Polskie Towarzystwo Materiałów Kompozytowych
Polish Society of Composite Materials
Kompozyty 11: 2 (2011) 125-129
KOMPOZYTY
COMPOSITES

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Otrzymano (Received) 31.01.2011

PROCESSING COPPER-INTERMETALLIC FIBROUS COMPOSITES FROM COPPER AND TITANIUM POWDERS

Copper-intermetallic fibrous composites were produced using the powder metallurgy method followed by extrusion. The mixtures of Cu powder with 1 wt.% Ti; 2.5 wt.% Ti and 5 wt.% Ti powder were cold pressed and sintered at a temperature of 850°C. The sintered material was extruded using the KOBO method. During extrusion the hard particles containing copper--titanium intermetallic phases undergo a plastic deformation assuming a fibrous shape as the processed composite consists of a copper matrix reinforced with fibrous particles of copper-titanium intermetallic phases. Metallographic examinations of the composites revealed uniform distribution of the reinforcing particles in the copper matrix. SEM investigations and X-ray microprobe analysis showed that as a result of sintering intermetallic phases were synthesized at the Cu-Ti interface. The Ti-Cu reaction products were composed of intermetallic phases in the external zone (at the copper-titanium interface) and the core containing a solid solution of copper in titanium. The microhardness of the reinforcing particles was 760 HV0.65. The samples of the composites and sintered unreinforced copper were examined in a compression test, parallel and perpendicular to the extrusion direction. The yield strength value of the composites increases with an increase in the number of reinforcing particles in the copper matrix. Mechanic anisotropy was observed for the Cu-2.5wt.% Ti and Cu-5wt.% Ti composites: the yield strength was higher for the composites loaded parallel to the extrusion direction than for those loaded perpendicular. The yield strength of the Cu-2.5wt.% Ti and Cu-5wt.% Ti copper-intermetallic fibrous composites was several times higher than that of the unreinforced copper.

Keywords: powder metallurgy, copper matrix composites, extrusion, compression test

WYTWARZANIE KOMPOZYTU WŁÓKNISTEGO MIEDŹ - FAZY MIĘDZYMETALICZNE Z PROSZKÓW MIEDZI I TYTANU

Do wytworzenia kompozytów włóknistych miedź-fazy międzymetaliczne zastosowano metodę metalurgii proszków a następnie wyciskanie. Mieszaniny proszku miedzi i proszku tytanu dodanego w ilości: 1 ; 2.5 i 5% były prasowane na zimno i spiekane w temperaturze 850°C. Następnie spieki poddano wyciskaniu metodą KOBO. Podczas wyciskania twarde cząstki wzmacniające ulegają deformacji plastycznej, przyjmując kształt włókien. Uzyskane kompozyty składają się z osnowy miedziowej, w której rozmieszczone są włókniste wydzielenia twardych faz międzymetalicznych miedziowo-tytanowych. Badania metalograficzne kompozytów pozwoliły stwierdzić, że cząstki wzmacniające kompozyt są rozmieszczone jednorodnie w miedziowej matrycy. Za pomocą mikroskopu skaningowego i mikroanalizatora rentgenowskiego zidentyfikowano fazy powstające w trakcie spiekania na granicy Cu-Ti. Produkty reakcji Ti-Cu składają się z warstwy zewnętrznej utworzonej na granicy miedzi z tytanem, w której stwierdzono fazy międzymetaliczne miedziowo-tytanowe i rdzenia o strukturze roztworu stałego miedzi w tytanie. Mikrotwardość cząstek wzmacniających wynosiła 760 HV0.65. Próbki kompozytów oraz spieku miedzi niewzmocnionego twardymi cząstkami poddano próbie ściskania. Próbki obciążone były równolegle i prostopadle do kierunku wyciskania. Stwierdzono, że wartość umownej granicy plastyczności wzrasta wraz ze zwiększeniem ilości cząstek wzmacniających w osnowie kompozytu. Zaobserwowano anizotropię własności mechanicznych kompozytów Cu-2.5%Ti i Cu-5%Ti: wartość umownej granicy plastyczności zmierzona równolegle do kierunku wyciskania była wyższa niż w kierunku prostopadłym. Kompozyty włókniste Cu-2.5%Ti i Cu-5%Ti posiadają kilkakrotnie wyższą wartość umownej granicy plastyczności niż spiek miedzi niewzmocniony włóknami zawierającymi fazy miedziowo-tytanowe.

