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# In-situ fabrication of TiC-Fe<sub>3</sub>Al cermet



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## ABSTRACT

Titanium carbide has high hardness, resistance to oxidation and abrasion while iron aluminide has proper ductility as well as good strength and excellent oxidation resistance up to high temperatures. Therefore, it can be expected TiC-iron aluminide cermet to have excellent mechanical properties as a cutting tool and a wear-resistance material. In this study, mechanical milling and hot press sintering processes were used to manufacture in-situ TiC-Fe<sub>3</sub>Al cermet, whose microstructure and mechanical properties were examined according to the changes in volume fraction of TiC and milling time. After 48 h of milling each mechanically alloyed powder crystallized in a TiC and Fe<sub>3</sub>Al biphasic material. The milled powder was hot-pressed at 1250 °C and 50 MPa for 30 min to obtain sintered bodies also consisting of only TiC and Fe<sub>3</sub>Al phases. The hard phase, TiC, had a size of 100–300 nm with overall uniform distribution decreasing as the volume fraction of TiC increased. The hardness of each sintered body showed a linearly increasing tendency according to the increase in TiC content, the hardness for 90 vol% TiC cermet being as high as 1813Hv. On the other hand, the bending strength was 1800 MPa and 1780 MPa when TiC volume fraction was 50% and 70%, respectively, while it showed an abrupt decrease up to 580 MPa at 90% TiC volume fraction. Fe<sub>3</sub>Al phase is effective to toughening of TiC-Fe<sub>3</sub>Al cermet and the volume fraction of Fe<sub>3</sub>Al phase significantly influences the bending strength of the cermet.

#### 1. Introduction

Cermet is a composite material with a ceramic-based hard phase comprising carbide, nitride, and oxide, and a metal-based binder phase including Ni, Co, or Fe. The material thus combines the high hardness and resistance to heat, oxidation, and abrasion of a ceramic with the toughness of a metal [1]. Typical examples include WC-Co-based cermet and TiC-metal-based cermet. WC-Co-based cermet is particularly widely used for the fabrication of various products such as cutting tools, molds, and abrasion tools, owing to its high hardness, excellent resistance to abrasion and heat, and the high ductility afforded by the excellent wettability between Co and WC [2]. However, WC-Co-based cermet is expensive and limited in it is availability because the raw materials used for its production are concentrated in a few localities. Much research has thus been devoted to developing substitute materials.

In the case of TiC-based cermet, TiC-Fe- and TiC-Ni-based composites are widely employed owing to their high hardness and elastic modulus, low density, and chemical stability [3]. Although TiC-Fe- and TiC-Ni-based cermets have been extensively studied as substitutes for WC-Co-based cermet [4], their applications are restricted by their lower toughness and hardness compared to WC-Co, attributable to the reduced wettability between the binder and reinforcement materials [5,6].

Iron aluminide-based intermetallic compounds have a low specific gravity and other attractive properties such as excellent resistance to oxidation and corrosion, as well as low cost. In addition, their good mechanical properties at elevated temperatures have attracted attention to their application in structures exposed to high temperatures [7–9]. It has further been reported that the wettability between aluminides and TiC is superior to that between Fe and Ni [10]. Iron aluminide-based intermetallic compounds thus have many merits for use as binders in cermets, and Schneibel et al. [10] reported the synthesis of TiC-(Fe-Al), TiB<sub>2</sub>-(Fe-Al), and WC-(Fe-Al) cermets with excellent mechanical properties through TiC preform and pressureless melt infiltration.

The methods for synthesizing composite materials are broadly divided into the use of in-situ and ex-situ processes. In an in-situ

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process, the reinforcing phase of the material is formed within the matrix phase during the synthesis. Conversely, in an ex-situ process, the reinforcing phase is produced by a separate process and subsequently added to the matrix phase during the synthesis of the composite material [11]. Because the reinforcing phase is formed during the synthesis of the composite in an in-situ process, it has the advantage of eliminating oxidation and external contamination of the interface between the matrix and the reinforcement phases, thereby increasing the interface strength and wettability. In addition, the process improves the toughness and strength of the composite, including at high temperatures, because the thermodynamically stable reinforcement phase is uniformly distributed through the matrix phase [12].

In the present study, TiC-Fe<sub>3</sub>Al-based cermet was produced by an in-situ process using powdered Ti, C, Fe, and Al as the raw materials. Mechanical milling was used to accelerate the in-situ reaction between the raw powders. The effects of the TiC volume fraction on the microstructures and mechanical properties of the milled powder and sintered cermet were examined.

