

## CONSOLIDATION AND PROPERTIES OF Cu-TiC COMPOSITE BY A REDUCTION SINTERING AND COLD EXTRUSION PROCESS

H. L. Vu<sup>1,2</sup>, L. Q. Tran<sup>1</sup>, B. N. Duong<sup>1</sup>, K. Q. Dang<sup>1\*</sup>

<sup>1</sup>*Department of Non-ferrous Metal Materials and Composite, Hanoi University of Science and Technology, No. 1, Dai Co Viet Street, Hanoi, Vietnam*

<sup>2</sup>*Faculty of Mechanical Engineering, Thai Nguyen University of Technology, 3-2 Street, Thai Nguyen, Vietnam*

*\*Email: khanhdq-fmmt@mail.hut.edu.vn*

**Keywords:** Cu-TiC, sintering, cold extrusion.

### Abstract

*In the present study, titanium carbide (TiC) reinforced copper (Cu) composite was fabricated via powder metallurgy route. Cu-TiC powder mixture was prepared by a high-energy planetary ball milling process for 2h with TiC content varied from 1 to 5 wt%. The powder mixtures were then cold compacted at 200 MPa and sintered at 900 °C for 2h. After sintering, sample were subjected to cold extrude and then characterized. The results obtained show that relative density decreases when TiC content increased. Result also shows significant improvement of wear resistance at higher TiC addition. An enhancing in tensile strength was observed at less than 2 wt% TiC, larger dose reduces tensile strength of the material. The higher TiC content, the lower electrical conductivity was recorded. Cold extrusion had improved mechanical properties and electrical conductivity of the material.*

### 1 Introduction

Copper is an attractive metal for a wide range of applications because of its high electrical and thermal conductivity. However, copper is a soft metal and possesses poor strength at elevated temperature [1-8]. Copper alloys like brass and bronze have better mechanical properties but also suffer from problems such as corrosion [9] and inferior high temperature properties due to the coarsening of the less stable microconstituents [2, 8]. Thus, the improvement of the elevated temperature properties of Cu and its alloys without adversely affecting their high thermal, electrical conductivities and corrosion resistance remains as an important issue.

Composite material with copper matrix and ceramic particle reinforcement provide basis for producing relatively high hardness and high electrical conductivity materials. Several research works on copper based composite has involved transition metal carbide reinforcement, which is introduced in the copper matrix through a powder metallurgy route. Copper matrix composites, such as Al<sub>2</sub>O<sub>3</sub>-Cu, SiC-Cu, TiC-Cu, WC-Cu, B<sub>4</sub>C-Cu, TiB<sub>2</sub>-Cu and so on [10-14], have been widely investigated. Among those reinforcements used, TiC is an attractive candidate to reinforce copper matrix due to its high modulus, hardness and melting temperature, and moderate electrical conductivity. Additional of TiC in copper matrix does not induce any detrimental effect on physical and electrical properties of the resulted Cu-TiC

composite [9]. The synthesis of TiC reinforced copper matrix composite has attracted numerous researchers over the years. Makoto et al. [15] has synthesized TiC-Cu composites by combustion synthesis in Cu-Ti-C System. The results showed that TiC-Cu composite with 20-80% TiC could be also fabricated by direct heating the powder mixture of Cu-Ti-C. Cu-TiC composite could be also synthesized by in situ method such as mechanical alloying, self-propagating high temperature synthesis or selective laser melting. The mixture of Cu, Ti and C powder was grinded in a high energy ball mill and then sintered [16] or scanned under laser beam [17] to in situ synthesis TiC in copper matrix. Akhtar et al. [18] attempted to fabricate Cu-based TiC reinforced composites with a high volume fraction of TiC particles (69 vol.% to 77 vol.%) and some alloying elements (Al, Ti, Ni and Co) were added to enhance the sintering and mechanical properties. However, until now, there is not any report on consolidation of Cu-TiC composite with small amount of TiC content (lower than 5 wt%) via powder metallurgy route. Powder metallurgy has been used for several years as a low-cost fabrication method for producing near-net shape composites. Thus, powder metallurgy is a cost-effective method to produce TiC-Cu matrix composites. In this study, Cu-TiC composite with small amount of TiC content (1 - 5 wt%) was fabricated via powder metallurgy route. Cold extrusion has been used to obtain a relatively dense Cu-TiC composite material. The electrical conductivity, mechanical properties and wear behaviour were investigated.

