

Research Article

Development of MoSi₂-SiC Component for Satellite Launch Vehicle

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Received 7 August 2012; Accepted 29 August 2012

Academic Editors: D. Escalera and P.-Y. Lee

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Intermetallic base MoSi₂-SiC composite, an excellent high temperature oxidation-resistant material meant for the aerospace structural applications between 1600°C and 1700°C under oxidizing environment, has been developed successfully using powder metallurgy techniques. Mechanically milled (MM) MoSi₂ powder, blended with SiC particulate was consolidated by vacuum hot pressing, yielded about 98.5% theoretical density. The composite has been characterized for physical, mechanical, and thermal properties. Properties were found satisfactory. Machining of semis to intricate shape was possible through electro-discharge machining (EDM) process. Plasma arc jet test (PAJT) under argon and argon + oxygen environment has proved its excellent high temperature oxidation resistance properties as it could sustain high heat flux up to 250 W/cm² under oxidizing environment. The component realized has full potential to be used in critical aerospace application. This paper highlights the details of experimental work carried out and its characteristic properties attained.

1. Introduction

Molybdenum disilicide (MoSi₂) is an intermetallic with tetragonal unit cell arrangement (Figure 1) and has strong potential for high temperature structural applications in aerospace industries due to its high melting point (2030°C) and ability to undergo plastic deformation above 1200°C. Molybdenum disilicide possesses outstanding oxidation resistance up to 1700°C due to the formation of an impervious film of SiO₂ on the surface which prevents further oxidation. Being electrically conductive, it could be machined by electro-discharge machine (EDM) to desired shapes [1–3]. Apart from wide use as heating element, it is also used in power generation components, heat exchanger, filters, turbine blades, vanes, combustion nozzles, turbocharger rotors, valves, and so forth [4]. However, its room temperature fracture toughness is poor compared to other high temperature metallic systems. Other mechanical properties such as yield strength and creep strength gradually decrease above 1100°C. Hence, reinforcement of the matrix with thermally stable ceramic particles or whiskers which can significantly improve the fracture toughness at room temperature as well as high temperature properties is recommended

[5–7]. It is chemically compatible with a number of ceramic constituents such as SiC, Si₃N₄, TiC, TiSi₂, ZrO₂, Al₂O₃, and so forth, which could be judiciously selected as reinforcement for particular use and temperature regime [8, 9].

In the present work, MoSi₂-20v% SiC composite discs were consolidated using vacuum hot press (VHP) to near theoretical density (TD) using mechanically milled (MM) MoSi₂ powder and SiC particulates. The composite was characterised for mechanical properties and was evaluated for its thermal property. Uniform distribution of SiC particulates in MoSi₂ matrix was confirmed by optical microscopy. Heat flux test on the composite carried out under oxidizing plasma indicated its excellent oxidation resistance and its ability to withstand high heat flux. Based on the technical properties achieved, a critical component of Indian space programme was fabricated out of this.

2. Processing

Mo and Si powders were blended in stoichiometric ratio as starting ingredients for mechanical milling (MM). The mixture along with grinding media (SS440C balls) in 1 : 10

