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Production of Ti-Fe Based $MgAl_2O_4$ Composite Material by Pressureless Infiltration Method

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ABSTRACT

In spite of their high temperature and corrosion resistance, ceramics show a brittle nature under applied loads and this limits their usage areas. In order to compensate the weakness of ceramics with metals, which have high ductility, the production of ceramic-metal composite materials has been recently started. In the present study, a ceramic-metal composite material was produced by means of pressureless infiltration method using spinel oxide ($MgAl_2O_4$) and Ti-Fe alloy. Atmospheric conditions, Ti-Fe/$MgAl_2O_4$ ratio, infiltration temperature and time were chosen as an experimental parameter. The experimenral results showed that a passivating oxide layer on the surface of the Ti-Fe alloy was observed in ambient atmosphere at 1600 °C for 120 minutes and therefore no reaction took place between the spinel and Ti-Fe alloy. Whereas, the Ti-Fe alloy was successfully infiltrated under argon atmosphere at 1550 and 1600 °C for 30, 60 and 120 minutes. The infiltrated Ti-Fe composites were characterized using SEM equipped with EDS. The hardness values of the Ti-Fe/$MgAl_2O_4$ composite increased up to 22.5 % Ti-Fe amount and further increase in the Ti-Fe amount led to a decrease in the hardness.

Keywords: Titanium alloys, pressurelles infiltration, cermet, spinel

1. INTRODUCTION

Ceramics are known for their brittleness. The incorporation of a ductile metal into the ceramics matrix leads to improved mechanical properties like hardness, wear strength and fracture toughness [1]. Ceramic-metal composites are also of considerable interest due to their high melting points, low densities and excellent oxidation and corrosion resistance. They are in use especially for lightweight applications such as automotive or aerospace components. Despite the advantages of low density and the availability of a suitable process technology, these materials often have the disadvantages of high temperature properties and corrosion resistance [2].

In cermet production, three main routes are commonly used; a) powder metallurgy route, b) self propagating high temperature synthesis and c) the melt infiltration route [3]. Melt infiltration is considered as a promising process to fabricate various composites using ceramic preforms. In this process, a molten alloy is introduced into a porous ceramic preform. Compared with other methods, such as traditional liquid phase sintering technique, this process has several advantages. It is easy to control the volume fraction of reinforcement. The distribution of reinforcement is quite uniform. Elimination of residual porosities and absence of interfacial reactions between the reinforcement and matrix can be ensured in the final product. The resulting properties of composite can be adjusted to the required values via addition of appropriate alloying elements [4,5].

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The crucial point in the case of infiltration techniques is the wetting behaviour of the system. The driving force for infiltration the liquid metal is dictated by the affinity between the melt/ceramic couple in terms of wettability. Generally this affinity is poor and worsened by the formation of oxide skins on the metal surfaces. When good wettability exists in a given metal/ceramic system, and under adequate conditions of temperature and atmosphere and liquid metal may be drawn into the ceramics preform simply by capillarity [6]. The wetting of the ceramics by liquid metal is, therefore, the main problem to overcome [2].

Recently, many investigations have been carried out on pressureless melt infiltration. Bilen et al. infiltrated Al metal into the spinel ($MgAl_2O_4$) under nitrogen at 900°C and found that the addition of Mg improves wetting of metal-ceramic systems by reducing the surface tension of the melt, reducing the solid/liquid interfacial energy or inducing chemical reactions at the interface [7]. Gürü et al. infiltrated Zn metal into the mixture of fly ash and spinel ($MgAl_2O_4$) under inert atmosphere at 500-550 °C and the experimental results showed that fly ash addition improved sinterability and hardness behavior of zinc based $MgAl_2O_4$ composite [8]. Pressureless infiltration method has been utilized to produce various composites in different studies such as Al_2O_3/Si [9], Al_2O_3/Cu-O [10] and Zn-Al based metal matrix composites [11].

In the present study, a ceramic-metal composite material was produced by means of pressureless infiltration method using Ti-Fe alloy and $MgAl_2O_4$ at different temperatures and time under ambient and argon atmosphere.

