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Processing and Properties of $MoSi_2$–Nb Composites

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Abstract: Powder processing techniques for $MoSi_2$ composites are described. The effect of Nb morphology on the fracture resistance of the composites was studied. Discontinuous random Nb particles or fibers deflected cracks that propagated through the $MoSi_2$ matrix. However, this did not result in any improvements in toughness (as measured from the area under flexural stress–displacement curves). Aligned Nb fibers oriented perpendicular to the direction of matrix crack propagation directly participated in the fracture of the composite; these fibers toughened $MoSi_2$. However, there was an effect of the diameter on the fracture behavior of the aligned fibers. The effect of fiber diameter on fiber–matrix separation could be explained through the load carrying capacity of the fibers and the ability of the fiber–matrix interface to separate.

1 INTRODUCTION

Many attempts to toughen brittle intermetallics with ductile phases have been reported in the literature.[1–5] The refractory metals Nb[4,5] and Ta[6] have been incorporated into $MoSi_2$ by several investigators. Both reinforcements react with $MoSi_2$ and are easily oxidized, so they would have to be coated to be effective. Niobium is much less dense than tantalum, and therefore has been studied more extensively. The purpose of the present investigation was to study the influence of niobium morphology on the mechanical behavior of $MoSi_2$.

2 EXPERIMENTAL PROCEDURE

Hot isostatic pressing (HIP) of pre-alloyed powders was used to prepare monolithic $MoSi_2$ and $MoSi_2$ reinforced with 20vol% Nb. The Nb was incorporated into the $MoSi_2$ as nominally 100μm diameter particles, 400 and 800μm diameter random short fibers, and 400 and 800μm diameter continuously aligned fibers.

The particulate and random short fibrous composites were introduced into the $MoSi_2$ by simply blending the $MoSi_2$ powder with the Nb powder or fiber. Appropriate amounts of $MoSi_2$ powder and Nb particles (or 10mm long Nb fibers) were weighed to produce a mixture corresponding to $MoSi_2$/20vol (25wt%) Nb. The composite mixtures (or monolithic $MoSi_2$ powder) were placed into a cylindrical polyurethane Cold Isostatic Press (CIP) mold bag lined with Nb foil (0·025mm thick). The mixtures were CIPed at 241 MPa.

Continuous fibers were aligned in $MoSi_2$ powder by first cutting the Nb wire to the nominal length (90mm) of the charge cavity of a CIP mold bag. The 90mm long Nb fibers and $MoSi_2$ powder were weighed to produce a nominal composition of 20vol% Nb fibers. A CIP mold bag lined with Nb foil was filled to one half its capacity with the $MoSi_2$ powder. The Nb fibers were then hand placed in the powder. Once all fibers were in place, the remainder of the $MoSi_2$ powder was poured into the CIP mold, infiltrating the exposed Nb fibers. The composite was then CIPed at 241 MPa to produce a green specimen.

The CIP bars were vacuum encapsulated in HIP cans made from Ti. Consolidation of the monolithic $MoSi_2$ and the $MoSi_2$/20vol% Nb composites was achieved by HIPing at 1350°C and 172 MPa for 3 h. Cooling and depressurization occurred over a four hour period.

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Right cylindrical specimens were produced by electro-discharge machining (EDM) from the HIP bars. The specimens were 6·35mm diameter by 30mm long. For each composite a minimum of two specimens was prepared. Once machined, the specimens were containerless HIPed using the cycle previously described.

3 RESULTS

3.1 Microstructural features

The materials produced were essentially fully dense. The aligned fibers tended to agglomerate together due to the difficulty in placement of the fibers. As a result, the actual volume fraction varied between bend specimens from 18 to 20 vol% Nb fibers.

During processing a reaction between the $MoSi_2$ matrix and Nb reinforcement occurred. The reaction zones between the 400 and 800μm Nb fibers and $MoSi_2$ matrix were approximately 20μm thick and were chemically identical. Electron microprobe analysis identified the phases of the reaction zone as N_5Si_3, $(Nb,Fe)_5Si_3$ and NbS_2. The reaction zone between the Nb particles and the $MoSi_2$ matrix was only 10μm thick and consisted of two phases: Nb_5Si_3 and $(Nb,Mo)_5Si_3$. Unlike the reaction zone of the fibrous composites, no Fe-containing phase was found around the particles. The Fe was probably a contaminant introduced into the Nb during wire drawing. The characterization of the reaction zones suggests that the same interphase region developed in the fibrous composites. Therefore, any differences in mechanical properties cannot be attributed to differences in the reaction zone chemistry.

