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Impact Fracture and Shear Strength Characteristics on Interfacial Reaction Layer of $Nb/MoSi_2$ Laminate Composites

Sang-Pill Lee*, Han-Ki Yoon** and Won-Jo Park***

*Institute of Advanced Energy, Kyoto University, Gokasho, Uji, 611-0011, Japan

**Dept. of Mechanical Engineering, Dong-Eui University, 24, Gaya-Dong, Pusanjin-Gu, Pusan, Korea

***Dept. of Mechanical and Ship Engineering, Gyeongsang National University, 445, In-Pyeng Dong, Tongyeong, Gyeongnam, Korea

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ABSTRACT: *The present study dealt with the relationships among the interfacial shear strength, the thickness of interfacial reaction layer and the impact value of $Nb/MoSi_2$ laminate composites. In addition, the tensile test was conducted to evaluate the fracture strain of $Nb/MoSi_2$ laminate composites. To change the thickness of the reaction layer, $Nb/MoSi_2$ laminate composites alternating sintered $MoSi_2$ layers and Nb foils were fabricated as the parameter of hot press temperature. It has been found that the growth of the reaction layer increases the interfacial shear strength and decreases the impact value by localizing a plastic deformation of Nb foil. There also exist appropriate shear strength and the thickness of the reaction layer, which are capable of maximizing the fracture energy of $Nb/MoSi_2$.*

KEY WORDS: $Nb/MoSi_2$ Laminate Composites, Impact Properties, Shear Strength, Interfacial Reaction Layer

1. Introduction

Molybdenum disilicide ($MoSi_2$) is considered to be an attractive material for high temperature uses such as future gas turbines and hypersonic engines, because it has excellent oxidation resistance, thermodynamical compatibility with many kinds of reinforcements, and lower density than conventional superalloys.(1,2) However, for practical applications of $MoSi_2$, several drawbacks need to be resolved, such its pest behavior, the insufficient fracture toughness at the room temperature and the reduced strength at temperature over 1200℃. Recently, in order to improve the damage tolerance of $MoSi_2$, most of studies are focused on composite process by the incorporation of ductile or brittle reinforcements.(3-7) In particular,. the addition of ductile phases like Nb or Ta represented a sufficient improvement in the fracture toughness of monolithic $MoSi_2$ material.(8-10) The previous study also showed that lamination of $MoSi_2$ powder with Nb foil dramatically improved the impact fracture energy of $MoSi_2$ material.(11,12) However, the interfacial reaction layer formed during fabricating process was a deteriorative factor to decrease the impact value of $Nb/MoSi_2$ laminate composites, since the excessive interfacial reaction extremely restrained the plastic deformation of Nb foil. Therefore, for the optimization of $Nb/MoSi_2$ laminate composites, it is desirable to investigate the appropriate thickness of the interfacial reaction layer associated with the bonding strength. Especially, a proper bonding strength, which is capable of maximizing the fracture energy of Nb foil must be found, in order to increase the fracture energy of $Nb/MoSi_2$ laminate composites.

The purpose of the present work is to investigate the relationships among the thickness of interfacial reaction layer, the interfacial shear strength and the impact value in $Nb/MoSi_2$ laminate composites, based on the examination for its fracture strain and the Nb fracture profile. In addition, the interfacial microstructure and the fracture surface of $Nb/MoSi_2$ laminate composites is analyzed by EPMA (Electron probe microanalyzer) and SEM (Scanning electron microspcopy).

