

FABRICATION OF CU-INTERMETALLIC FIBROUS COMPOSITES

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Abstract

The paper is concerned with two methods of fabrication Cu-matrix fibrous composites, their microstructure and properties. The composites were reinforced with particles synthesized from elemental Cu and Ti powders. The Cu powder mixed with 5 wt.% Ti powder was cold pressed. The next two steps of the fabrication process were: sintering and extrusion (method A) or extrusion and sintering (method B). In both cases, extrusion was performed by means of a KOBOL press. During sintering, the formation of hard particles containing intermetallic phases was a result of the reaction between the Cu powder and the Ti powder. The above methods can be used to produce composites with reinforcing particles of intermetallic phases elongated in the extrusion direction. It was found that the composite fabricated with method A with sintering followed by extrusion had lower porosity and higher yield strength.

Keywords: Powder metallurgy, Cu-matrix composites, Cu-Ti intermetallic phases, compression test

1. INTRODUCTION

Cu-matrix composite reinforced with non-continuous (short) fibers [1], continuous (long) fibers [2-5], nano fibers [6, 7] whiskers [8] and particulates [9, 10] can be produced using methods such as powder metallurgy, casting and mechanical alloying. In Cu-matrix composites, the reinforcement can be metallic or non-metallic. Combination of high strength and good electrical conductivity has made Cu-matrix composites attractive for a wide range of applications in the electrical industry. Conventional techniques are based on the addition of reinforcement to a Cu matrix while the latter is in a molten or powder state. In novel methods, on the other hand, the reinforcement is formed in a metallic matrix by a controlled reaction between the constituents during composite fabrications [11].

In this study a powder metallurgy method followed by plastic working was applied to fabricate of the Cu-matrix composites. The reinforcing particles in the Cu matrix were formed as a result of synthesis of phases that takes place during the sintering of the two metal powders (Cu and Ti). The binary phase diagrams of Cu alloys indicate that Ti is the alloying element used to obtain high-strength and high-conductivity Cu alloys. According to the Cu-Ti binary phase diagram [12], 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 compare two methods of fabrication Cu-matrix composites with reinforcing fibrous particles of Cu-Ti intermetallic phases elongated in one direction. The paper focused on the influence of the fabrication method on the microstructure and properties of the composites.

2. EXPERIMENTAL PROCEDURE

The Cu-intermetallic composites were fabricated using two methods based on powder metallurgy. The starting materials were Cu powder (with an average particle size of 50 μm) and Ti powder (with the particle size ranging 10-60 μm). The Cu powder particles had a dendritic shape, while the Ti powder showed an irregular shape. The powder morphologies are illustrated in **Fig. 1**.

Cu powder was blended with 5 wt.% Ti powder. The powder mixture was placed in a die (40 mm diameter) and cold pressed at 600 MPa using hydraulic press machine. In the first method (method A - **Fig. 2**), the

resulting cold pressed compacts were sintered at a temperature of 850 °C for 0.5 h. After sintering the samples were extruded applying the KOBO method. In the second method (method B - **Fig. 2**), the compacts after cold pressing were extruded (KOBO method) and then sintered at a temperature 850 °C for 0.5 h.

