Effect of Si element on the interfacial reaction of Ti$_3$SiC$_2$ reinforced Al matrix composites

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Abstract
The interfacial reaction behavior of Ti$_3$SiC$_2$/Al and Ti$_3$SiC$_2$/Al-10Si matrix composites at high temperature was studied by XRD and SEM. The results showed that the reaction between Ti$_3$SiC$_2$ and Al matrix is Ti$_3$SiC$_2$ + Al $\rightarrow$ TiCx + Si + Al$_3$Ti. Ti$_3$SiC$_2$ particles in the Ti$_3$SiC$_2$/Al composite were almost completely reacted after 700 °C/1 h treatment, while a large amount of Ti$_3$SiC$_2$ particles still existed in the Ti$_3$SiC$_2$/Al-10Si composite under the same treatment condition. In addition, the friction coefficient and wear amount of Ti$_3$SiC$_2$/Al composite were 0.54 and 0.167 g, respectively, and that in Ti$_3$SiC$_2$/Al-10Si matrix composites were 0.29 and 0.019 g. According to the critical condition of $\Delta G = 0$, it is calculated that when the activity of Si is greater than 0.57, the interfacial reaction between Ti$_3$SiC$_2$ and Al matrix is inhibited.

1. Introduction

The MAX phase is a layered ternary compound [1] with both metallic and ceramic properties. The molecular formula of MAX phase is M$_{n-1}$AX$_n$, and its crystal structure is formed by alternately stacking MX sheets and A atom layers. As a typical representative of MAX, Ti$_3$SiC$_2$ has good processability [2], high electrical conductivity (4.5 $\times$ 10$^6$ Ω m$^{-1}$) [3], high elastic modulus ($\sim$325 GPa) [4], high melting point ($\sim$3000 °C) good oxidation resistance [5], and low friction coefficient [6], making it a candidate material as a reinforcing phase for self-lubricating metal matrix composite [7].

However, similar to common MAX, Ti$_3$SiC$_2$ has a weak bond in the crystal structure, and the binding energy between Ti and C is much higher than that between Ti and Si [8] (in Ti$_2$SiC$_2$, the binding energies of Ti 2p, Si 2p, and C 1s are 454.9 eV, 99.5 eV, and 282.1 eV, respectively). Such microstructure makes the Ti$_3$SiC$_2$ reinforced metal matrix composites prone to have interfacial reactions during the preparation process. Even at temperatures far below the melting point of Ti$_3$SiC$_2$, it is still difficult to prepare pure Ti$_3$SiC$_2$ reinforced metal matrix composite. For example, at higher temperature, Ti$_3$SiC$_2$/Ti [9], Ti$_3$SiC$_2$/Cu [10], Ti$_3$SiC$_2$/Ni [11] metal matrix composites will undergo interfacial reactions, resulting in the disappearance of Ti$_3$SiC$_2$ particles and the formation of TiCx, Ti$_3$Si$_3$, and other compounds. Chen Xinhua et al. [12] and Li Mengqi [13] studied the high-temperature reaction behavior between the max phase of Ti-Al-C system and metals, and considered that the atom deblocking from the max particles led to the dissociation of Max grains, the formation of smaller particles and the in situ reaction to generate MX phase. At the same time, the metal atoms diffused into the max grains through vacancies. Therefore, the key to the preparation of Ti$_3$SiC$_2$/metal matrix composites is how to control the interfacial reaction between Ti$_3$SiC$_2$ and metal matrix by inhibiting the in situ embedding of Si atoms.

