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Effects of alloy elements on the compression properties of *in-situ* nano-Ti_5Si_3/TiAl composite

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The low ductility and insufficient strength of the TiAl alloy are still two major obstacles for its applications. It was demonstrated that Ti_5Si_3 could improve the ductility of TiAl alloy, while it can't obviously improve the strength. In order to further improve the strength of Ti_5Si_3/TiAl composite to obtain TiAl materials with excellent strength and ductility, the effects of alloying elements (Cu, Zn, W and Mo) on the compression strength of the *in-situ* nano-Ti_5Si_3/TiAl composite were investigated in this work. With the addition of 2 at.% Me (Me = Cu, Zn and W, respectively), the compression true yield strength (σ^y_{true}) of the composite increases from 613 MPa to 840, 660 and 706 MPa, respectively. Moreover, the addition of Zn results in the increase of the σ^y_{true} with no obvious sacrifice of ductility. Finally, the 4 vol.% nano-Ti_5Si_3/TiAl-2Zn composite obtained the best comprehensive properties. Compared with the TiAl alloy, the σ^y_{true}, $\sigma^{\text{UCS}}_{\text{true}}$ and $\varepsilon^f_{\text{true}}$ of the Ti_5Si_3/TiAl-2Zn composite increases by 195, 140 MPa and 2.6%, respectively.

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1. Introduction

TiAl alloys have attracted extensive attention as a candidate for the high-temperature automobile and aerospace material due to its low density, high melting point, low creep rate and excellent high-temperature oxidation resistance.[1,2] However, the low ductility and insufficient strength of the TiAl alloys are still two major obstacles for its applications.[3]

In recent decades, increasing researches and reports proved that composite technology is an effective approach to enhance the material strength.[4–6] Xiang *et al.*[7] fabricated the Al_2O_3/TiAl by a hot-press-assisted exothermic dispersion method. Their results showed that the flexural strength of sintered sample could reach up to 420 MPa, while the fracture toughness was about 4.22 MPa $m^{1/2}$. Yang *et al.*[8] fabricated the Ti_2AlC/TiAl composites by the method of spark plasma sintering (SPS) through a mixture powder. They pointed out that the compression yield strength of the sintered sample reached 2058 MPa, while the fracture strain was about 0.16%. These above results indicate that the strength improvement of the TiAl composites by the addition of ceramic particles was usually at the cost of the ductility. It has been proved in our previous work that among the usually used reinforcing particle in TiAl alloy, only the Ti_5Si_3 particle could improve the ductility of TiAl alloy.[9,10] However, the addition of Ti_5Si_3 can't obviously enhance the strength of the TiAl alloy. In order to obtain the TiAl alloy with excellent strength and ductility simultaneously, it is necessary to explore another method to further improve the strength of the Ti_5Si_3/TiAl composite. Element alloying has been proved as an effective method to improve the TiAl strength.[11] In general, the addition of alloying elements would form a solid solution or precipitate in the alloys on the basis of the elemental characteristic and processing procedure.[12,13] Nevertheless, the effect of alloying elements would be more complex in TiAl matrix composite and it is needed to further investigate that whether they are also beneficial to the strength of Ti_5Si_3/TiAl composite.

In this study, we tried to fabricate the Ti_5Si_3/TiAl composite with high strength and good ductility simultaneously via the combined method of composite technology and element alloying. The effects of Cu, Zn, W and Mo on the compression properties of Ti_5Si_3/TiAl composites were investigated.

