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Synthesis and mechanical properties of ternary molybdenum carbosilicide and its composite

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Among the compounds with general formula $M_{\leq 5}Si_3C_{\leq 1}$ where M = Zr, Nb, Mn or Mo, the molybdenum carbosilicide ($Mo_{\leq 5}Si_3C_{\leq 1}$), known as a Nowotny phase [1], is the only stable ternary phase in the Mo-Si-C system with a hexagonal $D8_8$ structure. There is a great possibility for $Mo_{\leq 5}Si_3C_{\leq 1}$ to be not only a reinforcement second phase but also a new matrix phase for composites based on Mo-Si-C systems [2–5], due to (a) its high melting point up to 2100 °C and (b) its relatively complex structure that may lead to excellent creep resistance. However, it has been reported that it is difficult to prepare $Mo_{\leq 5}Si_3C_{\leq 1}$ as a single-phase product owing to the small stability region in the phase diagram [6, 7]. Moreover, the understanding of its related properties is still poor, except some crystallographical parameters. Accordingly, in this investigation, $Mo_{\leq 5}Si_3C_{\leq 1}$ was synthesized by reactive hot-pressing (RHP) from elemental molybdenum, silicon and graphite powders. $Mo_{\leq 5}Si_3C_{\leq 1}$-based composites were also fabricated through addition of small amount of nano-SiC particles. Constituent phases were identified by X-ray diffraction and some mechanical properties were measured for Mo-Si-C system and its composites.

Commercially available high purity powders of molybdenum, silicon and graphite were used for preparations of compacts. They were weighted and mixed by wet ball-milling in ethanol for 24 hr in stoichiometric proportion to obtain $Mo_{\leq 5}Si_3C_{\leq 1}$. Wet ball-milled mixture was dried, ground to crush large agglomerates and finally uniaxially die-pressed under 400 MPa into platelets with dimensions of 36 mm × 18 mm × 10 mm. The compact was placed into carbon dice coated with BN and then reactively hot-pressed in vacuum at a respective temperature of 1500 and 1600 °C under an applied pressure of 15 MPa for 2 hr. Some compacts were also sintered at the same temperature without external pressure to obtain loose ones used for fabrication of $Mo_{\leq 5}Si_3C_{\leq 1}$-based composites after the sintered compact was pulverized into powders which were also used for X-ray diffraction. Nano-sized SiC particles (10 nm) were first dispersed by ultrasonic vibration in ethanol and then wet ball-milled together with the reactively sintered Mo-Si-C pulverized powders. The composite compact, where the weight percentage of SiC was 5%, was prepared and sintered using the same parameters as Mo-Si-C compacts. The flexural strength measurement was performed using a three-point bending test on the specimens with dimensions of 36 mm × 4 mm × 3 mm where the loading span is 30 mm, according to JIS 1601. The fracture toughness was measured by the single edge precracked beam (SEPB) method with a notch depth of 0.4 W (the width of specimen), according to JIS 1607. Both the flexural strength and fracture toughness tests were carried out on a Shimazu machine with 0.5 mm/min cross-head speed. At least three samples were measured for each kind of test. The variation between load and displacement was automatically recorded by a computer connected with the machine. The microstructure of sintered compacts and fracture surfaces after mechanical tests were observed using XL 30 Phillip scanning electron microscopy (SEM).

As shown in Fig. 1, X-ray diffraction patterns of samples synthesized at both 1500 and 1600 °C for 2 hr indicated Mo_5Si_3C as the major phase. In addition, small amounts of impurity phases were $MoSi_2$, Mo_3Si, Mo_2C, unreacted Mo, Si, C and some unidentified phases. However, the amount of Mo_2C decreased when the preparation temperature was raised to 1600 °C since its peak intensity became weak at this temperature. Suzuki and Niihara [8] have also observed the formation of Mo_2C in their preparations when the Si-content in the starting mixture was slightly less than that required for single-phase formation. Therefore, formation of Mo_2C in our preparations may be attributed to the volatilization of a small amount of Si from the reaction mixture during high temperature treatment.

