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Effect of microwave sintering over vacuum and conventional sintering of Cu based nanocomposites



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ABSTRACT

The blend compositions of $Cu_{99}Cr_1$ (All the compositions are in atom% until otherwise mentioned.), Cu_{94} - Cr_6 , $Cu_{99}Cr_1-4$ wt.% SiC (average particle size ~30 nm) and $Cu_{94}Cr_6-4$ wt.% SiC were ball-milled for 50 h in a stainless steel grinding media. The structural investigation and phase evolution during milling of the same compositions have already been reported in elsewhere ([28]). In the present study, we are reporting effect of microwave sintering on mechanical properties and electrical conductivity over vacuum and conventional sintering carried out at 900 °C. Relative densification and grain coarsening characteristics were investigated and compared. The microstructural features were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The mechanical properties, and electrical conductivity was tutability of the materials for electrical contact applications. The best combination of mechanical properties and electrical conductivity was obtained for microwave sintered specimens. This is possibly due to the enhanced densification and better grain size distribution achieved in microwave sintering technique. Difference in the properties is discussed in the light of electron scattering factor and densification in presence of ultra fine SiC particles in the nanocomposites.

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1. Introduction

Copper (Cu) and Cu-based alloys are used in industrial products due to its inherent properties like excellent electrical and thermal conductivity, low cost, good corrosion resistance and ease of fabrication [1]. Though, the poor mechanical properties like low yield strength, low hardness and very low wear resistance limits its applications at room and/or elevated temperatures. By incorporating immiscible elements like Cr or hard phase ceramic particles like carbides, borides, oxides into the Cu matrix can improve its mechanical properties and could be useful in structural applications [2]. The nanosize reinforcement and retention of nanosize grains in the bulk matrix might further improve the mechanical properties of Cu-based composites, which can provide unique combination of required electrical conductivity, and high hardness and wear resistance [1–4] especially for contact applications. This also may provide an excellent resistance to high temperature annealing. The Cr and/or second phase particle play an important role

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both in strengthening and in restricting grains coarsening of Cu matrix [5]. These materials can be used for spot welding electrodes, high-performance switches, rotating source neutron targets, combustion chamber liners, nozzle liners, aerospace propulsion systems and fusion power plants etc. [6,7]. Mechanical milling (MM) is one of the most promising techniques used to produce heat-resistant dispersion strengthened copper alloys, where homogenous distribution of nanosize second phase particles in the ultra fine grains matrix could be achieved successfully [8,9]. Recently, Suryanarayana and Al-Aqeeli [10] discussed the roles of different nanosize ceramic particles reinforcement in Cu matrix to improve mechanical properties and to modify the electrical behaviour of copper.

In conventional pressure less sintering it is difficult to obtain a full density for nanostructured materials retaining nanofeatures in it. It is a challenging job to get near net shape materials with improved physical and mechanical properties [11] by conventional sintering at high temperatures (0.6–0.8 Tm, Tm = melting temperature in absolute scale). To overcome grain growth during consolidation, various nonconventional techniques are implemented [12–17]. These techniques are used for better control over microstructural development and densification, e.g., microwave sintering [14,15], spark plasma sintering [16] and high pressure torsion



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[17] etc. These techniques reduce the sintering time and temperatures without much coarsening of grains. Microwave sintering is a promising method for consolidation of mainly ceramic materials because of it high dielectric constant [18]. Very few literatures are available on microwave sintering of metal based nanomaterials [11,19]. Anklekar et al. showed that the mechanical properties of microwave sintered specimens of copper and nickel steel powder were superior to those of conventionally sintered samples [14,20]. Roy et al. [21] reported that unlike bulk metals, which reflect microwave, metal in the form of finely divided powder can absorbs microwave. It is due to the increase in multiple scattering and eddy current loss. The microwave sintering, in this regard, is expected to be useful for fine grained metallic nanomaterials; especially, when microwave susceptive ceramic particles are uniformly distributed in the matrix. Rapid heating of microwave sintering may be beneficial to obtain high sintered density and retention of fine grain size over conventional sintering [22].