2. Experimental

The starting materials of Ti_5Si_3/TiAl-2Me composites (Me = Cu, Zn, W and Mo, respectively) were made from commercial powders of Al (99 wt.% purity, $\sim$47 μm), Ti (99.5 wt.% purity, $\sim$25 μm), Si (99.5 wt.% purity, $\sim$25 μm), Cu (99.5 wt.% purity, $\sim$47 μm), Zn (99.0 wt.% purity, $\sim$47 μm), W (99.0 wt.% purity, $\sim$3 μm) and Mo (99.0 wt.% purity, $\sim$3 μm). Elemental powder blends corresponding to Ti49Al-4 vol.% Ti_5Si_3 with the addition of 2 at.% Me (Me = Cu, Zn, W and Mo, respectively) were mixed sufficiently by ball milling for 8 h at a low speed ($\sim$35 rpm) in a cylinder ball-miller. Both the jar and the balls were made of zirconia and the mass ratio of ball to powders was 10:1. The jar was aerated with argon gas to protect the powder from oxidation. Then the mixtures were cold pressed into a cylindrical compact with a diameter of 40 mm and a height of 30 mm. The powder compact was embedded in a graphite mold, which was put into a self-made vacuum thermal explosion furnace. The heating rate of the furnace was 10 K/min and the temperature in the center of the compact was measured by Ni–Cr/Ni–Si thermocouples. When the temperature measured by thermocouples suddenly rose rapidly, indicating that the sample should be ignited, the sample was quickly pressed just when it was still hot and soft. The pressure was maintained at 50 MPa for 10 s and then the sample was cooled down to the ambient temperature at a cooling rate of 10 K/min.

The phase composition of the samples was identified by the X-ray diffraction analysis (XRD, Model D/Max 2500PC, Rigaku, Tokyo, Japan) with Cu $K\alpha$ ($\lambda = 0.154$ nm) radiation operating at 40 kV. Microstructure analyses of the samples were conducted in a scanning electron microscope (SEM, Model Evo18 Carl Zeiss, Oberkochen, Germany) equipped with an energy dispersive spectrometer (EDS). The morphology of the ceramic particles was observed using a field emission scanning electron microscope (FESEM, JSM 6700F, JEOL, Tokyo, Japan). The cylindrical samples with a diameter of 3 mm and a height of 6 mm were used for compression tests, and the loading surface was polished parallel to the other one. The uniaxial compression tests were carried out three times for each sample under a servo-hydraulic-materials testing system (MTS, MTS 810, USA) with a strain rate of 1×10^{-4} s^{-1}. The true stress–strain curves were obtained from engineering stress–strain curves according to the formularies: $\varepsilon_{\rm t} = -\ln(1 - \varepsilon_{\rm e})$ and $\sigma_{\rm t} = \sigma_{\rm e}$ $(1 - \varepsilon_{\rm e})$, where ε_t is the true strain, σ_t is the true strength, $\varepsilon_{\rm e}$ is the engineering strain and $\sigma_{\rm e}$ is the engineering strength.

3. Results and Discussion

3.1. *Phase identification and microstructures*

Figure 1 shows the XRD patterns for the 4 vol.% Ti_5Si_3/TiAl composite and the composite with the addition of Cu, Zn, W and Mo elements, respectively. The products in these samples mainly consist of γ–TiAl, α_2–Ti_3Al and Ti_5Si_3. A small amount of AlCu was found in the Ti_5Si_3/TiAl-2Cu composite and some metallic W was detected in the Ti_5Si_3/TiAl-2W composite. No metallic Zn, Mo or their compound trace were detected in the Ti_5Si_3/TiAl-2Zn and Ti_5Si_3/TiAl-2Mo composites. However, as indicated in the inset of Fig. 1, the peak positions of the TiAl (111) plane shift to higher 2θ values with the addition of these elements, implying a decrease in the interplanar spacing of the TiAl (111). As calculated by first-principle in our previous work,[14] the addition of the atoms of Cu, Zn, W and Mo all could change the lattice parameter of TiAl. The atoms of Cu, W and Mo prefer to occupy the Ti site, while the Zn atom prefers to occupy the Al site in TiAl. Therefore, the interplanar spacing decrease could be induced by the solid solution of Cu, Zn, W

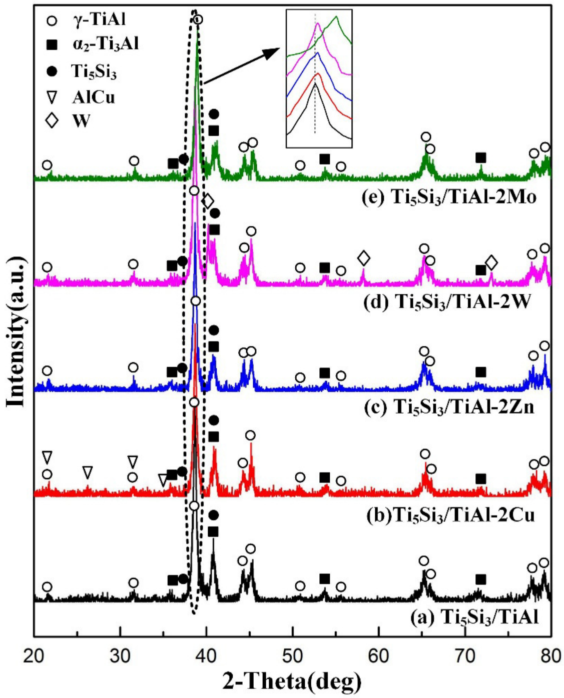

Fig. 1. (Color online) XRD patterns of these samples.

