Research Article

Microstructures and Mechanical Properties of Hot-Pressed MoSi₂-Matrix Composites Reinforced with SiC and Si₃N₄ Particles

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MoSi₂-matrix composites reinforced with Si₃N₄ and SiC particles were fabricated by means of wet-mixing and heat-pressing process. Scanning electron microscope (SEM), X-ray diffractometry (XRD), polarizing microscopy, Vickers hardness tester, with a universal materials testing machine were used to investigate the morphology, grain size, hardness, fracture toughness, and bending strength of the synthesized composites. Notable effects on the bending strength and fracture toughness of MoSi₂ caused by the addition of SiC and Si₃N₄ particles were found. The MoSi₂ composite with 20 vol.% SiC and 20 vol.% Si₃N₄ particles has the highest strength and toughness, which is about 100% and 340%, respectively, higher than that of pure MoSi₂. The grain size of MoSi₂ decreases gradually with the volume content of SiC and Si₃N₄ particles increasing from 0% to 40%, and MoSi₂-20 vol.% SiC-20 vol.% Si₃N₄ composite exhibits the minimum grain size of MoSi₂. The relationship between the grain size of MoSi₂ and bending strength is not entirely fit with Hall-Petch equation. The strengthening mechanisms of the composite include fine-grain strengthening and dispersion strengthening. The toughening mechanisms of the composite include fine grain, microcracking, crack deflection, crack microbridging, and crack branching.

1. Introduction

MoSi₂ has been investigated as potential material for high temperature structural applications and for application in the electronics industry. Its properties provide a desirable combination of a high melting temperature (2030°C), high modulus (440 GPa), good oxidation resistance in air, a relatively low density (6.24 g/cm³) [1], and the ability to undergo plastic deformation above 1200°C [2]. Combined with good thermal and electric conductivities, these properties have led to the utilization of MoSi₂ as a heating element material in high-temperature furnaces operating in air up to about 1700°C [3, 4].

However, as in the case of many such compounds, the current concern about these materials focuses on their low fracture toughness below the ductile-brittle transition temperature [5, 6]. To improve the mechanical properties of these materials, the addition of a second phase to form composites [7–10] has been found to be effective. Silicon nitride (Si₃N₄) has a high thermal shock resistance, due to

its low thermal expansion coefficient and good resistance to oxidation when compared to other structural materials [11–13]. The isothermal oxidation resistance of the NbSi₂-40 vol.% Si₃N₄ composite prepared by the spark plasma sintering (SPS) process in dry air at 1300°C was superior to that of monolithic NbSi₂ compact since the composite contained a larger amount of Si, which made it easier to form dense SiO₂ scale [14]. The fracture toughness of MoSi₂ + Si₃N₄ composites has been found to increase significantly with increasing temperature, reaching values as high as 15 MPa·m^{1/2} at 1300°C [15]. Also the addition of SiC had a significant influence on the behavior of MoSi₂, by forming a protective SiO₂ layer leading to its exhibiting outstanding oxidation resistance [16]. As a matter of fact, MoSi₂ and SiC are compatible and stable up to 1600°C. Bhattacharya and Petrovic [17] examined the hardness and indentation fracture toughness of a SiC-particle-reinforced MoSi₂ composite. Suzuki and coworkers [18] successfully improved the mechanical properties, the flexural strength in particular, of SiC-nanoparticle-reinforced MoSi₂-matrix

nanocomposites. Therefore, Si_3N_4 and SiC may be the most promising additives for use as reinforcing material for metal silicide-based composites. However, there are few papers [19] reporting the addition of SiC and Si_3N_4 particles to improve the strength, toughness, and decrease the possibility of pest disintegration of MoSi_2 .

