Combustion synthesis and mechanical properties of MoSi$_2$–ZrB$_2$–SiC ceramics

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MoSi$_2$–ZrB$_2$–SiC ceramics were synthesized using Mo, Zr, Si and B$_4$C powders by self-propagating high-temperature synthesis and densifying by spark plasma sintering. The effects of MoSi$_2$ content on the combustion synthesis process, microstructure, and mechanical properties of the ceramics were investigated. The results showed that combustion synthesis is an unstable mode, spiral combustion. The Gibbs calculations and combustion temperature curves indicate there are two reactions occurring at the same time. The volume fraction of the four different phases and their relative densities were also measured and calculated. Compared to pure MoSi$_2$, the 0.0MoSi$_2$–0.2ZrB$_2$–0.1SiC (M10) ceramic exhibits excellent mechanical properties with its maximum Vickers hardness and fracture toughness being 14.0 GPa and of 5.5 MPa m$^{1/2}$, respectively. The hardness is in agreement with the rule of mixture. The morphology of indentation cracks reveals that the fracture toughness improves as a result of toughening mechanisms such as crack bridge, crack deflection, and microcracks.

Key-words : Combustion synthesis, In situ, Spark plasma sintering, Mechanical properties, MoSi$_2$

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1. Introduction

Molybdenum disilicide (MoSi$_2$), as structural and functional material has widely potential applications in high-temperature environments$^{1-4}$ due to its high melting point (2030°C), relatively low density (6.24 g/cm$^3$) and good resistance to high-temperature oxidation (>1600°C). However, the actual applications of MoSi$_2$ are limited due to its low fracture toughness (2–3 MPa m$^{1/2}$) at room temperature, as well as poor strength at high temperatures. Moreover, MoSi$_2$ exhibits the “pest oxidation” behavior at approximately 773 K.$^{5-8}$ Recent studies have reported promising methods to overcome the low fracture toughness and improve the poor strength using alloying approaches where W, Cr, Re, and Al are commonly used to toughen and strengthen the silicide structure materials.$^{9-12}$

Additions of ceramic particle-reinforcements have been developed to toughen and strengthen MoSi$_2$. Reinforcements of MoB,$^{13,14}$ TiC,$^{15}$ CrB$_2$,$^{16}$ BN,$^{17}$ B$_4$C$^{18}$ and Si$_3$N$_4$$^{19}$ can improve fracture toughness of MoSi$_2$. Patel et al.$^{20}$ investigated the nanocrystalline SiC–MoSi$_2$ composite which was fabricated by liquid silicon infiltration and its average hardness and fracture toughness to 12 GPa and 4.3 MPa m$^{1/2}$, respectively. Moreover, the SiC phase can contribute to the toughness due to the second phase strengthening mechanism. Peng et al.$^{21}$ fabricated 15 vol % SiC–MoSi$_2$ nanocomposites using reactive hot pressing. The results showed the maximum Vickers hardness and fracture toughness were 10.9 GPa and 4.9 MPa m$^{1/2}$, respectively, while SiC particles can strengthen the MoSi$_2$ materials by fine-grain strengthening mechanism. Thus, it could be seen that the addition of SiC is an important method for reinforcing the MoSi$_2$ matrix ceramics.

Some studies about the mechanical properties of ZrB$_2$ matrix composites reinforced by MoSi$_2$ particles have already been published. Liu et al.$^{22}$ studied ZrB$_2$–30 vol % MoSi$_2$ platelet composites fabricated using reactive hot pressing of Mo, Si, Zr and B powders at 1550°C for 30 min and determined a fracture toughness of 5.4 MPa m$^{1/2}$ and Vickers hardness of 16.0 GPa. Sciti et al.$^{23}$ investigated the properties of a pressureless-sintered ZrB$_2$–20 vol % MoSi$_2$ ceramic composite and determined the microhardness (HV1.0) and fracture toughness were 16.0 GPa and 2.3 ± 0.2 MPa m$^{1/2}$, respectively. Meanwhile, MoSi$_2$ is the secondary phases and a sintering aid for the densification of ZrB$_2$ matrix composites.$^{24,25}$ However, in this study, MoSi$_2$ will be the major phase and ZrB$_2$ will be the secondary phase doping the MoSi$_2$ matrix composites.

