Influences of Cu content on the microstructure and mechanical property of Ti₃SiC₂/Cu composites

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Abstract

Ti₃SiC₂/Cu composites with different Cu content were prepared by spark plasma sintering (SPS) process in vacuum and the effect of Cu content on the microstructure and mechanical property was investigated. The axial displacement, temperature and current of the composites during the sintering process were recorded and discussed. The phase compositions of the original Ti₃SiC₂ and Cu powder before and after ball-milling, and the as-produced composites were studied by XRD analysis. The surface morphology and fracture surface of the composites were investigated by SEM. The influence of Cu content on the relative density, hardness and compressive strength of the composites was inquired. The results discovered that the phase composition of Ti₃SiC₂/Cu composites varied with the content of Cu. The phase composition of the Ti₃SiC₂/5 vol% Cu composite was composed of Ti₃SiC₂, Ti₅Si₃ and Cu₃Si, while that of Ti₃SiC₂/10 vol% Cu composite and Ti₃SiC₂/15 vol% Cu composite contained TiC, besides Ti₃SiC₂, Ti₅Si₃ and Cu₃Si. Moreover, the relative density of all Ti₃SiC₂/Cu composites was relatively high (>93%). With the increase of Cu content, the axial displacement, hardness and compressive strength of the Ti₃SiC₂/Cu composites increased. Conclusively, the Ti₃SiC₂/Cu composite with 15 vol% Cu exhibited better mechanical properties.

1. Introduction

MAX phases as novel structural ceramics had received intensive and widespread attention due to their unique characteristic, a combined property of metal and ceramic [1, 2]. MAX phases were the abbreviated term of general formula Mn+1AXn [2]. Among 60 family members of MAX phases, Ti₃SiC₂ ceramic was the representative one [3, 4]. The chemical bonding in Ti₃SiC₂ ceramic contained a mixture of ionic bonds, metallic bonds and covalent bonds, endowing it a range of superior performance, such as electric and thermal conductivity, machinability, high temperature oxidation resistance, corrosion resistance. Additionally, Ti₃SiC₂ ceramic had been demonstrated to possess the electromagnetic shielding performance [5–7] and radiation resistance [8–10]. However, its anti-wear behavior was not compelling to a certain extent because of its low hardness, which limited its widespread applications. Thus, researchers focused on the strengthening of Ti₃SiC₂ ceramic.

In literatures, methods of carburization [11], sintering [11] and boronization [12] were utilized to effectively improve the surface hardness of Ti₃SiC₂. Islak et al [13] studied the reinforcing effect of TiB₂ on the properties of as-formed Ti₃SiC₂ composites. They discovered that the addition of TiB₂ obviously improved the mechanical properties and anti-wear behavior of Ti₃SiC₂ matrix. Additionally, based on the fact that Si atoms can likely de-intercalated from the crystal structure of Ti₃SiC₂, certain substances such as Cu [14–16], Ni [17], Al [18] and molten cryolite [19], were used to enhance the de-intercalation of Si from Ti₃SiC₂, leading to the structural transformation of Ti₃SiC₂ from hexagonal structure to cubic structure and strengthening the surface hardness and compressive strength of the Ti₃SiC₂ matrix.