In this study, MoSi_2 -matrix composites reinforced with Si_3N_4 and SiC particles were fabricated by means of wet-mixing and heat-pressing process. The morphology, grain size, hardness, fracture toughness, and bending strength of the synthesized composites were evaluated. The effects of Si_3N_4 and SiC contents on these properties were examined. And the mechanisms of strengthening and toughening MoSi_2 by adding Si_3N_4 and SiC particles were also investigated.

2. Experimental Procedure

2.1. Materials and Preparation Process. A mixture of 99.9% pure MoSi_2 ($<2.5\ \mu\text{m}$), 98.5% pure SiC ($<0.5\ \mu\text{m}$), 99% pure Si_3N_4 ($<1\ \mu\text{m}$) was used for the preparation of monolithic MoSi_2 and its composites in the present study. The powder mixture was wet mixed in ethyl alcohol and milled for 48 h. The milling process was realized by rolling a 500 mL cylindrical container made of agate on a planetary ball-milling machine, in which the balls of $\Phi\ 10\ \text{mm}$ made of agate and the powder mixture had a mass ratio of about 20:1, and the gas atmosphere was argon. The compositions of the target composites were (a) MoSi_2 ; (b) MoSi_2 -20 vol% SiC ; (c) MoSi_2 -20 vol% SiC -10 vol% Si_3N_4 ; (d) MoSi_2 -20 vol% SiC -20 vol% Si_3N_4 ; (e) MoSi_2 -20 vol% SiC -30 vol% Si_3N_4 . The resulting powders were hot pressed in a graphite die at 1700°C for 30 min under 30 MPa pressure. The dimensions of the consolidated samples were 52 mm in diameter and 6–10 mm in thickness. Similar conditions were employed to prepare monolithic MoSi_2 . The bulk density of the sample was measured by the Archimedes method with an immersion medium of deionized water.

2.2. Microstructure Analysis. Crystal-phase identification of the synthesized samples was determined by X-ray diffraction (XRD, RIGAKU D/Max-rB) with Ni-filtered $\text{CuK}\alpha$ radiation ($0.1542\ \text{nm}$) at a scan rate of $4^\circ/\text{min}$. Using a Sirion200 field emission scanning electron microscope, the microstructures of the synthesized samples were analyzed. The grain sizes of MoSi_2 of the synthesized samples were analyzed by polarizing microscopy and the transversal method.

2.3. Mechanical Property Test. Vickers hardness of MoSi_2 and MoSi_2 -based composites was measured with a HV-10 A Vickers hardness tester under the load of 49 N for 10 s. Indentation fracture toughness (K_{1c}) was calculated by using an Anstis mode [20] and described as

$$K_{1c} = A \left(\frac{E}{H} \right)^{1/2} \left(\frac{P}{C^{3/2}} \right), \quad (1)$$

where A is a constant 0.016, H the hardness, P the indentation load, C the average of the four surface radial cracks, and

E the elastic modulus of MoSi_2 and MoSi_2 -based composites. The value of H is the Vickers hardness (H_v), given by [21]

$$H_v = \frac{P}{2b^2}, \quad (2)$$

where b is the length value of each indent diagonal. The average values of 16 indentation diagonals and 32 radial crack lengths from the eight indentations on the surface of sample were used for the calculation of fracture toughness and hardness under the same load. The composite specimens for measurement of three-point bend strength were of size $3\ \text{mm} \times 4\ \text{mm} \times 36\ \text{mm}$. A self-aligning bend fixture made of hardened tool steel was used for measurement of bend strength on an Instron test frame at room temperature.