or Mo in the TiAl matrix as they have smaller atomic radii leading to lattice shrink (The atomic radii of Ti, Al, Cu, Zn, W and Mo are 0.147, 0.143, 0.128, 0.137, 0.137 and 0.136 nm, respectively). According to these results, it is believed that all of these elements exist in the form of solid solution in the TiAl matrix. Moreover, some Cu and W are presented in the form of second phase in the TiAl matrix. To further confirm these results, the analyses of microstructures of these samples were conducted.

Figure 2 shows the microstructures of the Ti_5Si_3/TiAl, Ti_5Si_3/TiAl-2Cu, Ti_5Si_3/TiAl-2Zn, Ti_5Si_3/TiAl-2W and Ti_5Si_3/TiAl-2Mo composites. The EDS results of the TiAl matrix detected at points +1, +2, +3 and +4 are given in Table 1. With the addition of 2 at.% Cu, Zn, W and Mo elements, the actual concentrations of Cu, Zn, W and Mo in the TiAl matrix are 0.51, 1.51, 0.36 and 1.65 at.%, respectively. Thus, it can be proved that the Zn and Mo mainly exist in the form of solid solution in the TiAl matrix, while the Cu and W are partly presented in the form of solid solution in the TiAl matrix. The uniform distribution of the Ti_5Si_3 particles in the Ti_5Si_3/TiAl composites is shown in Fig. 2(a). Most of the Ti_5Si_3 particles distribute in the TiAl grains and a few of the Ti_5Si_3 particles distribute along the grain boundaries. Figure 2(b) gives a magnified image of the Ti_5Si_3 particles in the Ti_5Si_3/TiAl composites. The Ti_5Si_3 particles in the composite are in a near spherical shape with the size of about 60–90 nm.

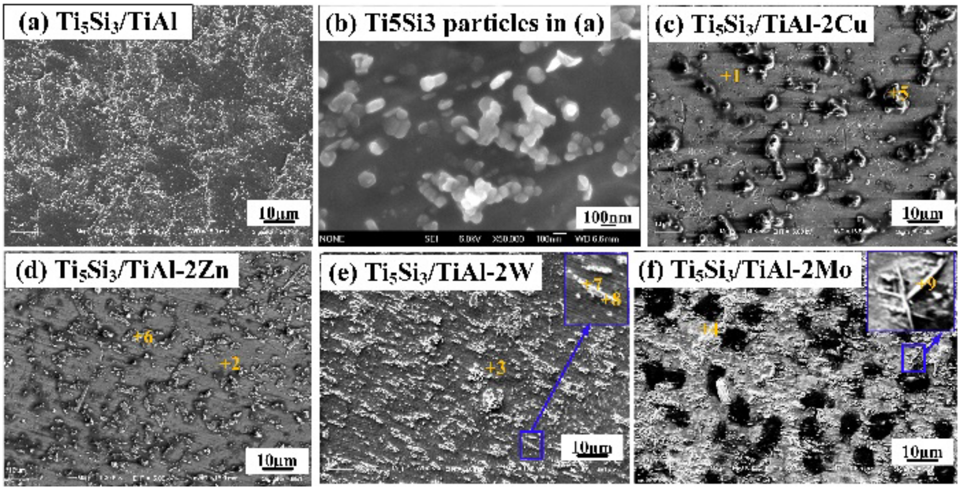

Fig. 2. (Color online) Microstructure images of these samples.