2. Experimental Procedures

2.1 Fabrication of laminate composites

The $MoSi_2$ sheet and the Nb foil were used to fabricate $Nb/MoSi_2$ laminate composites. The $MoSi_2$ sheet was sintered at 1773K under 30MPa for 3.6 ks, using a commercial $MoSi_2$ powder with an average particle size of 2.8μm. The relative density of $MoSi_2$ sheet was 96 %. The thickness of $MoSi_2$ sheets used in this experiment was 1.44 mm and 3.0 mm, respectively. The thickness of 99.99 % Nb foil induced in this laminate system was 0.2 mm and 0.5 mm, respectively. By alternating five layers of $MoSi_2$ sheet (1.44 mm thick) with four layers of Nb foil (0.2 mm thick), and then hot pressing in a graphite mould, $Nb/MoSi_2$ laminate composites for the impact test were fabricated. $Nb/MoSi_2$ laminate composites for the tensile test, which was composed of two layers of $MoSi_2$ sheet and one layer of Nb foil, were also fabricated Table 1 shows fabricating conditions of $Nb/MoSi_2$ laminate composites. The hot press temperature was selected as the main parameter for the diffusion bonding between $MoSi_2$ sheet and Nb foil. In order to evaluate the interfacial shear strength between $MoSi_2$ and Nb, Nb/$MoSi_2$ laminate composites, which comprised two layers of $MoSi_2$ sheet (3.0 mm thick) and one layer of Nb foil (0.5 mm thick), were produced by the fabricating condition shown in Table 1.

Table 1 Fabricating conditions of Nb/$MoSi_2$ laminate composites.

Volume fraction of Nb sheet	(%)	10
Fabricating temperature	(K)	1473, 1523, 1573, 1623, 1773
Fabricating pressure	(MPa)	30
Fabricating time	(ks)	3.6
Vacuum pressure	(Pa)	1.33×10^{-2}

2.2 Evaluation of impact, shear and tensile properties

Impact properties of laminate composites were evaluated at room temperature by an instrumented Charpy impact test machine. Fig. 1 shows the structure of laminate composites and the dimensions of an impact test specimen. The U-shaped notch in the impact specimen was machined by EDM(Electric Discharge machining). The impact test was carried out on the flat wise specimen, in which impact load was normal to the lamination.. The test velocity and the span length of the specimen were 3.3 m/sec and 40 mm, respectively.

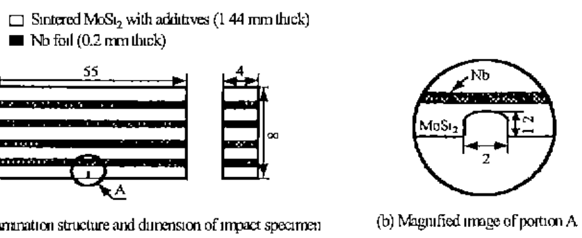

Fig. 1 Structure of laminate composites and dimension of the impact test specimen

The shear test and the tensile test for laminate composites were conducted at room temperature using the tensile test machine. The test velocity was 0.1 mm/min. The interfacial shear strength between MoSi2 and Nb was determined by a plunger method. The specimen was fixed in the frame of test machine, and then a shear force was applied by the plunger, which was mounted on the upper of load cell. In this work, the shear strength is calculated by dividing the fracture load into the fracture area of the specimen. The dimensions of the shear specimen were 4.0×6.5×5.0 mm3. Tensile properties of laminate composites were evaluated by the double notched specimen, which machined U-shaped notch in both sides of the specimen. The dimensions of the tensile specimen were 3.0×4.0×50.0 mm3. The gauge length of the tensile specimen was 10 mm. The width and the depth of a U-shaped notch were 1.0 mm and 1.2 mm, respectively.

3. Results

3.1 Interfacial microstructures of laminate composites

Microstructure constituents of the interfacial reaction zone between Nb and $MoSi_2$ were analyzed with the JEOL JXA-8900RL WD/ED Combined Microanalyzer. In particular, the thickness of the reaction layer was estimated by WDS (wave Dispersive Spectrometer) line analysis. Fig. 2 represents the interfacial microstructure and the WDS analysis result of Nb/$MoSi_2$ laminate composites fabricated at 1773 K. It was clearly found that reaction products such as $(Nb, Mo)Si_2$, $(Nb, Mo)_5Si_3$, Nb_5Si_3 formed at the interfacial region. The line analysis profile also shows that Si diffused far deeper into Nb region than Mo, due to the high diffusion rate of Si relative to Mo. The effect of fabricating temperatures on the thickness of interfacial reaction layer of Nb/$MoSi_2$ laminate composites is shown in Table 2. The thickness of reaction layer between $MoSi_2$ and Nb was defined as that region in which the composition of Nb was between 0 and 100 %, based on the result of the WDS line analysis (See Fig. 2). The reaction layer increased with increasing the fabricating temperature. The thickness of reaction layer created at 1773 K was about 17.5 μm, being about six times compared to that of 1473 K.