Hence, the aim of the present study is to investigate the efficiency of microwave sintering of Cu–Cr and (Cu–Cr)–SiC metalbased nanocomposites to that of the conventional and vacuum sintering. The microstructural characterizations were carried out by XRD, SEM and AFM analysis. Vickers hardness and wear resistance, and electrical conductivity of the sintered specimens were also investigated.

2. Experimental

The elemental powders of Cu, Cr (purity >99.0%) were used to blend the compositions Cu₉₉Cr₁, Cu₉₄Cr₆, Cu₉₉Cr₁-4 wt.% SiC and Cu₉₄Cr₆-4 wt.% SiC. Mechanical alloying is carried out of the blend composition for 50 h by using Fritsch Pulverisette-5 planetary ball mill in the stainless-steel grinding media at a mill speed of 400 rpm. The ball to powder weight ratio was maintained at 10:1. Toluene was used as the process control agent. Before this, laboratory grade SiC powder (purity >96.0%) were initially ball milled to prepare nanosize (~30 nm) powder particles. Uniaxial pressing was carried out for the as-milled powders at an applied pressure of 1500 MPa. These pellets were subjected to three different sintering techniques, namely, pressure-less conventional and vacuum sintering at 900 °C for a soaking period of 1 h, and microwave sintering at 900 °C for 30 min. Microwave sintering was carried out using a furnace of 2.45 GHz frequency and 900 W powers. Microstructural analysis of the sintered specimens was studied by XRD, SEM and AFM. Relative green density and sintered density measurements were performed by Archimedes' principle. The phase evolution of the milled samples and sintered compacts were studied by Philips X-pert PRO high-resolution X-ray diffractometer using Cu K α (α = 1.54059 Å) radiation. Average crystallite size of the Cu-rich solid solution was estimated from the broadening of 3 major peaks after stripping its Ka2 component using the Philips X'Pert high-score software. A VEECO di Innova atomic force microscope was used for AFM micrograph of sintered samples. Hardness values were measured using a Leco LV 700 Vickers hardness tester. Ball-On-Plate wear tester TR-208-M1 instrument was used to analyse the wear behaviour of sintered samples. The wear tester was operated under a constant load of 10 N at 15 rpm for 1200 s. The electrical conductivity was measured using 4 probe method in a SIGMATEST D 2.068. FOERSTER instrument.

3. Results and discussion

The percentage of densification obtained by conventional, vacuum and microwave sintering methods at 900 °C has been summarized in bar diagram as shown in Fig. 1. Actually, microwave sintering was carried out at 900 °C for 20 min, 30 min and for 1 h. But, we have reported the data (in Fig. 1) for the specimens, which were sintered at 900 °C for 30 min only. Because it was observed that the density of the specimens sintered at 900 °C for 1 h and for 30 min was almost same, especially for SiC containing composition; whereas, in case of 20 min duration, sintered density was lower than that of 30 min duration. This may be due to the saturation of microwaves effect achieved (under the present experimental conditions) in 30 min duration in the present study. After that, decoupling of phases with microwaves may take place [23]. Moreover, microwaves also start decoupling with the sintered metal parts after saturation [23]. Similar type of results has been reported



Fig. 1. Relative sintered density of $Cu_{99}Cr_{1}$, $Cu_{99}Cr_{6}$, $Cu_{99}Cr_{1}-4$ wt.% SiC and $Cu_{94}Cr_{6}-4$ wt.% SiC compositions for conventional and microwave sintering at 900 °C.

in [23], where the sintered density of the microwave sintered Cu-5 vol.% graphite composites was almost same for the soaking period of 20 min and 30 min. As the aim of the present study is to investigate the effectiveness of the microwave sintering of the metal based nanostructures over the other two methods, the maximum sintered density with a minimum soaking period (30 min) was reported for all characterizations.