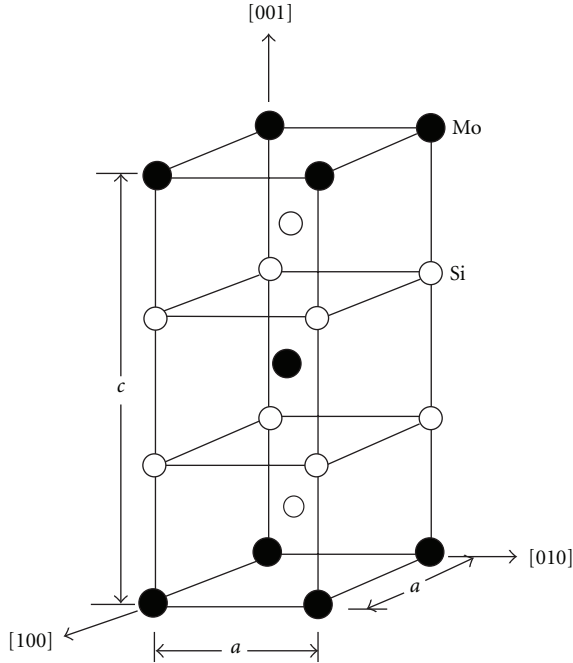


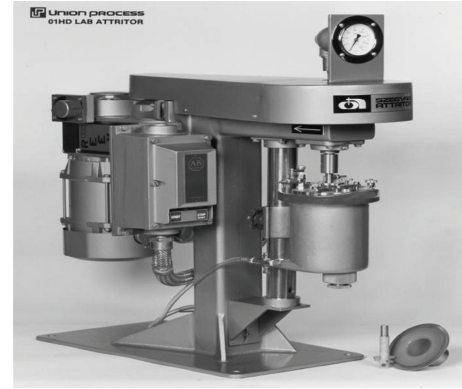
FIGURE 1: Schematic sketch of atomic arrangement of tetragonal unit cell structure of MoSi_2 .

ratio was charged in Union Process 01HD attritor mill (Figure 2). The milling time was optimized based on particle size measurement in periodic interval. Mo powder (99.8% purity) with $7\ \mu\text{m}$ (avg.) particle size was reduced at 800°C in H_2 prior to MM. Reduced Mo powder was sieved and blended with below-38-microns Si powder (99.6% purity). Milling was performed under protective (Argon) atmosphere at 400 rpm for an optimized time of 20 hrs (Figure 3). The particle size distribution of MoSi_2 using bimodal pattern and average size of particles observed under scanning electron microscope are shown in Figure 4.

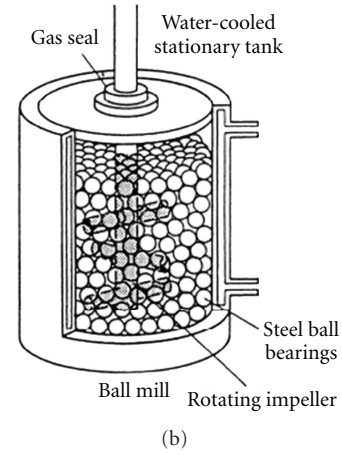
SiC powder of 21-micron particle size (avg.) was treated with 40% HF solution to remove SiO_2 prior to blending with mechanically milled (MM) MoSi_2 . Further processing involved vacuum degassing at 800°C for 2 hours and vacuum hot pressing (VHP) between 1500 and 1600°C for 1 hour using a pressure of 22 MPa. The heating rate was kept below $10^\circ\text{C}/\text{min}$ to maintain temperature uniformity, effective degassing, and full conversion to silicides.

The hot pressing was carried out in the range $1500/1600^\circ\text{C}$ with an intermediate soaking at 1200°C for 2 hrs, keeping in view of thermodynamics of the system and ensuring the following reactions, namely [10]: (i) reaction between Mo and Si to form MoSi_2 , (ii) transformation of lower silicides into MoSi_2 , and (iii) to completing and achieving near theoretical density (TD).

The probable reactions, which take place are [10, 11] as follows.



(a)



(b)

FIGURE 2: Photographs showing (a) attritor mill and (b) rotating impeller with mixture and grinder media.

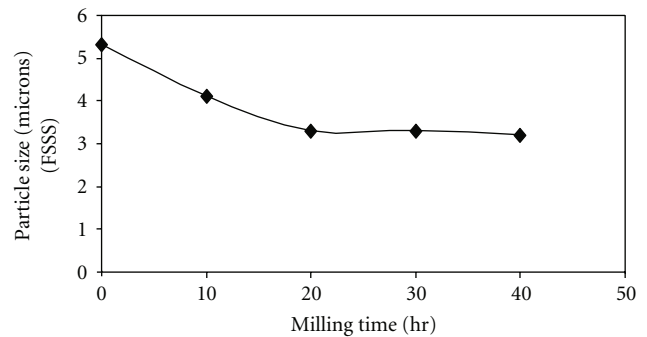
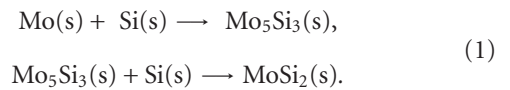


FIGURE 3: Effect of milling time on particle size reduction.