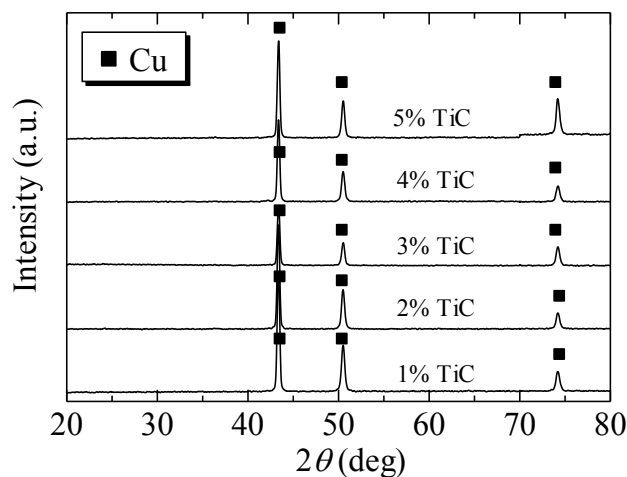
## **2 Experimental procedures**

Commercial copper powder (99,5%, Guangdong Guanghua Chemical Factory Co. Ltd., China) and TiC powder (98%, particle size of 325 mesh, Sigma-Aldrich Co., USA) were used as a starting materials. The copper and the reinforcement powders were firstly mixed in a high-energy planetary ball milling for 5h. The ball/powder weight ratio was 10:1 and the rotation speed was 150 rpm. The mixing powders with different amount of TiC content were put in stainless steel die ( $\phi 40 \times (\phi 10) \times 20$  mm) then cold compacted at 200 MPa. The reduction sintering was conducted by the muffle furnace (Linn High Therm, Germany) for evaluating an advantage of sintering process. A Cu-TiC powder mixture was sintered at sintering temperature of 900°C with heating rate of 10 K/min for dwelling time of 2h in carbon. The final rod-like samples were approximately 10 mm in diameter. After sintering, samples were subjected to several steps of cold extrusion until the samples have an average size of 6.5 mm in diameter.

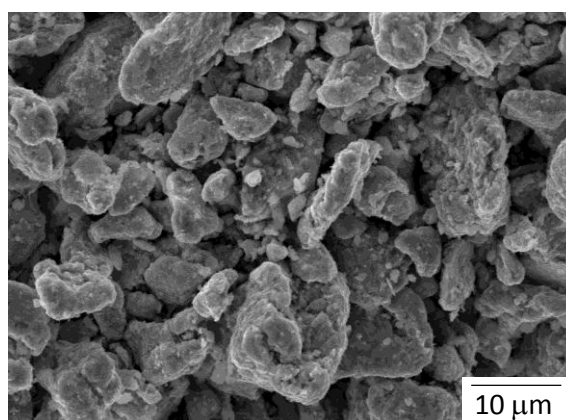
The powders were then characterized by X-ray diffraction (XRD, D5005, Siemens, Germany) and scanning electron microscopy (SEM, JSM 6490, JEOL, Japan). Bulk density of sample was measured according to the liquid displacement technique with distilled water. The relative density was calculated from the measured bulk density to the theoretical density. An MTS 809 axial testing (USA) was used to measure tensile and compression strength of the sample. Wear resistant and electrical properties were conducted at room temperature using a Tribotester (France) and Lakeshore HR6000 (USA) equipments, respectively.