Słowa kluczowe: metalurgia proszków, kompozyty o osnowie miedzi, wyciskanie, próba ściskania

INTRODUCTION

The powder metallurgy method is widely applied in the production of composite materials with a copper matrix. Currently, copper-based composites are produced by blending copper powders with reinforcing elements and then by cold pressing and sintering followed by plastic working (forging, extrusion). The combination of high strength, good wear resistance and high electrical and thermal conductivity has made copper matrix composites reinforced with hard particles attractive for applications in electrical contacts, resistance welding electrodes, electrical brushes, sliding contacts, elements of electronic systems and sliding rings [1]. Copper matrix composites can be reinforced with different hard particles: Al_2O_3 [2], TiO_2 [3], TiB_2 [4], AlN [5], SiC [6,7], Ti_3AlC_2 [8], Ti_3SiC_2 [9], TiC [10], WC [11], Ti_2SnC [12]. For metal matrix composites, the reinforcing phases are prepared separately prior to composite fabrication or they can be synthesised in a metallic matrix as a result of reactions between the elements during composite fabrications [13].

In this work the reinforcing particles were formed using the method of intermetallic phases synthesis during the sintering of two elemental metal powders (copper and titanium). According to the Cu-Ti binary phase diagram [14], Ti can react with Cu to form a solid solution - Cu_4Ti, Cu_3Ti_2, Cu_4Ti_3, CuTi and $CuTi_2$ intermetallic compounds.

The aim of this work was to obtain a copper-based composite with fibrous particles of copper-titanium intermetallic phases containing reinforcing particles of copper-titanium intermetallic phases elongated in one direction. Such a material can be produced in two steps by using the powder metallurgy method and an unconventional method of metal forming (KOBO Type Forming). The KOBO technology is used for materials that do not undergo deformation easily [15]. This paper describes the processing, microstructure and mechanical properties of the composite.

EXPERIMENTAL PROCEDURE

The materials used to prepare the composite were copper powder (average particle size 50 μm) and titanium powder (average particle size 60 μm). The copper powder particles were dendritic in shape while the titanium powder particles were irregular. The morphology of the raw powders is shown in Figure 1.

To fabricate the composite, three powder mixtures were prepared: Cu-1wt.%Ti; Cu-2.5%wt. Ti; Cu--5 %wt. Ti. The powder mixtures were poured into a die and cold pressed at a pressure of 600 MPa using a hydraulic press machine. The resulting compacts 40 mm in diameter were sintered at a temperature of 850°C for 0.5 h. After sintering, the samples were extruded using the KOBO method. The KOBO extrusion process combines conventional extrusion with cyclically reversible plastic twisting (cyclic change of the deformation path). The samples were extruded at a temperature of 300°C with additional reversible rotation of the die at a frequency of 5 Hz at an angle of ± 8°. The diameter of the extruded rods was 6 mm. The unreinforced sintered copper samples were also fabricated under identical processing conditions.

Structural examinations of the specimens were performed using a Neophot 2 optical microscope and a JMS 5400 scanning electron microscope equipped with an ISIS 300 Oxford Instruments microprobe. The microhardness (*HV* 0.065) of the reinforcing particles and the Cu matrix were measured using a Hanemann microhardness tester mounted on the Neophot 2 microscope. The compression test was carried out at a room temperature using an INSTRON testing machine. Cubic samples with a dimension of 4 mm were machined from the extruded composite rods and unreinforced copper rods. The samples were compressed parallel and perpendicular to the extrusion direction at a strain rate of $4 \cdot 10^{-4}\,s^{-1}$.

a) b)

