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Impact Fracture Characteristics on Fabricating Process of Nb/$MoSi_2$ Laminate Composite (I)

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Nb/$MoSi_2$ laminate composites have been successfully fabricated by hot pressing in a graphite mould. Lamination of Nb foil and $MoSi_2$ layer showed a sufficient improvement in the absorbed impact energy compared to that of monolithic $MoSi_2$ material. The impact value of Nb/$MoSi_2$ laminate composites obviously is reduced when sintered at temperatures higher than 1523K, even if the composite density contributing to impact load increased along with fabricating temperatures. Impact value of laminate composites was also drastically decreased with the growth of reaction layer after the heat treatment. However, it was effective to increase the pressure at the same sintering temperature for the improvement of the impact value.

Key Words : Nb/$MoSi_2$ Laminate Composite, Fabricating Process, Impact Fracture Characteristics, Reaction Layer

1. Introduction

Molybdenum disilicide($MoSi_2$) is considered to be an attractive candidate for future gas turbine and high performance engines in aerospace vehicles as well as various industrial applications. $MoSi_2$ has an excellent oxidation resistance compared to the most of other intermetallic compounds at the elevated temperature above 1273K and its density(6.3 g/cm^3) is lower than that of nickel based superalloy. Furthermore, $MoSi_2$ has a considerable potential for the improvement of mechanical properties due to its excellent chemical stability with many kinds of ceramic reinforcements(Meschter and Schwartx, 1989). However, practical applications of $MoSi_2$ have still been restricted by its pest oxidation behavior, the insufficient fracture toughness at room temperature and the reduced strength at higher temperature than 1473K. Several attempts have been focused on composite process in order to improve the critical damage tolerance of MoSi2. Recent works on the addition of ceramic reinforcements including SiC, ZrO_2, Al_2O_3, TiB_2 and TiC to $MoSi_2$ have shown small improvements in the fracture toughness at room temperature(Pan et al., 1998 ; Yi and Li, 1999 ; Newman et al., 1999 ; Tiwari et al., 1992 ; Yang et al., 1989). It has been also found that $MoSi_2$ based composites containing Nb short fiber shows a sufficient improvement in static fracture energy compared to that of monolithic $MoSi_2$(Alman and Stoloff, 1995). However, such a microstructural variation of $MoSi_2$ material has shown only a limited improvement effect in the fracture toughness and the fracture energy. Therefore, it is necessary to improve the damage tolerance of $MoSi_2$ through structural configurations. Lamination strategy is considered as another way to improve the fracture energy at room temperature, because it can delay the propagation crack through plastic deformation of component material and interfacial delamination(Yoon et al., 1995 ; Choi and Kinloch, 1998). In order to apply

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Table 1 Fabricating conditions for Nb/$MoSi_2$ laminate composites

Volume fraction of Nb foil (%)	10
Consolidation temperature (K)	1473,1523,1573,1623,1773
Consolidation pressure (MPa)	20, 30, 40
Consolidation time (ks)	0.9, 1.8, 3.6
Vacuum pressure (Pa)	1.33×10^{-2}

$MoSi_2$ as high temperature structures, it is required to estimate impact properties under dynamic load as well as fracture toughness at room temperature. Unfortunately, there have been few studies to investigate impact properties of $MoSi_2$ based composites.

The primary purpose of the present work is to investigate the effect of impact fracture characteristics on the fabricating condition for Nb/$MoSi_2$ laminate composites. The secondary goal is to estimate the influence of interfacial reaction layer between $MoSi_2$ and Nb on the impact properties of laminate composites after heat treat ment at different temperatures. In addition, the interfacial reaction product between $MoSi_2$ and Nb is analyzed by EPMA, and then the fracture mechanism depending on the growth of interfacial reaction layer is discussed.

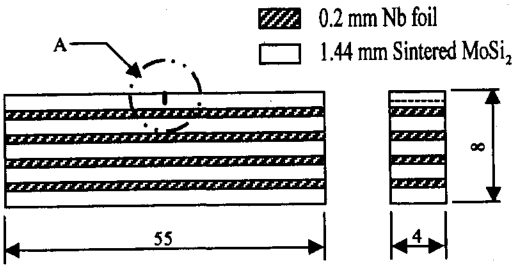
2. Experimental Details

2.1 Fabrication of Nb/$MoSi_2$ laminate composites

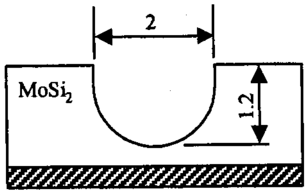
By alternating $MoSi_2$ powder layer with four layers of Nb foil, and then hot pressing in a graphite mould, Nb/$MoSi_2$ laminate composites were fabricated. The matrix material in this experiment was a commercial $MoSi_2$ powder supplied by Japan New Metal Corporation with an average particle size of 2.8μm. The thickness of 99.99% Nb foil in this system was 0.2mm. Table 1 shows the fabricating conditions for Nb/$MoSi_2$ laminate composites. The dimension of the as-pressed laminate composites was $8\times20\times80mm^3$.

