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Electrical and Mechanical Properties of Microwave Sintered $Cu-ZrO_2$ (8-YSZ) Nano-Composites

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Abstract: Metal matrix nano-composites containing copper and ZrO_2 (8-YSZ) were sintered by microwave using mixtures of Cu and 1 wt.%, 2 wt.%, 3 wt.%, 4 wt.% and 5 wt.% of zirconia powders for 20min. The zirconia content up to 5 wt.%, resulted in an increase of 62.5% and 21.9% in micro-hardness and compressive strength respectively, and slight decrease in relative density and electrical conductivity (% IACS). In addition, field emission scanning electron micros copy (FESEM) and SEM fitted with EDX were used to characterize the sintered nano-composites.

Keywords: $Cu-ZrO_2$, Metal Matrix Nano-Composite, Microwave Sintering, Electrical Conductivity

1. Introduction

Metal matrix composites (MMCs) with a uniform dispersion of particles smaller than 100 nm size exhibit more outstanding properties over MMCs and are termed metal matrix nano-composites (MMNCs).It has been reported that with a small friction of nano-sized reinforcements, MMNCs could obtain comparable or even far superior mechanical properties than MMCs. The main advantages of MMNCs include excellent mechanical performance, feasible to be used at elevated temperatures, good wear resistance, low creep rate, etc.[1]. In addition, The MMNCs are assumed to overcome the shortcoming of MMCs poor ductility.

Copper has been extensively used as matrix because of its superior thermal and electrical conductivity. On the contrary copper has inadequate mechanical properties from the structural application point of view. The incorporation of ceramic nano-particulate reinforcement, such as oxides, borides and carbides, can improve high-temperature mechanical properties, specific stiffness, specific strength, abrasion resistance, creep resistance and dimensional stability significantly, without severe deterioration of thermal and electrical conductivity of Cu matrix [2]. Stabilized zirconia nano-particles can be uniformly dispersed in a Cu matrix, providing unique characteristics, such as high degree of strength without great loss in plastic deformation. Zirconia as a ceramic material finds many applications as piezoelectric devices, ceramic condensers and oxygen sensors due to some of its unique properties such as high hardness, low coefficient of friction, high elastic modulus, chemical inertness and high melting point [3].

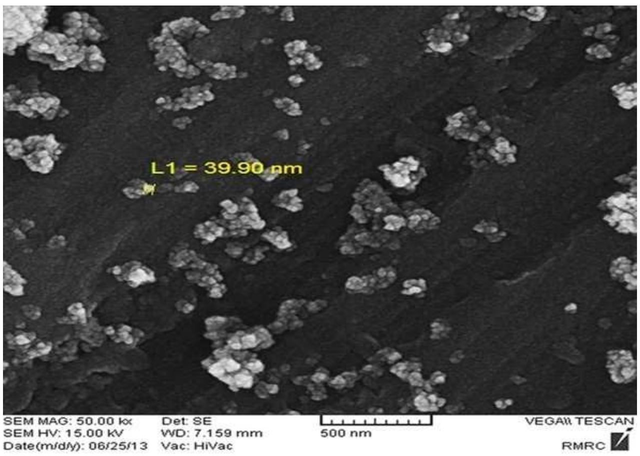
Various techniques are employed in the fabrication of MMNCs, which can be broadly classified into two major groups, namely liquid metallurgy (LM) and powder metallurgy (PM) [4]. LM route is one of the many production processes under investigation for fabrication of metal matrix nano-composites. The main argument against them is usually the claim of high reactivity of melt/filler systems. In fact this phenomenon does not prohibit the application of LM to a number of systems [5,6].PM is a process whereby a material powder is compacted s a green body and sintered to a net shape at elevated temperatures about 0.6-0.8 T_m. There are challenging demands from the PM industry for new and improved sintering process with finer microstructures and enhanced physical and mechanical properties. This is where the microwave technology is found to be advantageous [7].

