Production of Dispersion-strengthened Cu-TiB_2 Alloys by Ball-milling and Spark-plasma Sintering

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Abstract

Dispersion-strengthened copper with TiB_2 was produced by ball-milling and spark plasma sintering (SPS).Ball-milling was performed at a rotation speed of 300rpm for 30 and 60min in Ar atmosphere by using a planetary ball mill (AGO-2). Spark-plasma sintering was carried out at 650 ℃ for 5min under vacuum after mechanical alloying. The hardness of the specimens sintered using powder ball milled for 60min at 300rpm increased from 16.0 to 61.8 H_RB than that of specimen using powder mixed with a turbular mixer, while the electrical conductivity varied from 93.40% to 83.34%IACS. In the case of milled powder, hardness increased as milling time increased, while the electrical conductivity decreased. On the other hand, hardness decreased with increasing sintering temperature, but the electrical conductiviey increased slightly

Keywords : ball-milling, Cu-TiB_2 alloy, spark-plasma sintering

1. Introduction

High strength Cu alloys with good electrical conductivity at high temperatures can be achieved by dispersing fine particles such as oxides, carbides, nitrides and borides in the Cu matrix [1-6]. TiB_2 is well known for its high melting point, hardness and electrical conductivity [7]. Thus, TiB_2 reinforced matrix composites were investigated in this study.

High-energy ball-milling is applied to produce fine and nanosized particles. Spark-plasma sintering (SPS) is a newly developed process that enables sintering of high quality materials in short periods possible by DC-pulse charging between powder particles with relatively high sintering pressure. SPS systems offer many advantages (e. g. rapid sintering, less sintering additives, uniform sintering, low running cost, and easy operation) over conventional systems [8].

In the present study, commercial TiB_2 and copper powders were ball-milled to obtain composite powders with improved TiB_2 particles distribution by means of ball-milling microstructure and properties of spark-plasma sintered Cu-TiB_2 composites were investigated.

2. Experimental and Results

TiB_2 (99.5 %, <45 ㎛) and copper (99.5 %, <45 ㎛) powders were used as raw materials. To obtain the desired composite powders containing 2.5 wt% TiB_2, powders were perepared by two methods. First, Cu powder was mixed with the raw TiB_2 powder in a turbular mixer at a speed of 75 rpm for 2 hours. Next, the Cu and TiB_2 powders were milled by high-energy ball-milling (AGO-2, planetary ball mill type) for 30 min and 60 min at 300 rpm. Balls and vials were made of stainless steel. The diameter of the balls was 2.77 mm and the powder to ball ratio was 1:20. The vials were then evacuated and subsequently filled with argon up to 0.3 MPa.

Spark Plasma Sintering (SPS) was performed using a SPS apparatus (Sumitomo Coal Mining Co.Ltd, AUJ-1611) under vacuum. A graphite mold of 15 mm in inner diameter was used. The applied SPS-pressure and SPS-temperature were 50 MPa and 650-750 ℃, respectively. Holding time at the final temperature was 5 min. It should be noted that the effective temperature of the sample is usually 50 ℃ higher than the measured SPS-temperature.

X-ray diffraction patterns (XRD, Cu Kα, Rigaku, RAD-3C) were taken to conduct phase analyses of powders and compact. The particle size distribution was determined by a laser particles size analyzer (LPSA, Malvern, Mastersizer 2000). Then, a morphology observation and qualitative analysis (EDS) of powders were done by means of field emission-scanning electron microscopy (FE-SEM, JEOL, JSM-6500F). The relative density of the samples was measured by the Archimedes method and hardness (H_RB) values were measured by a Rockwell hardness tester. The electrical conductivity expressed in %IACS (International Annealed Copper Standard) was measured by a conductivity meter (Fischer, SIMASCOPE® SMP10).

Table. 1 Density, hardness and electrical conductivity of sintered samples

sample	Ball-milling		Sintering		Relative density (%)	Hardness (H_RB)	Electrical conductivity (%IACS)
	Speed (RPM)	Time (min)	Temp. (℃)	Time (min)			
1*	-	-	650	5	99.10	16.0	93.40
2	300	30	650	5	97.81	48.7	88.26
3	-	60	650	5	97.78	61.8	83.34
4	-	60	700	5	97.75	53.4	84.11
5	-	60	750	5	97.09	53.2	86.25

*** Using powder mixed with a turbular mixer**

Table. 1 shows the changes in the relative density, hardness and electrical conductivity of Cu–2.5wt.% TiB_2 consolidated by the SPS process using powders ball milled for 30min and 60min at 300rpm at various temperatures ranging from 650 to 750°C. The relative density of all the samples could be obtaine over 97% of the theoretical density after the SPS.

Even though the hardness of the sintered samples using ball-milled powder increased more than that of sample sintered using powder mixed with a turbular mixer, the electrical conductivity still remained high.

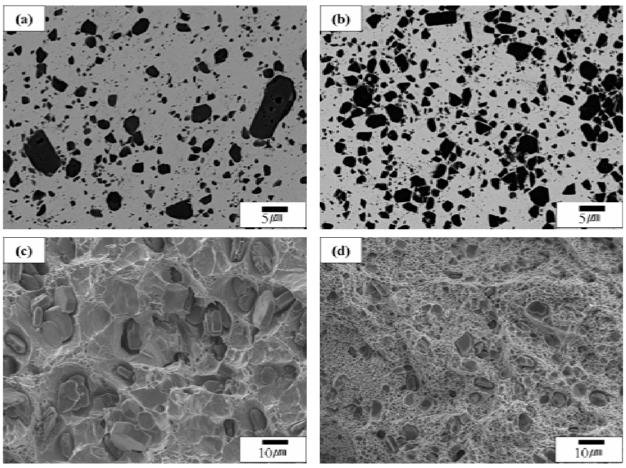

Fig. 1. SEM morphologies of cross-section and fracture surface of samples sintered for 5min at 650℃ by mixing ((a) and (c)) and ball milled powders ((b) and (c)).

Fig. 1 shows the SEM morphologies of cross-section and fracture surface of samples sintered for 5min at 650℃. In the case of simple mixing, the large and small particles in the copper matrix were observed together (Fig. 1(a) and (c)). The shape and size of TiB_2 paricles in sample sintered using mixed powde with turbular mixer did not show any change (Fig. 1(c)). On the other hand, the size of TiB_2 particles in sample that was sintered using powder ball-milled for 60min at 300rpm decreased (Fig. 1(b) and (d)). Furthermore, the shape of TiB_2 particles changed from flate and hexagonal to angular (Fig. 1(d)). Physical impacts on powders induced by ball-milling cause fractured in TiB_2 particles, consequently deforming their shape. Based on these observations, Partices are expected to become smaller with increasing of milling time

3. Summary

Dispersion-strengthened copper with TiB_2 was produced by ball-milling and spark plasma sintering. Hardness of specimens sintered using the ball-milled powder increased from 16.0 to 61.8 H_RB than that of specimen using the mixed powder, while the electrical conductivity changed from 93.40% to 83.34%IACS. For ball-milled powder, the hardness increased with increasing milling time, while the electrical conductivity decreased. On the other hand, hardness decreased with increaseing sintering temperature and the electrical conductivity increased slightly

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5. References

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