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Effect of spark plasma sintering temperature on the microstructure and mechanical properties of a Ti_2AlC/TiAl composite

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ABSTRACT

The effect of spark plasma sintering (SPS) temperature on the microstructure and mechanical properties of a bulk ultrafine structured Ti_2AlC/TiAl composite prepared by SPS of a mixture of mechanically alloyed Ti–Al powder with a composition of Ti–50Al (at%) and 10 vol% carbon nanotubes (CNTS) has been investigated. X-ray diffraction analysis showed that the sintered bulk material mainly composed of γ-TiAl and Ti_2AlC phases. The microstructures of the samples were examined using scanning electron microscopy and transmission electron microscopy. It was found that when an SPS temperature of 950 °C was used, the bulk Ti_2AlC/TiAl composite had a high density of 98.3%, and a microstructure consisting of a continuous interpenetrating network of Ti_2AlC phase and equiaxed TiAl grains with an average size of 300 nm. The compressive yield strength and hardness of the sintered samples reached 2058 MPa and 6.12 GPa respectively. With increasing the SPS temperature up to 1150 °C, the TiAl grains became significantly coarsened, leading to clearly reduced mechanical properties.

1. Introduction

In recent years, much attention has been paid to intermetallic compounds, and a representative of such compounds is titanium aluminides. These materials are attractive for use in advanced propulsion systems for aircraft and also in automobile engine parts due to their low density, high specific strength, high specific stiffness, good creep strength up to 700 °C and better high-temperature oxidation resistance than titanium alloys [1–3]. However, the low room temperature ductility and poor formability limit the extensive application of TiAl based alloys [4]. It has been found that the intermetallic matrix composites (IMCs) can provide the right combination of high-temperature strength and creep resistance, and overcome the problem of poor ductility and toughness at ambient temperatures [5]. In addition, a number of compounds, like TiB_2, Ti_5Si_3 and Ti_2AlC, have been identified as compatible and thermochemically stable reinforcement phases for the γ-TiAl matrix [5–9]. Therefore, considerable efforts have been devoted to prepare and investigate the γ-TiAl matrix composites with these reinforcement phases. For example, Lin and co-workers [10] found that both colony size and lamellar spacing of TiAl/TiB_2 composites prepared by powder metallurgy were reduced with increasing the volume fraction of TiB_2. Bingchu and Yoshinari [9] produced TiAl/Ti_2AlC composites by spark plasma sintering (SPS) of a mixture of Ti, Al and TiC powders, and showed that its bending strength can reach 900 MPa. Chen et al. [11] prepared a TiAl/Ti_2AlC composite by hot pressing compacts of a mixture Ti, Al and TiC powders and a mixture of Ti, Al and C, respectively, and demonstrated that the nature of the starting powder had clear effect on the pathway for forming TiAl/Ti_2AlC composite structure and the resulting microstructure of the composite.

Intermetallic matrix composites can be synthesized by combinations of a variety of materials synthesis methods such as mechanical alloying, hot isostatic pressing, physical vapor deposition, plasma spraying, melt infiltration, combustion synthesis and spark plasma sintering (SPS) [5,10–16]. Among them, the combination of mechanical alloying for preparing powders and SPS for consolidation of the powders offers many advantages such ultrafine microstructure, high purity and high relative density since the SPS can facilitate high heating rate (up to 600 °C/min) and high sintering rate (due to effective breaking of oxide surface layers on powder particles) [16–22]. In the present work, we utilized a combination of mechanical alloying and SPS to synthesize a bulk Ti_2AlC/TiAl composite with a novel microstructure consisting of an interpenetrating network of Ti_2AlC phase and equiaxed TiAl grains from a mixture of a mechanically alloyed Ti–Al powder with a composition of Ti–50Al (at%) and 10 vol% CNTS. The effect of SPS temperature on the microstructure and mechanical properties of the bulk Ti_2AlC/TiAl composite was investigated.

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Table 1
Ball milling condition used in mechanical alloying of the Ti–50Al powder mixture.

Milling item	Condition
Milling vial	Stainless steel
Grinding medium	Stainless steel, 9.5 mm in diameter
Starting powders	Ti (99.9% pure, 500 mesh), Al (99.9% pure, 325 mesh)
Milling speed	400 r/min
Milling time	10–50 h
Ball-to-powder weight ratio	20:1
Process control agent	Acetone (1.5 wt.%)
Atmosphere	Ar (99.99% pure)

Table 2
Ball milling condition used for mixing the mechanically alloyed Ti–50Al powder and carbon nanotubes.

