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Microstructure and mechanical properties of TiB_2/(Cu, Ni) interpenetrating phase composites

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TiB_2/(Cu, Ni) interpenetrating phase composites (IPCs) with ceramic volume content of 75–86% were prepared by melt infiltration. The microstructure of TiB_2/(Cu, Ni) IPCs is characterized by the three-dimensional interpenetration of TiB_2 and Cu–Ni phases. Bending strength and fracture toughness of CuNi/81.6%TiB_2 IPCs are as high as 640.5 MPa and 9.37 MPa $m^{1/2}$, respectively. The combination of the micro-crack and interfacial debonding and the cleavage of TiB_2 grains are the main fracture modes of TiB_2/Cu–Ni IPCs.

Keywords: Interpenetrating phase composite; Melt infiltration; Microstructure; Fracture

Recently research has progressed from traditional composite materials with discrete, dispersed additions and has focused on incorporating larger quantities of second phase reinforcements or the utilization of processing routes that result in composites with a connected second phase [1,2]. This new class of composite materials has been termed interpenetrating phase composites (IPCs) and has attracted much interest over the past 10 years [3–5]. Interpenetrating phase composites (IPCs) represent a family of materials whose microstructures are characterized by the continuity and interpenetration of two or more phases. In the context of ceramic–metal IPCs, much of the driving force for investigating interpenetrating microstructures has been the toughening of ceramics by addition of low concentrations of a metal phase. Therefore, IPCs of 60 vol.% ceramic or more are most common in the literature [6,7].

This new material is the subject of much interest in regard to lightweight structural, wear resistant components and/or armored applications. Presently, the Al_2O_3–metal (Al, Cu) [8,9], TiC–metal (Ni, Mg) [10] or TiC–intermetallic (FeAl, NiAl, Ni_3Al) [11–13] systems are proving to be interesting IPCs for either theoretical studies or practical applications. However, studies on TiB_2–metal or alloys IPCs are very limited.

TiB_2 is attractive for high temperature applications because of its high hardness at high and low temperature environments, high melting point (3253 K), high thermal and electric conductivity, good chemical stability and good thermal shock stability. Several recent reviews [14,15] give general methods to enhance the wettability in metal–nonmetal systems. The wetting angle of noble and base metals on oxide ceramics can be decreased significantly by small additions of alloy element. Previous studies showed that addition of an alloy element such as Ni or Al can improve the wettability of TiB_2 ceramic and Cu metal (the wettability angle of TiB_2/Cu is 140° in vacuum [16]).

Many techniques have been found to prepare ceramic–metal IPCs, e.g., the pressure regulated melt infiltration process [12], pressureless melt infiltration of a pre-sintered preform [10], the self-propagating high temperature synthesis [17] and so on. Of the many methods, pressureless melt infiltration is particularly interesting because it offers the advantages of low cost, ease of composite production and near net shape capability. In the present study pressureless melt infiltration was adopted to prepare fully dense TiB_2/Cu–Ni IPCs with high TiB_2 content (75–86 vol.%), and the interpenetrating microstructure and mechanical properties were investigated in detail.

The constituent materials used in the present study were TiB_2 powder produced by the Northwest Institute for Non-Ferrous Metal Research, Xi'an, China with a mean particle size of 2 μm, and commercial nickel powder (70–100 μm) and copper powder (70–100 μm).

TiB_2 powder was de-agglomerated in 2-propanol medium with WC/Co balls, then dried and sieved through an 80-mesh sieve before forming. TiB_2 preforms were first

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pressed by filling a cuboid-shaped rubber mould with suitable amounts of TiB_2 powder, followed by cold isostatic pressing up to 200 MPa (giving TiB_2 green densities of 50–70% of theoretical). The green-compact billets were sintered in a furnace, in a vacuum atmosphere, at a heating rate of 10 °C/min. The sintering temperature was from 1700 °C to 2000 °C. All specimens were held at the sintering temperature for 30 min and then cooled to room temperature at the same rate of 10 °C/min. A predetermined amount of Cu–Ni powder was placed on the TiB_2 pellets, which were positioned on high-purity Al_2O_3 powder (99.99%) within a high-purity graphite furnace under a dynamic vacuum of $\sim 10^{-1}$ Pa, at 1400 °C and 1500 °C, and the conditions were maintained for 60 min. The above different preforms were infiltrated with Cu–Ni in an indirect downward infiltration mode in the present study, which is schematically illustrated in Figure 1.

Figure 2 shows the microstructures of porous TiB_2 specimens sintered from different compacts at a temperature of 2000 °C. Interpenetrating voids and small flaws existed in the specimen, due to a loose connection between the TiB_2 grains. However, these voids and small flaws were reduced greatly with the increasing initial compact density, indicating a better connection between the TiB_2 grains. It can been seen that highly packed regions densified faster than the less-dense regions, as shown in Figure 2(a) and (b), where the ceramic grains clearly have grown in the neck area but the connections between the particles are still porous structures.

Figure 3 shows the dependence of relative density on the sintering temperature of the TiB_2 porous network. It can been seen that for the lower density initial green compacts, higher temperatures are required to reach the same density as that of the green compact with higher density.