Microwave energy is a form of electromagnetic energy with the frequency range of 300 MHz to 300 GHz. Microwave heating is a process in which the materials couple with microwaves, absorb the electromagnetic energy volumetrically, and transform into heat. This is different from conventional methods where heat is transferred between objects by the mechanisms of conduction, radiation and convection. In conventional heating, the material's surface is first heated followed by the heat moving inward. This means that there is a temperature gradient from the surface to the inside. However, microwave heating generates heat within the material first and then heats the entire volume [5]. This heating mechanism is advantageous due to the following facts: (i) enhanced diffusion processes, (ii) reduced energy consumption, (iii) very rapid heating rates and considerably reduced processing times, (iv) decreased sintering temperatures, (v) improved physical and mechanical properties, (vi) simplicity, (vii) unique properties, and (viii) lower environmental hazards. These are features that have not been observed in conventional processes [5-10].

In the present work, copper matrix composites reinforced with ZrO_2nano-particles were produced by microwave-assisted sintering technique and the effect of ZrO_2 amount on the mechanical and electrical properties of the copper matrix was studied.

2. Experimental

(a)



(b)

Figure 1. *SEM microstructures of Cu powder(a) and ZrO_2 reinforcement (b).*

The high purity copper powder (99.8%) with d< 50 μm (Powder Metallurgy co., Iran) and zirconia powder, 8% mol yttria-stabilized, with 99.5% purity and d< 40 nm were used as starting materials. In Fig.1, the SEM microstructures of Cu powder and ZrO_2 reinforcement are shown. As can be seen copper powder is in spherical shape and ZrO_2 particles are in irregular and angular shape and nano-size.

The powders in a range of composition of 100-99-98-97-96-95 wt.% Cu and 0-1-2-3-4-5 wt.% ZrO_2were mixed mechanically in aY-shape mixer. In order to obtain the proper mixing, ZrO_2 balls with the charge ratio of 4:1 were used. Prior of sintering, the mixed powders were compacted in a hydraulic cold press at a pressure of 500 MPa in a steel mould to obtain cylindrical and disc shaped specimens. The compacted samples were sintered in microwave oven (900 W, 2.45 GHz LG microwave oven having Ar atmosphere) as samples were heated for 20min. The specimens were supported inside two nested zirconia crucibles and the space between the crucibles was filled with ZrO_2particles to facilitate heating at low temperatures. Since ytria-stabilized zirconia reinforcement nano-particles were well wetted by Cu matrix, there was no need to use wetting agent.

The relative density of the sintered specimens was measured using Archimedes' method (ASTM B 311). Micro-hardness measurements were performed on the polished samples using a HVS-1000A automatic digital micro-hardness tester under a test load of 50 g and a dwell time of 10 s in accordance with the ASTM standard E 92.In order to obtain optimum results, micro-hardness values were determined by taking the average of six different measurements on each sample. The compression tests of the cylindrical shaped specimens having 10 mm diameter and 20 mm height were performed over an initial strain rate of 10^{-4} s^{-1} at room temperature using Zwick Royal Machine in compliance with ASTM E9-89a standard for measuring the compressive response of the matrix and composite materials [11]. A comparative method was used to measure electrical conductivity using DC 4probe method. The samples for 4 probe conductivity measurements were 10 mm height and 10 mm diameter after cutting and grinding. The two end faces of the specimens were ground flat, cleaned and painted with platinum paste (Hanovia Liquid Gold 6082). Platinum foil contacts for passing current were attached to these paste electrodes later on. A constant current of 0.5 to 200 μA (depending upon the specimen resistance) was supplied by a Keithley constant current source model 227. All the current ranges were pre-calibrated and correction was applied during calculation of the conductivity. Microstructural analyses were performed by field emission scanning electron microscopy (FESEM Hitachi S4160) and SEM fitted with EDX (VEGA-TESCAN).

3. Results and Discussion

3.1. Relative Density

Relative densities of pure copper and Cu-ZrO_2nano-composites, microwave sintered in 20 min at various zirconia contents were given in Table 1.Density measurements were carried out using the Archimedes water immersion method.