3.2 Mechanical properties

3.2.1 Monolithic $MoSi_2$

The room temperature flexural stress versus displacement curves for monolithic $MoSi_2$ were linear, indicating no plastic deformation. This was confirmed by the flatness of the fracture surface. The mean flexural strength was 345 ± 83 MPa. Fracture was predominantly intergranular.

3.2.2 Niobium

To simulate the composite processing conditions, as-received Nb fibers were heat treated at 1350°C for 6 h in vacuum. The microstructure of the Nb wires in the as-heat treated condition was similar to the as-composite processed condition. The grain size of the Nb wire appeared to be the same for both conditions, of the order of 250μm. The Nb fibers in the heat treatment condition were tested in tension at room temperature and were ductile. The hardness and grain size of the fibers in the heat treated and composite processed conditions were similar. Thus, the behavior of the as-heat treated wires simulates the unconstrained behavior of the Nb fibers in the as-composite processed conditions.

3.2.3 $MoSi_2$/20vol% Nb particles

Niobium particles did not improve the toughness of $MoSi_2$; in fact, they weakened the composite compared to monolithic $MoSi_2$ (Table 1). The flexural stress vs displacement curves were linear, indicating extreme brittleness. It appears that the Nb particles may have deflected the crack propagating through the matrix, as the fracture surfaces did not appear as flat as that of monolithic $MoSi_2$. The Nb particles failed by cleavage.

3.2.4 $MoSi_2$/20vol% Nb random short fibers

The addition of the discontinuous, randomly oriented Nb fibers could be considered to be similar in effect to the addition of very large Nb particles. The flexural stress–displacement curves were variable in nature (Fig. 1), with some curves showing no resistance to fracture (400μm specimen #2) and some curves displaying non-catastrophic failure (800μm specimen #2). The fracture surfaces of these composites clearly indicate that the Nb deflected matrix cracks, as it appeared that the matrix crack propagated through the interface between the fiber and matrix (Fig. 2). Some fibers fractured during testing. These fibers tended to be oriented perpendicular to the fracture surfaces, and thus directly participated in the fracture of the composite. Also, there was a correlation between the number of these fractured fibers and the fracture energy. Those composites that contained numerous fractured fibers had higher fracture

Table 1. Fracture energy of $MoSi_2$/20vol% Nb composites (measured from the area under flexural stress–displacement curves)

Composite	Energy (J/cm^2)	Normalized energy E
Monolithic $MoSi_2$	1·4 ± 0·7	1·0
$MoSi_2$/20vol% Nb particles	1·2 ± 0·6	0·9
$MoSi_2$/20vol% random short 400μm diameter Nb fibers		
Specimen #1	4·0	2·9
Specimen #2	0·9	0·6
$MoSi_2$/20vol% random short 800μm diameter Nb fibers		
Specimen #1	4·1	2·9
Specimen #2	12·3	8·8
Specimen #3	2·4	1·7
$MoSi_2$/20vol% continuous aligned 400μm diameter Nb fibers		
Specimen #1	9·3	6·6
Specimen #2	7·8	5·6
$MoSi_2$/20vol% continuous aligned 800μm diameter Nb fibers		
Specimen #1	44·1	31·5
Specimen #2	29·7	21·2