Figure 4(a) and (b) shows typical micrographs of TiB_2/Cu–Ni prepared by spontaneously infiltrating the TiB_2 preforms of the two groups with TiB_2 volume content of 75.5% and 86.4%, respectively. It should be noted that the bright phase is Cu–Ni, and the black is TiB_2 by energy dispersive X-ray spectroscopy analysis. The bond between TiB_2 and Cu–Ni looked good and strong. No obvious micro-defects such as large voids and separated

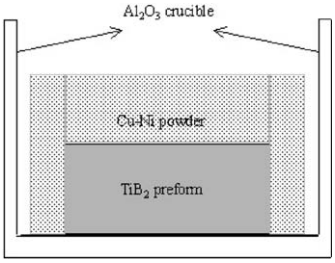

Figure 1. Scheme of indirect downward horizontal infiltration.

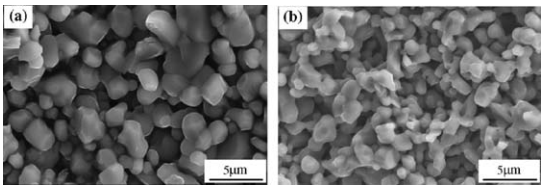

Figure 2. SEM micrographs of TiB_2 specimens sintered from (a) porosity 32.5% and (b) porosity 28.4% at 2000 °C/1 h.

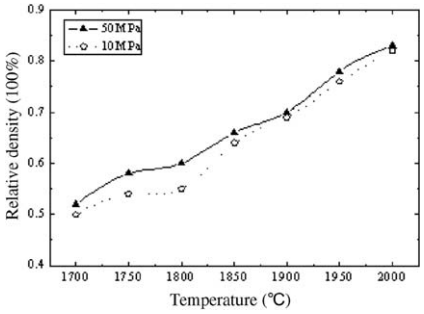

Figure 3. Dependence of relative density on sintering temperature of TiB_2 porous network.

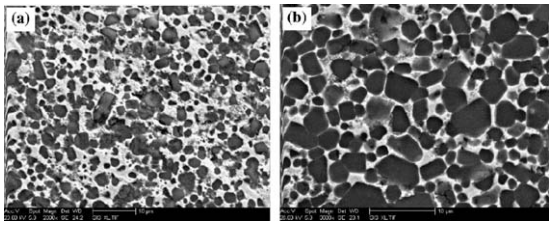

Figure 4. Typical micrographs of TiB_2/Cu–Ni composites infiltrated at 1500 °C: (a) with 75.5 vol.% TiB_2, (b) with 86.4 vol.% TiB_2, prepared by infiltrating the 2000 °C × 1 h pre-sintered TiB_2 preform.

interface of TiB_2 and Cu–Ni were observed. The infiltration ability of liquid Cu–Ni in porous TiB_2 preform was adequate to form fully dense and defect-free composites. It is interesting to see in Figure 4 that TiB_2 particles join each other. The joints between TiB_2 particles formed in the pre-sintering stage and were maintained after infiltration. Cu–Ni melt filled the channels within the three-dimensional network of the joining TiB_2 particles and formed also a continuous network of Cu–Ni.

Slightly higher densities were generally observed for materials processed at 1500 °C when compared to those processed at 1400 °C (shown in Fig. 5), particularly for the lowest binder contents (i.e., <20 vol.%). These materials exhibited minimal porosity for Cu–Ni contents of >20 vol.%, with retained pore sizes generally being sub-micrometer in magnitude.

Table 1 lists the mechanical properties of the prepared TiB_2/Cu–Ni IPCs. The TiB_2 volume fraction of the composite was estimated from the density of the composites, which was measured by the Archimedes method in ethyeneglycol, and is given by the following equation:

$$\rho_{(\mathrm{Cu,Ni})} \times \rho_{\mathrm{TD}}(1 - V_{\mathrm{TiB_2}}) + \rho_{\mathrm{TiB_2}} \times \rho_{\mathrm{TD}} \times V_{\mathrm{TiB_2}} = d_{\mathrm{measured}} \quad (1)$$

$$\rho_{(\mathrm{Cu,Ni})} = \frac{m}{\frac{m_{\mathrm{Cu}}}{\rho_{\mathrm{Cu}}} + \frac{m_{\mathrm{Ni}}}{\rho_{\mathrm{Ni}}}} \quad (2)$$

where $\rho_{(\mathrm{Cu,Ni})}$ and $\rho_{\mathrm{TiB_2}}$ are the densities of Cu–Ni alloy and TiB_2, respectively. The ρ_{TD} represents the relative density of the TiB_2/Cu–Ni composites. The density of Cu–Ni can be calculated from Eq. (2). In this study, 8.93 and 4.52 g/cm^3, respectively, are taken as the densities of Cu–Ni and TiB_2. From Eq. (1) and Eq. (2), the calculated volume fraction of TiB_2 in the composites is as listed in Table 1. We can see that the calculated volume fraction of TiB_2 after infiltration is higher than that of the preforms before infiltration. A major reason that caused the TiB_2 content to increase a little is that the