Table 1. *Presentation of relative densities of pure copper and Cu-ZrO_2MMNCs, microwave sintered in 20 min atvariouszirconia contents.*

ZrO2(wt.%)	Relative density (%)
0	94.9 ± 0.4
1	93.9 ± 0.5
2	93.4 ± 0.9
3	93.1 ± 0.9
4	92.8 ± 0.6
5	92.5 ± 0.9

The densities of the nano-composites decreased from 93.9% to 92.5% with increasing the amount of zirconia up to 5 wt.%. In the nano-composites with low ZrO_2weight%age, less Cu-ZrO_2 interface means less copper atom diffusion barrier, copper atoms can diffuse easily and spread between the ZrO_2nano-particles, thus leading to a higher densification of the nano-composites. In addition, decreasing of relative density with increasing zirconia content in the metal matrix probably could be due to the high volume of zirconia agglomerates and high volume of porosity [12].The creation of voids in the Cu matrix hinders the densification and impedes the continuity in intimacy contact of Cu and zirconia.

Comparing the results of this study with previous research [13] it can be concluded that with increment in ZrO_2 up to 5 wt.% in copper matrix, decreasing of density in microwave-assisted sintered samples is less than specimens that sintered by means of conventional method, 1.4 and 2.9%, respectively.

3.2. Electrical Conductivity

Electrical conductivity is a very useful property which is affected by chemical composition and the stress state of crystalline structures. It was known that the higher the relative density the higher the electrical conductivity [14]. Fig. 2 shows electrical conductivity values of the Cu-ZrO_2nano-composites, sintered for 20 min in microwave oven. The electrical conductivity of the samples ranged from 98.7% IACS to 59.6% IACS as a function of zirconia content. Electrical property of materials with base metal basically is influenced by the electron motion in the structure. Increasing of ceramic content in MMNCs, structure distorts and causes some impediment for metal electrons [15,16]. As a result, electrical conductivities of the nano-composites reduced with increasing ZrO_2%age.

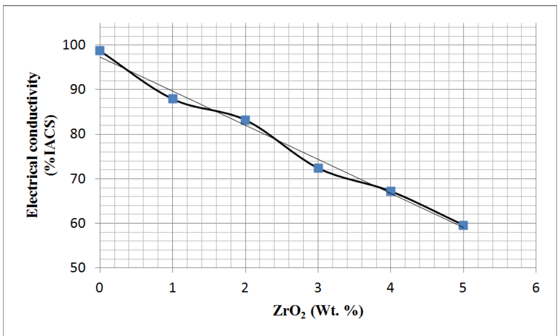


Figure 2. *Electrical conductivity values of the Cu and Cu-ZrO_2MMNCs as a function of zirconia content (wt.%) sintered for 20 min in microwave oven.*

3.3. Micro-hardness

Fig. 3 shows micro-hardness of specimens prepared by microwave-assisted sintering in 20 min. With regard to Fig. 3, it is revealed with increasing hard reinforcement content up to 5 wt.% in the ductile copper matrix, micro-hardness can be improved up to 62.5%. The micro-hardness of the nano-composites was found to be dependent on the zirconia contents rather than on the relative density. The gain in micro-hardness may be attributed to: (i) the presence of harder ceramic particulates in the metal matrix (1250 HV); (ii) decreased grain size [6]; and (iii) dislocations that are created by ceramic particles in the metal matrix [8].

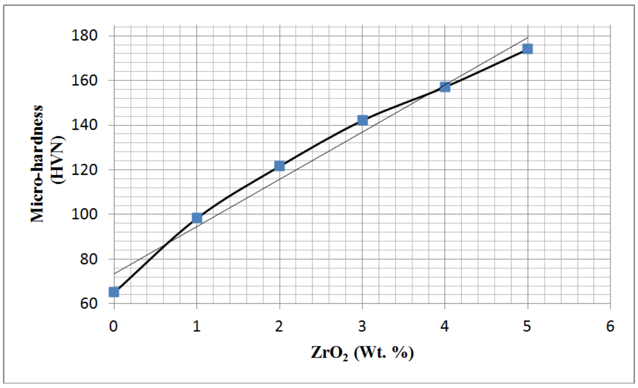


Figure 3. *Micro-hardness values of the Cu and Cu-ZrO_2MMNCs as a function of zirconia content (wt.%) sintered for 20 min in microwave oven.*