The maximum green density achieved was ${\sim}80\%$ for Cu-Cr compositions, whereas it was \sim 75% for the compositions which contains SiC. This difference in green density was due to the presence of hard and nondeformable nanosized SiC particles which impede the plastic deformation of the Cu matrix during pressing. Relative density was found to increase after sintering. Microwave sintering of Cu-Cr compositions at 900 °C resulted 94-95% of densification, whereas densification was 90-93% for SiC containing compositions. In case of microwave sintered compacts when compared with their corresponding green density, the enhancement of densification is much higher for SiC containing compositions rather than Cu-Cr compositions. This is mainly due to the microwaves interact rapidly with SiC as it is a high dielectric constant material and act as good susceptor of microwaves [24,25]. But in case of conventional and vacuum sintering methods, almost similar level of density, i.e., 81-82%, is achieved for Cu-Cr compositions; while 77-79% of densification is obtained for SiC containing compositions. This indicates that the SiC particles do not have any role in densification during conventional sintering as well as vacuum sintering of cold-pressed pellets. SiC is highly microwave sensitive because of its high dielectric constant. Moreover, because of the high effective surface area available due to ultra-fine size of SiC particles present in the matrix, microwave readily reacts with the surrounding matrix and generates local heating regions at the SiC/Cu-Cr interfaces. Thus, it enhances local diffusion, which eliminates pores, and provides more efficient bonding in the composites. In case of conventional and vacuum sintering, SiC particles present in the composites do not generate any extra local heating regions in the compacts. Moreover, they hinder the overall diffusion process during conventional and vacuum sintering. Thus, microwave sintering of Cu-alloy containing SiC particles showed higher sintered density of the nanocomposites.

Fig. 2(a–d) shows the XRD patterns and the phases present in conventional, vacuum, and microwave sintered $Cu_{99}Cr_1$, $Cu_{94}Cr_6$, $Cu_{99}Cr_1-4$ wt.% SiC and $Cu_{94}Cr_6-4$ wt.% SiC composites in comparison to the corresponding 50 h milled samples. From XRD phase analysis it can be noticed that Cr peaks are visible in all the 50 h milled samples as well as in all the sintered specimens except in the 50 h as-milled sample of $Cu_{99}Cr_1-4$ wt.% SiC. This is possibly due to the increase in dissolution of Cr up to 1 at.% in the presence



Fig. 2. XRD patterns of conventional sintered (CS), vacuum sintered (VS), and microwave sintered (MWS), (a) Cu₉₉Cr₁, (b) Cu₉₄Cr₆, (c) Cu₉₉Cr₁–4 wt.% SiC and (d) Cu₉₉Cr₁–4 wt.% SiC and (d) Cu₉₉Cr₁–4 wt.% SiC compacts combine with 50 h milled samples.

of 4% nanosize SiC particles during milling for 50 h. Recently, Sheibani et al. [26] also reported that due to the presence of nanosize Al₂O₃ particles solubility of Cr in Cu increases during mechanical milling. In the present study, 1 at.% Cr is possibly dissolved in Cu as revealed from the XRD data, i.e., free Cr was not detected in the XRD pattern of as-milled Cu₉₉Cr₁-4% SiC, whereas, free Cr is noticeable in the 50 h milled Cu₉₄Cr₆-4 wt.% SiC sample. Because high amount Cr added was not dissolved in Cr by the milling condition. Again it may be observed that the intensity and sharpness of the peaks decreased in case of Cu-Cr-SiC compositions as compared to the as-milled Cu-Cr alloys. It may be due to the additional grain refinement in presence of nanosize SiC particles during milling. It was also found that the peak intensity and sharpness increased after sintering of the compacts. The increase in the intensity is due to the increase in crystallite size after sintering processes. Microwave sintered samples showed less increase in the peak intensity as well as sharpness than that of the specimens sintered by other two methods. Grain coarsening of the microwave sintered samples containing SiC was found to be less compared to Cu–Cr alloys. This may be observed from XRD peaks of the corresponding specimens.

Moreover, grain growth is also hindered by fine SiC particles during sintering, which resulted in fine grained bulk composites. The less intense oxide peaks like Cu₂O, Cu(CrO₂) were also visible in the XRD patterns of all the sintered specimens due to oxidation in the presence of residual oxygen. In XRD patterns of sintered Cu₉₄Cr₆ and Cu₉₄Cr₆–4 wt.% SiC compositions, Cr₂O₃ peak was also observed. The presence of trace amount of SiO₂, CrSi/CrSi₂ is also revealed in the XRD patterns, which were produced due to the presence of free Si (as an impurity) along with SiC. But in case of sintered Cu₉₉Cr₁–4 wt.% SiC and Cu₉₄Cr₆–4 wt.% SiC, Cu(CrO₂) and SiO₂ phases were persistent. The oxides formed due to the reaction with residual oxygen present in argon atmosphere.