2.2 Instrumented Charpy impact test

Impact properties of monolithic $MoSi_2$ material and Nb/$MoSi_2$ laminate composites were evaluated at the room temperature by an instrumented Charpy impact test machine. The test velocity and the span length of the specimen were 3.3m/sec and 40mm, respectively. Figure 1 shows the geometry and the dimension of impact specimen. The U-shaped notch was introduced with EDM. The impact test was carried out on a flat wise specimen, and each load-displacement curve was directly monitored by the oscilloscope. Charpy impact value(Ec) was determined from the absorbed impact energy calculated from the area under load-displacement curve, divided by the fracture area of the notch of specimen ($4.0\times6.8\times$ mm^2).



(a) Overview of impact specimen



(b) Enlargement of portion A

Fig. 1 Geometry and dimension of the impact specimen

2.3 Interfacial reaction zone and fracture mechanism analysis

The microstructure constituent of the interfacial reaction zone between Nb foil and $MoSi_2$ was analyzed with the JEOL JXA-8900RL WD/ED Combined Microanalyzer. The thickness and the composition of the reaction region were estimated by WDS(Wave dispersive spectrometer) line analysis and semi-quantitative analysis processes. Moreover, the thickness of reaction layer produced by the heat treatment was measured from the WDS line analysis profile and then the

Table 2 Sintered density and impact results of Nb/MoSi2 laminate composite fabricated at various conditions

Consolidation temperature (K)	Consolidation time (ks)	Consolidation pressure (MPa)	Average density (mg/m^3)	Relative density (%)	Impact load (N)	Displacement (mm)
1423	3.6	3.0	5.18	79	405.5	2.31
1523			5.65	86	469.9	2.75
1573			5.91	90	471.0	2.02
1623			5.97	91	474.5	1.87
1773			6.17	94	324.0	1.43
1623	0.9	30	5.61	86	445.2	1.81
	1.8		5.95	91	466.2	1.85
	3.6		5.96	91	474.5	1.87
1623	3.6	20	5.85	89	423.9	1.92
		30	5.95	91	474.5	1.87
		40	6.07	93	481.0	1.87

effect of its thickness on the impact value of Nb/MoSi$_2$ laminate composites fabricated at 1773K was investigated. The heat treatment was conducted at 1873K and 1973K for 18ks. In addition, the plastic deformation of Nb foil and the interfacial delamination were macroscopically observed to explain the variation of absorbed impact energy.

3. Results and Discussion

3.1 Density depending on process conditions

Sintered densities of Nb/MoSi$_2$ laminate composites fabricated with a variety of process conditions are shown in Table 2. The theoretical density of laminate composites, 6.53mg/m^3, was calculated with the rule of mixture. The density of laminate composites increased along with consolidation temperature, consolidation pressure and consolidation time because of the high densitification of MoSi$_2$ powder. It can be seen that the most dense laminate composite is one fabricated at 1773K. Therefore, it is regarded that the optimum fabrication condition for Nb/MoSi$_2$ laminate composites may be 1773K, 30MPa and 3.6ks, considering sintered density and matrix strengthening.

3.2 Interfacial reaction zone

The interfacial microstructure of Nb/MoSi$_2$ laminate composites fabricated at 1773K and 30MPa for 3.6ks is shown in Fig. 2. It was found that two obvious phases differing from composi-

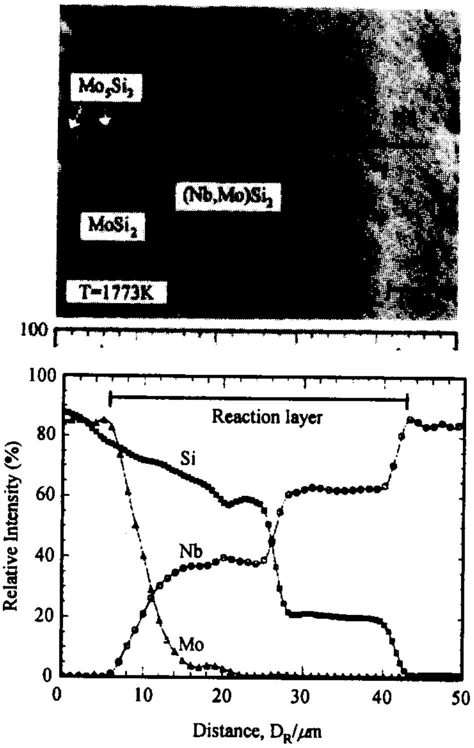


Fig. 2 SEM observation and WDS analysis for interfacial reaction layer of Nb/MoSi$_2$ laminate composites fabricated at 1773K