Similar to Ti$_3$SiC$_2$ particles, SiC is a commonly used strengthening phase for the preparation of particle-reinforced Al-based composites. SiC particles can also have interfacial reaction with Al, and the reaction products are Al$_4$C$_3$ and Si. At present, researchers at home and abroad have studied the inhibition of interface reaction by adding Si element in SiC/Al composites through experiments or thermodynamic calculation [13–15]. Similar to the reaction between SiC and Al, Si is also one of the main products between the max phase of Ti-Si-C system and Al matrix. Therefore, the interface reaction between Ti$_3$SiC$_2$ and Al matrix is also affected by the addition of Si. For the research on the interface reaction between Max phase and metal matrix, researchers...
mostly identify and analyze the phase types of reaction products [16], while the research on controlling the interfacial reaction between Ti3SiC2 and Al matrix by introducing the concept of element activity is rare. In this research, two kinds of composites Ti3SiC2/Al and Ti3SiC2/Al-10Si with no obvious interfacial reaction were prepared by SPS process. High temperature insulation treatment was performed on the composites, and the effects of composition and heat preservation conditions on the friction coefficient and wear loss under the same wear time were studied. Then, XRD was employed to research the interfacial reaction products between the added particles and the matrix. The morphology of the products in interfacial reaction was further characterized by SEM technique. The distribution characteristics of the main compositions of the composites were analyzed by EDS technique. Finally, the reason why Si element affected the interfacial reaction between Ti3SiC2 and Al matrix was preliminarily discussed through thermodynamic calculation.

2. Experimental method

Ti3SiC2 (<10 μm, 99.8%), Al powder (<75 μm, 99%) and Al-Si powder (<50 μm, 99.9%) were used as raw material to prepare the composites of Ti3SiC2/Al and Ti3SiC2/Al-10Si with a mass ratio of Ti3SiC2:Al = 1:9, and Ti3SiC2:Si:Al = 1:1:8, respectively. The powders were weighed according to the ratio, and then wetted with appropriate amount of alcohol on a PMQW planetary ball mill. The grinding ball was Zirconia, with a mass ratio of ball: powder = 3:1 and milling speed of 200 r min⁻¹ for 7 h. The milling process was protected by argon. The mixed powders were charged into a graphite mold and sintered in an ST-5 SPS sintering furnace. The vacuum degree in the furnace was 10⁻² Pa, the heating rate was 80 °C min⁻¹, and the sintering temperature was 50 °C ~ 60 °C below the liquidus temperature of the alloy, pressure of 40 MPa was applied, and the product was kept warm for 10 min, followed by cooling to room temperature within the furnace. The subsequent heat treatment was carried out in a muffle furnace at 500 °C/1 h, and 700 °C/1 h, respectively. Phase analysis was performed using an Empyean x-ray diffractometer (XRD), with Cu as the target, operating voltage of 30 kV, current of 40 mA, and scanning angle of 30 ~ 100°. The microstructure and morphology of the samples were observed by an FEI MLA650F field emission scanning electron microscope (SEM). The selected areas in the samples were analyzed by an energy disperse spectrometer (EDS).

The ring-block wear test was carried out on an M-2000 high-speed reciprocating wear tester. The friction pair used GCr15 steel with inner diameter of 20 mm, outer diameter of 40 mm and thickness of 10 mm. The wear test was dry wear, with load of 50 N, rotation speed of 200 r min⁻¹, and test time of 10 min.

3. Experimental results and analysis

3.1. Results of friction and wear test

Ti3SiC2 has a lamellar structure similar to that of graphite and MoS2 [17], and oxide film [18] can be formed on the surface of Ti3SiC2 at high speed sliding, thus reducing the friction coefficient. However, when tic and other compounds exist in Ti3SiC2, TiC particles are easy to fall off at high speed or high pressure, resulting in significant increase in friction coefficient and wear rate [19]. When the interface reaction of Ti3SiC2 occurs, Al3Ti, Al6SiC4, Al4C3 and other compounds will be formed [20], which will destroy the structure of Ti3SiC2, lose...
the self-lubricating effect, and increase the friction coefficient and wear loss. Based on this phenomenon, friction and wear tests of Ti3SiC2/Al Composites in different states were carried out.

Figure 1 shows the curve of friction coefficient versus time for pure Al, Al-10Si alloy, Ti3SiC2/Al composite and Ti3SiC2/Al-10Si composite. The pure Al and Al-Si alloys were obtained by melt casting, Ti3SiC2/Al and Ti3SiC2/Al-10Si composites were prepared by SPS process and heat-treated at 700 °C for 1 h. It can be seen from figure 1 that as the test time increases, the friction coefficient-time curve of all samples gradually becomes stable. The Ti3SiC2/Al and Ti3SiC2/Al-10Si composites both show low friction coefficient, and their steady-state friction coefficients are 0.23 and 0.17, respectively. After the heat treatment of 700 °C/1 h, the friction coefficients are increased to 0.54 and 0.29, respectively, an increase of 134% and 70%, respectively. Therefore, the results indicate that the addition of Ti3SiC2 causes a significant reduction in the friction coefficient of the sintered composites with pure Al and Al-10Si alloy as the matrix. However, after high-temperature heat treatment, the friction coefficient and wear amount of the samples both increase significantly.