In situ multiphase MoSi$_2$–ZrB$_2$–SiC composites were prepared by self-propagating high temperature synthesis (SHS) and spark plasma sintering (SPS). This in situ synthesis of MoSi$_2$–ZrB$_2$–SiC ceramics was first studied using the SHS method. The objective of this study is to expose the combustion behavior of the in situ fabrication of...
xMoSi$_2$–0.2ZrB$_2$–0.1SiC ($x = 2.0$, 1.5, and 1.0) composites. Meanwhile, the microstructure and mechanical properties of MoSi$_2$–ZrB$_2$–SiC composites were also analyzed and compared with pure MoSi$_2$. This study offered a promising method to toughen MoSi$_2$ matrix materials.

2. Experimental

Molybdenum (3.0–5.0 μm, 99.9% purity, Zhuzhou cemented carbide group Co. Ltd., China), silicon [3.0–5.0 μm, 99.9% purity, WODETAI (Beijing) Co. Ltd.], boron carbide (B$_4$C, 1.0–10.0 μm, 99.5% purity, Aladdin industrial, Co. China), and zirconium (–200 mesh, 99.9% purity, Aladdin industrial, Co. China) powder mixtures were used to prepare 1.0MoSi$_2$–0.2ZrB$_2$–0.1SiC (M10), 1.5MoSi$_2$–0.22ZrB$_2$–0.15SiC (M15), 2.0MoSi$_2$–0.2ZrB$_2$–0.1SiC (M20) and monolithic MoSi$_2$ (M100). The raw materials were mixed in a planetary ball mill for 240 min at 451 rounds per minute using agate jars and balls in alcohol medium. Subsequently, the mixtures were made into cylindrical compacts with 16 mm diameter and 15 mm height using 200 MPa pressure by cold-press. The combustion synthesis experiments were conducted in a steel combustion chamber in a pure argon (Ar, 99.99%, 0.1 MPa) atmosphere. The specimen was ignited by heating molybdenum wires using a 24 V and 28 A current at room temperature. The sintered samples were crushed into powder, and the powders were sintered by spark plasma sintering (Labox-125, Sinter Land Inc., Japan) at 1500°C for 3 min, using a 40 MPa uniaxial pressure and heating rate of 250 °C/min, in vacuum (<10 Pa). The hardness and fracture toughness were measured using a Vickers hardness tester (Laizhou Huayin HVS-50, Laizhou, China) with indentation load of 98 N and holding time of 15 s, the fracture toughness was calculated using the following formula:

$$K_{IC} = 0.0752 P (a/c)^{1/2}$$

(0)

where $P$ is the indentation load, $c$ is the length of the crack, and $a$ is the indentation half-diagonal length.

The combustion temperature was recorded by infrared temperature apparatus (Reteck, Marathon series, USA) and combustion videos were recorded using a high-speed camera. The microstructure was analyzed by scanning electron microscope (SEM, FEI Quanta TM 250) equipped with energy-dispersive spectroscopy (EDS, Quantax 400-10). The phase compositions were identified by X-ray diffraction (XRD, Bruker D8 Advance) with Cu Kα ($λ = 0.15406$ nm) radiation, and the densities were tested using Archimedes’ drainage method. The volume fractions of different phases were calculated using the Image-Pro software.