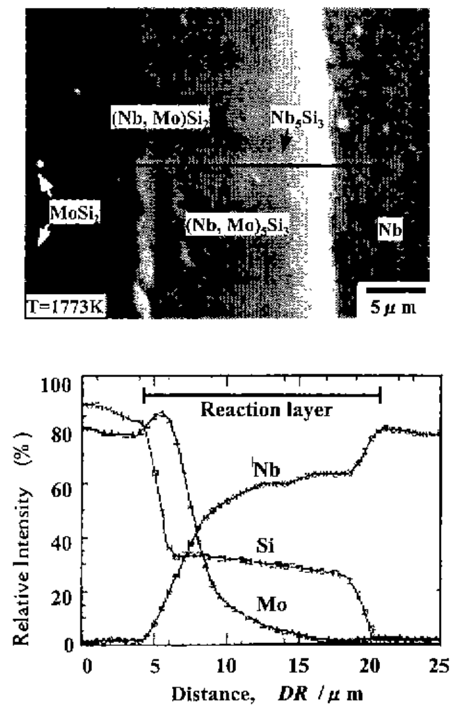

Fig. 2 SEM observation and WDS analysis for interfacial regions of Nb/$MoSi_2$ laminate composites fabricated at 1773K

Table 2 Impact properties and thickness of interfacial reaction layer of Nb/$MoSi_2$ laminate composites depending on fabricating temperatures

Fabricating temperature (K)	Thickness of reaction layer (μm)	Impact load (N)	Fracture displacement (mm)	Crack initiation energy (J)	Crack propagation energy (J)
1473	3.0	439.0	2.41	0.08	0.31
1523	5.0	474.8	2.51	0.15	0.45
1573	7.5	470.0	2.10	0.13	0.32
1623	9.5	461.4	2.03	0.11	0.28
1773	17.5	389.9	1.90	0.09	0.24

3.2 Impact fracture properties of laminate composites

The maximum impact load, the fracture displacement and the absorbed impact energy of Nb/$MoSi_2$ laminate composites fabricated at different temperatures are summarized in Table 2. In this table, the absorbed impact energy is divided into the crack initiation energy and the crack propagation energy. The former corresponds to the area under the impact load-displacement curve till the maximum load and the latter refers to this area after the maximum load.(13) The maximum impact load and the fracture displacement of laminate composites represent a peak value at the fabricating temperature of 1523K. The total absorbed impact energy consumed to fracture the impact sample of Nb/$MoSi_2$ laminate composites tends to decrease at a temperature higher than 1523K. In all fabricating temperature, the crack propagation energy is also higher than the crack initiation energy. Fig. 3 shows the effect of fabricating temperature on Charpy impact value of Nb/$MoSi_2$ laminate composites. As a comparison, the impact value of monolithic $MoSi_2$ sintered at 1753 K is also marked in the same figure. The Charpy impact value was determined from the absorbed impact energy calculated with the area under load-displacement curve, divided by the fracture area of impact specimen. The lamination of Nb foil represented a sufficient improvement in impact value with increasing the fabricating temperature. However, impact value of Nb/$MoSi_2$ laminate composites rapidly reduced at a process temperature higher than 1523 K. In detail illustrations, the impact value of the laminate composites fabricated at 1523 K was 23.8 kJ/m^2, compared to 12.4 kJ/m^2 for the composite at 1773 K.

3.3 Shear strength and tensile properties of laminate composites

The effect of fabricating temperature on interfacial shear strength, ultimate tensile strength and fracture strain of Nb/$MoSi_2$ laminate composites is shown in Table 3. The fracture strain was measured from the gauge length of 10 mm. The interfacial shear strength of laminate composites increased with increasing the fabricating temperature. The shear strength of the laminate composites fabricated at 1773 K was 37.0 MPa, increasing to about three times compared to 11.6 MPa for the composite at 1423 K. In addition, the ultimate tensile strength of laminate composites increased with increasing the fabricating temperature, but its fracture strain has a decreasing tendency.