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of Ti$_3$SiC$_2$. Especially, Cu was reactive and easy to form intermetallic compounds to strengthen the wear resistance of composites [20–22]. Interestingly, Lu et al [23] demonstrated that the wettability of Cu and Ti$_3$SiC$_2$ was good, which resulted from the formation of the interface layer between Cu and Ti$_3$SiC$_2$. Dang et al [16] prepared Ti$_3$SiC$_2$/Cu composites with 5–20 wt% Cu. It was found that the addition of Cu resulted in the breakdown of Ti$_3$SiC$_2$ to generate TiC$_x$, Ti$_5$Si$_4$C$_y$, Cu$_3$Si and TiSi$_2$C$_z$, which improved the wear resistance of the as-synthesized composites. Additionally, Zhou et al [14] synthesized Ti$_3$SiC$_2$/Cu composites with 20–70 vol% Ti$_3$SiC$_2$ and thoroughly studied the interfacial interaction between Ti$_3$SiC$_2$ and Cu. When the Ti$_3$SiC$_2$ content was low or reaction temperatures were $<$ 1000 °C, the reaction products of Ti$_3$SiC$_2$ and Cu were Cu(Si) solid solution and TiC$_x$. When the Ti$_3$SiC$_2$ content and reaction temperature were high, Ti$_3$SiC$_2$ reacted with Cu to generate some Cu-Si intermetallic compounds and TiC$_x$. In our recent publication [24], Ti$_3$SiC$_2$/Cu composites with 15 vol% Cu was prepared, and the mechanical and tribological behaviors of the composite were comparative with those of polycrystalline Ti$_3$SiC$_2$. It was discovered that the Ti$_3$SiC$_2$/Cu composite exhibited higher hardness, compressive strength and better wear resistance than polycrystalline Ti$_3$SiC$_2$. In addition, spark plasma sintering (SPS) technique had been demonstrated to be effective in synthesizing composites [25–27].

In this study, Ti$_3$SiC$_2$/Cu composites with different Cu content were synthesized by SPS technique in vacuum. Influence of Cu content on the microstructure and mechanical behaviors of the composites was explored and the corresponding sintering characteristic was discussed.

2. Experiment

2.1. Samples preparation

The Ti$_3$SiC$_2$/Cu composite was fabricated from powder mixture of Ti$_3$SiC$_2$ (400 mesh, ≥98% purity, 11 Technology Co., Ltd, Jilin, China) and Cu (200 mesh, 99.9% purity, Shanghai Macklin Biochemical Co., Ltd, Shanghai, China). The volume fraction of Cu in the Ti$_3$SiC$_2$/Cu system was 5 vol%, 10 vol% and 15 vol%, respectively. The mixture was firstly mixed by a planetary ball-milling machine (PMQD2LB, Nanjing Chishun Technology Development Co., Ltd, China) for 6 h at a rotatory speed of 150 rpm. Then, it was filled into column graphite dies with inner diameter of 25 mm. The Ti$_3$SiC$_2$/Cu composite was synthesized by a spark plasma sintering (SPS) equipment (Labox-350, SINTER LAND, Japan) under vacuum. The heating rate was 50 °C min$^{-1}$ from 570 °C to 1025 °C and the temperature was monitored by an infrared thermometer. The sample was sintered at 1025 °C for 5 min and then at 1000 °C for 15 min under a pressure of 35 MPa. Ultimately, it was cooled with the furnace.

2.2. Mechanical properties

Densities of the Ti$_3$SiC$_2$/Cu composites were measured according to Archimedes’ principle. Micro-hardness tests was investigated by a micro-hardness tester (HVT-1000A, Shanghai Zhongyan Instrument Factory, Shanghai, China) under a load of 1 kg and a dwell time of 10 s. Compressive strength was determined using a universal material tester (DWD-100, Chongqing Auto Optical Instrument Co., Ltd, China). The size of the sample for the compressive strength test was Φ 5 mm × 12 mm and the cross-head speed was 0.05 mm min$^{-1}$.

2.3. Analysis

The phase compositions of Ti$_3$SiC$_2$ powder and Cu powder before and after ball-milling, and the composites were determined by x-ray powder diffraction (XRD, DX-2700B, Dandong Haoyuan Instrument Co., Ltd, China) using Cu Kα radiation ($λ = 0.15418$ nm) with a 2θ range from 20° to 90° at a scanning rate of 7.2 °/min. The surface morphology and elemental composition of the polished composites was investigated by scanning electron microscopy (SEM, JSM-6510LA, JEOL Japan Electronics Co., Ltd, Japan) equipped with energy dispersive x-ray spectroscopy. The fracture morphology of the composites after compressive strength tests was investigated by SEM (JSM-6510LA, JEOL Japan Electronics Co., Ltd, Japan), too.