3. Results

3.1. Microstructure. Figure 1 shows the X-ray diffraction patterns of MoSi_2 , MoSi_2 -20 vol% SiC composite, and MoSi_2 -20 vol% SiC -20 vol% Si_3N_4 composite. As seen in Figure 1, the prepared composites mainly consist of the phases including MoSi_2 and reinforcing agents such as Si_3N_4 and SiC , and a small amount of Mo_5Si_3 . Among these phases, Mo_5Si_3 should be generated from the reaction (see (3)) between MoSi_2 and oxygen under high hot-pressing temperature (1700°C) [22]. The peaks of SiO_2 were not observed in Figure 1, which may be attributed to the low content of SiO_2 in the prepared samples

Figure 2 shows the backscattered electron images of the MoSi_2 -20 vol% SiC , MoSi_2 -20 vol% SiC -10 vol% Si_3N_4 , MoSi_2 -20 vol% SiC -20 vol% Si_3N_4 , and MoSi_2 -20 vol% SiC -30 vol% Si_3N_4 compacts. Figure 2 reveals a dense microstructure of the composite, which is consistent with the density analysis results of these samples (Table 1). Figure 2(a) indicates that the microstructure of monolithic MoSi_2 is composed of gray zone (MoSi_2), white zone (Mo_5Si_3), and black zone (SiO_2). It can be seen from Figures 2(b), 2(c), 2(d), and 2(e) that the microstructure of MoSi_2 -based composites consists of gray zone (MoSi_2), white zone (Mo_5Si_3), and black zone (Si_3N_4 and SiC) dispersed in the grain boundary of MoSi_2 . These results indicate that no chemical reaction occurs among MoSi_2 , SiC , and Si_3N_4 under the hot-pressing conditions; this is a precondition for preparing MoSi_2 -based composites with excellent properties.

3.2. Mechanical Properties and Densities of the Prepared Samples. The bulk densities and mechanical properties of the hot-pressed samples were listed in Table 1. The elastic modulus (E) of MoSi_2 - Si_3N_4 / SiC composites was calculated as follows:

$$E = E_m V_m + E_f V_f + E_w V_w, \quad (4)$$

where E_m is the elastic modulus of polycrystalline MoSi_2 (440 MPa), E_f the elastic modulus of Si_3N_4 (300 MPa), E_w the elastic modulus of SiC (448 MPa), V_m , V_f , V_w , the volume fractions of MoSi_2 , Si_3N_4 , and SiC , respectively [23].

TABLE 1: Mechanical properties and densities of the prepared samples.

Sample	Density/%	Bending strength/MPa	Fracture toughness/MPa·m ^{1/2}	Vickers hardness/GPa
MoSi ₂	98.6	216	3.1	8.1
MoSi ₂ -20 vol% SiC	98.3	335	8.2	9.3
MoSi ₂ -20 vol% SiC -10 vol% Si ₃ N ₄	97.6	388	9.4	10.5
MoSi ₂ -20 vol% SiC-20 vol% Si ₃ N ₄	96.3	427	10.4	11.4
MoSi ₂ -20 vol% SiC-30 vol% Si ₃ N ₄	95.2	396	8.9	11.8

FIGURE 1: XRD pattern of the prepared composites. (a) MoSi₂, (b) MoSi₂-20 vol% SiC, (c) MoSi₂-20 vol% Si₃N₄-20 vol% SiC.

FIGURE 2: SEM analysis on the microstructure of the prepared samples (backscattering imaging). (a) MoSi₂; (b) MoSi₂-20 vol% SiC; (c) MoSi₂-20 vol% SiC-10 vol% Si₃N₄; (d) MoSi₂-20 vol% SiC-20 vol% Si₃N₄; (e) MoSi₂-20 vol% SiC-30 vol% Si₃N₄.

It can be seen from Table 1 that the relative densities of the hot-pressed samples are above 95% and decrease slowly with the increasing of reinforcing agent contents, which means that high-density MoSi₂ could be obtained by the hot-pressing process. Table 1 also indicates that the bending strength, fracture toughness, and Vickers hardness of the MoSi₂-based composites are better than that of monolithic MoSi₂ and increase gradually with the increase of Si₃N₄ and SiC content in the range of 0–40 vol.%. Among these prepared samples, MoSi₂-20 vol% SiC-20 vol% Si₃N₄ composite exhibits the highest bending strength (427 MPa) and fracture toughness (10.4 MPa·m^{1/2}), which may be due to the fact that it has the smallest grain size of MoSi₂ (4.1 μm, see Table 2) and relatively high density. The bending strength and fracture toughness of the MoSi₂-20 vol%SiC-30 vol%Si₃N₄ composite are lower than that of the MoSi₂-20 vol%SiC-20 vol%Si₃N₄ composite, which may be attributed to its relatively large grain size of MoSi₂ (4.7 μm, see Table 2) and high Si₃N₄ content leading to the decreasing of its density.