3.4. Compressive Strength

As Fig. 4 shows, compressive strength of the nano-composite samples increased up to 21.9% with increasing ZrO_2 content up to 5 wt.% in the metal matrix. Two strengthening mechanisms were proposed for high compression strength of the materials, i.e., grain size and dispersion hardening effects. The main strengthening of metallic materials is based on preventing dislocation motion and propagation. However, there is a limit to this mode of strengthening in nano-composites. Grain sizes can range from about 100 µm to 1 µm in traditional materials whilst in nano-composite materials the grains of reinforcement are less than 100 nm. At grain size of about 10 nm only one or two dislocations can fit inside a grain [3,17]. This small numbers of dislocations inside the grains may prohibit the dislocation pile-up and grain boundary diffusion. The lattice would resolve the applied stress then by grain boundary sliding, resulting in a decrease in the material strength and increase in ductility. However, experiments on many nano-crystalline materials demonstrated that if the grains reached a small enough size, the compression strength would either remain constant or decrease with decreasing grains size. This phenomenon has been termed as the reverse or inverse Hall-Petch Relation [17]. In addition, the zirconia nano-metric particulates cause dispersion hardening effects that impede dislocation motion, increasing the compressive strength of the Cu matrix.

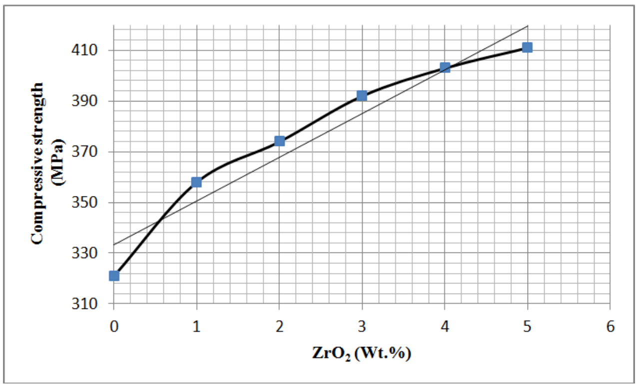


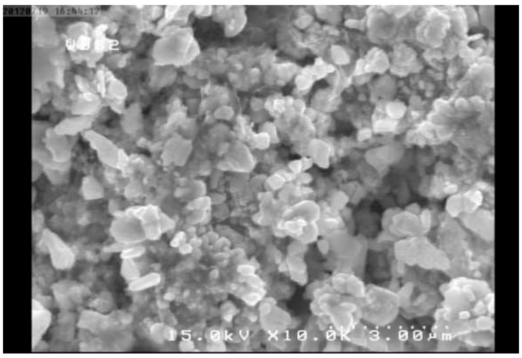
Figure 4. *Compressive strength values of the Cu and Cu-ZrO_2MMNCs as a function of zirconia content (wt.%) sintered for 20 min in microwave oven.*

3.5. Microstructure

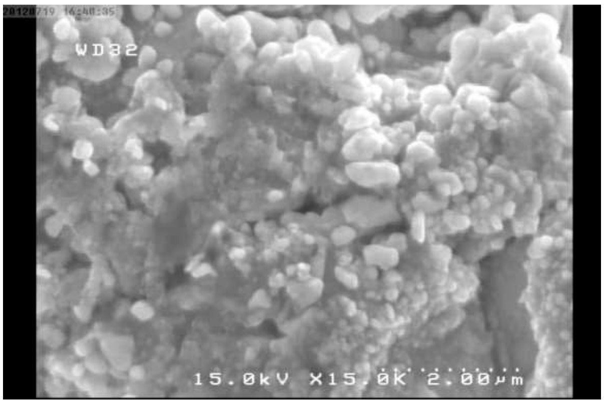
Microstructural morphologies of the copper matrix composites reinforced with 4 and 5 wt.% of zirconia nano-particles were produced by microwave-assisted sintering technique are shown in Fig. 5. The FESEM micrographs give abundant information about the reinforcement distribution, status of physical intimacy between Cu and $ZrO_2$and mechanical phenomena. For the composite materials, it is very important to obtain homogeneous distribution of reinforcement in the matrix in order to enhance mechanical properties. If reinforcement particles in the composites do not disperse uniformly, this affects the mechanical and electrical properties of composites negatively [18].