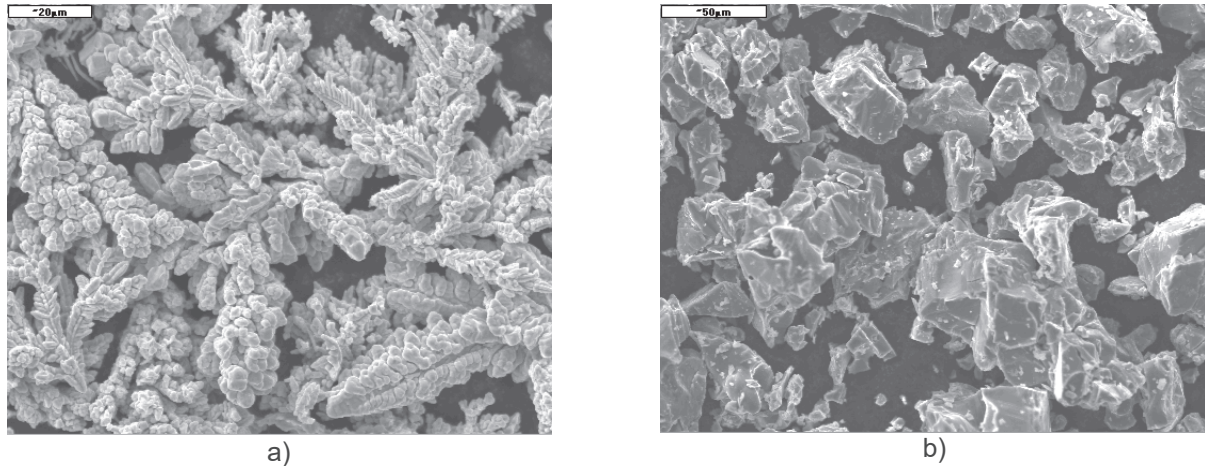


Fig. 1 SEM image of the powders: a) Cu, b) Ti

KOBO is an unconventional technique of metal forming used for materials that do not undergo deformation easily. This process combines conventional extrusion with cyclically reversible plastic twisting (cyclic change of the deformation path) [13]. The samples were extruded at 300 °C with additional reversible rotation of the die at a frequency of 5 Hz by an angle of $\pm 8^\circ$. The final diameter of the extruded rods was 6 mm.

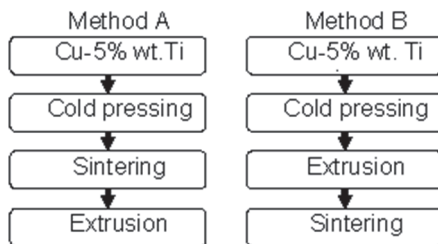


Fig. 2 The flowchart two fabrication procedures of Cu-intermetallic fibrous composites

The structural analysis of the specimens was performed using an OLYMPUS BX51M optical microscope, a JMS 5400 scanning electron microscope equipped with Oxford Instruments ISIS 300 energy dispersive X-ray analysis system. The compressive strength test was carried out at room temperature using an INSTRON universal testing machine. The cubic samples (4 mm side length) were deformed at a strain rate of $4 \cdot 10^{-4} \text{ s}^{-1}$.

3. RESULTS AND DISCUSSION

Fig. 3 shows the microstructure of the sintered Cu - 5 wt.% Ti compact (method A). The reinforcing particles synthesized from the powder mixture of Cu and Ti are uniformly distributed in the Cu matrix. Chemical reaction between the Cu matrix and the Ti particles during sintering at 850 °C for 0.5 h leads to the formation of intermetallic phases at the Cu-Ti interface. The reaction products are located around the Ti particles not fully consumed in the course of the reaction as shown in the high magnification SEM micrograph in **Fig. 4**. 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 Cu-Ti interface) and a core containing a solid solution of Cu in Ti. The regions marked 1 to 6 in **Fig. 4** were analysed by EDS. The chemical composition of the region marked by 1: 97.89 at.% Ti, 2.11 at.% Cu suggests the presence of a solid solution of Cu in Ti. The region marked by 2: 67.61 at.% Ti, 32.39 at.% Cu suggests that it is a Ti_2Cu intermetallic compound. The atomic ratios of Ti and Cu of the region marked by 3: 51.36 at.% Ti, 48.64 at.% Cu indicate CuTi according to the Cu-Ti binary diagram. The region marked 4: 45.65 at.% Ti, 54.35 at.% Cu suggests Cu_4Ti_3 intermetallic phase. The results of the X-ray microanalysis of

the region marked by 5 containing 18.58 at.% Ti, 81.42 at.% Cu suggests Cu_4Ti . These results are in agreement with those presented in the literature concerning the Ti/Cu interfacial reaction [14,15].

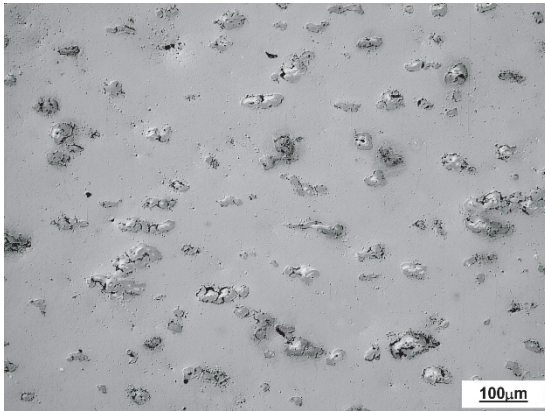


Fig. 3 Distribution of the reinforcing particles in the Cu-5 wt.% Ti compact after sintering at 850 °C for 0.5 h

