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Fabrication of Nb/$MoSi_2$ laminate composites and their thermal shock properties

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This paper dealt with the characterization of Nb/$MoSi_2$ laminate composites suffered from high temperature thermal shock history. Nb/$MoSi_2$ laminate composites consisted of single $MoSi_2$ matrix layers alternated with four layers of Nb foil. The $MoSi_2$ matrix region of Nb/$MoSi_2$ laminate composites was consolidated by a vacuum hot pressing, using a commercial powder with an average size of 2.8 μm. The flexural strength of Nb/$MoSi_2$ laminate composites has been investigated at the thermal shock temperature difference up to 600 °C. Nb/$MoSi_2$ laminate composites represented a dense morphology in the matrix region, accompanying the creation of two types of reaction layer at the interfacial region. The flexural strength of Nb/$MoSi_2$ laminate composites was greatly affected by the thermal shock test temperature. Nb/$MoSi_2$ laminate composites represented a rapid reduction of flexural strength at the thermal shock temperature difference higher than 300 °C.

Key words: Nb/$MoSi_2$ laminate composites, microstructure, sintered density, flexural strength, thermal shock temperature.

Introduction

Structural intermetallic materials have been extensively studied for a new approach to the production of turbojet and hypersonic engine system in aerospace vehicles. Molybdenum disilicide ($MoSi_2$) can be recognized as a promising material for high temperature components of advanced power generation and aircraft engine system, such as heat exchanger, turbine blade, vane and combustor. [1,2] $MoSi_2$ materials offer an excellent oxidation resistance up to the temperature around 1700 °C, a good mechanical properties, a lower density than nickel based superalloys. Especially, $MoSi_2$ materials have attractive potentials in the composite process for the strengthening of their microstructures, due to the thermodynamical compatibility with many kinds of ceramic reinforcements. [3,4] However, several drawbacks of $MoSi_2$ materials such as pest behavior, room temperature embrittlement and strength degradation at the elevated temperature still impose a severe limitation on extensive applications. Majority of the research on $MoSi_2$ materials was focused on the improvement of their toughness and fracture energy by the incorporation of ductile refractory metals like Nb or Ta, compared to that of ceramic reinforcements. [5-7] Especially, the lamination technique of $MoSi_2$ with high temperature refractory metals was very effective for the toughness strengthening through the variation of their configuration. [8-10] Previous studies also showed that the lamination of $MoSi_2$ with Nb foil could sufficiently improve the impact value of $MoSi_2$ materials, even if the brittle interfacial compounds were formed at the interfacial region. [11,12] The proper suppression of interfacial reaction was also required for maximizing the fracture energy of Nb/$MoSi_2$ laminate composites, since the interfacial reaction layer constrains the plastic deformation of Nb foil. In order to extend the practical applications of Nb/$MoSi_2$ laminate composites to high temperature components, it is essential to investigate the thermal damage resistance at the various critical atmospheres. Especially, the thermal shock properties of Nb/$MoSi_2$ laminate composites by the variation of operating temperature are important for the stability of thermal structures. Unfortunately, there are few studies for the thermal shock damages of Nb/$MoSi_2$ laminate composites.

This work is aimed to investigate high temperature thermal resistance properties of Nb/$MoSi_2$ laminate composites, in conjunction with the detailed examination of their microstructures and fractured surfaces. Especially, the effect of thermal shock temperature on the mechanical properties of Nb/$MoSi_2$ laminate composites was examined.

Experimental procedures

Nb/$MoSi_2$ laminate composites, which consisted of single $MoSi_2$ matrix layers alternated with four layers of Nb foil, were successfully fabricated at the temperature of 1350 °C, using a hot-press device. The applied pressure and its holding time for the consolidation of $MoSi_2$ matrix were 30 MPa and 1 hr, respectively. The $MoSi_2$ matrix was fabricated, using a commercial powder with an average size of 2.8 μm. In this laminate system, the thickness of commercial Nb foil was constant as 0.2 mm. The volume fraction of Nb foil was also fixed

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as 10%. The dimensions of as-pressed Nb/$MoSi_2$ laminate composites were 20(w) × 80(l) × 8(t) mm^3.

The sintered density of Nb/$MoSi_2$ laminate composites were determined by the Archimedes' method. The microstructure and its chemical composition for the interfacial region of Nb/$MoSi_2$ laminate composites were also analyzed, using a JEOL JXA-8900RL WD/ED Combined Microanalyzer. In particular, the thickness of the interfacial reaction layer between $MoSi_2$ and Nb was estimated by WDS (wavelength dispersive spectrometer) line analysis. The thermal shock test for Nb/$MoSi_2$ laminate composites was performed at the temperature difference range from 100 °C to 600 °C. The thermal shock test system is mainly composed of the heating furnace with driving motor and thermocouple and the control box for the measurement of heating and cooling time. The test sample was heated to the operating temperature for twenty minutes, prior to the dropping into the water. In order to investigate the strength degradation of Nb/$MoSi_2$ laminate composites suffered from the thermal shock, the three point bending tests were carried out at the room temperature. The bending load was applied to the lamination direction. The dimension of bending test samples was 3(w) × 25(l) × 8(t) mm^3. The span length and the crosshead speed for the bending tests were 16 mm and 0.5 mm/min, respectively.