From XRD analysis crystallite size of the sintered as well as milled samples was determined from the broadening of 3 major peaks after stripping its K α 2 component using the Philips X'Pert

high-score software and is shown in Table 1. Variation of measurement of the corresponding crystallite size is also mentioned in the same table (Table 1). From the table (Table 1) it seems that the variation of crystallite size measurement is very small and negligible. Therefore, comparative analysis was carried out on the basis of average crystallite size. Average crystallite size of the as-milled $Cu_{99}Cr_1$ was calculated to be 24 nm, while it was 17 nm for $Cu_{94}Cr_6$ alloy. Furthermore, obtained average crystallite size was 20 nm and 16 nm, respectively, for Cu₉₉Cr₁-4 wt.% SiC and Cu₉₄Cr₆-4 wt.% SiC. Therefore, presence of SiC and/or Cr helps in refining the microstructure although milling condition is same. After sintering, the crystallite size increased irrespective of the sintering methods followed. But, compared to pressure-less sintering, microwave sintered samples showed less increase in crystallite size, e.g., crystallite size increased to 91 nm from 24 nm in microwave sintered Cu₉₄Cr₆, whereas it was 151 nm for vacuum sintered compacts and 169 nm in case of conventional sintered samples of same composition. The crystallite size was 80 nm for microwave sintered Cu₉₄Cr₆-4 wt.% SiC, but it increased to120 nm by vacuum sintered specimens. During sintering, grain growth is normally hindered by the presence of hard particles [27]. So, the presence of uniformly distributed fine SiC particles, in the present investigation, possibly played an important role in controlling the fine-grain structures by pinning effect. This also could be corroborated with the XRD peak intensities of the milled samples and sintered specimens. As microwave sintering requires less sintering temperature and less soaking period than the conventional technique, it is possible to produce bulk size nanocomposites without much grain coarsening [28].

To investigate grain size of the sintered samples AFM analysis was carried out and Fig. 3(a and b) shows the representative structures for microwave sintered specimens of $Cu_{94}Cr_6$ and $Cu_{94}Cr_6$ -4 wt.% SiC, respectively. The estimated average grain size is more than 200 nm for $Cu_{94}Cr_6$ whereas, it is less than 200 nm for $Cu_{94}Cr_6$ -4 wt.% SiC compositions. It is clear from Fig. 3 that the grain size is much finer in case of Cu–Cr–SiC composition than that of $Cu_{94}Cr_6$. Moreover, AFM micrographs clearly indicate that the matrix grains are more uniform in case of SiC containing composition compared to Cu–Cr composition. Therefore, fine SiC particles not only hinder the coarsening of matrix grains by pinning but also control the final distribution of it. It may be noticed that the grain size determined from AFM micrograph is bit larger than that of calculated from the XRD analysis. But it showed a good comparative result.

Fig. 4(a and b) shows the SEM micrographs of the polished surface of sintered $Cu_{94}Cr_6-4$ wt.% SiC samples. It may be noted that the microwave sintered samples showed less amount of porosity and significantly finer grain than that of conventionally sintered specimens. The level of porosity can also be evidenced from the relative sintered density as shown in Fig. 1.

Vickers hardness values were measured for the all sintered specimens and average hardness values were presented in bar diagram in Fig. 5. Comparatively, higher hardness values in the range of 1.9–2.4 GPa were obtained for the microwave sintered specimens, compared to vacuum sintered (1.5–1.7 GPa) and conventionally sintered (1.27–1.63 GPa) specimens. The hardness of