Conversion of lower silicide (before melting of Si):



Direct reaction (after melting of Si):



It was found that the temperature for VHP processing (1500 – 1600°C) to achieve near TD is much lower compared

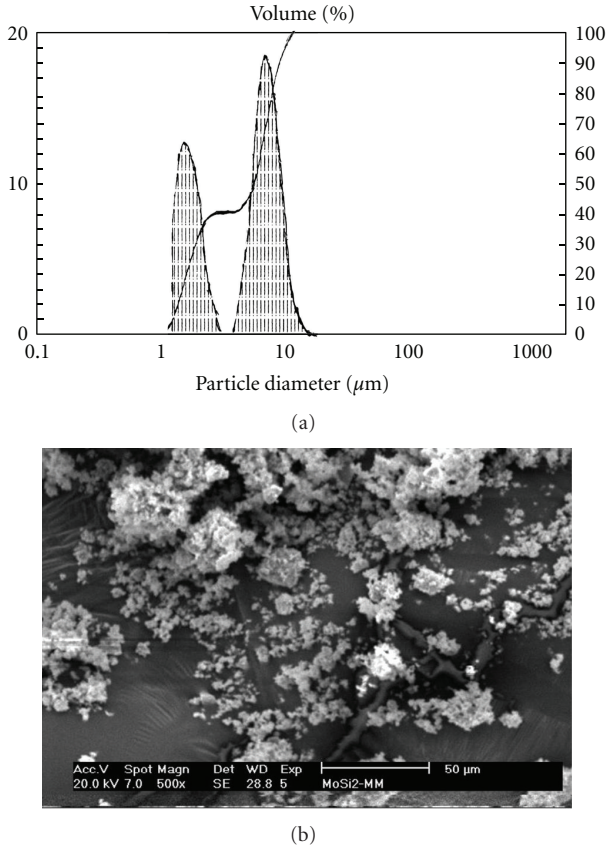


FIGURE 4: (a) Bimodal distribution pattern of mechanically milled MoSi_2 and (b) particles as such under SEM.

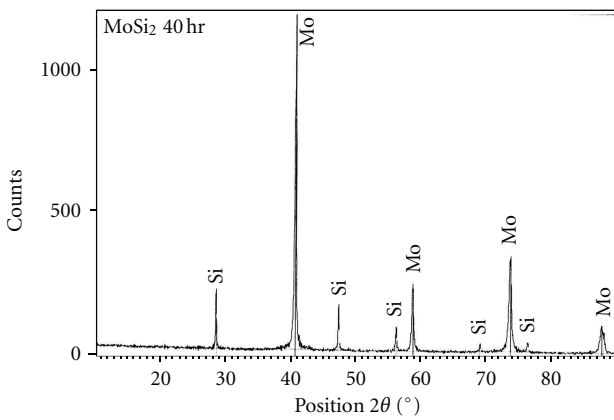


FIGURE 5: XRD pattern of powder specimen after 40 hrs of milling.

to conventional compaction and sintering ($>1800^\circ\text{C}$) route. The high energy state of MM powder and formation of liquid phase due to melting of Si caused lower sintering temperature and shorter time to achieve near theoretical density in the VHP process. The similar observation has been noticed by Schwarz et al. [12] for hot pressing of MoSi_2 powder made in high energy Spax mill.