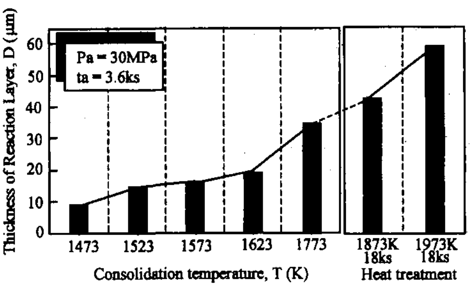


Fig. 3 Effect of consolidation temperature and heat treatment condition on the growth of the reaction layer for Nb/MoSi$_2$ laminate composites

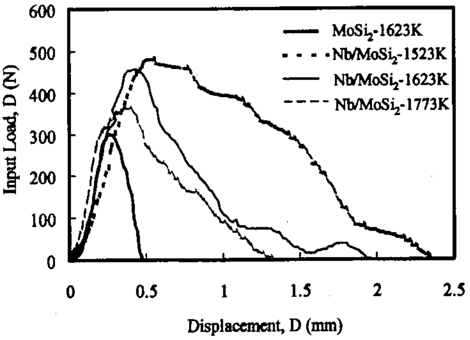


Fig. 4 Impact behavior of monolithic MoSi$_2$ material and Nb/MoSi$_2$ laminate composites depending on consolidation temperatures

tion proportion of Mo, Si and Nb formed at the interfacial region. The line analysis profile also shows that Si diffused far deeper into the Nb region than Mo. This is due to the high diffusion rate of Si relative to Mo at 1623K. The diffusion coefficient of Si and Mo in Nb was 6.16×10^{-13} and $8.37\times10^{-18}m^2/s$, respectively, which was calculated from the available literature data[10]. Interfacial reaction products for Nb/MoSi$_2$ laminate composites fabricated at this temperature were dominated by the diffusion of Si into Nb region, which resulted in forming other intermetallic compounds adjacent to Nb, such as (Nb, Mo)Si$_2$ and Nb5Si$_3$.

The Effect of consolidation temperature and heat treatment condition on the thickness of the reaction layer between Nb and MoSi$_2$ in Nb/MoSi$_2$ laminate composites is shown in Fig. 3. From the results of the EPMA-WDS line analysis, the thickness of the reaction layer defined as that region in which the composition of Nb was between 0 and 100%. The thickness of layer increased along with the consolidation temperature. The reaction layer created at 1773K was about 35m in thickness, being about four times compared to that of 1473K. In addition, its thickness increased to about 60m after the composite was heated at 1973K for 18ks. This is believed due to the high diffusion rate of Si at a higher fabricating temperature.

3.3 Impact behavior and absorbed impact energy

Figure 4 shows the effect of consolidation temperature on the impact behavior for Nb/MoSi$_2$ laminate composites. As a comparison, the impact behavior of the monolithic MoSi$_2$ material sintered at 1623K is shown in this figure. The monolithic MoSi$_2$ material displays a typical brittle behavior, that is, impact load catastrophically drops down at the maximum load. By contrast, the fracture behavior of laminate composites exhibits a stable crack propagation stage beyond the maximum load. Especially, this ductile behavior of laminate composites was obviously revealed in the case of lower consolidation temperature, together with the reaction layer reduction. The maximum impact load, the fracture displacement and the sintered density for Nb/MoSi$_2$ laminate composites fabricated at different conditions are summarized in Table 2. The lamination of Nb foil with MoSi$_2$ powder shows an obvious improvement in the maximum load and the fracture displacement, comparing to those of the monolithic MoSi$_2$ fabricated at 1623K. The maximum impact load increased to a peak value at 1623K and went dramatically down at 1773K. The fracture displacement of laminate composites has a decreasing tendency at a temperature higher than 1523K. Therefore, it can be found from Fig. 3 and Table 2 that the maximum load of Nb/MoSi$_2$ laminate composites depends on the sintered density and the thickness of the interfacial reaction layer, whereas the fracture displacement is mainly dominated by the reaction layer constraining the plastic deformation of Nb foil.