3. Results and discussion

3.1. Phase composition and microstructure

The phase composition of Cu powder, Ti$_3$SiC$_2$ powder and their mixture with 15 vol% Cu after ball-milling was shown in figure 1. It was seen from figure 1 that the mixture of Ti$_3$SiC$_2$ and Cu after ball-milling was composed of only Ti$_3$SiC$_2$ and Cu, and no other phase was detected. In the literature [16], Ti$_3$SiC$_2$/10 wt% Cu was mixed by a high energy ball-milling machine and the phase composition of the Ti$_3$SiC$_2$/10 wt% Cu after mechanical alloying included Ti$_3$SiC$_2$, Cu, TiC$_x$ and Cu (Si) solid solution. It was concluded that Cu reacted with Ti$_3$SiC$_2$ producing TiC$_x$ and Cu (Si) solid solution during high-energy ball milling. In our present study, the mixture of
Ti$_3$SiC$_2$ and Cu powder was mixed by a planetary ball-milling machine, not a high-energy ball-milling machine. Thus, there was no reaction between Ti$_3$SiC$_2$ and Cu during ball-milling.

Figure 2 showed the XRD patterns of the Ti$_3$SiC$_2$/Cu composites with different Cu content synthesized by SPS. It was seen from figure 2 that Ti$_3$SiC$_2$ reacted with Cu during the sintering process and no Cu peak was found in the XRD patterns of all the composites. When the Cu content was 5 vol%, the phase composition of the composite was composed of Ti$_3$SiC$_2$, Ti$_5$Si$_3$, and Cu$_3$Si. When the content of Cu increased to 10 vol% and 15 vol%, the composition of these two composite included TiC, besides Ti$_3$SiC$_2$, Ti$_5$Si$_3$, and Cu$_3$Si. Moreover, with the increase of Cu content, the intensity of Ti$_3$SiC$_2$ peak was weaker, while these of Cu$_3$Si and TiC peaks were stronger. It was deduced that the addition of Cu led to the break-down of Ti$_3$SiC$_2$, and the more the Cu content was, the more violent Ti$_3$SiC$_2$ decomposed. This result was consistent with that in literature [16].
Figure 3 showed the backscattered electron image and the mapping of elements of Ti$_3$SiC$_2$/Cu composites with different Cu content prepared by SPS. It was clearly indicated that the light grey regions distributed between the dark grey regions. In accordance with the mapping of element, the light grey regions abounded in Cu while the dark grey regions lacked Cu. It was guessed that the light grey regions were primarily made up of Cu$_3$Si, and the dark grey regions were consisted of the major Ti$_3$SiC$_2$ and the minor Ti$_5$Si$_3$ and/or TiC. The as-produced Cu$_3$Si distributed around the Ti$_3$SiC$_2$ grains. Additionally, with the increase of Cu content, the agglomeration of Si element was obviously restricted, in other word, the Ti$_3$SiC$_2$ grains got smaller. Therefore, it was inferred that the increase of Cu content was beneficial to the reaction between Ti$_3$SiC$_2$ and Cu.

The reaction mechanism of Ti$_3$SiC$_2$ and Cu was proposed as follows. As we all known, the crystalline structure of Ti$_3$SiC$_2$ can be depicted as a layer of Si atoms intercalated into the twin boundary of TiC and the interaction between Ti and Si was relatively weak in contrast with that between Ti and C. Such a structure contributed to the high reaction activity of Ti$_3$SiC$_2$. At the sintering temperature (1025 °C), the weak interaction between Ti and Si was favorable for the de-intercalation of Si atoms from Ti$_3$SiC$_2$, causing to the decomposition of Ti$_3$SiC$_2$. In other hand, the de-intercalated Si atoms diffused around Cu particles and combined with it to produce the only Cu-Si intermetallic compound, Cu$_3$Si. Guo et al. demonstrated that when Cu content was lower than the content of Si, the preferential outcome of Cu-Si system was Cu$_3$Si. The reason was that the Si content in Cu$_3$Si (22.2%–25.2%) was the highest among the Cu-Si system. This explained the fact that Cu$_3$Si was the only Cu-Si compounds in our present study. This was in accordance with the outcomes given by Zhou et al. The more detailed microstructural evolution can be found in reference 28.