3.3. Investigation of Strengthening and Toughening Mechanism of MoSi₂-Based Composites

3.3.1. Strengthening Mechanism. Figure 3 shows the fracture morphologies of the prepared MoSi₂ and MoSi₂-based composites. Figure 3(a) indicates that the grain size of pure MoSi₂ was very coarse, and the fracture surface of pure MoSi₂ was smooth, which suggested that the predominating fracture modes were transgranular for MoSi₂. Figures 3(b), 3(c), 3(d), and 3(e) show that the fracture surface of MoSi₂-based composites is rough and consists of a large amount of little irregular planes, which indicates that the predominating fracture modes were mixed transgranularly and intergranularly for MoSi₂-based composites. The reason for this may be that the addition of Si₃N₄ and SiC particles dispersed in the grain boundaries of MoSi₂ (see Figures 2(b), 2(c), 2(d), and 2(e)) has some influences on the intrinsic characters of MoSi₂ and then weakens the grain boundary of MoSi₂, subsequently leading to the increase of intergranular fracture modes. Thus, dispersion

FIGURE 3: SEM analysis of the three-point bending test fracture surface. (a) MoSi_2 ; (b) MoSi_2 -20 vol% SiC; (c) MoSi_2 -20 vol% SiC-10 vol% Si_3N_4 ; (d) MoSi_2 -20 vol% SiC-20 vol% Si_3N_4 ; (e) MoSi_2 -20 vol% SiC-30 vol% Si_3N_4 .

TABLE 2: MoSi_2 grain sizes of the prepared samples.

Sample	MoSi_2	MoSi_2 -20 vol% SiC	MoSi_2 -20 vol% SiC-10 vol% Si_3N_4	MoSi_2 -20 vol% SiC-20 vol% Si_3N_4	MoSi_2 -20 vol% SiC-30 vol% Si_3N_4
MoSi_2 grain size/ μm	18.6	10.2	6.5	4.1	4.7

strengthening of reinforced agents is an important reason for the increasing of bending strength of MoSi_2 -based composites.

The grain sizes of MoSi_2 of the synthesized samples were analyzed by polarizing microscopy and the transversal method. The results are shown as Figure 4 and Table 2, which indicate that the grain size of MoSi_2 decreases with the increase of Si_3N_4 and SiC content in the range of 0–40 vol.% and MoSi_2 -20 vol% SiC-20 vol% Si_3N_4 composite exhibits the smallest grain size (4.1 μm). Thus, the refinement of grain size of MoSi_2 is another important factor for the MoSi_2 -based

composites having higher bending strength than that of pure MoSi_2 .

The relationship between grain size of MoSi_2 and bending strength may be described by using a certain equation, such as Hall-Petch equation [24]. The effect of grain size (d) of MoSi_2 on the bending strength is shown as Figure 5.

Figure 5 indicates that the relationship between the grain sizes of MoSi_2 and bending strength is not entirely fit with the Hall-Petch equation because the bending strength not only is affected by the refinement of grain size of MoSi_2 but also is affected by the dispersion strengthening of reinforced agents.

FIGURE 4: Polarizing microscopy analysis of the prepared samples. (a) MoSi_2 ; (b) MoSi_2 -20 vol% SiC; (c) MoSi_2 -20 vol% SiC-10 vol% Si_3N_4 ; (d) MoSi_2 -20 vol% SiC-20 vol% Si_3N_4 ; (e) MoSi_2 -20 vol% SiC-30 vol% Si_3N_4 .

