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Investigation on microstructural and mechanical properties of Cu reinforced with Sic composites prepared by microwave sintering process

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Abstract: *In this present experimental studysix different metal matrix composites has been fabricated using pure copper (Cu) as a base material reinforced with silicon carbide (Sic) and graphite as one of the additives each time i.e., different volume fractions Sic and graphite. Powder state mixing technique was employed for the different constituent. After mixing, powders were poured in metallic die then specimens are sintered through Microwave sintering (2.45 GHz, 3.2 kW) and various tests such as hardness, compressive and microstructure of the composite specimens has been carried out.*

Keywords: Copper, silicon carbide, graphite, hardness test, compressive test, microstructure test.

1. INTRODUCTION

Metal matrix composites utilize the properties of the matrix (light weight, good thermal conductivity, ductility) and of the reinforcement, usually ceramic (high stiffness, high wear resistance, low coefficient of thermal expansion)[1]. By this way it is possible to obtain a material characterized, if compared to the basic metal component, by high values of specific strength, stiffness, wear resistance, fatigue resistance and creep, corrosion resistance in certain aggressive environments[2]. However, cause to the presence of the ceramic component, ductility, toughness and fracture to the coefficients of thermal expansion and thermal conductivity decrease.Many researchers attempted to sinter metal powder by microwave sintering [3] and they made a comparative evaluation on mechanical properties of sintered metal powders such as Fe–Ni, Fe–Cu and copper– TiC–graphite hybrid composites through microwave and conventional methods. Microwave sintered specimens exhibited better mechanical properties. Microwave sintered Fe–Ni exhibited a modulus of rupture which is 60% higher than the conventionally sintered ones. It has been reported that copper steel alloys could be sintered using microwaves. These alloys exhibited better mechanical properties than conventionally sintered ones[4]. Experimental; study made on microwave sintering response of copper–graphite[5]. The problem of negative densification (expansion) through conventional sintering is surpassed by microwave sintering. Positive densification (shrinkage) was attributed to faster sintering which hinder the diffusion of tin particles into copper lattice. Experimental investigation made on magnesium based composites for the improvement of mechanical properties of microwave sintered Al and Mg, to finer microstructure[6]. Microwave radiation is useful in material processing activities[7] it has been reported that microwaves are used for joining of ceramic and composites. Like lasers, microwaves are used for glazing of alumina–titania composite [8].Microwave sintering offers many advantages such as faster heating rate, lower sintering temperature, enhanced densification, smaller average grain size and an apparent reduction in activation energy in sintering. Some negative aspects of conventional sintering such as non-uniform heating, coarser microstructure and larger porosity can be minimized in microwave sintering[9]. The present study uses microwave hybrid heating for the development of metal matrix composites. Microwave hybrid heating comprises simultaneous actions of microwave and microwave coupled radiative external heating, to realize the uniform and rapid heating[10]. Though some literature exists in microwave sintering of metal, no literature is available presently in the area of microwave sintering of copper based composites.

Table 1. Composition of specimens in %

Composite specimens	Copper %	SiC %	Graphite %
Specimen 1	90	5	5
Specimen 2	85	10	5
Specimen 3	80	15	5
Specimen 4	85	5	10
Specimen 5	80	10	10
Specimen 6	75	15	10

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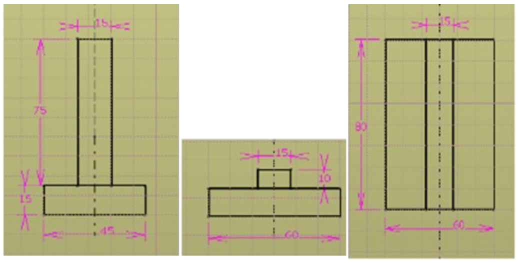


Plunger Base Sleeve

Photocopy of Die Die with ejection rod

Table 2. Dimension and photography of Die with ejection rod

2. EXPERIMENTAL

2.1. Materials

Copper-SiliconCarbide–graphite composites were manufactured through powder metallurgy (P/M) route. Electrolytic copper powder having average grain size of 47µm was mixed with the Silicon Carbide 37 µm size graphite powder of 50µm size.Copper powder was mixed with various volume fractions of Silicon carbide powder 5%, 10%, 15% And Graphite 5%,10% in manual pestle mortar for uniform mixing[11].Table.1 shows composition of specimens.

In order to obtain the proper mixing for duration of approximately 1 hr is maintain for mixing the powders in the pestle mortar. Mixed powders were preheated at 150 ^{o}C in a muffle furnace to evaporate any volatile matter.

