



# Synthesis and thermal behavior of Cu/Y<sub>2</sub>W<sub>3</sub>O<sub>12</sub> composite

Satyabati Das, Siddhartha Das, Karabi Das\*

Department of Metallurgical and Materials Engineering, Indian Institute of Technology-Kharagpur, Kharagpur 721302, India

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## Abstract

Cu metal matrix composite with Y<sub>2</sub>W<sub>3</sub>O<sub>12</sub> as a thermal expansion compensator was fabricated by high energy ball milling followed by compaction and sintering, and its thermal properties were explored for the potential applications as heat sinks in electronic industries, high precision optics, and space structures. The volume fraction of reinforcement was varied from 40% to 70% in order to tailor the composite for the simultaneous accomplishment of low thermal expansion and high thermal conductivity. The synthesis technique was optimized by varying the parameters like milling time from 1 to 20 h and sintering temperature from 600 to 1000 °C in order to achieve densified composites. The relative density of the composites is found to be around 90% for the 10 h milled powders followed by compaction at a pressure of 700 MPa and sintering at a temperature of 1000 °C. The thermal expansion of the composites exhibits linear behavior in the temperature range 200 to 800 °C and the low coefficient of thermal expansion (CTE) is found to be for Cu–70%Y<sub>2</sub>W<sub>3</sub>O<sub>12</sub> composite whose value,  $4.32 \pm 0.75 \times 10^{-6}/^{\circ}\text{C}$ , matches with that of Si substrate. The thermal conductivities are found to increase with a decrease in the volume fraction of the reinforcement and decrease with an increase in the temperature for all the samples. The experimentally determined CTE and thermal conductivity values are found to be comparable to those predicted by the thermal expansion based Kerner and Turner model and the thermal conductivity based Maxwell model, respectively. © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

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## 1. Introduction

Thermal expansion is a crucial property in many materials, as mismatch in thermal expansion can cause stress, fracture and detachment at interfaces resulting in failure of engineering applications. Therefore, tailoring of materials with a specific coefficient of thermal expansion (CTE) anywhere between the values of the pure components of the composite, i.e., positive, negative or zero is extremely essential in many fields such as in fiber optics as packaging material for refractive index grating, high precision optical mirrors, dental fillings, etc. In electronics industry, heat sinks that match the thermal expansion of Si chip or alumina substrate are indispensable to avoid the damage caused at the interfaces [1]. Materials having near zero thermal expansion which would be able to sustain the large thermal gradient and would have high thermal shock fracture resistance are the most challenging ones to be developed. Recently negative thermal expansion (NTE) materials have made a

significant contribution to the scientific and technological community since it can be used as thermal expansion compensator in metal matrix composite [2–5]. Furthermore, metal matrix composites having high thermal conductivity and low thermal expansion is essential due to their potential applications as heat sinks in electronic packaging, high precision optics, and space structures, etc.

Recent investigations demonstrate tunable thermal expansion in various composites having NTE material such as ZrW<sub>2</sub>O<sub>8</sub>, Sc<sub>2</sub>W<sub>3</sub>O<sub>12</sub>, etc. and positive thermal expansion materials as Al, Cu, Mg, ZrO<sub>2</sub>, etc. [6–8]. Tani et al. [9] have reported that by increasing Zr<sub>2</sub>WP<sub>2</sub>O<sub>12</sub> volume fraction from 0 to 75% in ZrW<sub>2</sub>O<sub>8</sub>/Zr<sub>2</sub>WP<sub>2</sub>O<sub>12</sub> composite, the CTE of the composite increases from  $-9.1 \times 10^{-6}$  to  $-3.1 \times 10^{-6} \text{ K}^{-1}$  and from  $-5.0 \times 10^{-6}$  to  $-1.9 \times 10^{-6} \text{ K}^{-1}$  over the temperature ranges of 323–373 and 473–673 K, respectively. Cu/ZrW<sub>2</sub>O<sub>8</sub> composite has been fabricated and is reported to exhibit a change in CTE value from about temperature 120 and 160 °C [1,10]. The anomaly in result has been explained by Yilmaz et al. [11] as due to the stress induced phase transformation of  $\alpha$  or  $\beta$ -ZrW<sub>2</sub>O<sub>8</sub> to  $\gamma$ -ZrW<sub>2</sub>O<sub>8</sub> phase (above 423 K). Many other NTE compounds are also reported to exhibit

\*Corresponding author. Tel: +91 9836895720; fax: +91 3222 25503.

E-mail address: [karabi@metal.iitkgp.ernet.in](mailto:karabi@metal.iitkgp.ernet.in) (K. Das).

phase transitions [12–14]. Therefore to overcome the problem of abrupt change of thermal expansion in composites due to phase transition,  $Y_2W_3O_{12}$  may be chosen as the NTE candidate from  $A_2M_3O_{12}$  ( $A$ -trivalent, and  $M$  is  $W^{+6}$  or  $Mo^{+6}$ ) family. This is because,  $Y_2W_3O_{12}$  has more stable phase at room temperature as compared to  $ZrW_2O_8$  and shows no structural phase transition over a wide range of temperature [15]. It has the highest average linear NTE coefficient  $\alpha$  ( $-7.0 \times 10^{-6} K^{-1}$ ) from 15 to 1373 K. This NTE behavior is due to the transverse thermal vibrations of oxygen atom, in  $Y-O-W$  chain, which causes the decrease in gap between ‘ $Y$ ’ and ‘ $W$ ’ atoms and as a result there is an overall lattice shrinkage [16]. At room temperature,  $Y_2W_3O_{12}$  has an orthorhombic structure belonging to  $Pnca$  space group with  $a=10.098 \text{ \AA}$ ,  $b=13.315 \text{ \AA}$  and  $c=9.691 \text{ \AA}$  [17]. Near zero thermal expansion of the order of  $-0.06 \times 10^{-6} \text{ }^\circ\text{C}$  is observed in  $ZrSiO_4/Y_2W_3O_{12}$  composite in the temperature range 25 to  $1000 \text{ }^\circ\text{C}$  [18]. Cu has been chosen as a metal matrix, because it has the second highest thermal conductivity ( $397 \text{ W m}^{-1} K^{-1}$ ) among all the metals and hence has many potential applications in electronics industry. Therefore, this work explores the feasibility of processing a densified  $Cu/Y_2W_3O_{12}$  composite having a low thermal expansion along with good thermal conductivity.

