂, which corresponds to approximately 65 vol% Ti₃SiC₂. This means that beyond this intersecting point, SPS sintering cannot further increase the sample density. This phenomenon is easy to understand. The density increase after sintering is due to the shrinkage effect of Cu and the applied pressure during SPS sintering. However, the presence of Ti_3SiC_2 particles, which is metallurgically incompatible with Cu, will impede the diffusion and grain growth of Cu; as a result, its densification process will be retarded. As Ti_3SiC_2 concentration increases, this effect becomes more obvious. When Ti_3SiC_2 concentration reached at 50 vol% and higher, the shrinkage effect of Cu exerts very little effect on reducing the overall volume of the sample since the hard incompressible Ti_3SiC_2 particle is the dominant constituent, and applied pressure (50 MPa) has no effect on sample densification either.

Figs. 5 and 6 show the effect of Ti_3SiC_2 content on electrical resistivity and hardness of the sintered composites, respectively. Experimental results show that electrical resistivity and hardness both increased with increasing Ti_3SiC_2 content. The electrical resistivities of the composites sintered at 750 °C (WC+SPS) are very close to those of the composites sintered at 950 °C (SPS). It is worth to note that although the 750 °C sintered samples have lower density, their resistivity is relatively lower, as shown in Figs. 4 and 5. The most probable reason for this peculiar phenomenon (usually higher density would lead to a lower resistivity) is that the interfaces between the Ti_3SiC_2 particles and the Cu matrix are cleaner; unlike those samples that are sintered at 950 °C, there is no impurity phase formed during 750 °C sintering.

Fig. 7 shows the effect of Ti_3SiC_2 content on the bending strength of the sintered composites. From Fig. 7, it can be seen that the bending strength decreases with increasing Ti_3SiC_2 content. This decrease is mainly due to the increase in void concentration and the destruction of matrix continuity. Fig. 8(a) and (b) shows the literature reported tensile strength and elongation percentage data, respectively, of Cu– Ti_3SiC_2 composites.

Gao et al. [12] used both conventional powder metallurgy method and HP technique to sinter Cu–Ti₃SiC₂ composites at 900 °C. Their results indicated that the hot pressed samples have much higher density than those of the conventionally pressureless sintered samples. The electrical resistivity of the conventionally prepared composites increases rapidly as the Ti₃SiC₂ content increase. Sun et al. [10] use HP temperature of 980 and 1200 °C to prepare Cu–Ti₃SiC₂ (60, 70 and 80 mass% Ti₃SiC₂) composites. They found that the 60 mass% Ti₃SiC₂ composite sintered at 980 °C for 120 min has a bending strength of 516 MPa, and the 70 mass% Ti₃SiC₂ composite sintered at 1200 °C for 30 min has an electrical resistivity of 0.155 μ Ωm, which is a pretty low value for a composite with a



Fig. 4 (a) Green and sintered density and (b) sintered RD, of $Cu-Ti_3SiC_2$ composites; solid symbols are data from this study; semi-solid symbols are data from the present research group. NP=pressureless sintering and DP-DS=double press-double sintered.



Fig. 5 Electrical resistivity of $Cu-Ti_3SiC_2$ composites; solid symbols are data from this study; semi-solid symbols are data from the present research group. NP=pressureless sintering.



Fig. 6 Hardness of $Cu-Ti_3SiC_2$ composites, solid symbols are data from this study; semi-solid symbols are data from the present research group. DP-DS=double press-double sintered.

high Ti₃SiC₂ content of 70 mass%. The present authors have reservation about their reported resistivity data since pure 100% dense Ti₃SiC₂ has a resistivity of 0.227 $\mu\Omega$ m, which is significantly higher than that of their 70 mass% Ti₃SiC₂ sample (corresponding to a 82.09 vol% Ti₃SiC₂). Wang [14] prepared Cu–Ti₃SiC₂ (40 vol% Ti₃SiC₂) composites by HP at 1000 °C for 30, 60, 90 min under 39 MPa. All of his reported



Fig. 7 Bending strength of Cu–Ti₃SiC₂ composites; solid symbols are data from this study. NP=pressureless sintering.

relative density data are higher than 100%, which is quite doubtful. Chen [15] studied the effects of Ti₃SiC₂ content, sintering temperature and holding time on the physical and mechanical properties of Cu-Ti₃SiC₂ composites. He obtained Cu-Ti₃SiC₂ (30-50 vol% Ti₃SiC₂) composites with a bending strength of about 1100 MPa by HP at 1000 °C for 120 min under a pressure of 300 MPa. His reported bending strengths are more than double of the other reported literature data, but they are somewhat higher than expected, since the material they prepared contains a 50 vol% of ceramic particles which usually require a sintering temperature higher than 1000 °C. Gao [13] found that the optimal processing parameter for HP is sintering at 900 °C with a pressure of 30 MPa for 2 h, and the composites prepared by using coated Ti₃SiC₂ have better performance. His results indicated that resistivity and Brinell hardness of the composite increased significantly as Ti₃SiC₂ content increased. The density of their hot-pressed composites with high Ti₃SiC₂ content (larger than 20 vol%) is pretty low. Zhang and Chou [8] successfully prepared Cu-Ti₃SiC₂ composites (with a low Ti₃SiC₂ content of 0.5-5 vol%) by highenergy ball milling the mixed powders and then HP at 900 °C under 40 MPa for 30 min. Their studies showed that vield strength and hardness improved linearly with increasing Ti₃SiC₂ content. They did not measure the electrical conductivity of the composites. Lu et al. [16] studied the impacts of Ti₃SiC₂ content (50, 60 and 70 vol% Ti₃SiC₂) and sintering temperature (920, 940, 1000 and 1020 °C) on the properties of hot pressed Cu-Ti₃SiC₂ composites. Their results showed that