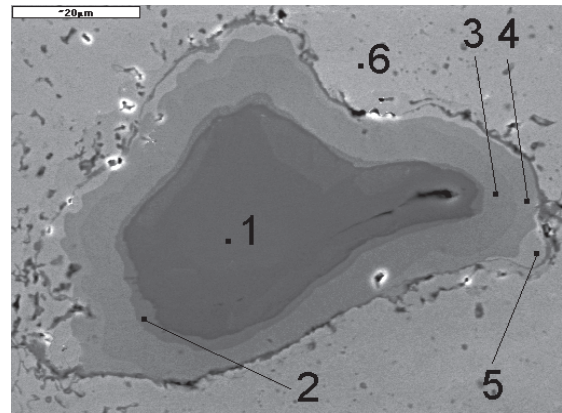
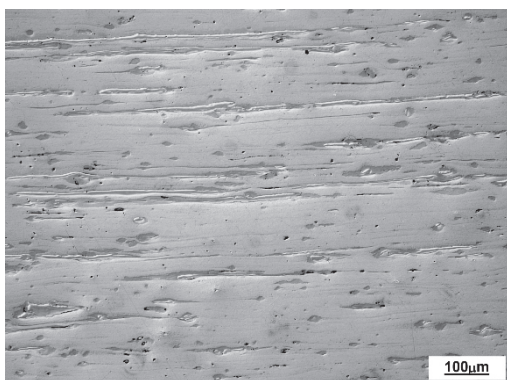


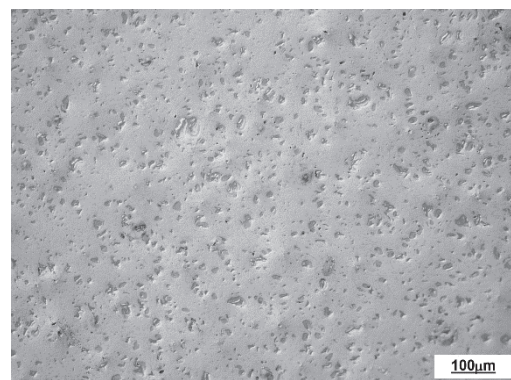
Fig. 4 SEM image of the reinforcing particles

The above results clearly indicate that the Ti particles were not fully consumed in the Ti-Cu reaction. The sintered compacts contained pores which were observed at the matrix-reinforcement interface and near the reinforcing particles. The EDS point analysis of the region marked 6 (near the reinforcing particles): 3.27 at.% Ti, 96.73 at.% Cu indicates that Ti diffused into Cu.

The last step in method A was extrusion using the KOBO method. **Fig. 5** shows optical micrographs of the longitudinal and transversal cross-sections of the extruded samples. The microstructural analysis of the extruded composite (**Fig. 5a**) reveals that the hard reinforcing particles of the Cu-Ti intermetallic phases, initially spherical and equiaxed in shape, elongate in the extrusion direction and take a fibrous form. The high magnification microphotograph of the extruded composites (the fiber cross-section) in **Fig. 6** shows good bonding between the matrix and the reinforcing particles. No cracks were observed in the reinforcing particles. From the observations it is evident that intermetallic phases are not too hard or brittle for extrusion KOBO method. The low porosity was observed in extruded composites.



a)



b)

Fig. 5 Microstructure of Cu - 5 wt.% Ti composite after extrusion (method A): a - longitudinal and b - transversal sections

In method B a compacts Cu - 5 wt.% Ti after cold pressing were extruded using KOBO method. **Fig. 7** shows the microstructure of the longitudinal section of the extruded rods. The Ti particles are elongated in the extrusion direction. Then the extruded rods were sintered at a temperature 850 °C for 0.5 h. **Fig. 8** shows of the longitudinal and transverse cross-sections of the extruded composite after sintering. Microstructural examinations reveal that intermetallic phases were formed at the Cu-Ti interface during sintering.

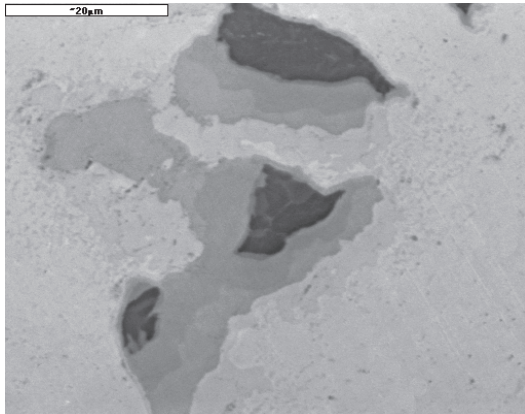


Fig. 6 Micrograph of the composite showing good bonding between the matrix and the particles- transverse section