3. Results and discussion

3.1 Combustion synthesis

Figure 1 shows typical combustion images from the M10 specimen. It could be seen that when $t = 0.24$ s, the top-right edge of the specimen was ignited by the heated molybdenum coil, which was in contact with the sample. Subsequently, the combustion wave propagated from the top-right to the left edge and spread downwards to the entire sample. As revealed in Fig. 1, the area behind the combustion front was incandescent. Yeh et al. studied the combustion synthesis of Ta–Si compounds and explained the high-brightness of the burned region behind the reaction front, indicating that the reaction not only occurs in the combustion front, but also continues vigorously in bulk. The front of the combustion flame moved along a spiral trajectory in a localized reaction point. The combustion points could be found at 2.46 and 3.20 s, respectively. Thus, combustion synthesis of M10 can be considered an unstable combustion mode, spiral combustion.27)

Figure 2 shows the combustion temperature-time curve of pure MoSi$_2$ and xMoSi$_2$–0.2ZrB$_2$–0.1SiC ceramics ($x = 2.0$, 1.5, and 1.0). The minimum combustion temperature was 1840 K for M100 and increased to a maximum of 1935 K from M10 specimen. The actual combustion temperature of M100 is 1840 K, which is close to 1943 K the adiabatic temperature ($T_{ad}$) of pure MoSi$_2$.28) The actual combustion temperature was lower than $T_{ad}$, as the result of the lack of adiabatic conditions due to heat loss into the environment.29) All the combustion temperatures were higher than the melting point of Si (1687 K) and lower than those of the other components (2890 K for Mo, 2125 K for Zr and 2623 K for B$_4$C). It has been reported that liquid Si can dissolve Zr, B$_4$C, and Mo until they form saturated liquid solutions.30–32) Feng et al.30) reported the effects of boron addition on the formation of MoSi$_2$ by combustion synthesis as Mo and B can be dissolved into liquid Si, while boride and silicide are precipitating from the saturated liquid. Hayun et al.31) researched the B$_4$C–SiC–Si infiltrated composites where B$_4$C can dissolve into liquid Si until its saturation with B and C. Meanwhile,
Bertolino et al.\textsuperscript{32)} indicated that the precipitates of the Zr–Si compound come from the saturated liquid solution in the combustion synthesis procedure. This implies that molten silicon can dissolve the Mo, Zr and B\textsubscript{4}C particles in the combustion synthesis process and form a saturated solution which can cause the precipitation of silicide and boride. Thus, the combustion synthesis mechanism of Mo\textsubscript{Si}_2–ZrB\textsubscript{2}–SiC composites is considered a dissolving-precipitating mechanism.

The transformation of the Mo, Si, Zr, and B\textsubscript{4}C powders into Mo\textsubscript{Si}_2+0.2ZrB\textsubscript{2}+0.1SiC composites can be described according to reaction (1) during the SHS process:

\[
x\text{Mo} + (2x + 0.1)\text{Si} + 0.2\text{Zr} + 0.1\text{B}_4\text{C} = x\text{MoSi}_2 - 0.2\text{ZrB}_2 - 0.1\text{SiC}
\]

\[
\text{Mo} + 2\text{Si} = 2\text{MoSi}_2
\] \hspace{1cm} (2)

\[
0.2\text{Zr} + 0.1\text{Si} + 0.1\text{B}_4\text{C} = 0.2\text{ZrB}_2 + 0.1\text{SiC}
\] \hspace{1cm} (3)

\[
0.4\text{Mo} + 0.1\text{Si} + 0.1\text{B}_4\text{C} = 0.4\text{MoB} + 0.1\text{SiC}
\] \hspace{1cm} (4)

Reaction (1) was the designed and expected reaction, while reaction (2) is the established procedure. The other possible reactions, (3) and (4) have been reported in the literature.\textsuperscript{33),34)} It should be noted that Eqs. (3) and (4) are based on the minimum stoichiometric number of B\textsubscript{4}C.

Zhang et al.\textsuperscript{33)} studied the reactive hot pressing of ZrB\textsubscript{2}–SiC composites from Si, Zr and B\textsubscript{4}C powders. Meanwhile, Zhu et al.\textsuperscript{34)} studied the Mo\textsubscript{Si}_2–MoB–SiC system synthesized by SHS using Mo, Si, and B\textsubscript{4}C powders. It can be considered that Mo, Si, Zr and B\textsubscript{4}C powders convert into Mo\textsubscript{Si}_2 matrix composites following reaction (1) during SHS.