pure Cu is ~0.48 GPa [29], whereas, hardness of microwave sintered Cu₉₉Cr₁ specimen is 1.9 GPa (Fig. 5). So, addition of 1 at.% of Cr in pure Cu enhanced the hardness to a value of 1.9 GPa with a sintered density of 95%, when sintered by microwave sintering method. Hardness of vacuum sintered specimen of the same composition is 1.5 GPa, with a sintered density of 82.4%. Hardness of microwave sintered Cu-Cr compositions is in the range of 1.9-2 GPa; whereas it is 1.5-1.54 GPa for vacuum sintered and 1.27-1.39 GPa for conventional sintered specimens. It can be noted that addition of Cr content increased the hardness values. For example, hardness of Cu₉₄Cr₆ was 1.39 GPa compared to 1.27 GPa for Cu₉₉₋ Cr₁. It is to be noted that a small amount reinforcement of SiC plays an important role in enhancing the hardness values of Cu-Cr alloys. The measured hardness of microwave sintered Cu₉₄Cr₆-4 wt.% SiC specimen is enhanced to 2.4 GPa. This is in contrast to the hardness of same composition sintered by other methods in the present study. The corresponding hardness values are 1.7 GPa and 1.63 GPa, respectively, for vacuum sintered and conventional sintered specimens of same composition (Cu₉₄Cr₆-4 wt.% SiC). The lower hardness is possibly due to the coarsening of grains during longer soaking period in conventional and vacuum sintering methods. This can also be substantiated with the crystallite size as shown in Table 1. As microwave sintered samples contains very less and small voids and also less grain growth occurred than conventional sintered specimens, the hardness values are expected to be higher than that of the specimens sintered by the other methods.

Ball-On-Plate wear test was carried out for the microwave sintered samples because of the best combination of hardness and sintered density obtained in the present investigation. Fig. 6 shows the wear depth vs. test time (s) plot for microwave sintered specimens sintered at 900 °C. It is clear from this figure that wear resistance of Cu-Cr composition is much lower than that of SiC containing composition. With increase in the Cr content from 1 to 6 at.% wear resistance increases as indicated by lower wear depth as shown in Fig. 6. But with addition of only 4 wt.% of SiC in the above composition wear depth decreases to a value (12-17 um), which is much less than that of only Cu-Cr compositions $(\sim 30 \ \mu m)$. As porosity level on the surface of the specimen is high, which may be due to improper bonding between particles and oxidation on the surface during sintering, initially it increases the wear depth for all the cases. With progress of time when indenter reaches in the subsurface level, a constant wear depth was observed. This is because of sufficient lubrication is generated itself due to plastic deformation during wear.

Electrical conductivity of the sintered specimens were measured and compared with the conductivity as per the International Annealed Copper Standard (IACS) (100%) and this is shown in Fig. 7. It can be noted (Fig. 7) that the electrical conductivity increases from the range of 38–46% to 60.3–63% when the sintering method was changed from conventional to microwave sintering. The electrical conductivity is 46.1% and 44% of IACS, respectively, for Cu₉₉Cr₁ and Cu₉₄Cr₆ specimens sintered by vacuum sintering. The measured conductivity is only 40.6% and 39.5%, respectively, for Cu₉₉Cr₁–4% SiC and Cu₉₄Cr₆–4% SiC when sintered by same method (vacuum). It is well known that electrical conductivity

Table 1

Average crystallite size of Cu-rich solid solutions for different compositions after conventional, vacuum and microwave sintering at 900 °C. It also shows the variation of corresponding crystallite size measurement.

Compositions	Average crystallite size (nm)			
	50 h milled	Conventional sintered	Vacuum sintered	Microwave sintered
Cu ₉₉ Cr ₁	24 ± 1.5	190 ± 4	164 ± 3.5	95 ± 2
Cu ₉₄ Cr ₆	17 ± 2	169 ± 3.5	125 ± 3	82 ± 2.5
Cu ₉₉ Cr ₁ -4 wt.% SiC	20 ± 1.5	181 ± 3.5	151 ± 3.5	91 ± 3
Cu ₉₄ Cr ₆ -4 wt.% SiC	16 ± 1.5	160 ± 3	121 ± 3	80 ± 3.5



Fig. 3. AFM micrographs of Cu₉₄Cr₆-4 wt.% SiC composition for (a) conventional sintered and (b) microwave sintered specimens respectively.