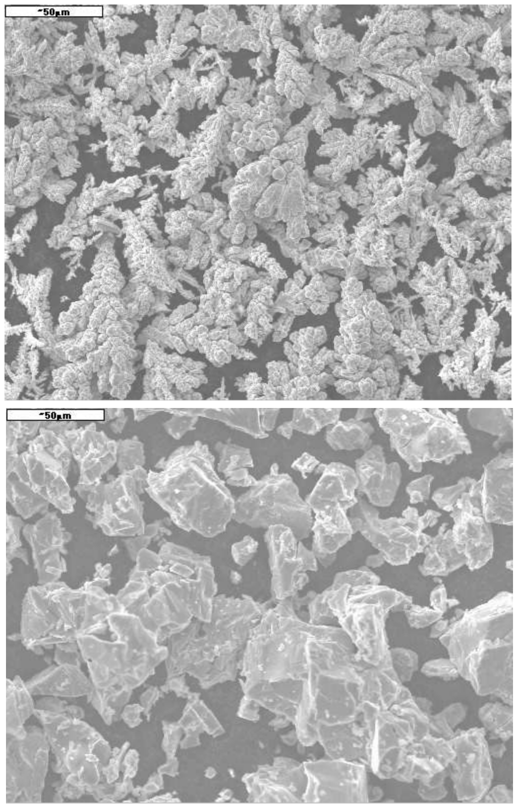

Fig. 1. SEM microstructure of powders: a) Cu powder, b) Ti powder

Rys. 1. Mikrostuktura proszków obserwowanych w mikroskopie skaningowym: a) proszek miedzi, b) proszek tytanu

RESULTS AND DISCUSSION

Figure 2 shows the distribution of the reinforcing particles synthesized from the powder mixture of Cu and Ti in the composite containing 5% Ti after sintering at a temperature of 850°C for 0.5 h. The reinforcing particles are distributed relatively uniformly in the copper matrix.

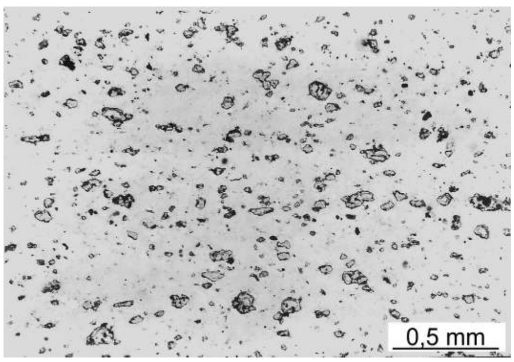

Fig. 2. Microstructure of the Cu-Ti powder containing 5% Ti after sintering at 850°C for 0.5 h

Rys..2. Mikrostruktura spieku Cu-Ti zawierającego 5% Ti. Spiekanie prowadzono w temperaturze 850°C w czasie 0,5 h

Figure 3 shows the microstructure of the reinforcing particle together with the profiles of the Cu and Ti concentration across the particle and Cu matrix. The products of the Ti-Cu reaction after sintering at a temperature of 850°C are located around the Ti particles not fully consumed in the course of the reaction at the Cu-Ti interface.

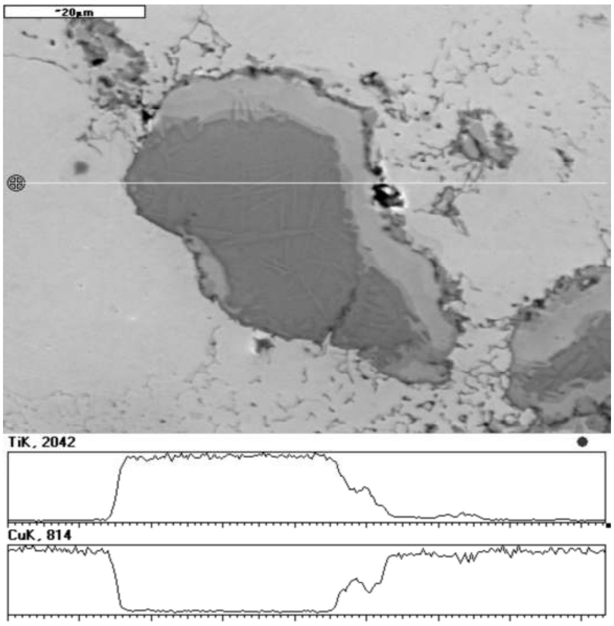

Fig. 3. Microstructure of particle as sintered from Cu and Ti powders and concentration of Ti and Cu across the particle

Rys. 3. Mikrostruktura cząstki, będącej produktem reakcji między proszkiem Ti a proszkiem Cu i analiza liniowa przez tę cząstkę

The composites contained pores which were particularly visible at the copper-reinforcing particles interface, and they were probably due to the powder metallurgical process.