Fig. 2 showed the microstructures of sintered Mo-Si-C compact and its composites reinforced by nano-sized SiC at 1600 °C under an applied pressure of 15 MPa for 2 hr. It was evident that both of them were fully dense. Comparatively, the latter exhibited finer microstructure than the former indicating the addition of nano-sized SiC inhibited to some extent the growth of related grains at high temperatures. However, the distribution of SiC particles was not so homogeneous in the matrix microstructure. As seen from the typical load-displacement curves in Fig. 3, the behavior of both materials was linear elastic. Monolithic $Mo_{\leq 5}Si_3C_{\leq 1}$ compacts showed relatively good flexural strength and

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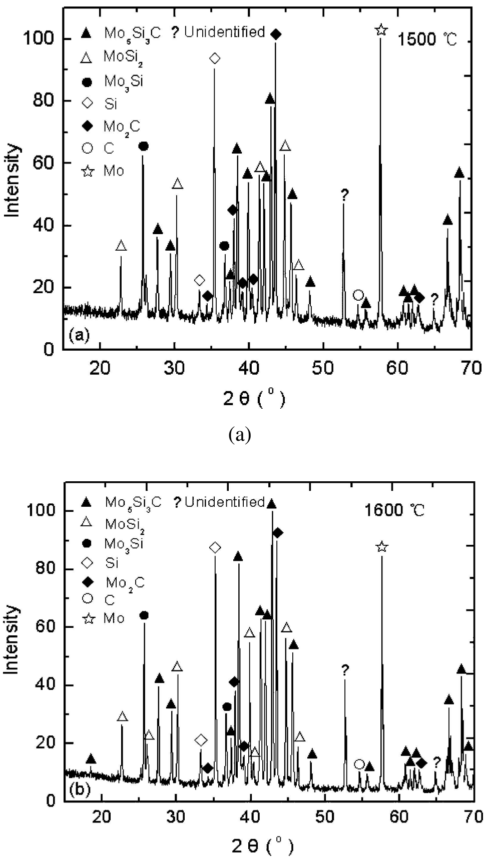

(a)

(b)

Figure 1 XRD patterns of the specimens prepared by hot pressing at (a) 1500 and (b) 1600 °C under 15 MPa for 2 hr.

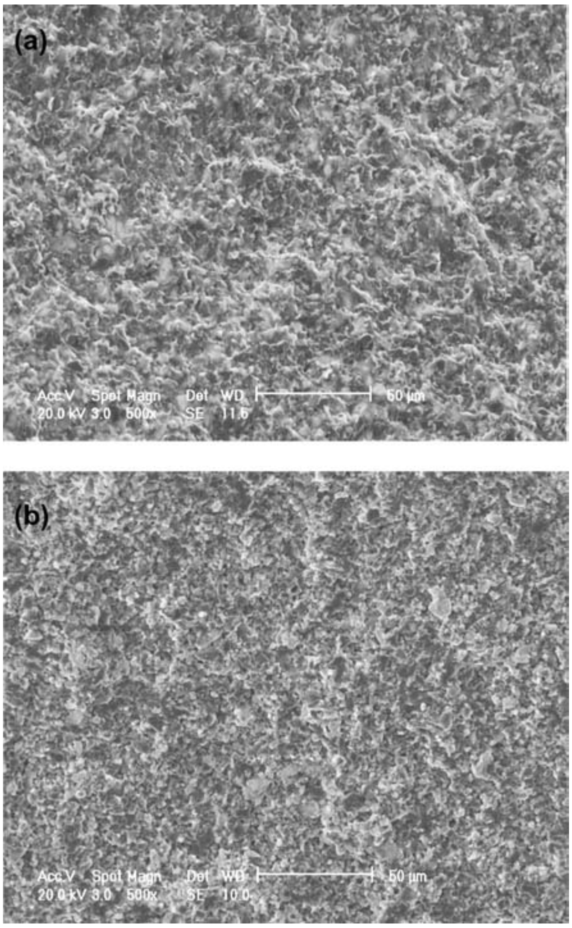

Figure 2 SEM photographs of (a) ternary molybdenum carbosilicide and (b) its composite reinforced by 5 wt.% nano-sized SiC particles.