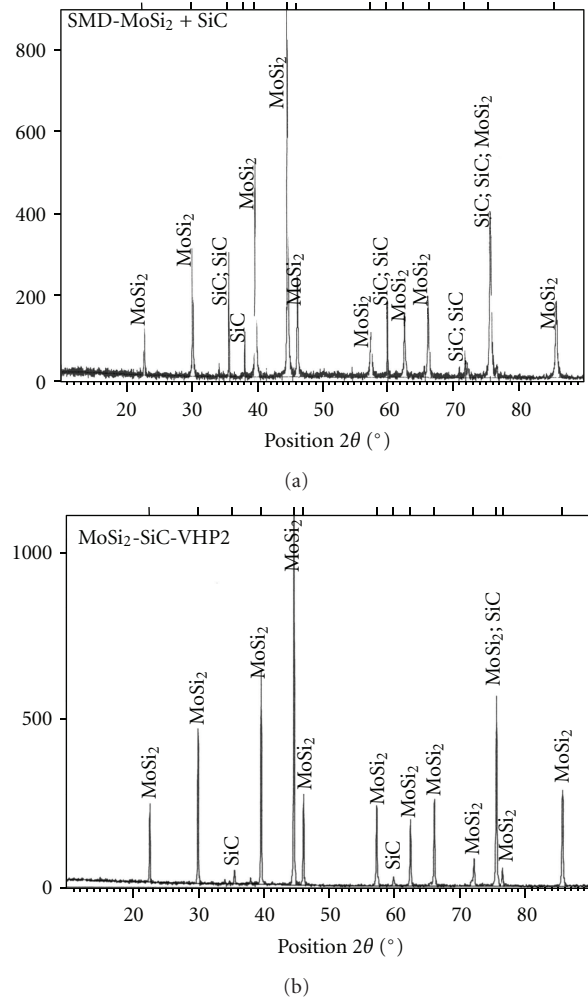


FIGURE 6: XRD pattern of MoSi_2 -SiC composite specimen, vacuum hot pressed at (a) 1500°C and (b) 1600°C .

3. Characterisation

Density measurement of composite was carried out by Archimedes' principle. Optical metallography was conducted with and without etching to assess grain size of MoSi_2 and dispersion of SiC particulate in the matrix. Phases have been identified through XRD analysis. Powder as well as polished specimen from hot pressed compact were analysed by using $\text{CuK}\alpha$ radiation. Hardness was measured on the metallographically polished specimens using Vicker's microhardness tester on individual particles, with diamond indenter at a load of 300 g. Vicker's macro-hardness was also measured on the composite to estimate its bulk hardness.

Mechanical properties were carried out at room temperature. Flexural strength specimens with a span (L) of 40 mm, a thickness (B) of 6 mm and width (W) of 8 mm were prepared by utilizing diamond cutter followed by utilizing smooth finish. Flexural test, a 3-point bend test, was carried out using a loading rate 0.5 mm/min in INSTRON machine as per ASTM C-1211. The flexural strength (σ) was evaluated by the expression, $\sigma = 3 PL/2BW^2$, where P was the

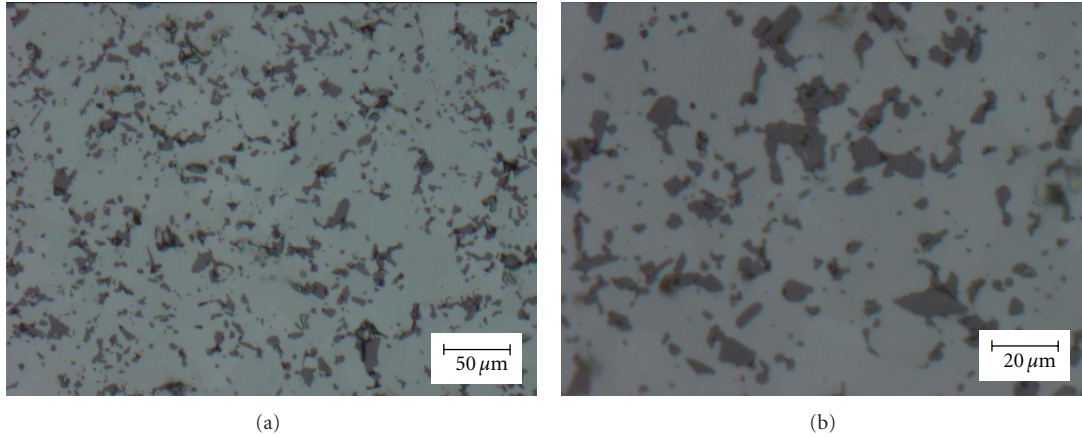


FIGURE 7: Optical microphotographs of MoSi₂-SiC composite specimen duly polished and unetched showing (a), (b) uniform distribution of SiC (gray) in MoSi₂ matrix at two different magnifications.