2.2. Composite fabrication

The D-2 Steel material was used for fabrication of die. D-2 Steel having high chromium content gives it corrosion resisting properties in the hardened condition and it possess extremely high wear resisting properties. Dimension specification and fabricated die as shown in table 2. By using this die six differentComposition of specimens (shown in table 3) has been fabricated. The preheated powders were uniaxially compacted in a CTM machine at a load of 20ton to obtain cylinder shaped specimens having dimension of 15mmdiameter and 23 mm height with approximate pressure starts from 500Mpa to 1000Mpa for the plunger diameter of the die. Prior to sintering;the mixture was cold pressed into a cylindrical compact in a metal die of 15 mm in diameter under an axial pressure of 280 MPa[12].

2.3. Microwave sintering and characterizations

The green compacts were kept inside the hybrid microwave sintering setup. Preliminary sintering trials were conducted for the sintering temperature range of 850 ˚C and 900 ˚C and isothermal holding time range of 10–60 min. Sintering condition which emerged from the trial experiment has been extended to copper with various volume fractions of Sic and graphite composite. The microwave simultaneously interact with the green body sample, i.e. couple with green body sample and generates the heat internally due to penetrating feature of microwave in powder compacted samples.. Two sets of green samples were sintered for each condition in order to assess the variation in processing and for the reproducibility of final properties. In all the cases, the power of microwave was controlled to 20% of maximum available power and the heating rate was also set within 25^{o} C /min for one set of samples for 60 mins. at 850 ^{o} C and another samples at 50^{o} C /min for 10 mins at 900 ^{o} C that is rapid fast. After the definite isothermal holding time, the samples were allowed to cool in the furnace[13].A 3.2 kW industrial microwave furnace (2.45 GHz) was used in this study. Microwave sintering setup was designed to have two layers of elements, i.e. one is transparent to microwave and the other is absorber of microwaves for uniform heating. The alumina wool (transparent to microwave) was used for preserving the heat inside the crucible SiC fencing was used as a susceptor (microwave absorbing element). SiC fencing not only provides hybrid heating facility but also reduces the thermal gradient and this promotes crack free components[14-18]. Accurate temperature of samples was monitored using a 'K' type thermocouple. The microwave sintering setup used in the present investigation and Sintered specimens are shown in table 3.

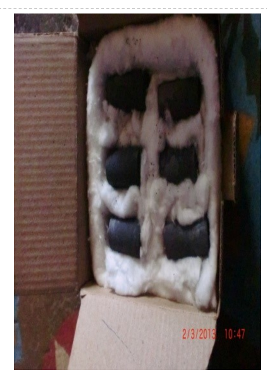
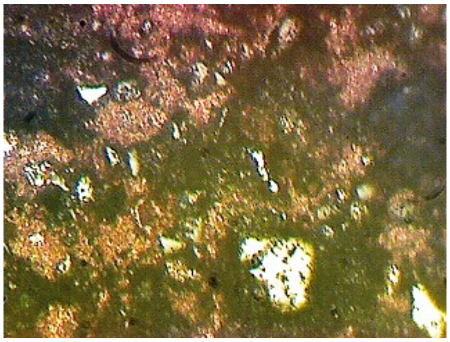
			
CTM (compact) machine for green composite fabrication	Fabricated green compact composite specimens	Microwave sintering of composite specimens	Sintered specimens

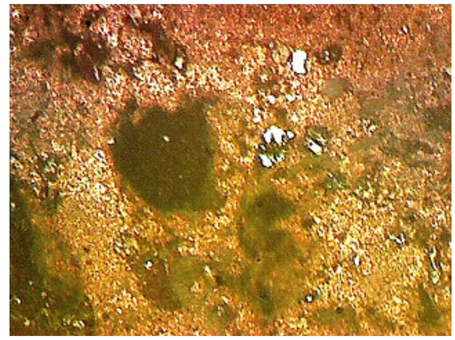
Table 3. Fabrication and sintering of composite specimens

At 850^{o}C - 5% of SiC



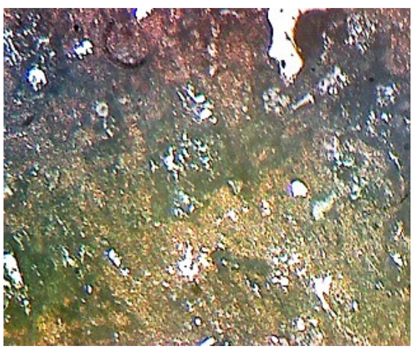
At 850^{o}C **-** 5% of SiC Two fields have been captured for images of the microstructure. The photo micrographs shows the completely fusion areas during sintering with negligible quantum of Free copper left un-fused. The matrix also shows the presence of grey particles of Sic and dark agglomerated particles of free graphite in the copper matrix. The sintered matrix of copper shows very fine equi-axed grains of copper