However, it can be seen from Fig. 2(c) that some large bulk phases of size 6 μm are distributed in the Ti_5Si_3/TiAl-2Cu composite. The EDS result [Table 1, pointed as +5 in Fig. 2(c)] shows that large bulk phases mainly consist of Al, Ti and Cu. Therefore, combined with the XRD result, the large bulk phases are confirmed as AlCu compounds. It can be seen from Fig. 2(d) that some particles with a cluster form in size of $\sim$2 μm are distributed in the Ti_5Si_3/TiAl-2Zn composite. The particle groups at the point 6 mainly consist of the Ti and Si, there is no any trace of Zn. Hence, the particle groups can be confirmed as Ti_5Si_3. In the Ti_5Si_3/TiAl-2W composite, the precipitates in fine-bar shape with a length of $\sim$10 μm are surrounded by some particle clusters [Fig. 2(e)]. By analyzing the EDS [Table 1, pointed as +7 and +8 in Fig. 2(e)] and XRD results, the fine-bar and particle clusters are identified as metallic tungsten and Ti_5Si_3 particles, respectively. As shown in Fig. 2(f), Ti_3Si_5 particles also gather with the addition of 2 at.% Mo. The inset in Fig. 3(f) shows some aggregate dendritic structures with the size of

Table 1. EDS results in the Ti_5Si_3/TiAl-2Cu, Ti_5Si_3/TiAl-2Zn, Ti_5Si_3/TiAl-2W and Ti_5Si_3/TiAl-2Mo.

	Ti (at.%)	Al (at.%)	Si (at.%)	Cu (at.%)	Zn (at.%)	W (at.%)	Mo (at.%)
Point 1	74.79	24.7	—	0.51	—	—	—
Point 2	72.15	26.34	—	—	1.51	—	—
Point 3	71.11	28.53	—	—	—	0.36	—
Point 4	68.84	29.51	—	—	—	—	1.65
Point 5	7.29	55.33	—	37.38	—	—	—
Point 6	77.57	4.44	17.99	—	—	—	—
Point 7	1.27	0.81	0.57	—	—	97.35	—
Point 8	69.86	3.61	26.53				
Point 9	71.03	1.85	27.12	—	—	—	—

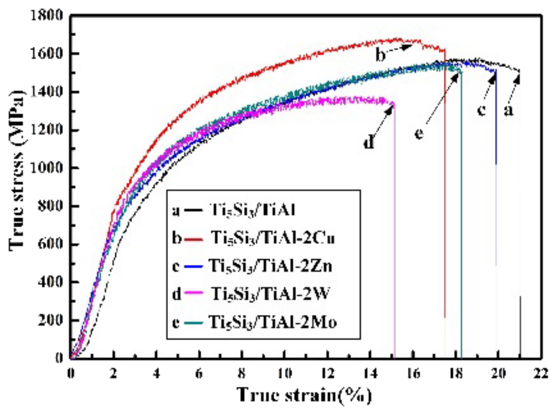

Fig. 3. (Color online) Compression true stress–strain curves.

8–10 μm. The EDS result reveals that these aggregates are also Ti_5Si_3 [Table 1, pointed as +9 in Fig. 2(f)].

3.2. *Compression properties*

Figure 3 shows the compression true stress–strain curves of the Ti_5Si_3/TiAl composites with and without the element addition. Their compression properties are summarized in Table 2. It can be seen that the addition of Cu, Zn and W could enhance the compression true yield strength (σ^y_{true}) of the Ti_5Si_3/TiAl composites. With the addition of 2 at.% Me (Me = Cu, Zn and W, respectively), the σ^y_{true} of the composites increase from 613 MPa to 840, 660 and 706 MPa, respectively. Moreover, the ultimate compression true strength ($\sigma^{\text{UCS}}_{\text{true}}$) of the composites increase from 1586 MPa to 1685 MPa with the addition of Cu. The addition of Zn has no effect on the $\sigma^{\text{UCS}}_{\text{true}}$ of the Ti_5Si_3/TiAl composite. While, with the addition of W, the $\sigma^{\text{UCS}}_{\text{true}}$ of the composites decreases to 205 MPa.

The addition of Zn could increase the σ^y_{true} with no obvious sacrifice of ductility of the composite (Table 2). However, with the addition of 2 at.% W, the fracture strain ($\varepsilon^f_{\text{true}}$) of the composite decreases from 20.9% to 15.2%. In our previous first-

Table 2. Compression properties of the Ti_5Si_3/TiAl, Ti_5Si_3/TiAl–2Cu, Ti_5Si_3/TiAl–2Zn, Ti_5Si_3/TiAl–2Mo and Ti_5Si_3/TiAl–2W composites.