With the increase in weight percentage of ceramic reinforcement in metal matrix the efficiency of distribution becomes remarkably better. The density difference between the matrix and reinforcement also leads to the formation of clusters sometimes at high wt.% of the reinforcement[19]. The physical contact of the zirconia nano-particles with the matrix can be attributed to the high atomic diffusivity of the nano-particles. The stabilization of the surface energy of nano-particles is a thermodynamic driven phenomenon; hence it is quite obvious that the physical adherance of Cu with $ZrO_2$is proper in the nano-composites.

In addition, EDX analyses were performed in order to analyse the various elements present in the Cu-ZrO_2 MMNCs. Example of these analyses has been shown in Fig. 6.

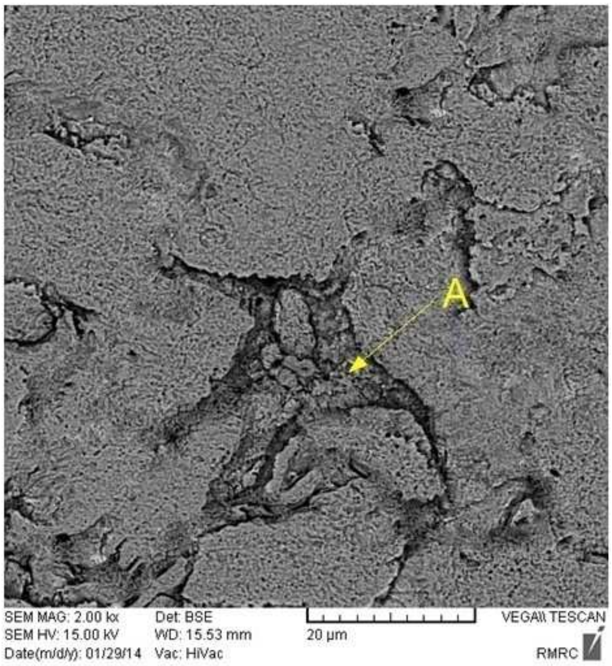


(a) Cu-4Wt.% ZrO_2

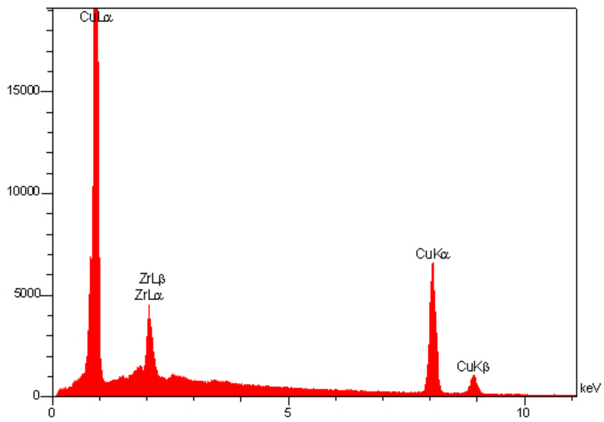


(b) Cu-5Wt.% ZrO_2

Figure 5. *FESEM images of the Cu-4 and 5 wt.%ZrO_2MMNCs, sintered for 20 min in microwave oven (Figure (a), (b)).*



(a)



(b)

Figure 6. *SEM-EDX analysis of Cu-3Wt.% ZrO_2 MMNCs, microwave-sintered for 20 min.*

4. Conclusion

From microwave sintering of the Cu-ZrO_2 (8-YSZ) nano-composites for 20 min in microwave oven, the following results can be concluded:

- Cu matrix reinforced with different weight % of the zirconia (1, 2, 3, 4 and 5 wt.%), were densified by microwave-assisted sintering method successfully.
- Increment in the weight %age of ZrO_2nano-particles up to 5 wt.% in the samples, caused the slight reduction in the densification (1.4%) and electrical conductivity (28.3%) of the nano-composites.
- The highest micro-hardness (174.1 HVN) and compressive strength (411 MPa) of the nano-composites is related to the Cu-5 wt.% ZrO_2.

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