## **3 Results and discussion**

Figure 1 shows XRD patterns of the Cu-TiC powder mixture with various amount of TiC content for 5h. The sample phase was obtained after mixing process. No contamination or other chemical reaction has occurred. Figure 2 shows the microstructure of the Cu-TiC powder mixture made by planetary ball milling. The microstructural observation of the powder mixture shows good agglomeration.



**Figure 1.** XRD pattern of Cu-TiC powder mixture.

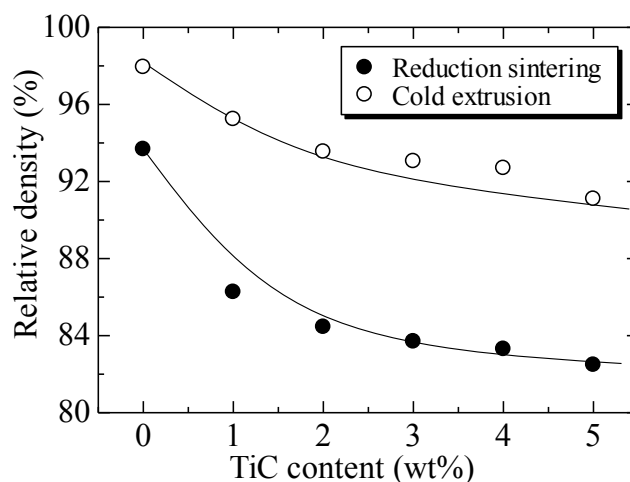


**Figure 2.** Microstructure of the Cu-TiC powder mixture after mixing process.

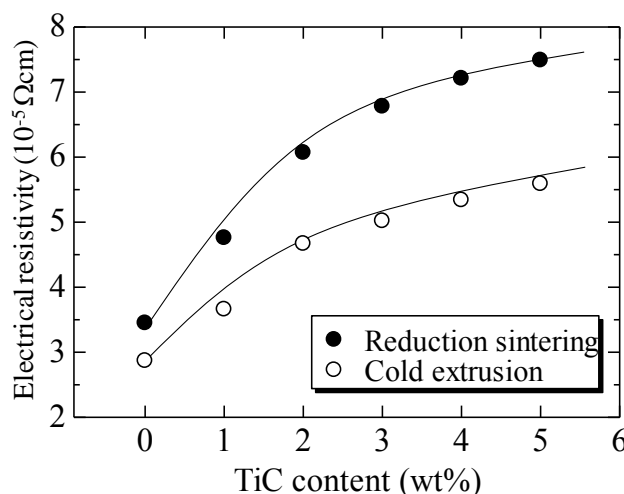
The relationship between the relative density of sintered body and an amount of TiC content is shown in Figure 3. The relative density is decreased with increasing TiC content. The results also show that the relative density of sample subjected to cold extrusion has significantly improved. At all TiC contents, relative densities of above 90% are obtained. Larger TiC content has showed significant effect on relative density of the sample. The lower relative density is obtained at the higher TiC content. It can be explained by the comparison between the natural stiffness of TiC and a relatively soft copper matrix. The addition of fined TiC powder into copper powder somewhat decreases the powder compressibility and thus, results in lower relative density.

Figure 4 shows the relationship between TiC content and electrical resistivity of the sample for both of reduction sintering and cold extrusion process. The higher TiC dose, the higher electrical resistivity were recorded. At the same TiC content, the electrical resistivity of cold extruded sample was higher than that in the reduction sintered sample. The primary reason is that the homogeneously distributed electrically conductive TiC phase in the sample also has an impact. An increase of electrical resistivity in the sample was caused by the larger electrical resistivity of TiC compared to copper. Additionally the clean interface between TiC grains reinforced Cu-matrix also improved the electrical conductivity. Results obtained show a double of electrical resistivity when TiC contents rise from 1% to 5%. Furthermore the higher electrical resistivity could be explained by the lower density of the sample. As

discussed above, higher TiC dose decreases density of the sample and thus, increases electrical resistivity. It proves that the effect of cold extrusion process on electrical resistivity is greater than that of the reduction sintering.