Table 1 shows the wear amount of the test materials. It can be seen that the variation of the wear amount is consistent with the change of the friction coefficient, that is, the addition of Ti3SiC2 leads to a significant decrease in the wear amount of the sintered material relative to the cast material, and the high-temperature heat treatment causes a re-increase of the wear amount. After the 700 °C/1 h treatment, the wear of Ti3SiC2/Al and Ti3SiC2/Al-10Si composites increase by 1013% and 111%, respectively, relative to that of the sintered material. The change of tribological properties under high temperature may result from the interfacial reaction between Ti3SiC2 particles and the matrix. It is worth noting that the addition of Si significantly reduces the damage to the tribological properties of the interface due to the interfacial reaction. So, it can be deduced that Si may have an effect of suppressing the interfacial reaction between Ti3SiC2 and the Al matrix.

3.2. XRD results

Figure 2 shows the XRD patterns of Ti3SiC2/Al and Ti3SiC2/Al-10Si composites treated at different temperature for 1 h: (a) Ti3SiC2/Al composite and (b) Ti3SiC2/Al-10Si composite.

| Steady-state friction coefficient | Wear amount (g) |
|----------------------------------|-----------------|
| SPS sintered Ti3SiC2/Al          | 0.23            | 0.015          |
| Ti3SiC2/Al after 700 °C/1 h treatment | 0.34            | 0.167          |
| Pure Al                          | 0.83            | 0.45           |
| SPS sintered Ti3SiC2/Al-10Si     | 0.17            | 0.009          |
| Ti3SiC2/Al-10Si after 700 °C/1 h treatment | 0.29           | 0.019          |
| Al-10Si alloy                    | 0.51            | 0.15           |

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diffraction peak of Ti$_3$SiC$_2$ shifts to a low angle, and the diffraction peak of Si appears at $2\theta = 47.3^\circ$ and $69.3^\circ$. At this time, the lattice constant of the Al matrix changes from 0.40499 to 0.40565, and the temperature is increased to 700°C. The diffraction peaks corresponding to the crystal planes (104), (008) and (105) of Ti$_3$SiC$_2$ disappear completely, and the phases under this state are Al phase, Al$_3$Ti phase, Si, and a TiCx phase. As shown in figure 2(b), the phase composition of the Ti$_3$SiC$_2$/Al-10Si composite does not change during the treatment at 500°C for 1 h. When the temperature increases to 700°C, the diffraction peaks corresponding to the crystal plane (105) of Ti$_3$SiC$_2$ disappears, and the diffraction peaks of TiCx and Al$_3$Ti appear, but the diffraction peaks corresponding to the crystal plane (104) and (008) of Ti$_3$SiC$_2$ still exist. Therefore, the SPS process can be used to prepare the interfacial reaction-free Ti$_3$SiC$_2$/Al and Ti$_3$SiC$_2$/Al-10Si composites. After the 700°C/1 h treatment, the interfacial reaction occurs in both composites, with the products being TiCx, Al$_3$Ti and Si. Compared with Ti$_3$SiC$_2$/Al composite, Ti$_3$SiC$_2$ phase exists in Ti$_3$SiC$_2$/Al-10Si composite with high temperature treatment, indicating that Si have the effect of suppressing rather than preventing the interfacial reaction between Ti$_3$SiC$_2$ and Al.