Results and discussion

The resultant densities and room temperature strength of Nb/$MoSi_2$ laminate composites were shown in Table 1. The relative density of this material was expressed as the ratio of experimental density determined by Archimedes' method and theoretical density calculated by the rule of mixture. Nb/$MoSi_2$ laminate composites represented an average density of about 6.13 Mg/m^3, which corresponded to about 94% of theoretical density. This material also possessed a porosity of about 6.1% in the matrix region. Moreover, Nb/$MoSi_2$ laminate composites represented an average flexural strength of about 310 MPa at the room temperature.

Fig. 1 shows the interfacial microstructure of Nb/$MoSi_2$ laminate composites. The WDS line analysis result for the interfacial region of Nb/$MoSi_2$ laminate composites (the black line on the SEM micrograph) is shown in the same figure. The qualitative analysis result for interfacial products is also shown in Table 2. Large amounts of pores were observed at the side of $MoSi_2$ matrix region. It is found from the line analysis profile for the interfacial region that Si diffuses far deeper into Nb region than Mo, due to the high diffusion rate of Si relative to Mo. [10] As a result, two sorts of reaction products such as (Nb, Mo)Si_2 and Nb_5Si_3 obviously created at the interfacial region of Nb/$MoSi_2$ laminate composites, due to the chemical reaction of $MoSi_2$ and Nb. In this composite system, a uniform band type of interfacial reaction layers was entirely formed at the interface between $MoSi_2$ and Nb. Such a thickness of interfacial reaction layer was measured as about 20.0 μm. From the result of the WDS line analysis, the thickness of interfacial reaction layer was defined as that region in which the composition of Nb was between 0 and 100%.

Fig. 2 shows the effect of thermal shock temperature difference on the sintered density of Nb/$MoSi_2$ laminate composites. The porosity of Nb/$MoSi_2$ laminate composites by the variation of thermal shock temperature difference was shown in this figure. Nb/$MoSi_2$ laminate composites represented a slight reduction of sintered density, after maintaining a similar density level to that of as-pressed materials without a thermal shock history up to the thermal shock temperature difference of 300 °C. The

Table 1. Properties of Nb/$MoSi_2$ laminate composites.

Theoretical density (Mg/m^3)	6.46
Sintered density (Mg/m^3)	6.13
Relative density (%)	93.8
Porosity (%)	6.1
Flexural strength (MPa)	310

Table 2. Composition for interfacial products of Nb/$MoSi_2$ laminate composites determined by WDS analysis.

Phase	Mo	Si	Nb
$MoSi_2$	30.6	69.4	0.0
(Nb, Mo)Si_2	0.86	72.2	26.9
Nb_5Si_3	0.0	43.3	56.7

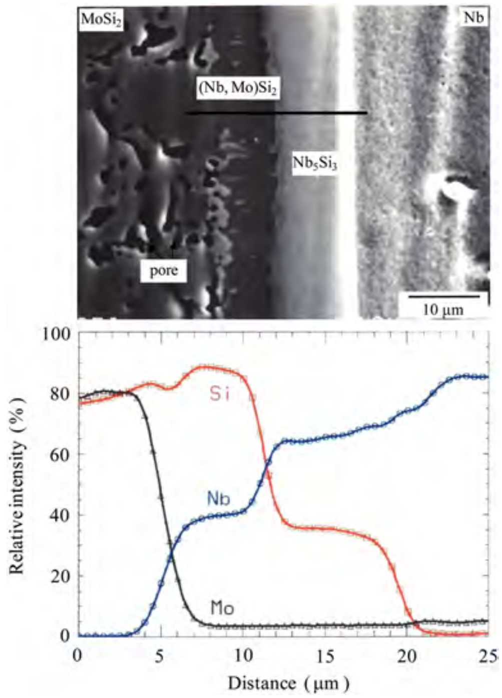

Fig. 1. Interfacial microstructure and WDS analysis results of Nb/$MoSi_2$ laminate composites.

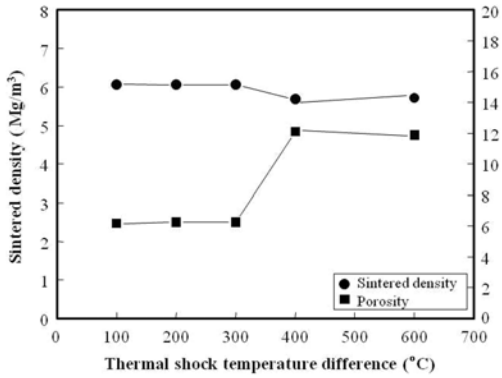

Fig. 2. Effect of thermal shock temperature difference on the sintered density of Nb/$MoSi_2$ laminate composites.