**Figure 3.** The relationship between the relative density of sintered body and an amount of TiC content.



**Figure 4.** The relationship between TiC content and electrical resistivity of the Cu-TiC sample.

The dependence of the TiC content on the tensile strength of sample is presented in Figure 5. The addition of TiC into copper matrix has significantly improved the matrix strength. As at 1% TiC, tensile strength of the sintered sample was around 180 MPa and increased to 220 MPa in extruded sample. Before cold extrusion, tensile strength of the sample has peaked at 1% TiC, higher TiC contents do not show any improvement. In theory, the sample with higher TiC content should have higher tensile strength since TiC has better tensile strength compared to copper. The first sample at 1% TiC has agreed with this trend. However, when TiC content increased above 1%, tensile strength decreased slightly after showing a maximum at 1%. This might be caused by the extra additive making compressibility and sinterability of powder mixture decreased. Lower TiC content sample is easier to compact, as can be seen in Figure 3, the lower TiC content sample has better relative density than others. As depicted in Figure 5, tensile strength of extruded sample is higher than those formed by reduction sintering. The highest value was recorded at 2% of TiC content. Cold extrusion has improved relative density, thus enhancing tensile strength of the material.

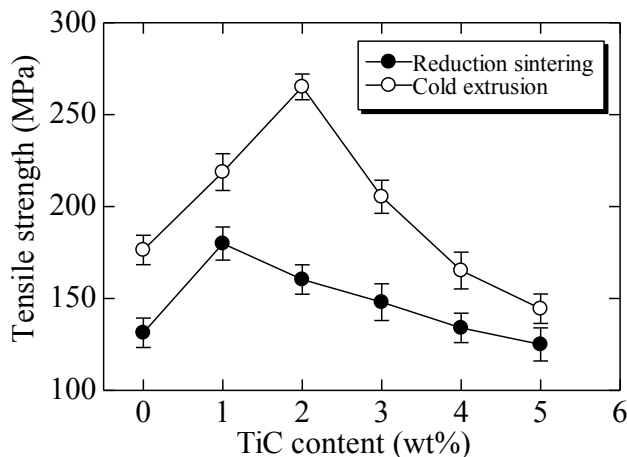


Figure 5. The dependence of the TiC content on the tensile strength of sample.

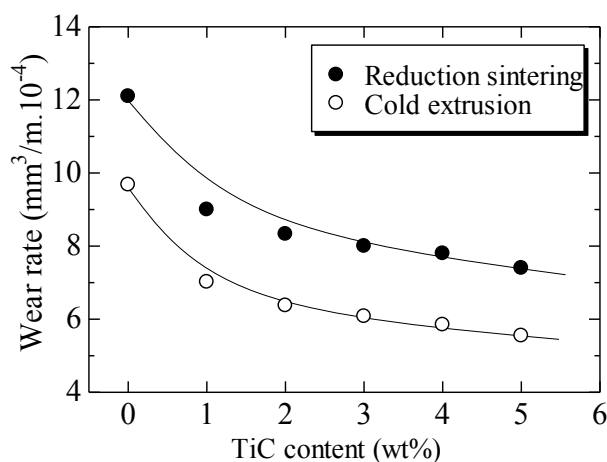


Figure 6. The wear resistance of Cu-TiC sample with various TiC content.

The variation of wear rate with TiC content of the sample at applied load of 18 N is shown in Figure 6. It has been observed that the wear rate reduces with TiC content up to 5 wt% under different process used in the current study. The results also show that the wear resistance of sample subjected to cold extrusion has lower than that of reduction sintering at the same TiC content. During sliding, the hard TiC particles do not easily come out in the debris because of their reasonably good bonding with the matrix. Furthermore, perhaps the formation of oxide layer appeared on the wear surface of the sample and reduced wear resistance. The cold extruded Cu-TiC sample had the best wear resistance of all the Cu-TiC considered herein.