Figure 3 shows the Gibbs free energy of reactions (2), (3) and (4) from 273 to 2000 K. Reaction (4) shows the highest Gibbs energy compared with reactions (2) and (3) over the whole calculated temperature range, which implies that when the combustion reactions begin, reactions (2) and (3) tend to get precedence. Reaction (1), the designed and expected reaction can consist of reactions (2) and (3). The curve of the combustion temperature depicts a single obvious crest in Fig. 2 indicating that reactions (2) and (3) happen simultaneously in the combustion synthesis process.

3.2 Combustion products

The XRD patterns of SHS-SPS products are shown in Fig. 4. It can be seen that single C\textsubscript{11b}–Mo\textsubscript{Si}_2 was synthesized from Mo\textsubscript{2}Si powders and the strongest intensity peaks of components were observed at 2\textdegree=44.87° for Mo\textsubscript{Si}_2. The peaks at 2\textdegree=35.57, 41.80 and 65.87° can be indexed to SiC (Moissanite-51R, JCPDS 51-0639) and the low relative intensities are the result of the low SiC content and weak crystallization.\textsuperscript{35)} On the other hand, these results indicate a good dispersion of the SiC phase in the composites. With the addition of Zr and B\textsubscript{4}C particles, the intensity of ZrB\textsubscript{2} becomes more distinct from M\textsubscript{20} to M\textsubscript{10}, while Mo\textsubscript{Si}_2 is still the dominating phase. The XRD results are in agreement with the anticipatory design and calculation of Gibbs free energy.

3.3 Microstructure and volume fraction

The backscattering electron diffraction (BSED) images of pure Mo\textsubscript{Si}_2 and xMo\textsubscript{Si}_2–ZrB\textsubscript{2}–SiC ceramics are shown in Fig. 5. Four different phases could be found: white spot, grey phase, light grey matrix phase and black phase, respectively. The white spot was 2–3 μm in size and had a low volume fraction (under 1%, negligible in the relative density), while the black phase had good dispersion and a
tinny size (under 3 μm). Table 1 shows the relative densities and the volume fractions of the four different phases measured and calculated using the Image-Pro software.

From the volume fraction and XRD results, it can be inferred that the light grey matrix phase is MoSi₂ and the grey phase is ZrB₂, while the volume fraction of the black phase is low (2.6–5.8%) which is in agreement with the analysis of the XRD patterns for SiC, thus the black phase should be SiC. The relative densities of all samples are in the range of 95 to 97%. To confirm the inference, the four phases were investigated by EDS analysis.

Figure 6 shows the BSED and EDS mapping images of M10. The Mo, Si, B, Zr, C and O elements were detected and the four phases were marked by blue and hollow arrows, and white and blue boxes, respectively in Fig. 6(a). The concentration of Zr was high, while the other elements had low concentration in the blue arrows in Fig. 6(e). Moreover, B had a dense distribution area indicated by the blue arrows in Fig. 6(d). The concentrations of Mo and Si were high on the hollow arrows in Figs. 6(b) and 6(c). High concentrations of Si, C and the small concentration of O were noted in the white boxes in Figs. 6(c), 6(f) and 6(g). The white spot showed high concentrations of Mo and B in the blue box in Figs. 6(b) and 6(d).

To further infer the composition of the white spots, a big sample was selected and evaluated using spot-scanning EDS analysis. Figures 6(h) and 6(i) shows the strongest intensity for Mo. Using the results of EDS mapping, the white phase was identified as the Mo–B compound, which is usually found at low volume fractions in the Mo–Si–B system. According to the XRD results, it could be confirmed that the light grey matrix phase marked by hollow arrows is MoSi₂ and the grey phase marked by blue arrows is ZrB₂, while SiC and SiO₂ are labeled in the white boxes. It should also be noted that the information mechanism for the SiO₂ phase in the MoSi₂ matrix composites and the effects on XRD have been reported in the literature. The
EDS results are in agreement with the previous inferences on the microstructure and volume fraction parts.