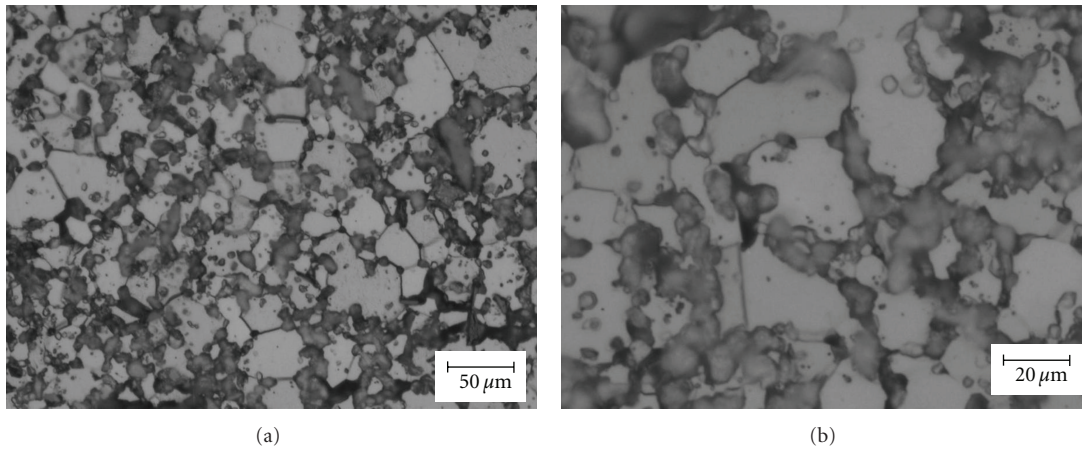


FIGURE 8: Optical microphotographs of MoSi₂-SiC composite specimen duly polished and etched specimen showing (a), (b) uniform distribution of SiC in MoSi₂ matrix at different magnifications. Fine equiaxed grains were also seen.

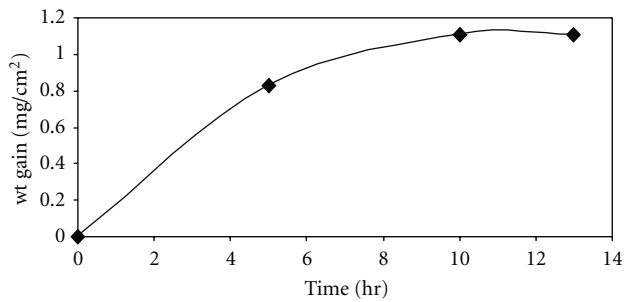


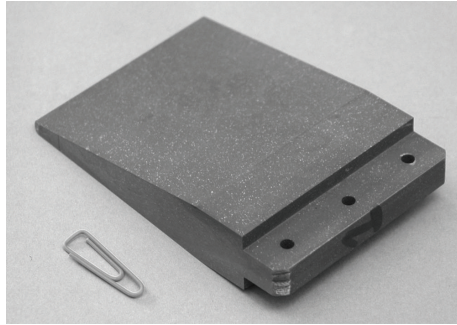
FIGURE 9: Oxidation behavior of MoSi₂-SiC composite exposed to 1400°C for 14 hrs.

maximum load in load-displacement curve. Compression test was conducted as per ASTM C-773 on 4 mm dia × 8 mm long test specimens using Instron machine at a loading rate of 0.5 mm/min. Modulus of elasticity was measured by ultrasonic nondestructive evaluation procedure. Longitudinal and shear waves have been used for determination of