Fig. 4. SEM micrographs of Cu₉₄Cr₆-4 wt.% SiC for (a) conventionally sintered and (b) microwave sintered sample sintered at 900 °C.



Fig. 5. Hardness (GPa) of different specimens sintered by conventional and microwave sintering techniques at 900 $^{\circ}$ C.

should be >50% of IACS for contact applications [1]. Therefore, the conductivity of the vacuum sintered and conventional sintered specimens does not reach to the desired level for practical applications. This is due to the poor sintered density (Fig. 1) obtained by the sintering methods. This can be corroborated with the large number of pores in SEM micrograph (Fig. 3) of conventionally sintered specimens. As pores are non-conductive, the charge carriers face large numbers of non-conductive sites on their way and thus decreased the conductivity value [30]. The electrical conductivity of microwave sintered specimens was in the range of 62.4-63% for Cu–Cr compositions, and 60.3-61.2% of IACS for Cu–Cr–SiC compositions. The better electrical conductivity is plausibly because of proper interface bonding and higher sintered density (Fig. 1) obtained in microwave sintered specimens. It can also be



Fig. 6. Wear depth vs. time (s) plot for microwave sintered specimens sintered at 900 $^\circ\text{C}.$

noticed that though the electrical conductivity of microwave sintered Cu–Cr–SiC and Cu–Cr specimens is almost the similar, the later exhibited a bit less sintered density (from Fig. 1). SiC particles in the composites practically decrease electrical conductivity of the composites as electron scattering sites increase due to the presence of SiC particles. But in the present study, the microwave sintered SiC particles reinforced composites showed comparable electrical conductivity as that of Cu–Cr alloys. This is due to fact that the better sintered density obtained in microwave sintered specimens of Cu–Cr-SiC compositions as explained earlier (in Fig. 1). Increasing Cr content from 1 to 6 at.%, the conductivity decreased slightly and it was further decreased with the addition of 4% of nanosize



Fig. 7. Electrical conductivity of different specimens sintered by conventional and microwave sintering techniques at 900 $^\circ$ C.

SiC particles with the same composition, although the relative sintered density almost remained in the similar level (Fig. 1). This is expected because of the electrical conductivity of Cr and SiC is much lower than that of Cu [2]. But, in the present investigation amount of these elements added is very less. As particle size reduced to nanometer level after 50 h of milling, it would generate more interface area. These interfaces possibly act as a source of additional electron scattering and might be a major factor in reducing of electrical conductivity [31,32] further. Due to much finer particle size in case of SiC compositions, and higher amount of Cr content, the reduction of electrical conductivity in case of Cu₉₄Cr₆–4 wt.% SiC is higher than that of Cu₉₉Cr₁–4 wt.% SiC compacts for a particular sintering temperature in a particular sintering method.

In the present investigation, the highest hardness values of 1.9– 2.4 GPa, better wear resistance and 60–63% of IACS electrical conductivity were obtained by microwave sintering of Cu–Cr–SiC compositions. The corresponding densification level of the sintered specimens was also good (91–95%). Therefore, the present materials may be useful in field of electro-technology. The combination of desired mechanical properties and electrical conductivity can further be improved by increasing the sintered density of the materials by employing proper sintering techniques, like, spark plasma sintering. Desired level of densification could also be achieved by adjusting the sintering parameters (temperature, pressure, time) properly without much deteriorating the mechanical properties. Therefore, the materials can be interesting and useful, where the devices require high electrical conductivity as well as high mechanical strength [16,33].

4. Conclusions

The microwave sintering is found to be most effective in consolidation of ball-milled Cu–Cr and/or Cu–Cr–SiC nanocomposite powders compared to the other methods used in the present investigation. Suitable combination of mechanical and electrical properties was obtained using this technique. SiC reinforced nanocomposites showed finer structure compared to that of Cu–Cr alloys after completion of sintering process. Microwave sintered specimens showed good combinations of hardness in the range of 1.9–2.4 GPa and electrical conductivity 60–63% of IACS. Wear resistance of microwave sintered specimens increased with addition of Cr. It was further enhanced by addition of 4 wt.% nanosize SiC particles. The material developed in the present investigation could be successfully used for thermo-electric applications.

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