Fig. 8 (a) Tensile strength and (b) elongation of Cu-Ti₃SiC₂ composites; semi-solid symbols are data from the present research group.

by using a pressure of 30 MPa, composites (50 vol% Ti₃SiC₂) with high relative density (99.7%), high electrical conductivity and high bending strength can be obtained by 940 °C sintering, while for composites with 70 vol% Ti₃SiC₂, the optimal HP sintering temperature is 1000 °C. Lu et al. [16] and Chen [15] belong to the same research group and similar to Chen's reported results the bending strength data reported by Lu et al. [16] are somewhat higher than expected. Li [21] studied the melt infiltration process. He found that between 980 and 1020 °C, copper can melt into the porous ceramic completely. This provides a feasible way to prepare Cu-Ti₃SiC₂ composites with a high volume fraction of Ti₃SiC₂, but the presence of liquid Cu will lead to a reaction between Cu and Ti3SiC2 for sure. Ni et al. [27] invented a series of Cu-Ti₃SiC₂ materials $(0.01-80.0 \text{ mass}\% \text{ Ti}_3\text{SiC}_2)$ by the powder metallurgy method and they claimed that the invented composites can be widely used as electrical contact materials, such as high and low voltage electrical contacts, brushes, and pantograph. However, no practical application has been reported so far.

As seen from the previous paragraphs, the costly HP process (at 900 °C or over) was adopted by almost all of the researchers since it can provide high density material easily, but due to the equipment restriction, the hot pressed products are limited both in size and shape. In order to solve the above problems, the research group of the present study employed a simple warm compaction forming technique (based on the conventional powder metallurgy technique, but a moderate temperature of around 150 °C was applied during powder compaction), followed by conventional sintering at 1000 °C to prepare the Cu-Ti₃SiC₂ composites (1.25, 2.5, 5, 10 and 15 mass% Ti₃SiC₂), and found that warm compaction has significant effect on enhancing the composite's density, mechanical property and electrical conductivity. However, these beneficial effects diminish as the Ti₃SiC₂ content increases [22-24].

In order to further improve density, double press-double sinter and rolling techniques were employed by the present research group to fabricate Cu–Ti₃SiC₂ composites. When both warm compaction and rolling processes were employed, Cu–5 mass% Ti₃SiC₂ composites with a density of 8.28 g/cm³ (97.5% RD), a resistivity of $7.0 \times 10^{-8} \Omega$ m, an ultimate tensile strength of 288 MPa and a hardness of HB 110 can be obtained [25]. With double press-double sintering, Cu–10 mass% Ti₃SiC₂ composites with a density of 7.712 g/cm³ (94.98% RD) and a resistivity of $21.65 \times 10^{-8} \Omega$ m can be obtained [26]. Experimental results obtained from the present research group showed that good quality composites with Ti_3SiC_2 concentrations less than 15 mass% Ti_3SiC (about 25 vol% Ti_3SiC) can be achieved by using the warm compaction technique [22–26]. Although the mechanical properties of the composites prepared by the warm compaction and conventional powder metallurgy techniques are not as good as those of the HP or SPS sintered composites, the overall mechanical properties and electrical conductivity are good enough for electrical applications including sliding contact applications.

4. Concluding remarks

Warm compaction forming and SPS sintering were employed to fabricate a series of Ti_3SiC_2 particulate reinforced copper matrix composites. Experimental results show that density and bending strength of the sintered composite decreased linearly with increasing Ti_3SiC_2 content, while hardness and electrical resistivity increased with increasing Ti_3SiC_2 content. Although the composites sintered at 750 °C (WC+SPS) have lower density when compared with those of the composites sintered at 950 °C (SPS), their electrical resistivity values are very close to each other and even lower.

The present experimental results indicate that the reinforcing particles in Cu–Ti₃SiC₂ composites are not stable at and above 800 °C. The decomposition of Ti₃SiC₂ will lead to the formation of TiC and/or other carbides and silicides, which will degrade the lubrication characteristic of the composites. If purity is the major concern, the processing temperature (if SPS or HP is employed) and the servicing temperature of the Cu–Ti₃SiC₂ composites should be limited to 750 °C or lower. Furthermore, since the high energy arc will decompose the reinforcing Ti₃SiC₂, it seems risky to use Cu–Ti₃SiC₂ composites as the high voltage vacuum contactor material, as suggested by some researchers.

The reaction between Cu and Ti_3SiC_2 may bring about a stronger interface between the matrix and the reinforcing particles, which is beneficial to the overall properties, especially the mechanical properties, of the composites. If lubrication is not an issue, then the reaction between Cu and Ti_3SiC_2 may be tolerated during the composite preparation. Actually, high temperature (over 900 °C) sintering is preferred, since the density and mechanical properties of the Cu– Ti_3SiC_2 composites can be improved significantly while their conductivity is not substantially reduced.

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