2. EXPERIMENTAL STUDY

The Ti-Fe/$MgAl_2O_4$ composites were produced by pressureless melt infiltration of the Ti-Fe alloy into the $MgAl_2O_4$ preform. The chemical composition of the Ti-Fe alloy obtained from Erkunt Casting C. is given in Table 1. The spinel used in this study was obtained from KÜMAŞ. The chemical analysis of the spinel was performed by X-ray fluorescence (Shimadzu, XRF-1700) and is given in Table 2. The mean particle size of the spinel powder determined by means of Malvern 2600 particle sizer was around 18µm (Fig. 1).

Table 1: Chemical composition of Ti-Fe alloy

Elements	Ti	Fe	F	Al	Mo	Mn	Si	Cr	Na
($^{w}/_{w}$) %	70.48	22.77	2.73	2.54	0.39	0.34	0.29	0.24	0.21

Table 2: Chemical composition of spinel powder

Compounds	Al_2O_3	MgO	Fe_2O_3	SiO_2	CaO
($^{w}/_{w}$) %	68.22	29.87	0.78	0.08	0.76

Cylindrical spinel preforms, 5 mm height and 13 mm diameter, with different porosity were prepared by cold pressing the powders with stearic acid ($CH_3(CH_2)_{15}$.COOH) at about 10 tons. The compacts were heated up to 950 °C at a heating rate of 5°C/min so as to remove the binder and presinter them (Fig.2). The porosity of the preforms were determined from dimension and weight loss measurements. Infiltration was carried out in a tube furnace at 1550 and 1600 °C by placing the preforms in an alumina crucible together with the Ti-Fe alloy and holding at the soaking temperatures for 30, 60 and 120 minutes under in ambient atmosphere and under argon atmosphere. After reaction, the specimens were cut and examined by means of scanning electron microscopy (JEOL-JSM 6360LV) equipped with an Energy Dispersive Spectroscopy. Hardness measurements were made by Vickers tester (Instron Wolpert) using 30 kg load and 15 s dwelling time.

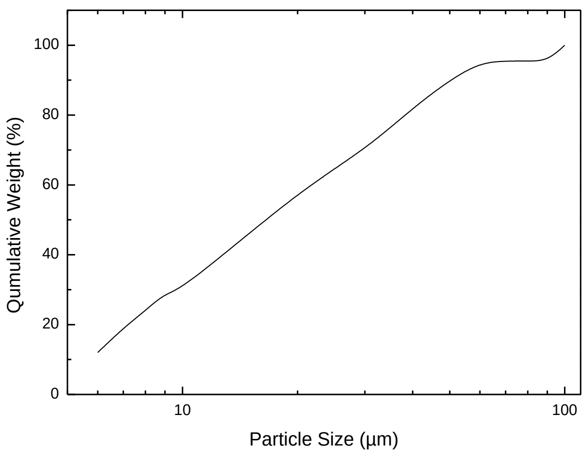

Figure 1. Particle size distribution of spinel

Figure 2: Spinel preform before infiltration powder.

3. RESULTS and DISCUSSION

For preliminary studies, it was tried to infiltrate the Ti-Fe alloy into a preform prepared from spinel oxide with 10% porosity in ambient atmosphere at 1600 ^{o}C for 120 minutes. The infiltration depth is defined as the depth to which the preform is completely infiltrated with metal, i.e, the vertical distance from surface to bottom of preform. As shown in Figure 3, the infiltration of the Ti-Fe alloy into the spinel preform was not complete in ambient atmosphere at 1600 ^{o}C for 120 minutes and only a thin infiltrated layer at the top and subsurface of preform was obtained. On the other hand, a thick passivating oxide layer at the top of the Ti-Fe alloy formed due to a tendency of Ti-Fe alloy against oxidation. Insufficient infiltration was caused by the presence of a thick oxide layer at the surface of the Ti-Fe alloy. This oxide layer prevented wetting of the spinel preform by the molten Ti-Fe alloy. As reported by Tomsia et al, a good infiltration is dependent on porosity amount of preform, critical infiltration temperature and oxygen amount in atmosphere [12].