3.3. SEM and EDS results
Figure 3 shows the SEM image and surface scanning result of the SPS sintered Ti$_3$SiC$_2$/Al composite. From the SEM image, we can see the dark, nearly circular matrix with a grain size of about 1.9–28.6 μm and bright white,
irregularly-shaped particles with a size of less than 6.9 μm and mainly distributed at the grain boundaries. It can be seen from the distribution characteristics of Ti, Si, C and Al elements that Al is the main element of the composite matrix, and the boundary between the black region and the Al matrix at the grain boundary is clear. In addition, the aggregation regions of Ti, Si and C elements are almost complete and the aggregation area is located at the grain boundary, with no obvious distribution of Al element. It is preliminarily concluded that these bright color particles are Ti3SiC2.

Table 2 shows the EDS results of SPS sintered Ti3SiC2/Al composite (atomic percent).

|   | Ti  | Si   | C     | Al  |
|---|-----|------|-------|-----|
| 1 | —   | —    | 20.91 | 79.09 |
| 2 | 52.1| 14.46| 31.04 | 2.38 |

Figure 4. SEM image and surface scanning result of Ti3SiC2/Al composite treated at 700 °C/1 h (b) Al element (c) Si element (d) C element (e) Ti element.

Table 2 shows the EDS results of SPS sintered Ti3SiC2/Al composite. At position 1, Al has the highest content, with Ti and Si undetected, indicating that position 1 is the matrix. At position 2, Ti has relatively high content, followed by C, and Al has the lowest content. However, C is a light element, and the EDS results cannot accurately determine its content, only with qualitative or semi-quantitative analysis. By calculating the atomic
ratio of Ti element and Si element, it is found that the Ti/Si ratio is basically consistent with the Ti/Si ratio in Ti$_3$SiC$_2$. Summarizing the XRD and EDS results, it can be deduced that the bright color particles at position 2 in figure 3(a) are Ti$_3$SiC$_2$.

Figure 4 shows the SEM image and surface scanning result of the Ti$_3$SiC$_2$/Al composite treated at 700 °C/1 h. Compared with the SPS sintered Ti$_3$SiC$_2$/Al composite, the grain size of the matrix in Ti$_3$SiC$_2$/Al composite becomes larger (15 ~ 36 μm), and there are two kinds of second phase particles in the structure, with one having a relatively regular morphology (size of 2 ~ 8 μm) and mainly distributed inside the grain and the other not having a regular morphology. It can be seen from the distribution characteristics of the constituent elements that there is a significant Ti segregation at the location of the second phase particles of the regular morphology, and

|   | Ti   | Si   | C    | Al   |
|---|------|------|------|------|
| 1 | 0.10 | 2.05 | 27.94| 69.61|
| 2 | 14.77| 2.75 | 39.30| 43.18|
| 3 | 3.49 |18.62 | 37.52| 40.37|

**Table 3.** EDS result of Ti$_3$SiC$_2$/Al composite treated at 700 °C/1 h (atomic percent).
the Ti segregation area coincides with the range and shape of the second phase particles, indicating that Ti element is the main composition of the phase. Si element is segregated in the same area, but the segregation area is significantly smaller than that of Ti element. C element has no segregation characteristics at the same position. The second phase with irregular shape is mainly distributed between the grains of the matrix, and the Ti, Si and C elements are all segregated at the position of the particles. It is speculated from the segregation characteristics of the elements that the two phases are the reaction products between Ti$_3$SiC$_2$ and Al.

Table 3 shows the EDS results of Ti$_3$SiC$_2$/Al-10Si composite treated at 700 °C/1 h. At position 1, Al has the highest content, indicating the matrix here. The atomic contents of Ti and Si are 0.1% and 2.05%, respectively, which is a significant increase compared with the matrix of SPS sintered Ti$_3$SiC$_2$/Al composite. At position 2, the

|       | Ti  | Si  | C   | Al  |
|-------|-----|-----|-----|-----|
| 1     | 0.68| 5.7 | 15.67| 77.95|
| 2     | 46.55| 10.51| 26.02| 6.92 |
| 3     | 0.75| 34.14| 7.5 | 57.61|

**Figure 6.** SEM image and surface scanning result of Ti$_3$SiC$_2$/Al-10Si composite treated at 700 °C/1 h (b) Al element (c) Si element (d) C element (e) Ti element.
Al with regular morphology particles has the highest content, followed by Ti, and the Al/Ti ratio is about 3:1. Besides, there is also a certain amount of Si (2.75). By referring to the XRD results, it is considered as Al, Ti(Si) phase, which is consistent with the interfacial reaction product described in [13]. At position 3, Al has the highest content, followed by Si element, and Ti has the lowest content. Combined with the XRD results, it is deduced that the phase may be an elemental Si phase, which is small in size and inevitably subjected to the effect from the surrounding matrix during energy spectrum analysis. Therefore, the energy spectrum analysis results indicate a large amount of Al element.