Detailed observation shows that the reaction between the titanium particles and the copper matrix leads to the formation of intermetallic phases at the Ti-Cu interface. After sintering at 850°C for 0.5 h, the Ti-Cu reaction products were composed of intermetallic phases in the external zone (at the copper-titanium interface) and a core containing a solid solution of copper in titanium. Figure 4 shows the details of the structure presented in Figure 3. The chemical composition of the region marked by 1: 44.6% at. Ti, 55.4% at. Cu and the region marked by 2: 51.3% at. Ti, 48.7% at. Cu suggests that it is a CuTi intermetallic compound. The region marked by 3 contains: 70.98% at. Ti, 29.02% at. Cu. The Ti:Cu ratio does not very greatly from that of Ti_2Cu. The results of an X-ray microanalysis of the region marked by 4 containing 97.09% at. Ti, 2.91% at. Cu, suggest the presence of a solid solution of copper in titanium. The above results clearly indicate that the Ti particles were not fully consumed in the Ti-Cu reaction. The aim of the work was to prepare a composite characterized by high strength and good electrical conductivity. That is why it was essential to maintain the purity of the copper in the composite matrix. The EDS point analysis near the reinforcing particles shows that the depth of titanium diffusion into the copper matrix is about 20 μm.

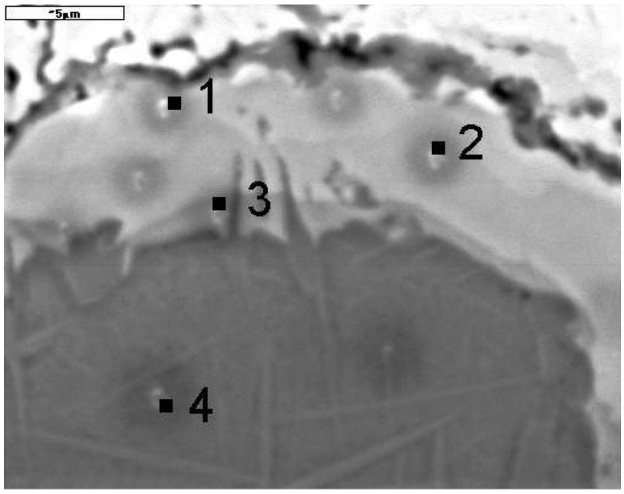

Fig. 4. Microstructure of Ti-Cu reaction zone

Rys. 4. Mikrostruktura w strefie rekcji Ti-Cu

The microhardness of the reinforcing particles measured after sintering was 760 HV0.65, while the microhardness of the copper matrix was 69 HV0.65.

The microstructural observations of the extruded composite reveal that the hard reinforcing particles of the intermetallic phases, initially spherical and equiaxised in shape, elongate in the extrusion direction. The microstructure of the longitudinal section of the Cu-5%wt. Ti composite after extrusion is shown in Figure 5 and Figure 6 shows the reinforcing particles elongated in the extrusion direction. It should be added that the characteristic feature of the composite matrix is a fine-grained structure (Fig. 6). Furthermore, good bonding between the copper matrix and the reinforcement was observed. The pressure applied during extrusion reduced the porosity of the powder compact.