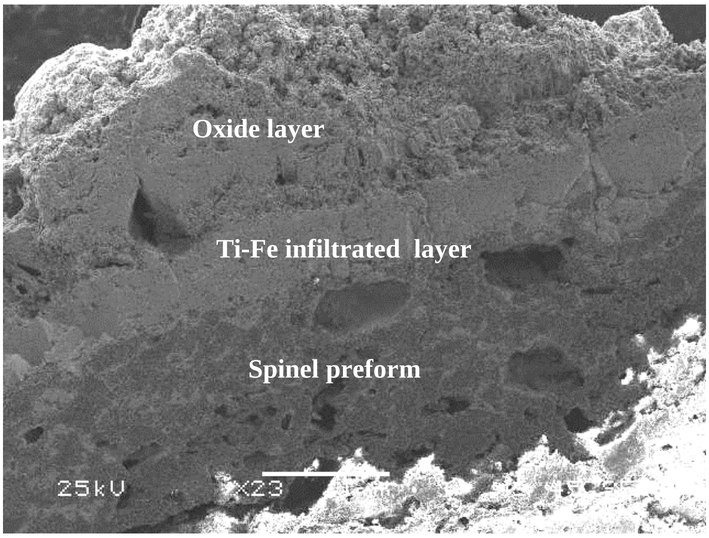

Figure 3. SEM micrograph of the cross section of the Ti-Fe/$MgAl_2O_4$ composite infiltrated in ambient atmosphere at 1600 ^{o}C for 120 minutes

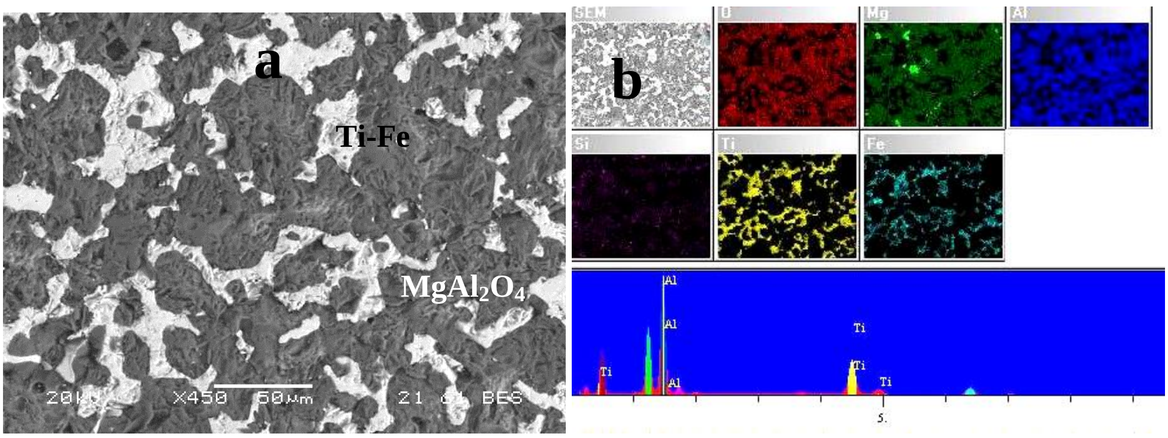

Figure 4. (a) SEM micrograph of the Ti-Fe/$MgAl_2O_4$ composite with 10% porosity infiltrated at 1550°C for 30 minutes under argon atmosphere, (b) EDS mapping of O-Mg-Ti-Si-Al-Fe elements.