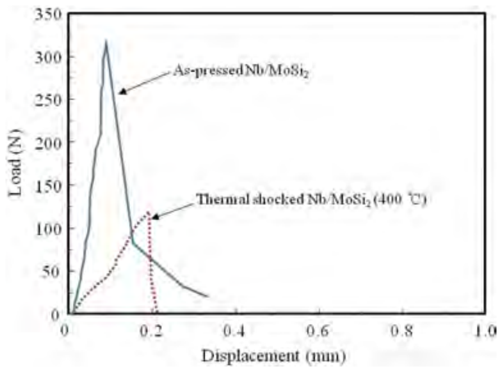

Fig. 3. Representative load-displacement curve of Nb/$MoSi_2$ laminate composites depending on the variation of thermal shock temperature difference.

porosity of Nb/$MoSi_2$ laminate composites was greatly affected by the thermals shock history. In other words, the porosity of Nb/$MoSi_2$ laminate composites tended to increase with the increase of thermal shock temperature difference. Especially, Nb/$MoSi_2$ laminate composites possessed a porosity of about 12% at the thermal shock temperature difference of 400 °C, which corresponded to about 2.0 times of that of as-pressed materials. This is maybe caused by the large formation of matrix pores by the oxidation behavior at the elevated temperatures.

Fig. 3 represents the representative load-displacement curve of Nb/$MoSi_2$ laminate composites depending on the variation of thermal shock temperature difference. Nb/$MoSi_2$ laminate composites displayed a non-catastrophic fracture behavior with stable crack propagation beyond the maximum load. This is probably caused by the variation of the crack propagation path associated with the interaction of interfacial delamination and plastic deformation of Nb layer. However, Nb/$MoSi_2$ laminate composites represented a different fracture behavior, after the thermal shock at the elevated temperature. Nb/$MoSi_2$ laminate composites suffered from the thermal shock history at the temperature difference of 400 °C displayed a great reduction of bending load and displacement.

Fig. 4 shows the effect of thermal shock temperature

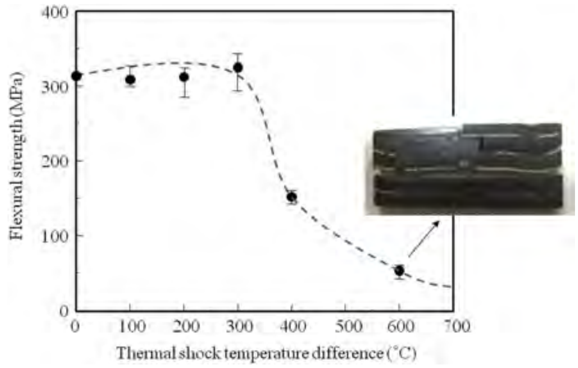

Fig. 4 Effect of thermal shock temperature difference on the flexural strength of Nb/$MoSi_2$ laminate composites.

difference on the flexural strength of Nb/$MoSi_2$ laminate composites. The fracture profile of Nb/$MoSi_2$ laminate composites tested at the thermal shock temperature difference of 600 °C was shown in this figure. Nb/$MoSi_2$ laminate composites maintained a similar strength up to the thermal shock temperature difference of 300 °C. However, the flexural strength of Nb/$MoSi_2$ laminate composites greatly decreased with the increase of thermal shock temperature difference. Especially, Nb/$MoSi_2$ laminate composites possessed a low flexural strength of about 150 MPa at the thermal shock temperature difference of 400 °C. Such a strength level corresponded to about one-half time of that without a thermal shock history. This is related with the interfacial degradation and the increase of porosity in the matrix region. In other words, it is considered that large amount of matrix pores by the thermal shock history lead to the reduction of flexural strength, as shown in Fig. 2. The interfacial degradation of Nb/$MoSi_2$ laminate composites can also decrease the capacity of load before the enough strengthening of strength owing to the extensive delamination between $MoSi_2$ and Nb, as shown in the fractured profile.

Conclusions

Nb/$MoSi_2$ laminate composites possessed a good density of about 6.13 Mg/m^3, accompanying a porosity of about 6.1% and represented a flexural strength of about 310 MPa at the room temperature. Nb/$MoSi_2$ laminate composites created two sorts of reaction layers with the thickness of about 20.0 μm at the interfacial region, due to the chemical reaction of $MoSi_2$ and Nb. The interfacial reaction products were identified as (Nb, Mo)Si_2 and Nb_5Si_3 phases. The density properties of Nb/$MoSi_2$ laminate composites were greatly affected at the thermal shock temperature difference higher than 300 °C. Nb/$MoSi_2$ laminate composites possessed a porosity of about 12% at the thermal shock temperature difference of 400 °C. Such a porosity level corresponded to about 2.0 times of that of as-pressed materials. Nb/$MoSi_2$ laminate composites represented a great reduction of flexural strength at the thermal shock temperature difference higher than 300 °C, owing to the excess increase of porosity in the matrix region and the extensive interfacial delamination by the thermal shock history.

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