Figure 5 shows the absorbed impact energy for

Nb/MoSi$_2$ laminate composites fabricated at different temperatures. The absorbed impact energy is divided into the crack initiation energy and the crack propagation energy. The former corresponds to the area under load-displacement curve till maximum load and the latter is one behind the maximum load. By laminating with Nb foil, the crack initiation energy and the crack propagation energy increased more than three times and seven times, respectively, comparing to those of monolithic MoSi$_2$ sintered at the same temperature(1623K). In laminate composites, the crack initiation energy has an analogous level with the increase of consolidation temperature, but the crack propagation energy reduces remarkably when fabricated at a process temperature higher than 1523K. This is because laminate composites fabricated at 1523K has a larger impact load and fracture displacement compared to those of laminate composites at 1773K in the crack propagation behavior as shown in Fig. 3, even if t displacement corresponding to the maximum lo has the same level. It can be considered from tl figure that the deformation behavior of Nb f and the interfacial delamination mainly contr utes to the crack propagation energy of Nb/Mo laminate composites.

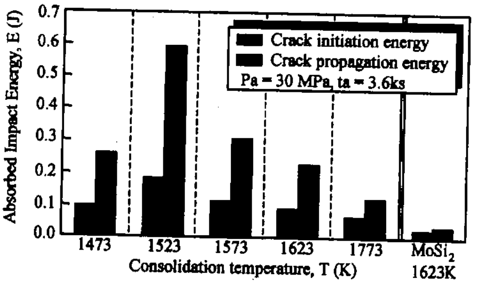


Fig. 5 Absorbed impact energy for monolithic MoSi2 material and Nb/MoSi$_2$ laminate composites depending on consolidation temperatures

3.4 Charpy impact value of Nb/MoSi$_2$ lam nate composites

Figure 6 represents the effects of consolidati temperature and heat treatment condition on t Charpy impact value of Nb/MoSi$_2$ laminate co posites. The impact value increased more th five times by laminating Nb foil, comparing that of monolithic MoSi$_2$ material sintered 1623K. However, impact values of Nb/Mo laminate composites rapidly reduced at a proce temperature higher than 1523K.

It could be seen that the impact values of lam nate composites were independent on composi density shown in Table 2, even if the densi increased along with consolidation temperature In detail illustrations, the impact value of th laminate composites fabricated at 1773K was 7kJ • m^{-2}, decreasing to a one third compared t 25.2kJ • m^{-2} for the composite at 1523K. It ha been found that the variation of the impact valu was influenced by the growth of the interfaci reaction layer related to the fabricating tempera ture, since this layer constrains the deformatio behavior of Nb foil and the interfacial delamina tion and results in the reduction of the fractur

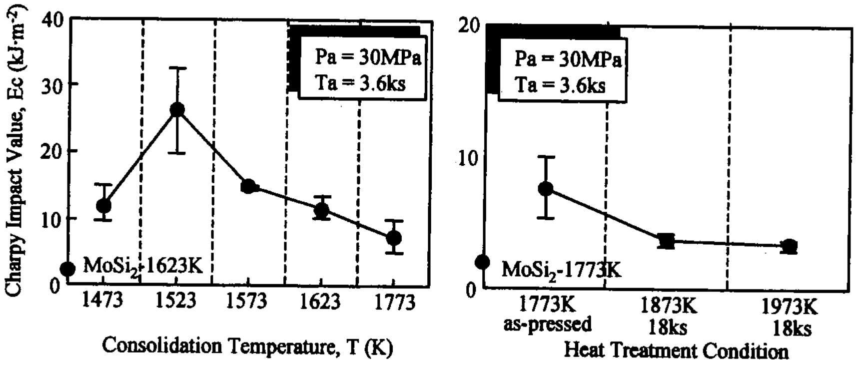
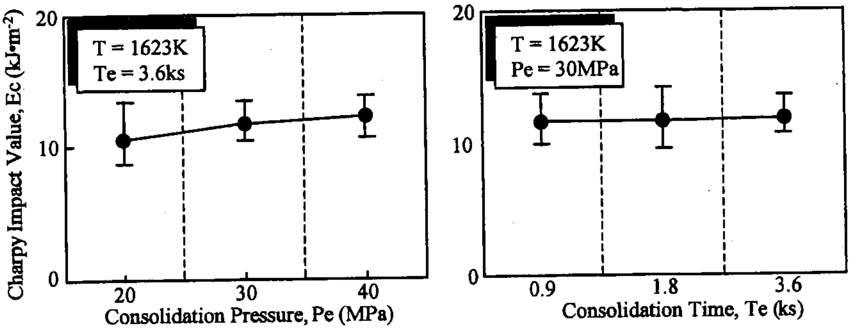