In order to prevent the formation of passivating oxide layer and obtain successful infiltration, the experiments were carried out under flowing argon atmosphere which went through an oxygen trapper. Pressureless melt infiltration of the molten Ti-Fe alloy into spinel preform was successfully achieved at 1600 ^{o}C under flowing argon atmosphere. Also, the infiltration of the Ti-Fe alloy was uniform throughout the spinel preform. Figure 4(a) shows a typical SEM micrograph of the resulting composite. The dark phase corresponds to the $MgAl_2O_4$ and the gray phase to the Ti-Fe alloy. In addition, no passivating oxide layer at the top of the Ti-

Fe alloy was observed. The results of scanning electron microscopy indicated good interfacial bonding between the Ti-Fe alloy and spinel preform, such voids or other discontinuities were not observed at the Ti-Fe alloy and spinel interface. To determine optimum infiltration time, the experiments were carried out at the above test conditions for 30 and 60 minutes. The cross section of the preforms showed that an uniform and complete infiltration were also obtained at the infiltration times of 30 and 60 minutes. The fact that there was no chipping at the edge of preform shows a complete wetting of the spinel oxide by the Ti-Fe alloy. Similar results were also observed at 1550 ^{o}C under argon atmosphere for different infiltration times. The elements in the composite were detected by a qualitative X-ray element dispersion analysis by using an energy dispersive spectrometer (EDS). Uniform distribution of O, Al, Mg, Si, Ti and Fe existing in the chemical composition of the composite was also proved by EDS analysis (Fig. 4b). The infiltration of the Ti-Fe amount into spinel preform changes depending on porosity amount. The hardness measurements were made on the spinel preforms with various porosity contents (10, 20 and 30 %) infiltrated at 1550 ^{o}C for 30 minutes. Figure 5 shows the effect of Ti-Fe amount

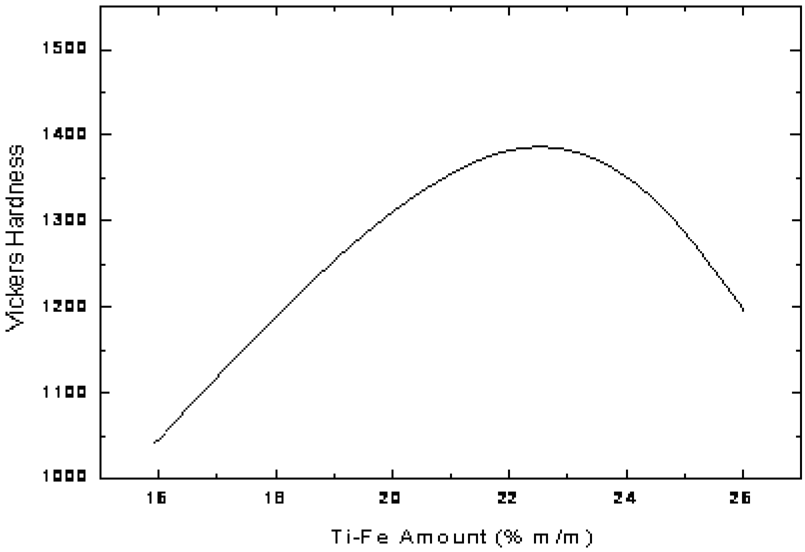

Figure 5. The effect of the Ti-Fe amount on the hardness of the Ti-Fe/$MgAl_2O_4$ composite

on the hardness of the composite. It can be seen from this figure that the hardness values increased up to 22.5 % Ti-Fe amount and further increase in the Ti-Fe amount led to a decrease in the hardness. The increase in hardness values could be due to the formation of good cohesion between spinel and Ti-Fe alloy. In addition, at this Ti-Fe amount, hard spinel oxide phase was dominant in the composite. However, after the optimum Ti-Fe amount of 22.5 %, softer Ti-Fe phase became the matrix phase and this caused the decrease in hardness values.

4. CONCLUSION

Infiltration studies indicated that the Ti-Fe alloy did not infiltrate into the spinel in ambient atmosphere at 1600 ^{o}C and 120 minutes. However, the Ti-Fe alloy was successfully infiltrated into the spinel preform in argon atmosphere at 1550 and 1600 ^{o}C for 30, 60 and 120 minutes. The optimum hardness value was observed at the Ti-Fe amount of 22.5 %.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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