At 900^{o}C-5%SiC



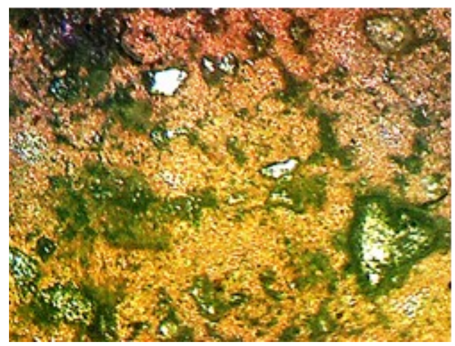
At 900^{o}C-5%SiC Two fields have been captured for images of the microstructure. The photo micrographs shows the completely fusion areas during sintering with negligible quantum of Free copper left un-fused. The matrix also shows the presence of grey particles of Sic and dark agglomerated particles of free graphite in the copper matrix. The sintered matrix of copper shows very fine equi-axed grains of copper. As more percentage of SiC is mixed large and fine particles of SiC is distributed in the matrix.

At 850^{o}C - 10% of SiC



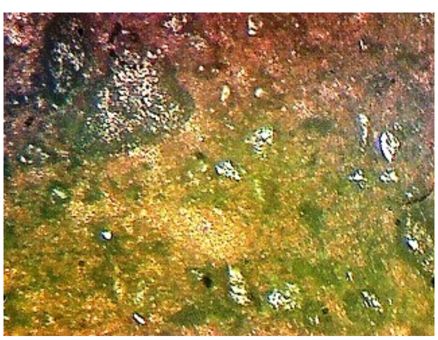
At 850^{o}C - 10% of SiC Two fields have been captured for images of the microstructure. The photo micrographs shows the completely fusion areas during sintering with negligible quantum of Free copper left un-fused. The matrix also shows the presence of grey particles of Sic and dark agglomerated particles of free graphite in the copper matrix. The sintered matrix of copper shows very fine equi-axed grains of copper. As more percentage of SiC is mixed large and fine particles of SiC is distributed in the matrix.

At 900^{o}C -10%SiC



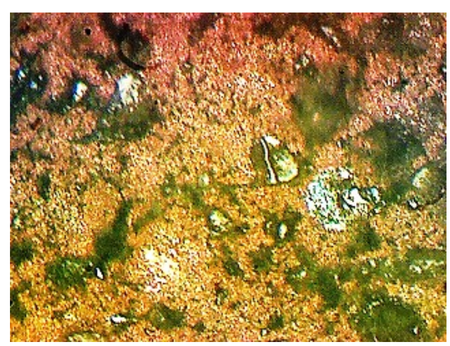
At 900^{o}C -10%SiC Two fields have been captured for images of the microstructure. The photo micrographs shows the completely fusion areas during sintering with negligible quantum of Free copper left un-fused. The matrix also shows the presence of grey particles of Sic and dark agglomerated particles of free graphite in the copper matrix. The sintered matrix of copper shows very fine equi-axed grains of copper.

At 850^{o}C -15% of SiC



At 850^{o}C -15% of SiC Two fields have been captured for images of the microstructure. The photo micrographs shows the completely fusion areas during sintering with negligible quantum of Free copper left un-fused. The matrix also shows the presence of grey particles of Sic and dark agglomerated particles of free graphite in the copper matrix. The sintered matrix of copper shows very fine equi-axed grains of copper. As more percentage of SiC is mixed large and fine particles of SiC is distributed in the matrix.

At 900^{o}C - 15%SiC



At 900^{o}C - 15%SiC Two fields have been captured for images of the microstructure. The photo micrographs shows the completely fusion areas during sintering with negligible quantum of Free copper left un-fused. The matrix also shows the presence of grey particles of Sic and dark agglomerated particles of free graphite in the copper matrix. The sintered matrix of copper shows very fine equi-axed grains of copper. Only large grains of copper are left unfused.