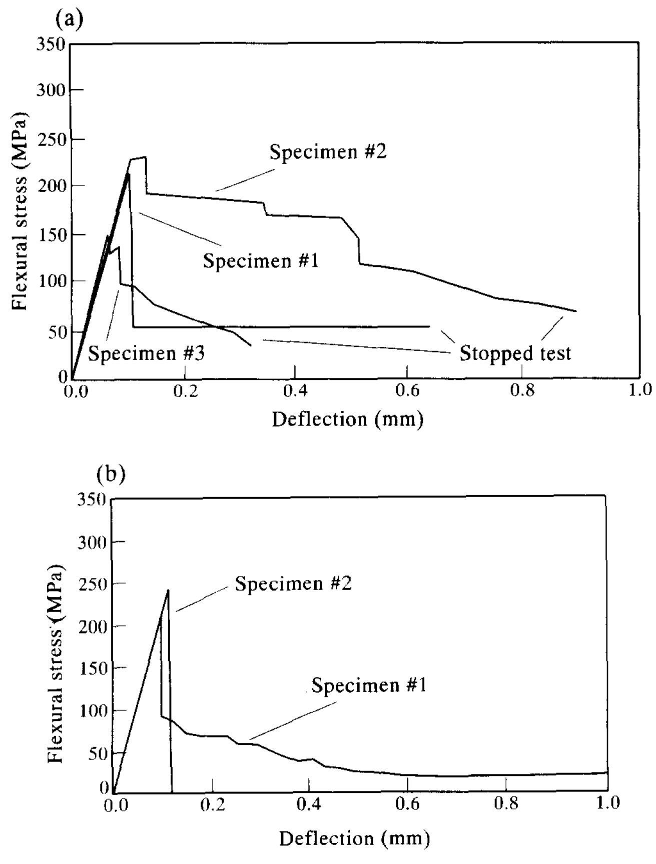

Fig. 1. Flexure curves for randomly oriented fibrous $MoSi_2$–Nb composites; (a) 800μm fibers, (b) 400μm fibers.

energies (400μm specimen #2 and 800μm specimen #2, Table 1 and Fig. 3). Clearly, when the fibers directly participated in the fracture process (through fiber fracture), the energy required to fracture the composite was greatly enhanced.

3.2.5 $MoSi_2$/20vol% continuously aligned Nb fibers

Composites containing aligned continuous fibers displayed the greatest resistance to failure (Table 1 and Fig. 4). The flexural stress–displacement curves of these composites showed significant resistance to fracture with no large drop in stress at the point of matrix fracture. Aligned fibrous composites containing the larger diameter fibers displayed the greatest enhancement in toughness as the normalized fracture energies of these composites ranged from 20 to 30. The addition of the smaller diameter fibers did not significantly enhance the toughness of $MoSi_2$, as these composites had normalized fracture energies of about 6. The extent of toughening derived from the addition of the fibers can be related to the fracture behavior of the fibers in the composites. The fracture surfaces revealed that the large Nb fibers had deformed prior to fracture, with portions of fractured matrix (and/or reaction zone) attached to the fiber. The smaller diameter Nb fibers did not extensively deform during testing, with some fibers failing by cleavage (see Fig. 3). Cross-sections of the fracture surfaces revealed that the larger diameter fibers had extensively separated from the matrix through cracking of the reaction zone adjacent to the fibers, Fig. 5(a), whereas the smaller diameter fibers remained strongly bonded to the matrix, as no cracks were observed in the reaction zone, Fig. 5(b). This behavior is consistent with previously reported observations that maximum toughness occurs when fiber deformation occurs simultaneously with fiber–matrix separation[7,8].

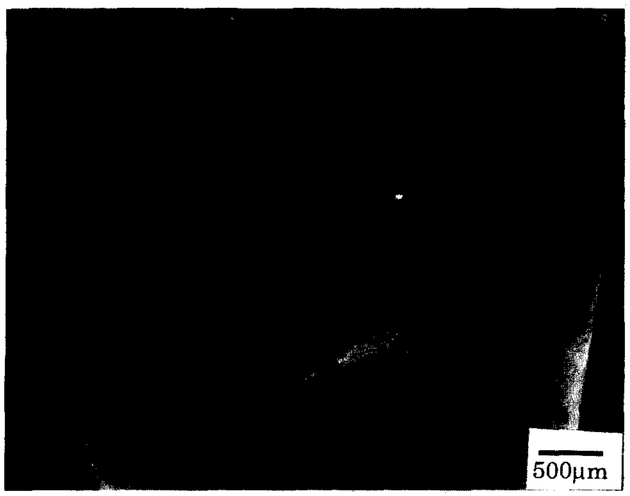

Fig. 2. Fracture surface of randomly oriented $MoSi_2$–Nb composites; 800μm fibers.