Sample	σ^y_{true} (MPa)	$\sigma^{\text{UCS}}_{\text{true}}$ (MPa)	$\varepsilon^f_{\text{true}}$ (%)
TiAl[11,16]	465 ± 41	1415 ± 20	17.3 ± 0.03
Ti_5Si_3/TiAl	613 ± 7	1586 ± 7	20.9 ± 0.10
Ti_5Si_3/TiAl-2Cu	840 ± 14	1685 ± 15	17.5 ± 0.23
Ti_5Si_3/TiAl-2Zn	660 ± 17	1555 ± 18	19.9 ± 0.33
Ti_5Si_3/TiAl-2W	706 ± 11	1380 ± 14	15.2 ± 0.14
Ti_5Si_3/TiAl-2Mo	603 ± 23	1549 ± 9	18.3 ± 0.16

principle calculation[14] and an other researcher's study,[15] the addition of W was confirmed to be beneficial to the ductility of TiAl alloy. But for the Ti_5Si_3/TiAl composite, the distribution of Ti_5Si_3 particles is a very important parameter for its ductility. Thus, the reason for the decrease of the ductility of the Ti_5Si_3/TiAl composite with the addition of W should be mainly due to the aggregation of Ti_5Si_3 particles around the fine-bar W phase at the TiAl matrix. The worse ductility would cause the decrease in the $\sigma_{\text{true}}^{\text{UCS}}$ of the Ti_5Si_3/TiAl-2W composite. The addition of Mo does not improve the compression strength and ductility of the Ti_5Si_3/TiAl composite. Similarly, the addition of Mo leads to the dendritic aggregation of Ti_5Si_3 particles in the TiAl matrix.

The σ_{true}^{y}, $\sigma_{\text{true}}^{\text{UCS}}$ and $\varepsilon_{\text{true}}^{f}$ of the TiAl alloy fabricated in our previous study are 465, 1415 MPa and 17.3%, respectively.[10,16] Compared with the compression properties of the TiAl alloy, it can be seen that the combined addition of *in situ* nano-Ti_5Si_3 particles and the elements of Zn could significantly improve the strength and ductility of TiAl alloy simultaneously. The σ_{true}^{y}, $\sigma_{\text{true}}^{\text{UCS}}$ and $\varepsilon_{\text{true}}^{f}$ of the 4 vol.% Ti_5Si_3/TiAl-2Zn composite are 195, 140 MPa and 2.6% higher than those of TiAl alloy. It is confirmed that the combined method of nano-ceramic particle reinforcement and element alloying is an effective way to improve both the strength and ductility of TiAl alloys.

4. Conclusions

The existence form of the addition alloying elements and the effect of alloying elements on the distribution of Ti_5Si_3 particles are the two main effective factors for the compression properties of Ti_5Si_3/TiAl composite. The Cu exists in the form of solid solution in the TiAl matrix and Al-Cu precipitates have the size of 6 μm. With the addition of Mo, Ti_5Si_3 gathers as dendritic structures with the size of 8–10 μm and the Mo mainly exists in the form of solid solution. The W mostly exists in the form of fine-bar metallic tungsten phase at the length of 10 μm and the Ti_5Si_3 particles gathered around the fine-bar W phase in the TiAl matrix. The Zn primarily exists in the form of solid solution. And some Ti_5Si_3 particles with a cluster form in size of 2 μm are uniformly distributed in the TiAl matrix with the addition of Zn. Hence, the addition of Zn enhances the strengths of Ti_5Si_3/TiAl composite with no obvious sacrifice of ductility. Compared with TiAl alloy, the σ_{true}^{y}, $\sigma_{\text{true}}^{\text{UCS}}$ and $\varepsilon_{\text{true}}^{f}$ of the 4 vol.% Ti_5Si_3/TiAl-2Zn composite increases from 465 MPa, 1415 MPa and 17.3% to 660 MPa, 1555 MPa and 19.9%, respectively. It is demonstrated that the combined method of addition nano-Ti5Si3 particles and Zn element is an effective way to improve the strength and ductility of TiAl alloy simultaneously.

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