Table 4. Microstructure test result on composite specimens

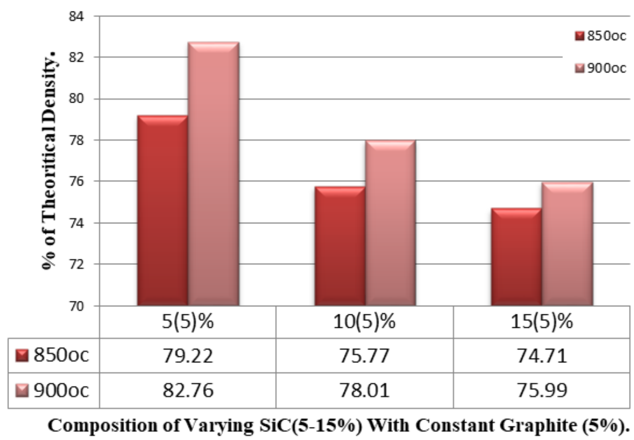


Figure 1. Density Result For Constant Graphite 5%

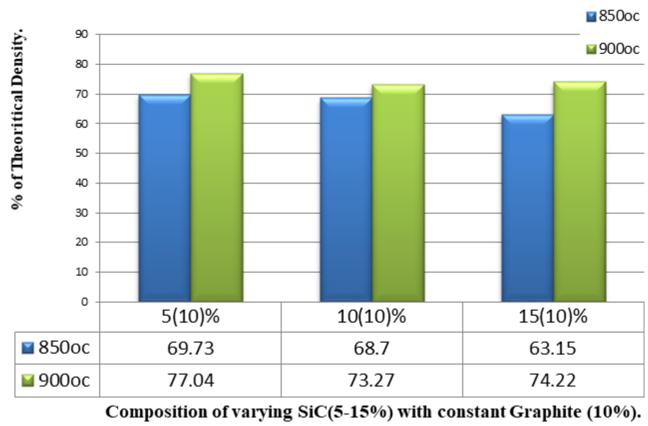


Figure 2. Density Result For Constant Graphite 10%

2.4. Physical Property

Density test is done for finding the theoretical density and sintered density using the following formula to find out the percentage of the theoretical density of the every samples for different composition.

Theoretical density = (ρ of Cu x Vd fraction of the Cu) + (ρ of SiC x Vd fraction of the SiC) + (ρ of Gr x Vd fraction of the Gr)

Sintered Density = (Weight of sample in air / loss of sample weight) x Density of Water. Or Density = mass /volume

Density of the composites was measured by using Archimedes principle, First the dry weight

Hardness test was carried out on Vickers's micro hardness testing machine. Prior to the hardness test both side of the specimens were polished with emery paper to ensure for even distribution load. Testing load range: 10 grams to 1 Kg Load; Vernier caliper least count: 0.01 mm; Available Hardness testing Scale: HV, HRA, HRC, 15N, 30N etc., are specifications of the machine.

Compression test was carried out on Universal testing machine. Prior to the compression test both side of the specimens were polished with emery paper to ensure for even distribution load. Lubrication was done by applying the grease on both sides of the sample so that the load could transfer without any Discontinuity and to minimize the friction at the interface. Loads of 400.800,1200 Kg etc with a constant interval 400 kg were applied until fracture appear on the surface of the specimen. And finally change in dimensions along the length and diameter was noted at the load and the corresponding true stress verses true strain data was plotted to obtain flow curve in accordance with equation (1). Testing load range: Max 5 Ton; Digital Encoder: Auto Instruments; Gear rotation speed (for gradual loading): 1.25. 1.5 & 2.5 mm /min are specifications of the machine.

$$\sigma = k\ \varepsilon n \quad (1)$$

Where, σ = True stress in MPa ; k= True strength; n= Strain Hardening exponent

Microstructure test

Internal micro structure of composite specimens was studied by optical microscope. Prior to this test Potassium Dichromate Solution as Reagent (for copper) applied on both sides of the samples

for 10-30 sec[15].Table.4. shows microstructure test result on composite specimens.

3. RESULTS AND DISCUSSION

3.1. Density

Density Study Shows and decreasing trend with increase in content of Silicon carbide.The percentage of theoretical density increased with increasing in sintering temperature soaking time for a particular composition may be due to better bonding as shown in Fig.1. and Fig.2.

3.2. Hardness test results

Hardness Study Shows an increasing trend with increase in content of Silicon carbide. The temperature without soaking time shows the decreasing in Hardness value compare to low temperature with soaking time This Effect is due to the better diffusion bonding between the materials during low temperature with soaking time during the microwave sintering. The Vickers's Hardness Number (VHN) increases with the increase in SiC composition at 850^{o}C sintering temperature. Few deviations occurred like slight decrease in hardness with increase in SiC percentage at the same sintering temperature mainly due to the presence of unwanted impurities like oxygen, nitrogen, etc. The Vickers's Hardness Number also increased with the increase in sintering temperature for a particular composition of SiC with 5% of graphite with slight exceptions at 900^{o}C possibly due to impurities present or due to non-uniform heat distribution of SiC throughout the copper matrix Fig.3.