Fig. 6 Effect of consolidation temperature and heat treatment condition on the Charpy impact value of Nb/MoSi$_2$ laminate composites



g. 7 Effect of consolidation pressure and consolidation time on the Charpy impact value of Nb/MoSi$_2$ laminate composites (Consolidation temperature:1623K)

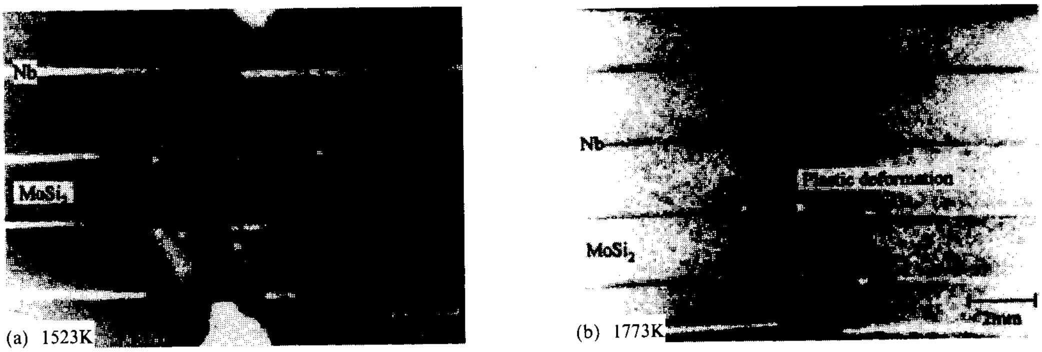


Fig. 8 Typical fractographs of Nb/MoSi$_2$ laminate composites fabricated at 1523K and 1773K

splacement for laminate composites (see Table . The effect of reaction layer thickness on the pact value obviously reveals in laminate com- sites heated at high temperatures. The impact lue of the laminate composites fabricated at 73K significantly decreased after the heat treat- ent and displayed the same level as that of onolithic matrix material. Therefore, it is essen- al to suppress the growth of the reaction layer etween MoSi$_2$ and Nb foil.

The Effect of consolidation pressure and con- lidation time on the Charpy impact value of b/MoSi$_2$ laminate composites is shown in Fig. . The Impact values of laminate composites have n increasing tendency along with consolidation ressure, but its impact value according to consol- lation time is nearly constant as about 12kJ • $^{-2}$. This is because the sintered density of the minate composites contributing to the maxi- um load increases, as shown in Table 2.

3.5 Fracture mechanism

From macroscopic observation of Nb/MoSi$_2$ laminate composites fabricated at 1523K and 1773K as displayed in Fig. 8, it can be seen that both the brittle fracture of MoSi$_2$ layer and the ductile fracture of Nb foil coexist in fracture profiles of laminate composites. The laminate composites fabricated at 1523K dominantly exhibits the plastic deformation of Nb foil and the interfacial delamination, whereas the laminate composites at 1773K displays the straight crack propagation in front of notch and have a smaller deformation of Nb foil compared to that at 1523K. Such a different fracture mode is resulted from the reaction layer growth according to process temperatures, since the interfacial constraint reduces the plastic deformation of Nb foil, which leads to the interfacial delamination. In addition, it can be illustrated from the view point of fracture profiles that the difference of absorbed impact energy is related to the extent of Nb deformation and interfacial delamination depending on consolidation temperatures. Therefore, these results suggest that the suppression of interfacial reaction layer is very effective to improve impact properties.

4. Summary

The lamination of Nb foil with MoSi$_2$ powder was an excellent strategy to improve impact properties for monolithic MoSi$_2$, but represented an anisotropic properties in the impact value associated with process temperatures. The optimum processing temperature of Nb/MoSi$_2$ laminate composites can be selected to 1773K from the view point of the sintered density, but the interfacial reaction layer obtained at this temperature was a detrimental factor for deteriorate the impact value. It can be stated that a critical process condition to increase the impact value of Nb/MoSi$_2$ laminate composites was 1523K, 30MPa and 3.6ks in this study, considering the ability of the plastic deformation of Nb foil and the interfacial delamination. In addition, it is effective in the improvement of the impact value to increase the consolidation pressure at the same process temperature.

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