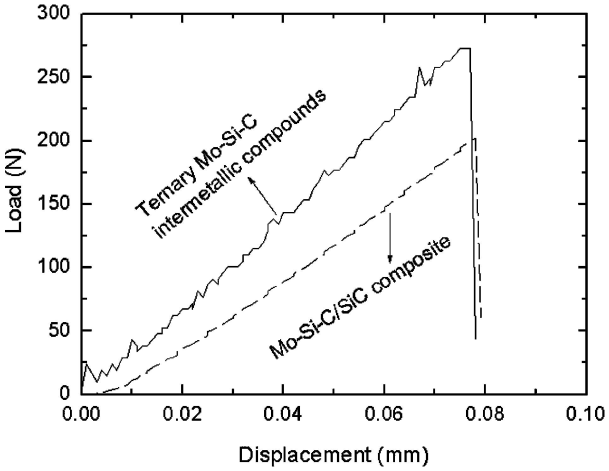

Figure 3 Load–displacement curves of ternary molybdenum carbosilicide and its composite at room temperature.

fracture toughness with a respective value of 360 MPa and 6.3 $\text{MPa}\sqrt{m}$. In contrast, Mo$_{\le 5}$Si$_3$C$_{\le 1}$/nano-SiC composites did not exhibit any superiority in mechanical properties to Mo$_{\le 5}$Si$_3$C$_{\le 1}$ compacts since its flexural strength and fracture toughness were 330 MPa and 4.7 $\text{MPa}\sqrt{m}$, respectively. This was consistent with the results of *in-situ* Mo$_{\le 5}$Si$_3$C$_{\le 1}$-based composites with

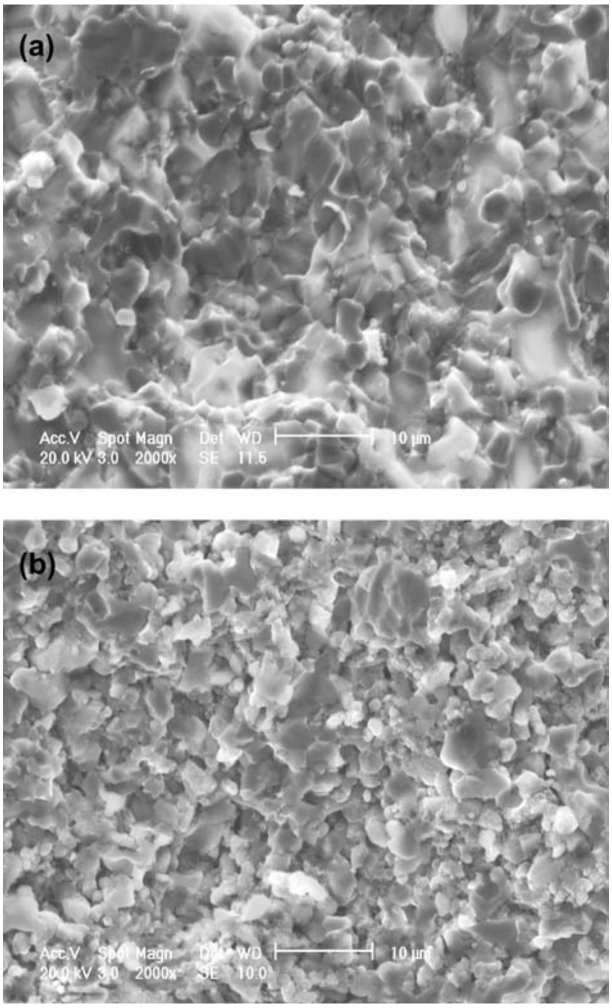

Figure 4 Fracture surfaces of (a) ternary molybdenum carbosilicide and (b) its composites with SiC after bending test at room temperature.

Mo_2C obtained by Suzuki *et al.* [8]. The decrease in mechanical properties of the composites in our investigation may be due to the inhomogeneous distribution of nano-sized SiC particles through the matrix as shown in Fig. 2.

Fig. 4 showed the fractographs of the base intermetallic compound and its composite. Both of them exhibited a typical brittle fracture mode. However, the former showed a flatter surface than the latter. Again, agglomeration of nano-sized SiC can be found on the fractograph of $Mo_{\leq 5}Si_3C_{\leq 1}$-based composites.

In summary, $Mo_{\leq 5}Si_3C_{\leq 1}$ was formed as the major phase by reactive hot pressing of elemental powders at both 1500 and 1600 °C under 15 MPa for 2 hr. The addition of nano-sized SiC inhibited the growth of grains at high temperatures. Monolithic $Mo_{\leq 5}Si_3C_{\leq 1}$ compacts showed relatively good flexural strength and fracture toughness with a respective value of 360 MPa and 6.3 $\text{MPa}\sqrt{m}$. However, $Mo_{\leq 5}Si_3C_{\leq 1}$/nano-SiC composites did not exhibit any superiority in mechanical properties to $Mo_{\leq 5}Si_3C_{\leq 1}$ compacts, which was mainly attributed to the agglomeration of SiC particles in the matrix microstructure.

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