## 2. Experimental procedure

Stoichiometric amounts of  $Y_2O_3$  (99.92%) and  $WO_3$  (99.61%) powders were milled by using a Fritsch Vario-Planetary Ball Mill (pulverisette-4) machine for 10 h. The transmission ratio, ball-to-powder ratio and milling speed were fixed at  $-2.25$ ,  $10:1$ ,  $300 \text{ rpm}$ ,

respectively and the toluene was used as process controlling agent. It was subsequently heat treated at  $800 \text{ }^\circ\text{C}$  to form  $Y_2W_3O_{12}$ . The details about the synthesis and characterization of  $Y_2W_3O_{12}$  have been reported elsewhere [19]. The  $Y_2W_3O_{12}$  powders, thus formed, were mixed with Cu powder by using ball milling. The above mentioned milling parameters were used for milling of  $Cu/Y_2W_3O_{12}$  powder with different milling time such as 1, 5, 10 and 20 h. The volume fraction of  $Y_2W_3O_{12}$  was varied from 40% to 70% with an interval of 10% and the respective mixtures were abbreviated as Cu60, Cu50, Cu40 and Cu30 accordingly. The prepared powder mixtures of  $Y_2W_3O_{12}$  and Cu were shaped into 13 mm diameter and 3 mm thick compacts by uniaxial pressing at 700 MPa and then sintered at temperatures of 600, 800 and  $1000 \text{ }^\circ\text{C}$  for 1 h in vacuum. The sintered samples were examined by X-ray diffractometer (XRD), using  $Cu K\alpha$  radiation ( $\lambda=1.5406 \text{ \AA}$ ). The surface morphology of the synthesized powders and sintered compacts was investigated by using a field emission scanning electron microscopy (FE-SEM, Zeiss Supra 40 and FEI). The thermal expansion measurement was carried out using a Perkin Elmer diamond thermo mechanical analyzer (TMA) at a heating rate of  $10 \text{ }^\circ\text{C}/\text{min}$ . Thermal diffusivity was measured by the laser flash method using a LFA457 machine (Netzsch).

## 3. Results and discussion

### 3.1. Characteristics of milled powder

The initial intent is to refine and homogenize the Cu and  $Y_2W_3O_{12}$  powders through ball milling so as to improve the

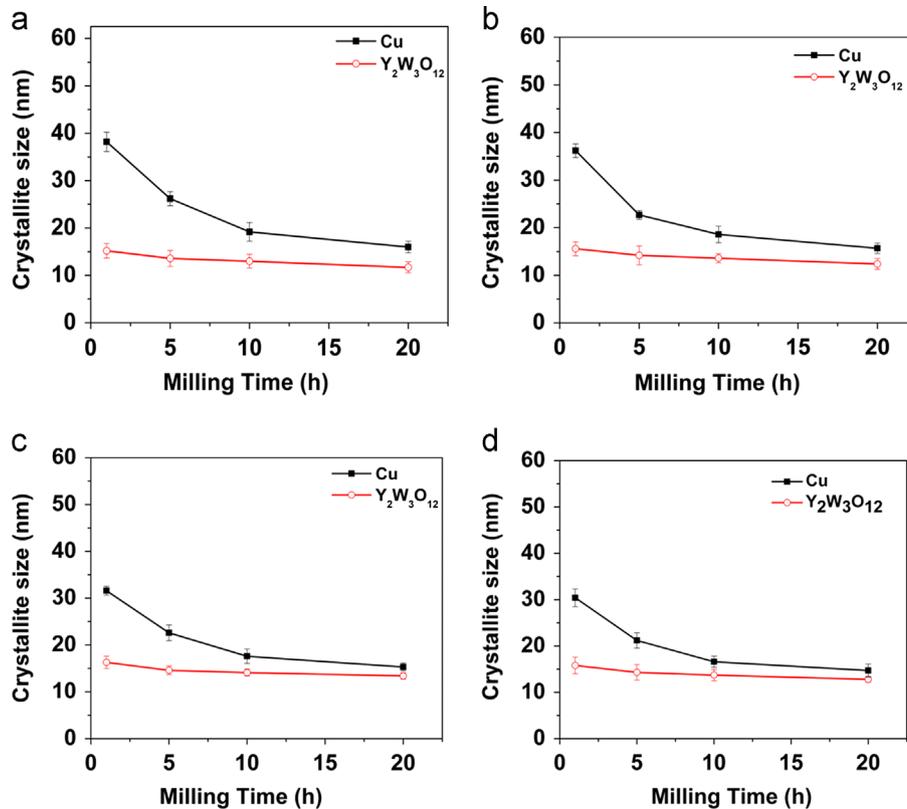


Fig. 1. Variation of crystallite size of Cu and  $Y_2W_3O_{12}$  with milling time in Cu30, Cu40, Cu50, and Cu60 powder.

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