3.3. Compressive test results

Compressive strength of each composition is proved through the compressive strength machine it is also known as crush testing machine which is computerized one with the help of FEI software the breaking loads are calculated and compared and the displacement analysis also taken for the comparison numerical analysis.

Maximum compressive strength is obtained at 15 vol. % SiC and const. 5 vol% of graphite reinforced MMC at 850^{o}C with soaking time. It is observed that the compressive strength at 850 degrees with soaking time has produced more effective strength than the 900 degree with rapid fast that is without soaking time.

From the value It is observed that the compressive strength at 850 degrees with soaking time has produced more effective strength than the 900 degree with rapid fast that is without soaking time (Fig.4).

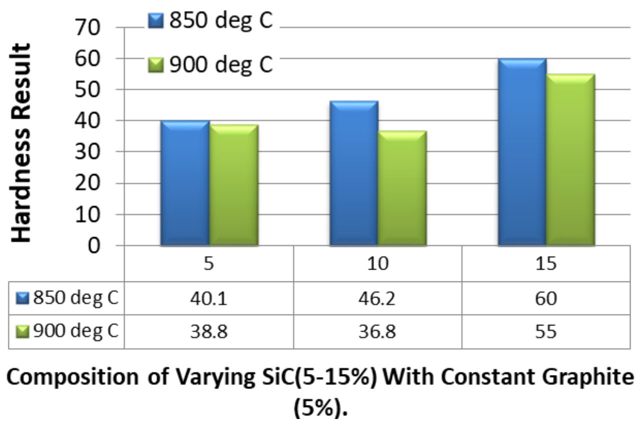


Figure 3. Hardness test Result

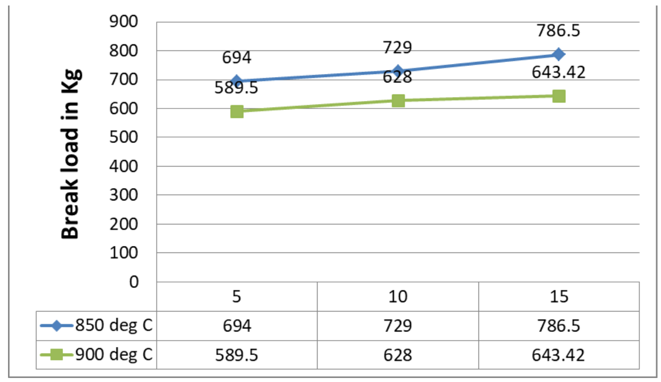


Figure 4. Compressive test result for Constant graphite 5%

4. CONCLUSIONS

The following conclusions can be drawn from the present investigation

1. The copper-Silicon carbide-graphite composites were successfully fabricated by microwave sintering process.

2. (a) The percentage theoretical density decreased with the increase in SiC and graphite content of the different samples sintered at a low temperature with soaking time where the percentage theoretical density is decreased too short at a high temperature without soaking time.

(b) The percentage theoretical density increased with the soaking time with sintering temperature for a particular composition of the composite due to better bonding between the particles and reduction in the number of voids at low temperature with soaking time.

3. (a) The Vickers's Hardness Number (VHN) increases with the increase in SiC composition at 850^{o}C sintering temperature. Few deviations occurred like slight decrease in hardness with increase in SiC percentage at the same sintering temperature mainly due to the presence of unwanted impurities like oxygen, nitrogen, etc.

(b) The Vicker's Hardness Number also increased with the increase in sintering temperature for a particular composition of SiC with 5% of graphite with slight exceptions at 900^{o}C possibly due to impurities present or due to non-uniform heat distribution of SiC throughout the copper matrix.

4. The mixing of composite powder mixture results in very fine and homogeneously distribution of reinforcement throughout the matrix is studied through the microstructure test. It is observed that the sintering at 850 has produced more effective fused matrix compared to rapid sintering at 900 degrees.

5. Maximum compressive strength is obtained at 15 vol. % SiC and const. 5 vol% of graphite reinforced MMC at 850^{o}C with soaking time. It is observed that the compressive strength at 850 degrees with soaking time has produced more effective strength than the 900 degree with rapid fast that is without soaking time.

6. Due to the presence of unwanted impurities like oxygen, etc are some of the reasons for the result variation caused in experimental analysis while compare displacement through the Ansys 14.0 Software. From that Displacement Analysis result concluded that the percentage error is decreased while the SiC content is increased with 850 degree temperature with soaking time.

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