FIGURE 5: Relationship between bending strength and grain size of MoSi_2 .

3.3.2. Toughening Mechanism

(1) *Fine Crystal Toughening.* The fracture toughening results mentioned above can be explained by the various toughening

mechanisms. It is well known that the fracture toughness of brittle ceramic materials increases with the decrease of the grain size [25]. The reason for this may be that the grain refining will be a benefit to decrease stress concentration and crack formation for it will increase the binding force between closed grains, then the deformation of inner crystal grain is transferred to adjacent crystal grain. On the other hand, grain boundary can restrain crack propagation, and furthermore, the energy consumed by crack propagation increases with the decrease of grain size. Usually, (5) is used to judge whether the crack is destabilizing propagation or not [26]:

$$(\sigma_0 d^{1/2} + k_y)k'_y \geq \beta\mu\gamma, \quad (5)$$

where σ_0 is the frictional resistance of dislocation, d grain size, k_y , k'_y , β constant, μ shear modulus, and γ specific area. When the value of $(\sigma_0 d^{1/2} + k_y)k'_y$ is higher than that of $\beta\mu\gamma$, the crack is destabilizing propagation; therefore, decreasing grain size will be difficult for crack generating destabilizing propagation.

FIGURE 6: Secondary electron SEM images of the crack propagating behavior in the MoSi_2 - SiC - Si_3N_4 composite. (a) Microcracking, (b) crack deflection, (c) crack microbridging, and (d) crack branching.

(2) *Crack Deflection, Branch and Microcrack Toughening.* Microcracks and crack propagation in MoSi_2 and MoSi_2 -20 vol% SiC -20 vol% Si_3N_4 composite are shown in Figure 6.

Figures 6(a), 6(b), 6(c), and 6(d) show the microcracking, crack deflection, crack microbridging, and crack branching in the composite. Rice [27, 28] has reviewed the mechanisms that can toughen ceramic composite. These mechanisms include matrix microcracking, crack branching, crack deflection, crack bowing, and fiber pull out. Carter and Hurley [29] suggested crack deflection as an important toughening mechanism in SiC -whisker-reinforced MoSi_2 . Bhattacharya and Petrovic [17] provided evidence for crack-interface grain bridging (crack microbridging) in 20 vol.% SiC - MoSi_2 composite and crack branching in 40 vol.% SiC - MoSi_2 composite. In the present study, grain pullout does not appear. Microcracking, crack deflection, crack microbridging, and crack branching were observed in the composite, which can be explained by the presence of a complex residual stress field [30] and the high resistance to crack propagation because the addition of SiC and Si_3N_4 particles in the matrix will absorb more energy and thus improve the fracture toughness value.

4. Conclusions

- (1) MoSi_2 -matrix composites doped by SiC and Si_3N_4 particles were successfully prepared by hot-pressing techniques. Notable effects on the bending strength and fracture toughness of MoSi_2 caused by the

addition of SiC and Si_3N_4 particles were found. When adding about 20 vol.% SiC and 20 vol.% Si_3N_4 particles, the strength and toughness value of the composite reaches the highest, which is about 100% and 340%, respectively, more than that of pure MoSi_2 .

- (2) When the volume content of reinforced agents is below 40 vol%, the grain size of MoSi_2 decreases with increase of the volume content of reinforced agents, and MoSi_2 -20 vol.% SiC -20 vol.% Si_3N_4 exhibited the minimum grain size of MoSi_2 . The relationship between the grain sizes of MoSi_2 and bending strength is not entirely fit with the Hall-Petch equation.
- (3) The strengthening mechanisms of MoSi_2 - SiC - Si_3N_4 composites include fine-grain strengthening and dispersion strengthening; the toughening mechanisms of it include fine-grain, microcracking, crack deflection, crack microbridging, and crack branching.

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