3.2. Sintering characteristics of the Ti$_3$SiC$_2$/Cu composites

Figure 4 showed the relationship of the axial displacement, the temperature, and the current with sintering time for the Ti$_3$SiC$_2$/Cu composites with different Cu content sintered by SPS. As seen in figure 4(a), as the temperature increased, the axial displacement of the Ti$_3$SiC$_2$/Cu composites showed a maximum at about 300 s, which was owing to the thermal expansion of the Ti$_3$SiC$_2$/Cu system. From 570 °C, the temperature was monitored by an infrared thermometer and the heating rate was adjusted to 50 °C/min. Because from room temperature to 570 °C, the heating rate was higher than 50 °C/min, thus, the current was suddenly decreased to a low value from the initial monitor temperature of the infrared thermometer (570 °C). Therefore, the temperature exhibited a down trend until the current increased to a relative high value (see figures 4(b)–(d)) and the axial displacement exhibited a similar down trend as the temperature (see figure 4). The decrease of the axial displacement resulted from the effect of cold shrinkage of the Ti$_3$SiC$_2$/Cu system. As the temperature increased from a critical temperature (900 °C for Ti$_3$SiC$_2$/5 vol% Cu and Ti$_3$SiC$_2$/10 vol% Cu, and 800 °C for Ti$_3$SiC$_2$/15 vol% Cu) to 1025 °C, the axial displacement of the Ti$_3$SiC$_2$/Cu system quickly decreased. When the temperature was stable at 1025 °C for 5 min and at 1000 °C for 15 min, the decrease rate of the axial displacement of the Ti$_3$SiC$_2$/Cu system was also rapid, but it was apparently not higher than that before 1025 °C. The decrease of the axial displacement from the critical temperature to the sintering temperature mainly originated from the
densification action because of the successive pressure at the sintering temperatures, and partly owing to the reaction between Ti$_3$SiC$_2$ and Cu. The axial displacement of the Ti$_3$SiC$_2$/15 vol% Cu rapidly decreased at a lower temperature and much earlier than that of Ti$_3$SiC$_2$/5 vol% Cu and Ti$_3$SiC$_2$/10 vol% Cu. It was deduced that the higher electrical conductivity of Cu was helpful in the SPS sintering of the Ti$_3$SiC$_2$/Cu system. It had been proved that the formation of intermetallic compounds can change the electricity of AlMgSiCu alloys [29, 30]. Moreover, the higher the Cu content was, the more significant the effect of Cu worked. Therefore, the axial displacement of the Ti$_3$SiC$_2$/15 vol% Cu was the earliest and the biggest among the three Ti$_3$SiC$_2$/Cu system, and the axial displacement of the Ti$_3$SiC$_2$/5 vol% Cu was the smallest.

3.3. Mechanical property
The relative density, hardness and compressive strength of the Ti$_3$SiC$_2$/Cu composites with different Cu content were shown in figure 5. As can be observed in figure 5, the relative density of all Ti$_3$SiC$_2$/Cu composites was relatively high, indicating its good compactness. The Ti$_3$SiC$_2$/10 vol% Cu composite showed the highest relative density (96%) and the Ti$_3$SiC$_2$/15 vol% Cu composite showed the lowest one (93%). The Cu content in the Ti$_3$SiC$_2$/15 vol% Cu composite was highest among three Ti$_3$SiC$_2$/Cu composites. There was such more Cu in the Ti$_3$SiC$_2$/15 vol% Cu composite that it induced the intense decomposition of Ti$_3$SiC$_2$ and violently reacted with the de-intercalated Si to form Cu$_3$Si, causing the generation of opening holes.