Figure 5 shows the SEM image and surface scanning result of SPS sintered Ti3SiC2/Al-10Si composite. It can be seen from the SEM image that the grains of the matrix in the composite are approximately circular and have a size of about 9–24 μm. In addition, the image shows two differently distributed second phase particles, one of which are bright and large particles concentrated near the grain boundary with size of less than 8 μm, and the other of which are fine particles with darker brightness evenly distributed in the matrix. It can be seen from the distribution characteristics of the elements that Al element is still the main constituent element of the matrix, and that the Si element at the grain boundary is basically the same as the distribution of Ti and C elements at the grain boundary. So, it is speculated that the bright large particles are Ti3SiC2, and the phase with small particles in the crystal has a significant amount of Si element. Combined with XRD results and considering that one of the raw materials used is Al-Si alloy powder, it is deduced that the particle is Si phase.

Table 4 shows the EDS result of SPS sintered Ti3SiC2/Al-10Si composite. The content of Al At position 1, Al has the highest content, and the contents of Ti and Si are lower, indicating the matrix at this position. At position 2, there are large particles near the grain boundary, in which Ti has the highest content, followed by C element. Besides, the Ti/Si ratio is about 3:1, which is consistent with the Ti/Si ratio in Ti3SiC2. It is inferred that these bright and large particles are Ti3SiC2. At position 3, there are small particles in the matrix, where Si content is second only to Al, and the content of Ti and C is extremely low. Combined with XRD results, it is deduced that these bright small particles are elemental Si phase, which is consistent with the surface scanning result.

Figure 6 shows the SEM image and surface scanning result of Ti3SiC2/Al-10Si composite after 700 °C/1 h treatment. Compared with the SPS sintered Ti3SiC2/Al-10Si composite, the grain size of the matrix has increased (about 14 ~ 44 μm), and there are second phase particles extending along the grain boundary and extending in the structure, as indicated by the arrow 2. The portion in contact with the crystal has a regular shape, and the shape of the other side substantially coincides with the shape of the grain boundary. The distribution characteristic of Ti element coincides with the range and shape of the particle. Therefore, Ti element is the main constituent element of the phase, Si element also segregates here, and C element is not segregated here. A large amount of irregular second phases are distributed at the grain boundaries, as shown in position 3, where there is significant segregation of Ti, Si and C elements. Combined with XRD results, it is deduced that these particles are elemental Si phase, which is consistent with the surface scanning result.

Table 5 shows the EDS result of Ti3SiC2/Al-10Si composite treated at 700 °C/1 h. At position 1, Al has the highest content, indicating the matrix. At position 2, the Al/Ti ratio is about 3:1, which is the same as the atomic ratio of Al/Ti. Besides, there is Si element (1.75%) at this position. Considering that the distribution areas of Si element and Ti element are substantially coincident, the phase is deduced as Al,Ti(Si) phase. At position 3, the Ti/Si ratio is about 3:1, which is close to the Ti/Si ratio in Ti3SiC2. It can be considered as residual Ti3SiC2 particles.