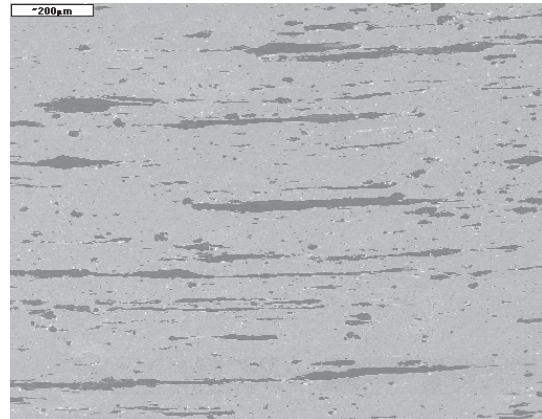
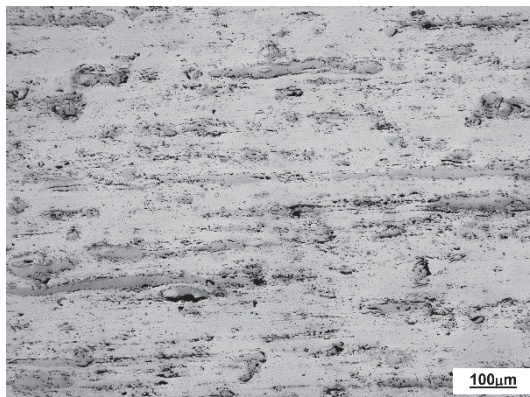
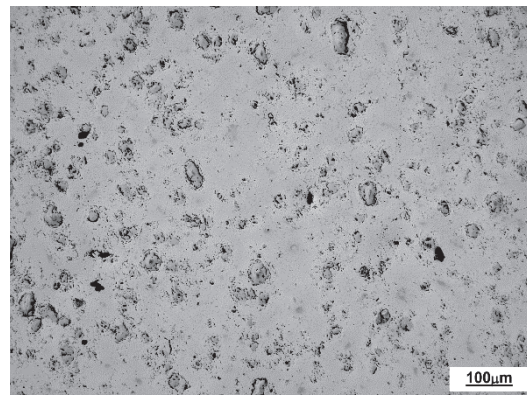


Fig. 7 Microstructure of the longitudinal section of the extruded rods (method B)



a)



b)

Fig. 8 Microstructure of the Cu - 5 wt.% Ti composite after sintering (method B): a) longitudinal, b) transversal sections

The microstructure of the reinforcing particles is presented in **Fig. 9**. EDS point analysis shows that the regions marked from 1 to 5 in **Fig. 9** contain 1: 98.15 at.% Ti, 1.85 at.% Cu, 2: 68.22 at.% Ti, 31.78 at.% Cu, 3: 51.36 at.% Ti, 48.64 at.% Cu, 4: 44.05 at.% Ti, 55.95 at.% Cu and 5: 21.85 at.% Ti, 78.11 at.% Cu respectively, which implies the following phases: a solid solution Cu in Ti, Ti_2Cu , $CuTi$, Cu_4Ti_3 and Cu_4Ti from the Ti side, respectively. The analysis at point 6 was: 4.04 at.% Ti, 95.96 at.% Cu. The main drawback of method B is high porosity of the final composites. The porosity was observed near the reinforcing particles and in the Cu matrix. The high magnification observations revealed a high level of porosity in the Cu matrix (**Figs. 9** and **10**). The microstructural analysis shows that these pores are surrounded by Ti rich intermetallic phases. The regions marked in **Fig. 10** contained 1: 54.56 at.% Ti, 45.44 at.% Cu, 2: 31.36 at.% Ti, 68.64 at.% Cu, 3a: 2.99 at.% Ti, 97.01 at.% Cu and 3b: 2.20 at.% Ti, 97.80 at.% Cu.

Microstructural analysis of the composites fabricated with method B suggests that this method can also be used to obtain Cu-matrix composite with reinforcing particles of Cu-Ti intermetallic phases elongated in one direction. However, this composite contains porosity and the reinforcing particles are less coherent.