Milling item	Condition
Milling vial	Stainless steel
Ball-to-powder weight ratio	2:1
Milling speed	100 r/min
Grinding medium	Stainless steel, about 2 mm in diameter
Milling time	20 h

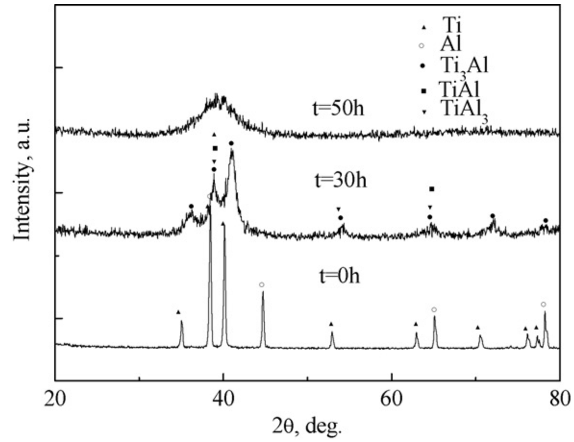

Fig. 1. X-ray diffraction patterns of Ti–50Al (at%) powder mixture ($t = 0$ h) and mechanically alloyed powders produced by high energy mechanical milling for 30 and 50 h, respectively.

2. Experimental procedures

The mechanically alloyed Ti–Al powder was produced by ball milling a mixture of titanium powder (99.9% purity, −500 mesh) and aluminum powder (99.9% purity, −325 mesh) in a planetary ball mill under an argon atmosphere with the addition of acetone (1.5 wt.%) as a process controlling agent. Before milling, the powders were well mixed, and the mixture had a composition of Ti–50Al (at%). The ball milling conditions are given in Table 1. The mechanically alloyed Ti–50Al powder prepared using 50 h of milling was mixed with 10 vol% of CNTS (density 1.3–1.5 g/cm^3, diameter 20–50 nm, length 0.5–500 μm) using the same planetary ball mill but with much lower rotation speed and very small stainless steel balls to prevent mechanical alloying between Ti–50Al particles and CNTS. The ball milling condition for mixing the Ti–50Al (at%) powder and CNTS is given in Table 2. Samples of Ti–50Al/CNTS powder mixture were sintered using SPS at 750, 950, 1050 and 1150 °C, respectively, under a vacuum of 6 Pa and with a holding time of 15 min and uniaxial pressure of 60 MPa.

Compressive tests with cylindrical specimens with dimensions of Φ4 mm × 6 mm were conducted at room temperature using an Instron testing machine with a strain rate of about 5.56×10^{-4} s^{-1}. X-ray diffraction (XRD) analysis of milling powders and sintered samples was conducted using Philips X'Pert machine with Cu K_α radiation ($\lambda = 0.154157$ nm) to determine the phase constitution of the materials. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to examine the microstructures of sintered compacts. The polished surfaces of samples for SEM examination were etched in a modified Kroll's reagent of 10 vol% HF, 4 vol% HNO_3 and 86 vol% H_2O. The specimens for TEM observation were prepared using a twin-jet electropolishing method with a solution of 60 vol% methanol, 34 vol% normal butanol and 6 vol% perchloric acid which was cooled to −20 °C and a voltage of 40 V. The Archimedes technique was used to measure the density of sintered samples.

3. Results and discussion

3.1. Characterization of the mechanically alloyed Ti–50Al powders and CNTS

The XRD patterns of the mechanically alloyed Ti–50Al powders produced with different milling times are shown in Fig. 1. After milling for 30 h, all the diffraction peaks became broader, with the shift of Ti peak to higher end. The Al peaks were absent, and the Ti_3Al, $TiAl_3$ and TiAl peaks appeared in the XRD pattern, indicative of the formation of the intermetallics phases. In addition, the XRD patterns also indicated that the amount of Ti_3Al phase in

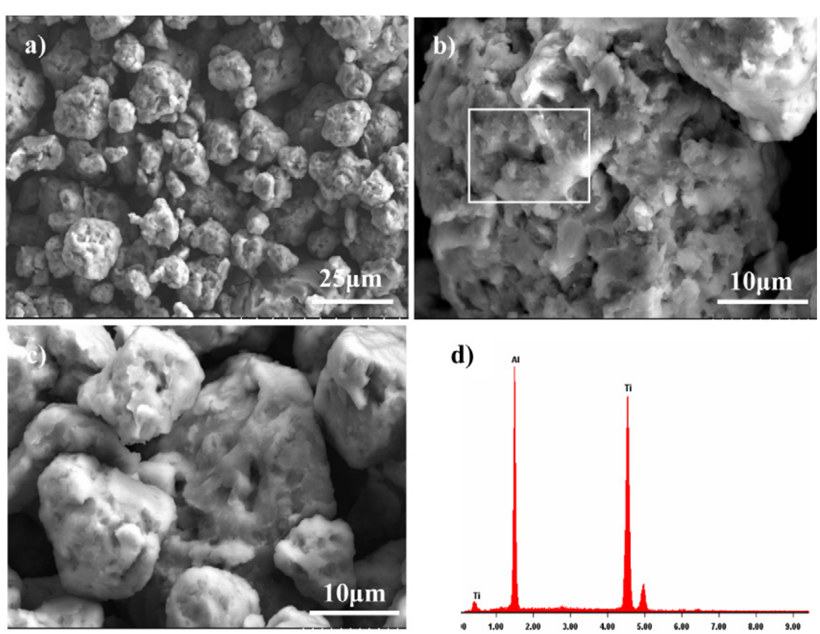

Fig. 2. (a)–(c) SEM micrographs showing morphologies of the particles in the mechanically alloyed Ti–50Al (at%) powder produced by milling for 50 h and (d) an EDS spectrum from the selected area shown in (b).