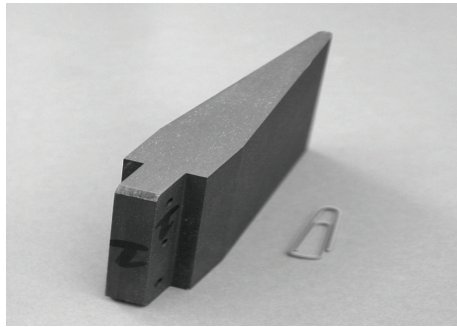
E value. Another approach, that is, estimation of E value from formula $E = V_L^2 \ell (1 + \gamma)(1 - 2\gamma)/(1 - \gamma)$ where V_L is longitudinal wave velocity, ℓ = density, and γ = poisson's ratio, has also been used. Coefficient of thermal expansion was measured on $5 \times 5 \times 5$ mm test specimens up to 450°C. Evaluation of machinability of the composite was carried out by drilling a 2 mm dia. through hole by EDM. The specimen was evaluated for high temperature oxidation resistance. Heat flux studies were performed on 15 mm dia × 5 mm thick specimens at 250 W/cm² flux under argon and oxidizing atmosphere for 15 sec in plasma arc jet test facility.

4. Result and Discussion

Density measurement shows that the VHP processed composites had 99.6% and 98.6% TD when processed at 1600°C and 1500°C, respectively. Such a high densification by hot pressing for 1 hr, under 22 MPa pressure, indicates that MM processed powder had faster rate of densification. It can be inferred here that finer particle sizes (3.3 μm, Fisser's



(a)



(b)

FIGURE 10: Critical shaped component machined out of MoSi₂-SiC composite.

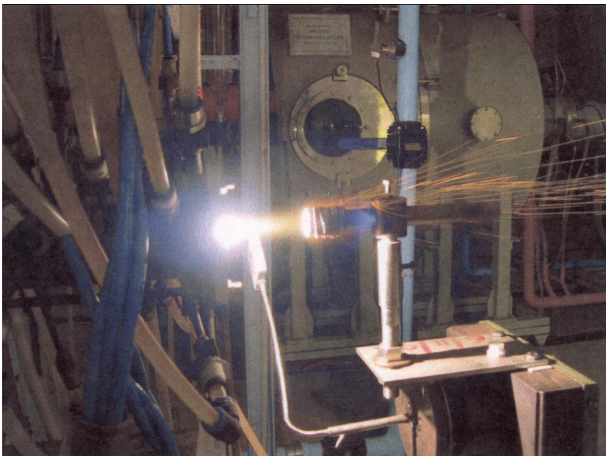


FIGURE 11: Plasma arc jet setup used for study.

subsieve size) of mechanically milled MoSi₂-SiC is densified more readily during hot pressing due to more homogenous mixing, a higher driving force available from high particle surface area and shorter diffusion distances for both Nabarro Herring and Coble creep deformation to occur [14, 15]. It was observed that even after mechanical milling for 40 hrs, MoSi₂ or other silicides of molybdenum could not form. On the other hand, particle refinement and intimate mixture of Mo and Si could take place with the progress of milling. Particle size analysis data (Figure 4) and presence of Mo and

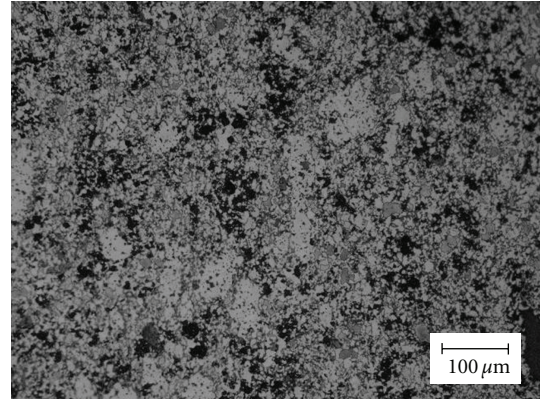


FIGURE 12: Optical photomicrograph of MoSi₂-SiC composite after plasma arc jet test showing stable microstructure with little grain coarsening.