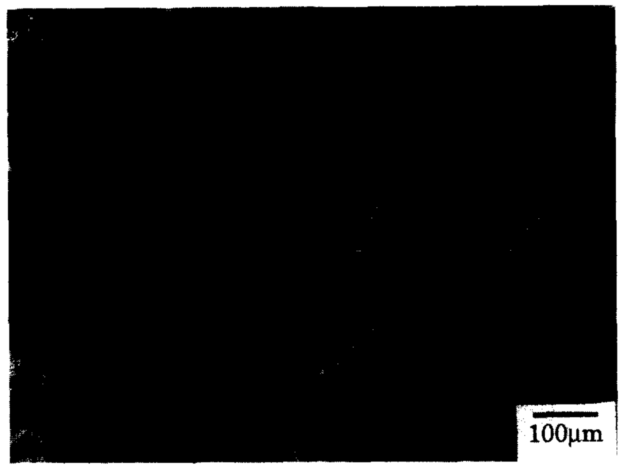

Fig. 3. Fracture surface of $MoSi_2$–Nb, 400μm diameter fibers.

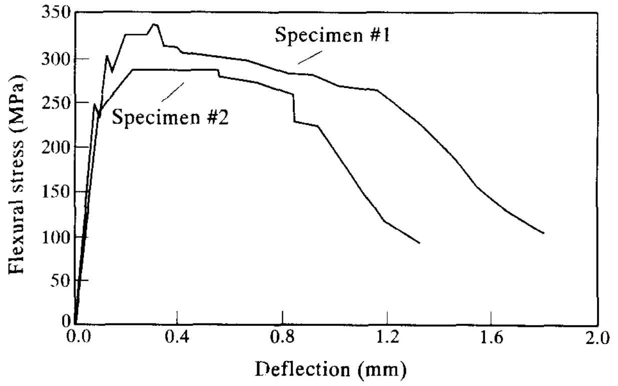

Fig. 4. Flexure curves for 800μm diameter continuous fiber $MoSi_2$–Nb composites.