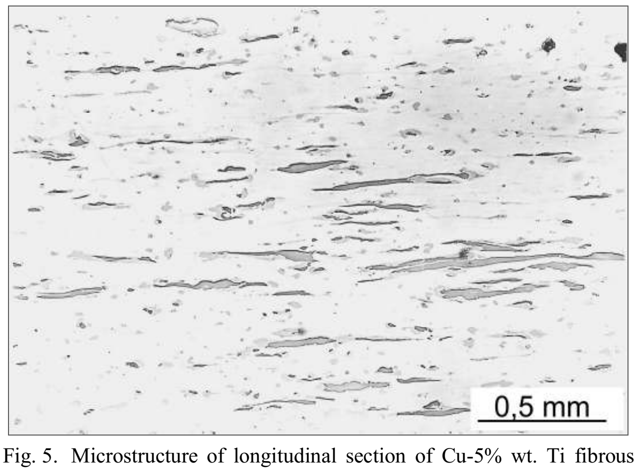

Fig. 5. Microstructure of longitudinal section of Cu-5% wt. Ti fibrous composite

Rys. 5. Mikrostruktura przekroju wzdłużnego kompozytu włóknistego Cu-5% Ti

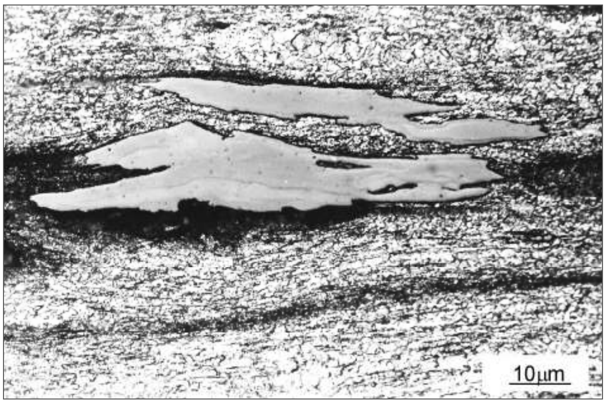

Fig. 6. Microstructure of reinforcing particles and copper matrix in etched composite. Plastic deformation of reinforcing particle in extrusion direction is observed

Rys. 6. Mikrostruktura cząstek wzmacniających i osnowy miedziowej po trawieniu. Widoczne odkształcenie plastyczne w kierunku wyciskania cząstek wzmacniających kompozyt

A compression test was carried out for the extruded samples to compare the mechanical properties of the composites and the sintered unreinforced copper. The mean values of the yield strength of the copper-intermetallic fibrous composites with different titanium content are summarized in Table 1.

TABLE 1. **Yield strength of copper-intermetallic fibrous composites**

TABELA 1. **Umowna granica plastyczności kompozytu włóknistego miedź-fazy międzymetaliczne**

Material	Compression parallel to extrusion direction	Compression perpendicular to extrusion direction
	$R_{0,2}$	
Cu-1wt.% Ti	115	118
Cu-2.5 wt.% Ti	212	162
Cu-5 wt.% Ti	381	243

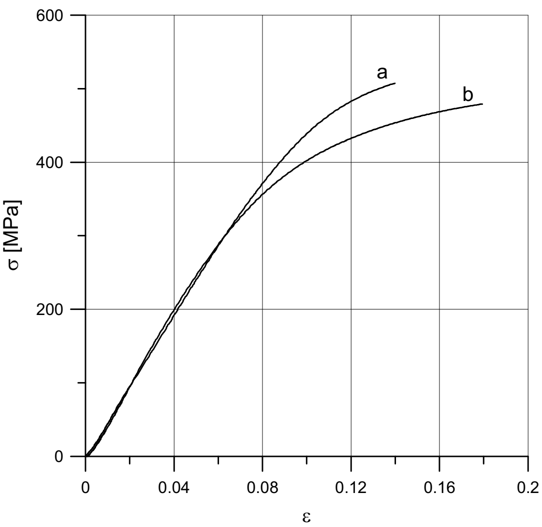

Fig. 7. True stress-strain curves obtained for Cu-5wt.% Ti composite: compression parallel to extrusion direction (a), compression perpendicular to extrusion direction (b)

Rys. 7. Krzywe naprężenie-odkształcenie rzeczywiste dla kompozytu Cu-5% Ti: próbka ściskana równolegle do kierunku wyciskania (a), próbka ściskana prostopadle do kierunku wyciskania (b)

It can be seen that the yield strength increases considerably with an increase in the Ti content. For the Cu-2.5wt.% Ti and Cu-5wt.% Ti composites mechanical anisotropy was observed. The yield strength was higher for the composites loaded parallel to the extrusion direction than for the composites loaded perpendicular to the extrusion direction. Figure 7 shows the true stress--strain curves for the Cu-5 wt.% Ti composite loaded parallel and perpendicular to the extrusion direction. The yield strength for the Cu-1wt.% Ti composite was similar in both directions. As shown in Table 1, the composite Cu-5wt.% Ti loaded parallel to the extrusion direction exhibits a yield strength of 381 MPa. The yield strength of the compression tested specimens of sintered unreinforced copper was 70 MPa.

CONCLUSIONS

1. The powder metallurgy method combined with extrusion of the sintered products can be successfully used for processing copper-intermetallic fibrous composites from Cu and Ti powders.
2. The reinforcing particles synthesised during sintering are composed of intermetallic phases in the external zone (at the matrix - Ti particle) and a core containing a solid solution of copper in titanium.
3. During extrusion, the hard reinforcing particles, initially spherical in shape, are plastically deformed in the extrusion direction, assuming a fibrous shape.
4. The yield strength of the copper-intermetallic fibrous composites Cu-2.5 wt.% Ti and Cu-5 wt.% Ti is several times higher than that of the unreinforced copper. The composites exhibit anisotropy of yield strength.

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