As shown in figure 5, the hardness and compressive strength of the Ti$_3$SiC$_2$/Cu composites increased with the increase of the Cu content. The hardness of the Ti$_3$SiC$_2$/Cu composites increased from 3 GPa for Ti$_3$SiC$_2$/5 vol% Cu composite to approximate 7 GPa for Ti$_3$SiC$_2$/15 vol% Cu composite and the compressive strength of the Ti$_3$SiC$_2$/Cu composites increased from 300 MPa for Ti$_3$SiC$_2$/5 vol% Cu composite to 1500 MPa for Ti$_3$SiC$_2$/15 vol% Cu composite. Compared with polycrystalline Ti$_3$SiC$_2$ (5.5 GPa for hardness and 1232 MPa for compressive strength) [31], the hardness and compressive strength of both Ti$_3$SiC$_2$/5 vol% Cu and Ti$_3$SiC$_2$/10 vol% Cu composites exhibited lower values, while these of Ti$_3$SiC$_2$/15 vol% Cu composite showed higher values. The higher hardness of Ti$_3$SiC$_2$/15 vol% Cu composite was perhaps related to the formation of higher amount of hard TiC product (see figure 2), while the higher compressive strength of Ti$_3$SiC$_2$/15 vol% Cu composite was possibly in connection with the denser structure consisting of relatively uniform grain sizes (see figure 6(d)).
Figure 6 illustrated the compressive stress-strain curves of Ti$_3$SiC$_2$/Cu composite with different content of Cu. As shown in figure 6(a), all the Ti$_3$SiC$_2$/Cu composite only suffered from elastic deformation before fracture, which was a typical characteristic of brittle fracture mode. The fracture surface of Ti$_3$SiC$_2$/5 vol% Cu composite was covered by loosely scattered grains with different sizes. The weak interaction among these grains led to the lowest compressive strength of Ti$_3$SiC$_2$/5 vol% Cu composite (see figure 5). In comparison, the fracture surface of both Ti$_3$SiC$_2$/10 vol% Cu composite and Ti$_3$SiC$_2$/15 vol% Cu composite was denser than that of Ti$_3$SiC$_2$/5 vol% Cu composite (see figures 6(b)–(d)). Additionally, the grain size of the Ti$_3$SiC$_2$/15 vol% Cu composite was relatively uniform while the Ti$_3$SiC$_2$/10 vol% Cu composite contained abnormally grown grains. Therefore, the compressive strength of the Ti$_3$SiC$_2$/15 vol% Cu composite was the highest among three.
Ti$_3$SiC$_2$/Cu composites. From the fracture morphology of the composites (see figure 6(b)), it was apparently seen that both the intergranular fracture and transgranular fracture were present among the composites with different Cu contents, which indicated that the fracture mode of the composites was independent with the Cu contents.

4. Conclusions

Ti$_3$SiC$_2$/Cu composites with different Cu content were sintered by spark plasma sintering (SPS) process. The phase composition of Ti$_3$SiC$_2$/Cu composites varied with the content of Cu. The phase composition of the Ti$_3$SiC$_2$/5 vol% Cu composite was composed of Ti$_3$SiC$_2$, Ti$_5$Si$_3$ and Cu$_3$Si, while that of Ti$_3$SiC$_2$/10 vol% Cu composite and Ti$_3$SiC$_2$/15 vol% Cu composite included TiC, besides Ti$_3$SiC$_2$, Ti$_5$Si$_3$ and Cu$_3$Si. The relative density of all Ti$_3$SiC$_2$/Cu composites was relatively high ($\geq$93%) and Ti$_3$SiC$_2$/10 vol% Cu composite exhibited the highest relative density. With the increase of Cu content, the axial displacement, hardness and compressive strength of the Ti$_3$SiC$_2$/Cu composites increased. In short, the optimum Cu content was 15 vol% and the corresponding Ti$_3$SiC$_2$/Cu composite exhibited better mechanical properties.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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