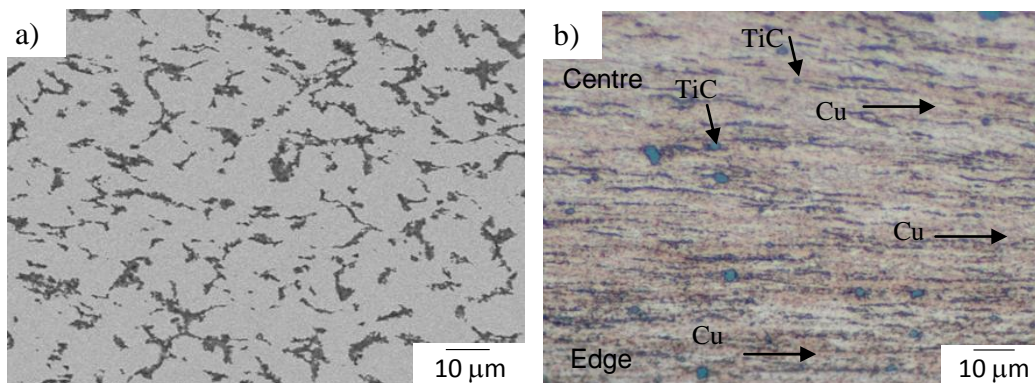


Figure 7. SEM images of cross section surface of Cu - 2%TiC sample: a) Reduction sintering; b) Cold extrusion.

Figure 7(a) displays SEM image of the microstructures of cross section surface of Cu-TiC sample with 2 wt% TiC content after sintering. The results gave direct evidence for the high concentration and homogenous of TiC particles in microstructure following that mechanical properties improved. This is a main reason for reducing mechanical properties which come from enhancing of porosity. As depicted in figure 7(b), cold extrusion resulted in the shape of copper grains changed. Long and narrow copper grains were found close to the edge while larger grains were observed at the centre. It can be also seen from these micrographs that TiC reinforcing particles reside in the sample after extruding. No particle cracking and separation of particle from the matrix can be observed. Small TiC particles were homogeneously distributed in the copper matrix. By decreasing the particle size, bonding takes place better and the wear resistance increases. It is clear that cold extrusion offers significant advantages over conventional reduction sintering for the densification and properties of Cu-TiC sample.

#### 4 Conclusions

The Cu-TiC powder mixture with various TiC content consolidated by reduction sintering and cold extrusion process were investigated. The final densification and their properties resulting from cold extrusion had in all cases greater influence than those resulting from the reduction sintering. When a small amount of TiC was added and the powder mixture was sintered at 900°C for 2h and then cold extruded, the wear resistance of Cu-TiC sample was significantly improved. Furthermore, by increasing TiC content higher than 2 wt%, the tensile strength was reduced. It was caused by extensive concentration of TiC particles in the sample. The cold extrusion process is a promising process for consolidating high densified Cu-TiC composite without resorting to specialized powders or complicated sintering temperature.