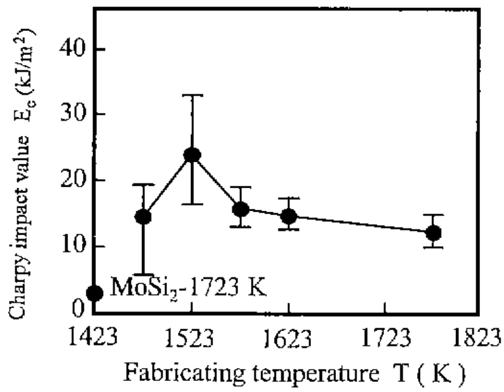

Fig. 3 Effect of fabricating temperature on Charpy impact value of Nb/$MoSi_2$ laminate composites

Table 3 Effect of fabricating temperature on interfacial shear strength, ultimate tensile strength and fracture strain of Nb/$MoSi_2$ laminate composites.

Fabricating temperature (K)	Shear strength (MPa)	Tensile strength (MPa)	Fracture strain (%)
1473	11.6	63 8	6.1
1523	22.4	65.5	5.4
1573	24.6	69.0	4.9
1623	26.2	74.5	3.4
1773	37.0	85.3	2.5

4. Discussion

Fig. 4 shows the shear strength and the fracture strain of laminate composites depending on the thickness of reaction layer. The growth of reaction layer increased the shear strength, but resulted in the decrease of the fracture strain. Such an increase of the shear strength is caused by the increase of the bonding area between $MoSi_2$ and Nb, together with the growth of reaction layer. In other words, as seen in the shear fracture surfaces of Fig. 5, the laminate composite fabricated at 1250 ℃ represents $MoSi_2$ and (Nb, Mo)$_5$Si$_3$ phases, whereas the laminate composite at 1500 ℃ dominantly displays interfacial products such as (Nb, Mo)$_5$Si$_3$ and Nb_5Si_3. Especially, the laminate composite at 1250 ℃ exhibits a large amount of $MoSi_2$ portion relative to that at 1500

℃. It can be considered from these results that the interfacial shear strength decreases with the reduction of net area for the interfacial bonding, since it is easy to create some voids at the bonding interface owing to an insufficient interfacial reaction in the low fabricating temperature (1250 ℃). In addition, the reduction of the fracture strain is because the plastic deformation of Nb foil is localized by the increase of the shear strength. As shown in Fig. 6, laminate composites have different fracture modes for weak and strong interfacial bonding. The laminate composite fabricated at 1523 K dominantly exhibits an obvious plastic deformation of Nb foil and an extensive interfacial delamination (Fig. 6(a)) On the contrary, the laminate composite at 1773 K displays a straight crack propagation in front of notch and has a smaller Nb foil deformation relative to that at 1523 K, due to the increase of the shear strength (Fig. 6(b)). It was obvious from tensile fracture profiles of laminate composites that the shear strength increase, which was associated with the thickness of reaction layer, led to a decrease in the plastic deformation of Nb foil. Such shear strength change affects the impact value of laminate composites.

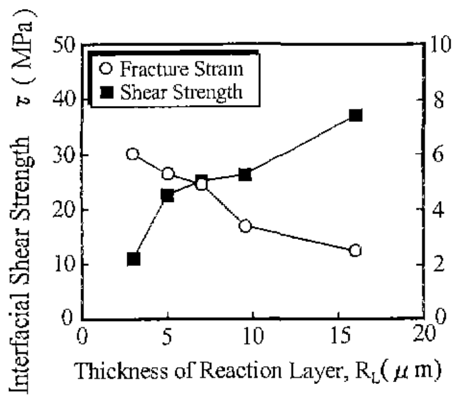

Fig. 4 Interfacial shear strength and fracture strain of Nb/$MoSi_2$ laminate composites depending on the thickness of reaction layer

Fig. 7 shows the effect of the shear strength and the Charpy impact value of Nb/$MoSi_2$ laminate composites on the thickness of reaction layer. The impact value of laminate composites represents a peak value at the shear strength of about 22.4 MPa (1523 K). This is because fracture modes of laminate composites, accompanied with the plastic deformation of Nb foil, changes at this shear strength (Fig. 6). When the shear strength is smaller than 22.4 MPa, Nb foils mounted in laminate composites are not fractured due to the shear strength being too low, accompanying with an extensive interfacial delamination. Oppositely, when the shear strength is higher than 22.4 MPa, the localized fracture of Nb foils emerges. Such a different shear strength results from the variation in the impact value of laminate composites. It is obviously found from these results that there is an appropriate shear strength optimizing the fracture energy of Nb foil in Nb/$MoSi_2$ laminate composites. Therefore, in order to improve the impact value of Nb/$MoSi_2$ laminate composites, it is effective for controlling the shear strength, which is associated with the thickness of reaction layer and the fabricating temperature.