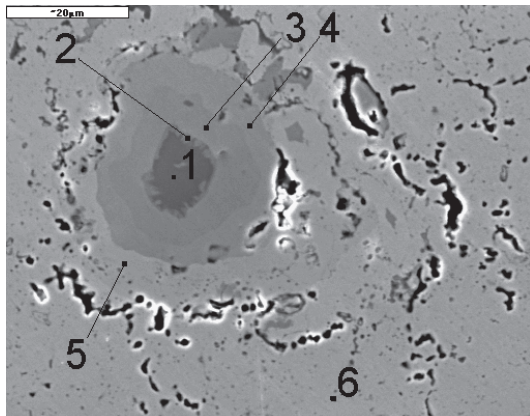


Fig. 9 Microstructure of the reinforcing particles - transverse section (method B)

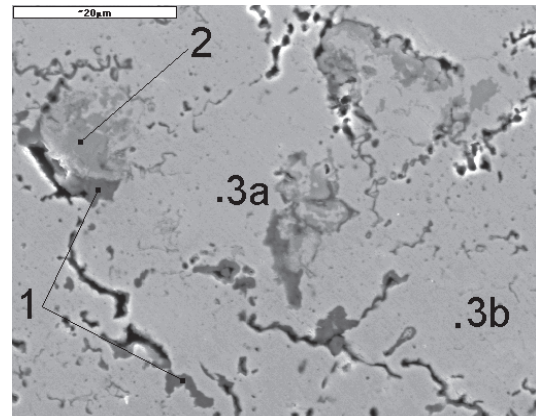


Fig. 10 Porosity in the Cu matrix (method B)

Compressive strength tests were carried out to study the effects of the fabrication method on the mechanical properties of the composites. The mean values of the yield strength of the Cu-intermetallic fibrous composites fabricated with the two methods are presented in **Table 1**. For the composites mechanical anisotropy was observed. The yield strength was higher for the composites loaded parallel to the direction of extrusion than for the composites loaded perpendicular to the direction of extrusion. The yield strength of the composites fabricated with method A measured in both directions was 40% higher than that prepared using method B. **Fig. 11** shows the true stress-strain curves for the Cu - 5 wt.% Ti composite, obtained with method A. **Fig. 12** demonstrates the true stress-strain curves for the Cu - 5 wt.% Ti composite, obtained with method B. The yield strength of the compression tested specimens of sintered unreinforced copper was 70 MPa.

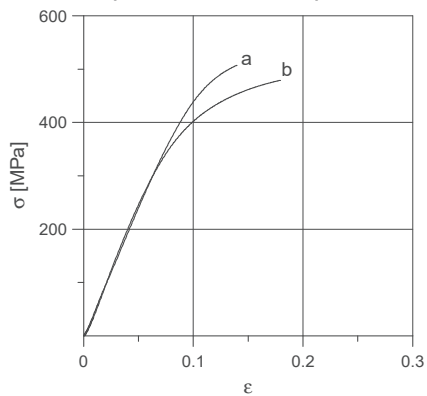


Fig. 11 True stress-strain curves obtained for composite (method A): compression parallel to the extrusion direction (a) compression perpendicular to the extrusion direction (b)

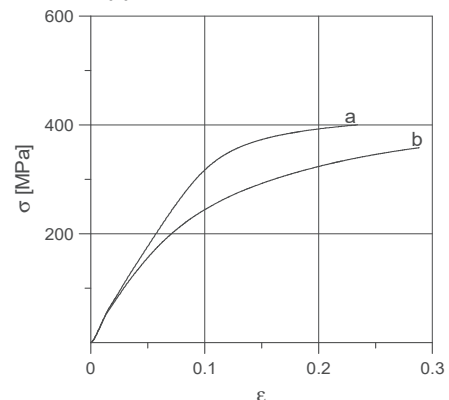


Fig. 12 True stress-strain curves obtained for composite (method B): compression parallel to the extrusion direction (a) compression perpendicular to the extrusion direction (b)

From the experiments it is clear that the mechanical properties of the Cu-intermetallic fibrous composites are greatly affected by the fabrication method.

Table 1 The mechanical properties of Cu-intermetallic composites made by two methods

Material	Compression parallel to extrusion direction	Compression perpendicular to extrusion direction
	$R_{p0.2}$ (MPa)	
Cu - 5 wt.% Ti. Method A	381	243
Cu - 5 wt.% Ti. Method B	236	143

CONCLUSIONS

The experimental results show that method A (blending → cold pressing → sintering → extrusion) is more suitable for the fabrication of Cu-intermetallic fibrous composites than method B (blending → cold pressing → extrusion → sintering). Composites produced with method A are characterized by:

- good bonding between the matrix and the reinforcing particles,
- low porosity,
- higher yield strength.

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