#### References

- [1] Chrysanthou A., Erbaccio G. Enhancing the dispersion of TiC in copper. *J. Mater. Sci. Lett.*, **15**, pp. 774-775 (1996).
- [2] Chrysanthou A., Erbaccio G. Production of Copper-Matrix Composites by In Situ Processing. *J. Mater. Sci.*, **30**, pp. 6339-6344 (1995).
- [3] Besterci M., Ivan J., Kovac L., Weissgaerber T., Sauer C. Strain and Fracture Mechanism of Cu-TiC. *Mater. Lett.*, **38**, pp. 270-274 (1999).
- [4] Zarrinfar N., Kennedy A.R., Shipway P.H. Reaction synthesis of Cu-TiC<sub>x</sub> master-alloys for the production of copper-based composites. *Scripta Mater.*, **50**, pp. 949-952 (2004).
- [5] Liang Y.H., Wang H.Y., Yang Y.F., Wang Y.Y., Jiang P.C., Evolution process of the synthesis of TiC in the Cu-Ti-C system. *J. Alloys Compd.*, **452**, pp. 298-303 (2008).
- [6] Xu Q., Zhang X., Han J., He X., Kvanin V.L. Combustion Synthesis and Densification of Titanium Diboride-Copper Matrix Composite. *Mater. Lett.*, **57**, pp. 4439-4444 (2003).
- [7] Tu J.P., Wang N.Y., Yang Y.Z., Qi W.X., Liu F., Zhang X.B., Lu H.M., Liu M.S. Preparation and properties of TiB<sub>2</sub> nanoparticle reinforced copper matrix composites by in situ processing. *Mater. Lett.*, **52**, pp. 448-452 (2002).
- [8] Dudina D., Kwon D.H., Huynh K.X., Nguyen T.D., Kim J.S., Kwon Y.S. *Nanoscale TiB<sub>2</sub>-Dispersed Cu-matrix composite produced by a high-energy milling and self-propagating high-temperature Synthesis Process* in "Proceedings of the 9th Russian-Korean International Symposium", KORUS, Novosibirsk State Technical University, Russia, (2005).
- [9] Rathod S., Modi O.P., Prasad B.K., Chrysanthou A., Vallauri D., Deshmukh, Shah A.K. Cast in situ Cu-TiC composites: Synthesis by SHS route and characterization. *Mater. Sci. Eng. A*, **502(1-2)**, pp. 91-98 (2009).

- [10] Park J.Y., Oh S.J., Jung C.H., Hong G.W., Kuk I.H. Al<sub>2</sub>O<sub>3</sub>-dispersed Cu Prepared by the Combustion Synthesized Powder. *J. Mater. Sci. Lett.*, **18**, pp. 67-70 (1999).
- [11] Kwon Y.J., Makoto K., Choh T., Naoyuki K. Fabrication of TiC/Cu Composites by Combustion Synthesis in Cu-Ti-C System. *J. Jpn Inst. Metals*, **65(4)**, pp. 273-278 (2001).
- [12] Baikalova Y.V., Lomovsky O.I. Solid state synthesis of tungsten carbide in an inert copper matrix. *J. Alloys Compd.*, **297**, pp. 87-91 (2000).
- [13] T. Maruyama, S. Onose, Fabrication and Thermal Conductivity of Boron Carbide/Copper Cermet, *J. Nuclear Sci. Tech.*, **36(4)**, pp. 380-385 (1999).
- [14] Lee J., Kim N.J., Jung J.Y., Lee E.S., Ahn S. The influence of reinforced particle fracture on strengthening of spray formed Cu-TiB<sub>2</sub> composite. *Scripta Mater.*, **39(8)**, pp. 1063-1069 (1998).
- [15] Makoto K., Takao C., Naoyuki K., Kwon Y.J. Fabrication of TiC/Cu Composites by Combustion Synthesis in Cu-Ti-C System. *J. Jpn Inst. Metals*, **65(4)**, pp. 273-278 (2001).
- [16] Nur Hawadah M.S., Zuhailawati H., Othman R. Formation of titanium carbide reinforced copper matrix composite by in situ processing. *Adv. Mater. Res.*, **173**, pp. 111-115 (2011).
- [17] Lu L., Fuh J.Y.H., Chen Z.D., Leong C.C., Wong Y.S. In situ formation of TiC composite using selective laser melting. *Mater. Res. Bulletin*, **35(9)**, pp. 1555-1561 (2000).
- [18] Akhtar F., Askari S.J., Shah K.A., Du X., Guo S. Microstructure, mechanical properties, electrical conductivity and wear behavior of high volume TiC reinforced Cu-matrix composites. *Mater. Charact.*, **60**, pp. 327-336 (2009).