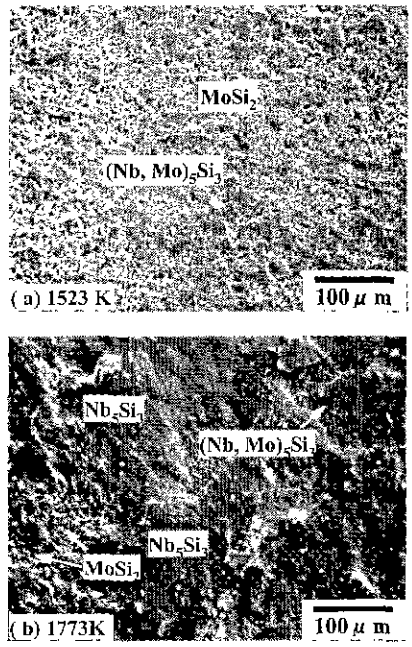

Fig. 5 SEM micrographs on the $MoSi_2$ side in the shear fracture surface of Nb/$MoSi_2$ laminate composites fabricated at different temperatures

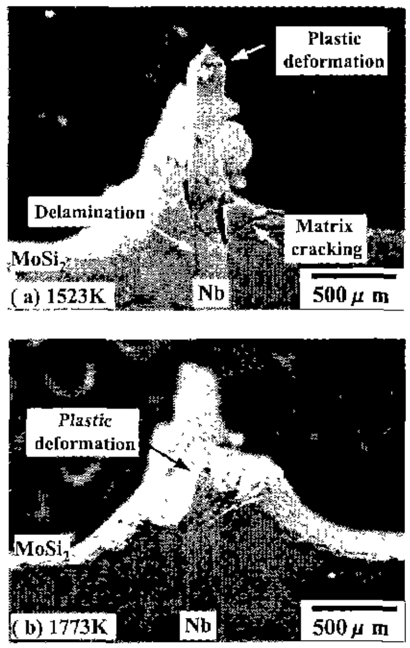

Fig. 6 SEM micrographs on the tensile fracture profile of double notched Nb/$MoSi_2$ laminate composites fabricated at different temperature

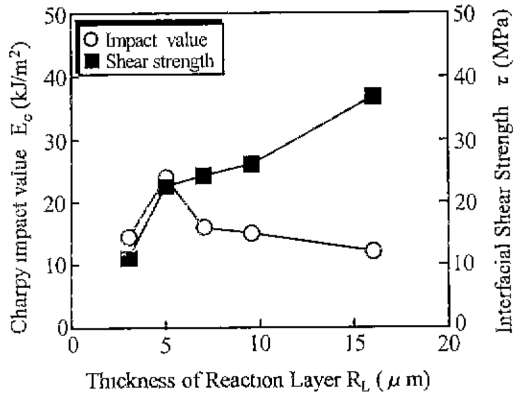

Fig. 7 Interfacial shear strength and the Charpy impact value of $Nb/MoSi_2$ laminate composites on the thickness of reaction layer

5. Conclusion

(1) $Nb/MoSi_2$ laminate composites represented the maximum impact value at the reaction layer thickness of about 5.0 μm (Fabricating temperature of 1523K) in which the interfacial shear strength was about 22.4 MPa.

(2) The thickness of interfacial reaction layer between $MoSi_2$ and Nb increased with increasing the fabricating temperature. Moreover, the growth of reaction layer resulted in the increase of the interfacial shear strength.

(3) The increase of the shear strength localized the plastic deformation of Nb foil and hence decreased the impact value of $Nb/MoSi_2$ laminate composites.

(4) An appropriate thickness of interfacial reaction layer promoting the maximum fracture energy of Nb foil must be considered to optimize the impact value of $Nb/MoSi_2$ laminate composites.

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