#### 2. Experimental procedure

Powdered Fe (5-10 µm, 99%), Al (3 µm, 99.9%), Ti (45 µm, 99.9%), and C (5 µm, 99.7%) were used as the starting materials. The ratio of Fe to Al was set to 84.15:15.85 to obtain Fe-28at%Al after the reaction between Fe and Al. The cermet samples were formulated to contain 50%, 70%, and 90% TiC volume fractions, respectively. The chemical compositions and designations of the samples are summarized in Table 1. The elemental Ti, C, Fe, and Al powders were mechanically alloyed using a ball mill. A stainless steel canister and stainless steel ball of diameter 9.525 mm were used for the attrition milling. The mixed powders were milled at a rate of 320 rpm in an Ar atmosphere (Ar purity of 99.999%) for 6, 12, 24, and 48 h, respectively. The mechanically milled powders were then poured into a graphite die and hot pressed. During the hot pressing, the samples were heated to 1250 °C in a vacuum at a rate of 10 °C/min, followed by pressing at 50 MPa for 30 min. The sintered samples were then cooled to room temperature in the vacuum.

Identification of the phases in the milling powders and the sintered samples was performed by X-ray diffraction (XRD) (D8, Broker, Germany). The microstructures of the milled powders and sintered samples were characterized by a scanning electron microscope equipped with an energy dispersive X-ray spectrometer (SEM-EDS) (Nova SEM, FEI, USA). The hardness of the sintered samples was measured using a Vickers hardness tester (HM-21, Mitutoyo, Japan). The specimens prepared for the hardness tests measured  $3\times4\times25$  mm. Three-point bending tests were also conducted using a Universal Testing Machine (UTM) (DTU-900MHA, Daekyung Tech, Korea). The bending strength of the sintered material was determined as the average result of six tests for each composition.

Table 1
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Designations	and	chemical	compositions	of	the	cermet	sami	ples
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Nominalcomposition	Designation	Amo	Amounts of elemental addition [at% (wt%)]					
_		Ti	С	Fe	Al			
(Fe–28at%Al)–50vol%	50%TiC	28.0	26.2	33.0	12.8			
TiC		(34.8)	(8.2)	(48.0)	(9.0)			
(Fe–28at%Al)–70vol%	70%TiC	37.9	35.5	19.2	7.4			
TiC		(51.6)	(12.2)	(30.5)	(5.7)			
(Fe–28at%Al)–90vol%	90%TiC	47.2	44.3	6.1	2.4			
TiC		(70.7)	(16.6)	(10.7)	(2.0)			

#### 3. Results and discussion

Fig. 1 shows the XRD results for 50% TiC powder with respect to the milling time. The peaks of the elemental powders and TiC can be observed for 6-h milling. Milling for 12 h produces a Fe<sub>3</sub>Al peak in addition to the Fe and TiC peaks, while milling for 24 h causes the disappearance of the elemental powder peaks, with only the TiC and Fe<sub>3</sub>Al peaks retained. The phases that were formed in the Fe-Al-Ti-C system included Fe2Al5, Al3Fe, FeTi, Fe3C, Fe3AlC, Al3Ti, TiAl, as well as TiC and Fe<sub>3</sub>Al [9,13,14]. However, when a Fe-Al-Ti-C alloy containing Fe-28at%Al/15 vol%TiC is synthesized by an in-situ casting process, only TiC and Fe<sub>3</sub>Al are present at the end of the process. This can be explained by the pseudo-binary phase diagram of Fe<sub>2</sub>Al and TiC [15]. As in the casting process, each elemental powder was apparently transformed into two thermodynamically stable phases during the milling process. The mechanical milling of multiple powered elements has been reported to result in the formation of amorphous or supersaturated solid solutions or intermetallic compounds due to the reactions among the elements [16]. There are also instances in which an amorphous or a supersaturated solid solution is formed at the beginning of the milling process, with a stable intermetallic compound subsequently appearing after a sufficient period of milling. The phases identified after milling depends on not only the change in the free energy due to the reactions among the elements, but also on the milling method and conditions [17]. The formation of solid solutions has also been reported for the milling of the same elements (Ti, C, Fe, and Al) using a vibratory ball mill [18]. However, in this previous study, the stable phase of the intermetallic compounds was directly formed during the milling. It is generally considered that attrition ball milling transfers more energy to the milled powders than vibratory ball milling. The employment of the higher-energy ball milling in the present study was apparently the reason for the formation of a stable intermetallic compound phase rather than a solid solution, because higher mechanical and thermal energies were applied to the powders.

However, as can be observed from the XRD results, the TiC peaks were preferentially detected after 6 h of milling, with the Fe<sub>3</sub>Al peaks appearing after 12 h of milling. From this formation of TiC before Fe<sub>3</sub>Al despite the 50:50 vol ratio of the two phases, it can be inferred that there is a difference between the formation rates of the phases for given milling conditions. The formation rate of each phase is determined by the activation energy required for the formation reaction. The activation energies of  $3Fe + Al \rightarrow Fe_3Al$  and  $Ti + C \rightarrow TiC$  are 929 and 340 kJ/mol, respectively [4,5]. Hence, from a kinetic viewpoint, TiC is preferentially formed [19,20].

To investigate the effect of the milling time on the microstructure of



Fig. 1. XRD results for the milled powder containing 50 vol% TiC for different milling times.

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