3.4 Vickers hardness and fracture toughness

Figure 7 shows the fracture toughness and Vickers hardness of xMoSi₂–0.2ZrB₂–0.1SiC composites. The M10 composite showed the highest fracture toughness of 5.5 MPa m¹/₂ and Vickers hardness of 14.0 GPa. Vickers hardness varied from 11.6 to 14.0 GPa from M100 to M10. The hardness was in compliance with the rule of mixture,³⁸ as seen in the Eq. (5):

\[
H = v_{f1} H_{c1} + v_{f2} H_{c2} + (1 - v_{f1} - v_{f2}) H_m
\]

where \(H_{c1}\), \(H_{c2}\) and \(H_m\) are the hardness of the ZrB₂, SiC and MoSi₂ phases, while \(v_{f1}\) and \(v_{f2}\) are the volume fractions of ZrB₂ and SiC, respectively. The hardness is 25 GPa for ZrB₂,³⁹ 26 GPa for SiC⁴⁰ and 11.6 GPa for MoSi₂ (experimental value in this study). The red dash line in Fig. 7 represents the hardness of the xMoSi₂–0.2ZrB₂–0.1SiC composites calculated from Eq. (5). This shows the same tendency as the black line. The hardness of the xMoSi₂–0.2ZrB₂–0.1SiC composite ceramics increases with the increasing volume fraction of the ZrB₂ and SiC phases.

Compared to pure MoSi₂ (3.2 MPa m¹/₂ in this study), the toughness of M10 (5.5 MPa m¹/₂) improved by approximately 73%. The SEM and BSED images of the indentation crack propagation paths for M10 are shown in Figs. 8(a) and 8(b), respectively. As shown in Figs. 8(b), the crack bridge was visible in part A, the crack deflection was observed in part B and the formation of microcracks were impeded by the second phase.

As shown in Figs. 8(b)–8(d), Zr and B had dense distribution on the hollow arrows, which implies that the ZrB₂ phase causes crack deflection in MoSi₂–ZrB₂–SiC composites. Evans et al.⁴¹ have reported the formation of microcracks primarily induced by the additional and residual stress caused by the mismatch between the thermal expansion coefficients. The morphology of the microcracks clearly indicates their formation is due to compressive stress.⁴² As second phase, ZrB₂ and SiC can reinforce the MoSi₂ matrix composites through crack bridge, crack deflection and microcracks mechanisms. These mechanisms can complicate and obstruct the indentation crack propagating paths⁴³ by absorbing energy during crack propagation.⁴²,⁴⁴ Thus, by introducing higher volume fractions of ZrB₂ and SiC into MoSi₂ matrix composites, fracture toughness can be improved.

4. Conclusion

The in situ combustion behavior and mechanical properties of MoSi₂–ZrB₂–SiC composites were studied by SHS-SPS. The combustion synthesis of MoSi₂–ZrB₂–SiC composites occurs via spiral combustion with a dissolving-
precipitating mechanism. The M10 ceramic had the highest Vickers hardness (14.0 ± 0.7 GPa) and the best fracture toughness (5.5 ± 0.8 MPa m^{1/2}), exceeding the fracture toughness of pure MoSi_{2} ceramic by about 73.3%. The increasing volume fraction of ZrB_{2} and SiC particles in the xMoSi_{2}−0.2ZrB_{2}−0.1SiC ceramics can increase the Vickers hardness of the composites and the variation of Vickers hardness is in agreement with the rule of mixture. ZrB_{2} and SiC particles can improve toughness via the crack deflection and crack bridge, which benefit the second-phase strengthening mechanism, as well as micro-cracks, which are caused by compressive stresses.

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