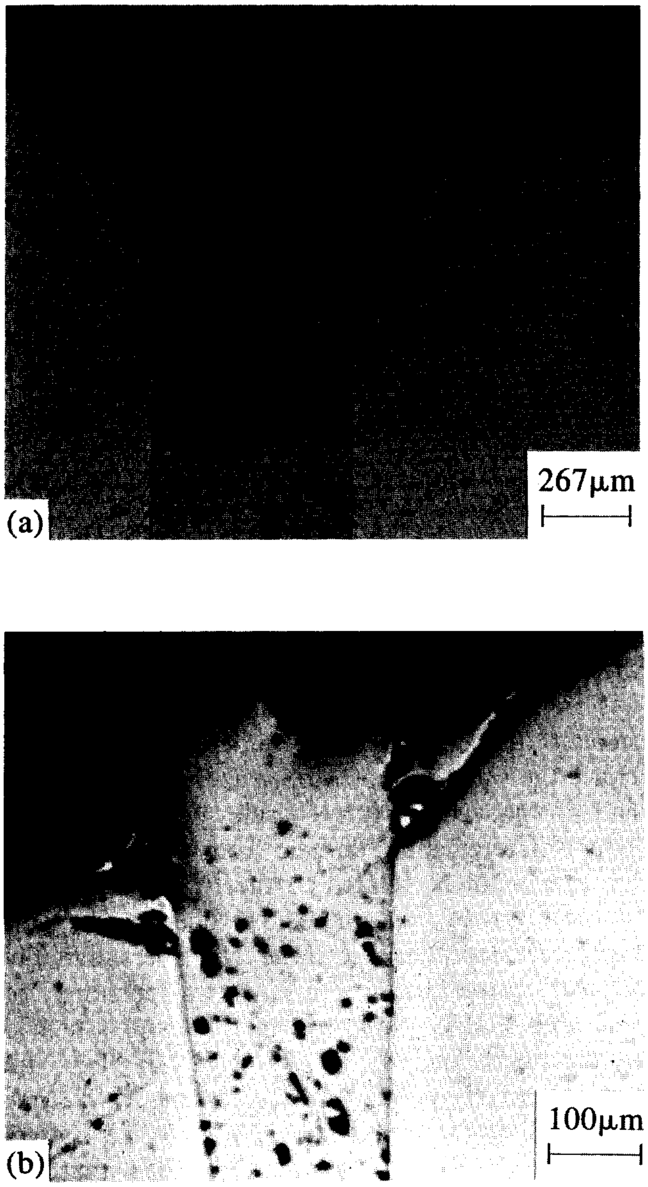

Fig. 5. Longitudinal sections of $MoSi_2$–Nb composites; (a) 800μm diameter fibers, (b) 400μm diameter fibers.

4 DISCUSSION

The results clearly demonstrate that the morphology of Nb added to $MoSi_2$ significantly affected the fracture behavior of the composite. This confirms the conclusions of earlier investigators that particle or fiber size and shape are important factors in determining toughness.[9-11] Two toughening mechanisms were observed, crack deflection and ductile phase toughening. Which mechanism was operational was dependent upon the morphology of the Nb incorporated into the $MoSi_2$. The addition of small Nb particles resulted in crack deflection. The incorporation of large particles (the random short fibers) resulted in both mechanisms operating simultaneously. The addition of oriented fibers aligned perpendicular to the direction of matrix crack propagation resulted in ductile phase toughening.

The Nb was not effective in enhancing the toughness of $MoSi_2$ via crack deflection. This may have been a consequence of the brittle nature of the reaction zone, as evidenced by the ease with which cracks propagated from hardness indentations in the reaction zone. Matrix cracks may preferentially propagate through the reaction zone; thus, the Nb does not enhance the toughness of $MoSi_2$ through crack deflection. Some of the randomly oriented fibers fractured during testing. Composites consisting of these fibers displayed intermediate resistance to fracture. Clearly, Nb was most effective in toughening $MoSi_2$ through a deformation–fracture or a ductile phase toughening mechanism.

5 SUMMARY AND CONCLUSIONS

The morphology of Nb added to $MoSi_2$ significantly affected the fracture behavior of the composite. Two toughening mechanisms were observed, crack deflection and ductile phase toughening. Which mechanism was operative was dependent on the morphology of the Nb incorporated into the $MoSi_2$. The addition of random or discontinuous Nb particles or fibers resulted in toughening through crack deflection. This mechanism was not effective in improving the toughness of $MoSi_2$. The addition of oriented fibers aligned perpendicular to the direction of matrix crack propagation resulted in ductile phase toughening.

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REFERENCES

1. HING, P. & GROVES, G. W., *J. Mater. Sci.*,7 (1972) 427.
2. CHERMANT, J. L. & OSTERSTOCK, F., *J. Mater. Sci.* **11** (1976) 1939.
3. FITZER, E. & REMMELE, W. In *Proc. 5th Int. Conf. on Composite Materials*, ICCM-V, ed. W.C. Harrigan, Jr., J. Strife & A. K. Dhingra. TMS, Warrendale, PA, 1985, p. 515.
4. DEVE, H. E. & MALONEY, M. J., *Acta Metall.*, **39** (1991) 2275.
5. DEVE, H. E., WEBER, C. H. & MALONEY, M., *Mater. Sci. Engng*, **A153** (1992) 668.
6. CARTER, D. H. & MARTIN, P. L., *Intermetallic Matrix Composites, MRS Symp. Proc.*, **194** (1990) 131.
7. XIAO, L. & ABBASCHIAN, R., *Metall. Trans.*, **24A** (1993) 403.
8. ASHBY, M. F., BLUNT, F. J. & BANNISTER, M., *Acta Metall.*, **37** (1989) 1847.
9. XIAO, L. & ABBASCHIAN, J. R., *Metall. Trans.*, **24A** (1993) 403.
10. NARDONE, V. C. & STRIFE, J. R., *Metall. Trans.*, **22A** (1991) 403.
11. VENKATESWARA RAO, K. T., SOBOYEJO, W. O. & RITCHIE, R. O., *Metall. Trans.*, **23A** (1992) 2249.