Si peaks in XRD after 40 hrs of milling (Figure 5) supported the above statement.

XRD analysis of MoSi₂-SiC composite specimen, vacuum hot pressed at 1500°C and 1600°C after vacuum hot pressing at 1500–1600°C (Figure 6), confirmed presence of MoSi₂ and SiC only.

The light optical photomicrographs of hot pressed MoSi₂-SiC under unetched (Figure 7) and etched conditions (Figure 8) revealed presence of two phases, namely, MoSi₂ and SiC. The uniform distribution of SiC particles in MoSi₂ matrix was confirmed. The grain size in the composite has been found to be between 15 and 20 microns.

The room temperature physical, mechanical, and thermal properties of MoSi₂-SiC composite were evaluated. The flexural strength, compressive strength, modulus of elasticity, hardness, and coefficient of expansion data are given in Table 1. The fracture in flexural test has been found to have taken place in a mixture of intergranular and transgranular mode. Elastic modulus was found to be 410 GPa which is in between individual elastic modulus of MoSi₂ and SiC and in general obeys rule of mixture (ROM). It was inferred that blending of ingredients and processing temperature have significant influence on final properties achieved in the composite. Microhardness measured on individual phases and found to be about 11 GPa on MoSi₂ and about 12.7 GPa on SiC particles. Bulk hardness on the composite was found to be about 11.5 GPa which was in agreement with values approximated by ROM from individual hardness of MoSi₂ and SiC. Flexural and compressive strengths are found to be on higher side than predictable by ROM. This was attributed to better particle-particle bonding and uniform distribution of SiC particle in MoSi₂ matrix. Machinability study gave confidence that critical shapes including through holes could be made by EDM. Study evaluating high temperature resistance of this composite at 1400°C indicated initial weight gain of 1.12 mg/cm² for 10 hrs exposure. However, beyond 10 hrs of exposure at 1400°C, it was almost stable (Figure 9). Few MoSi₂-SiC composite components machined through EDM are shown in Figure 10. Plasma arc jet test under argon and argon +

TABLE 1: Physical/mechanical/thermal properties (RT) of MoSi₂-SiC composite (measured) with respect to pure MoSi₂ and SiC (literature) [13].

Properties	MoSi ₂ -20v% SiC (measured)	MoSi ₂ (literature)	SiC (literature)
Relative density (%TD)	98.8	100	100
Vicker's hardness (GPa)	11.5	11*	12.7*
Mod of elasticity (GPa)	410	395	462
Flexural strength (MPa)	338	257	468
Compressive strength (MPa)	2710	2600	3500
Co-eff of th. exp. (/°C)	6.4×10^{-6}	7.79×10^{-6}	4.63×10^{-6}

* Measured on individual particles.

oxygen environment carried out (Figure 11) indicated that composite had excellent oxidation resistant property and was able to withstand high heat flux (250 W/cm²) under the above environmental conditions. The cross-section of the component subjected to high temperature oxidation was polished and observation under optical microscope confirmed retention of contour. Microstructure was stable with little grain coarsening (Figure 12).

5. Conclusion

(1) MoSi₂-SiC composite has been consolidated by vacuum hot pressing of MM blend of Mo, Si, and SiC particulates between 1500 and 1600°C. A temperature of 1500°C and pressure of 22 MPa are found to be adequate to achieve composite having near theoretical density of 98.5%.

(2) Mechanical properties at RT are found to be greater compared to estimated values due to better bonding and uniform distribution of reinforced particles.

(3) Machining to desired shapes could be achieved through EDM process.

(4) The composite has excellent high temperature oxidation resistance properties. It successfully withstood high heat flux (250 W/cm²) under oxidizing atmosphere, thus proving potential for use in high temperature oxidizing atmosphere.

Acknowledgment

The authors wish to express their sincere gratitude to Shri P. S. Veeraraghavan, Director, VSSC, for giving permission to publish this paper.

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