In summary, both Ti3SiC2/Al and Ti3SiC2/Al-10Si composites can have interfacial reaction. At 700 °C, Ti3SiC2/Al composite shows slight interfacial reaction and Ti3SiC2 particles still exist. By contrast, the interfacial reaction in Ti3SiC2/Al composite is stronger as the presence of Ti3SiC2 cannot be detected by XRD and SEM after the same treatment (700 °C/1 h). Therefore, it can be confirmed that the addition of Si hinders the interfacial reaction between Ti3SiC2 and the matrix Al. According to the energy spectrum analysis of the matrix and the second phase of the particles before and after the heat treatment, we can summarize the reaction between the Ti3SiC2 phase and the Al matrix as follows. The Si atoms in the Ti3SiC2 particles are deintercalated into the Al matrix to form solid solution elements and elemental Si phase. The Ti3SiC2 particles have in situ dissociation reaction to form TiCx phase. At the same time, the Al atoms diffuse into the Ti3SiC2 particles and

| Position | Ti    | Si    | C     | Al    |
|----------|-------|-------|-------|-------|
| 1        | 0.28  | 0.29  | 45.58 | 53.84 |
| 2        | 9.77  | 1.75  | 57.41 | 31.08 |
| 3        | 31.78 | 10.93 | 46.08 | 11.20 |
react with the Ti atoms to form the Al$_3$Ti phase. Therefore, the deintercalation of Si atoms is a key process for the reaction between Ti$_3$SiC$_2$ and Al. In SiC/Al composites, Handwerker [21] investigated the stability of SiC/Al system and found that the activity of Si in Al matrix affected the change of free energy of reaction, and then the activity of Si could be used to control the interfacial reaction. Xie et al [16] studied the process of preparing SiC/Al-7Si-5Mg-based composites by pressureless infiltration. They found that the increase of Si activity could significantly inhibit the formation of harmful interfacial reaction product Al$_4$C$_3$.

According to the XRD and EDS results, the interfacial reaction equation between Ti$_3$SiC$_2$ and Al is expressed as:

\[
2\text{Ti}_3\text{SiC}_2 + 6\text{Al} = 4\text{TiCx} + 2\text{Si} + 2\text{Al}_3\text{Ti(Si)}
\]  

(1)

It is assumed that in a closed system with constant temperature and pressure, there is a chemical reaction expressed by:

\[
a\text{A} + b\text{B} = c\text{C} + d\text{D}
\]

When a unit reaction occurs, the Gibbs function variable $\Delta G$ of the system is given by:

\[
\Delta G = RT \ln \frac{\alpha^3\text{Si}}{\alpha^{3\text{Al}}} + 4\Delta \alpha G^0(\text{TiC}) + 2\Delta \alpha G^0(\text{Al}_3\text{Ti}) - 2\Delta \alpha G^0(\text{Ti}_3\text{SiC}_2)
\]

(3)

where $\Delta \alpha G^\theta$, $\Delta \alpha G^\theta(\text{Ti}_3\text{SiC}_2)$, $\Delta \alpha G^\theta(\text{Al}_3\text{Ti})$ and $\Delta \alpha G^\theta(\text{TiC})$ are the Gibbs free energy generated at 700 °C for 1 h, Ti$_3$SiC$_2$ phase in Ti$_3$SiC$_2$/Al composites under the same treatment conditions. When the activity of Si exceeds 0.57, the interfacial reaction between the reinforcing particles Ti$_3$SiC$_2$ and the Al matrix will become positive and the reaction will be inhibited.

4. Conclusions

The main conclusions of this research are summarized as follows.

(1) After treatment at 700 °C for 1 h, Ti$_3$SiC$_2$ phase in Ti$_3$SiC$_2$/Al Composite completely disappeared, and TiCx, Al$_3$Ti and Si reaction products were produced. However, Ti$_3$SiC$_2$ phase still exists in Ti$_3$SiC$_2$/Al Si composites under the same treatment conditions.

(2) The addition of Si can improve the tribological properties of Ti$_3$SiC$_2$/Al composites, and the friction coefficient and wear loss of Ti$_3$SiC$_2$/Al composites decreased from 0.54 g and 0.167 g to 0.29 g and 0.019 g, respectively.

(3) The reaction between Ti$_3$SiC$_2$ and Al matrix is Ti$_3$SiC$_2$ + Al → TiCx + Si + Al$_3$Ti. According to the critical condition of $\Delta G = 0$, the $\alpha_{\text{Si}}$ activity after Ti$_3$SiC$_2$ reacting with Al is calculated as 0.57. When the activity of Si exceeds 0.57, the interfacial reaction between the reinforcing particles Ti$_3$SiC$_2$ and the Al matrix is inhibited.

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