ARTICLE IN PRESS

Advanced Powder Technology

Advanced Powder Technology xxx (2017) xxx-xxx

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

An investigation on the effect of sintering mode on various properties of copper-graphene metal matrix composite

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ARTICLE INFO

Article history:
 Received 7 October 2016
 Received in revised form 22 February 2017
 Accepted 20 April 2017
 Available online xxxx

- 22 *Keywords:* 23 Composites
- 23 Composites 24 Powder proce
- 24 Powder processing 25 Electrical propertie
- 25 Electrical properties26 Mechanical properties
- 27 Microstructure
- 28 Optical microscopy

ABSTRACT

The present work investigates the effect of sintering mode and graphene addition on the microstructural, mechanical and electrical properties of copper–graphene metal matrix composites reinforced with varying amounts (0.9, 1.8, 2.7 and 3.6 vol%) of graphene particles fabricated through powder metallurgy route. Sintering was carried out at 900 °C in 95%N₂-5%H₂ (forming gas) atmosphere with a heating rate of 5°/min for conventional and 20 °C/min for microwave with a holding time of 60 min in both cases. All the composites were found to couple well with microwave field and had resulted in 63% reduction in the processing cycle time as compared to the conventional process. Micro-structural analysis revealed the homogeneous distribution of graphene in copper matrix. Copper-graphene composites exhibited excellent wear resistance due to higher hardness and excellent lubricating nature of graphene. It was observed that porosity has a significant effect on the electrical conductivity values.

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46 1. Introduction

47 Copper metal powder has been widely used in engineering applications because of its high electrical and thermal conductivi-48 49 ties combined with excellent corrosion resistance and ease of fab-50 rication [1,2]. Pure copper has wide variety of applications such as electrical contacts in relays, magnetrons in microwaves, electro-51 magnets, vacuum tubes, heat sinks, welding electrodes, semi-52 conductors, microchips, piping systems, automobiles, etc. [3-5]. 53 Copper is not considered as a good material for structural applica-54 55 tions due to its poor mechanical properties especially at elevated 56 temperature [6]. Therefore the researchers have been investigating 57 the effect of different ways to improve its mechanical properties. 58 The most effective method is making a Cu based composite by 59 reinforcement addition of a suitable material [7]. Graphene is a 60 2-D allotrope of carbon and is considered as the strongest material ever tested with a tensile strength of 130 GPa, Young's modulus of 61 1 TPa and excellent lubricating properties in both dry and humid 62 63 conditions [8-11]. It is considered an excellent reinforcement 64 material because of its excellent mechanical strength, thermal con-65 ductivity (5300 W/mK), electrical conductivity, and large specific 66 surface area (SSA- 500-1200 m²/g) [12]. The dispersion of graphene

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Researchers at the Korean Advanced Institute of Science and 72 Technology recently reported that graphene reinforced composite 73 materials exhibited up to 500 times tensile strength than the raw 74 or monolithic material. Muhammad Rashad et al. investigated 75 the effect of graphene on magnesium based metal matrix nano 76 composites and reported uniform dispersion of multilayer gra-77 phene in the matrix that led to enhanced mechanical properties 78 [13]. Varol et al. fabricated multilayer graphene copper nano com-79 posites by employing flake powder metallurgy and conventional 80 sintering process. They reported that the composite resulted in 81 reduced density, improved hardness and electrical conductivity 82 due to non-homogeneous distribution of multilayer graphene in 83 the copper matrix [14]. Zhang et al. studied the strengthening 84 effect of graphene nano platelets and reduced graphene oxide in 85 copper matrix through a modified molecular level mixing process 86 87 and spark plasma sintering process [15]. Kim et al. fabricated Cu and Ni graphene nano layered composites with layer thickness of 88 70 nm and 100 nm for Cu-Gn and Ni-Gn nano composites respec-89 tively. The Cu-Gn and Ni-Gn nano composites exhibited strength 90 of 1.5 and 4 GPa respectively. These values were the highest values 91

http://dx.doi.org/10.1016/j.apt.2017.04.013

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Please cite this article in press as: C. Ayyappadas et al., An investigation on the effect of sintering mode on various properties of copper-graphene metal matrix composite, Advanced Powder Technology (2017), http://dx.doi.org/10.1016/j.apt.2017.04.013



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in copper matrix is challenging due to the relatively high difference in densities of the matrix and reinforcement phase. The high interfacial contact area of graphene and the mis-match in thermal conductivities result in complex behavior of the resultant composite. An investigation to all these aspects is still in its infancy.

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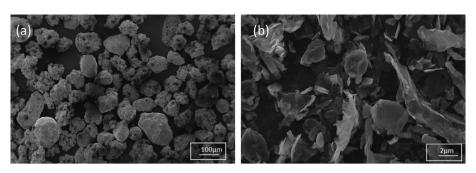


Fig. 1. Scanning electron micrographs of as received (a) copper powder and (b) graphene powder.

 Table 1

 Powder characteristics of the as received powders.

Characteristics	Copper	Graphene
Apparent density (g/cc)	4.5	0.015
Tap density (g/cm)	5.3	0.12
Flow rate in secs, 50 g	30	Non flowing
Particle size (µm)		
D ₁₀	13.82	3.90
D ₅₀	30.53	8.84
D ₉₀	62.02	17.77
Theoretical density, g/cm ³	8.96	2.2
Surface area, m ² /g	0.246	0.842
Shape	Spherical	Flaky
Purity	>99.5%	99.99%

reported for any MMCs ever fabricated [9]. Literature presented here mainly depicts the improvement in properties of the composites as compared to the monolithic material as a result of graphene addition. The major strengthening mechanisms in Cu-Gn composites are also identified.

97 Copper graphene composites in general exhibit superior electri-98 cal and thermal conductivity with low coefficient of thermal 99 expansion (CTE), good lubricating properties and improved 100 mechanical properties [16-18]. Copper-graphene composites find 101 application in bearing materials, heat spreaders, electro-friction 102 materials and a variety of applications which require low thermal 103 expansion coefficient, high electrical and thermal conductivity 104 with higher hardness values [19]. The challenge to develop graphene based metal matrix composites is to address the agglomer-105 106 ation tendency and poor wettability of graphene and metal matrix [20]. Microwave heating is a rapid sintering technique for the con-107 solidation of various materials. The major advantage of microwave 108 sintering over conventional sintering is that it provides rapid heat-109 ing resulting into much finer microstructure [21,22]. Material get 110 111 heated up as a result of the coupling reaction between samples and the electromagnetic wave. In addition to this, the grain bound-112 ary diffusion is promoted by the decrease in the activation energy 113 for sintering [23,24]. Microstructural homogeneity is preserved 114 115 even though the heating rates are high. Though sintering process are many, microwave sintering is projected as environment 116 117 friendly and energy efficient technique [24-30]. Zheng et al. 118 reported that microwave sintering could result in enhanced densification, smaller grain size due to faster heating rate and lower sin-119 120 tering temperature [31]. Limited numbers of researches are reported on metal based composite materials processed by powder 121 122 metallurgy route [32-35]. Manoj Gupta et al. synthesized alu-123 minium based composites by employing microwave sintering. It 124 was reported that the process parameters used in powder metal-125 lurgy route coupled with microwave sintering which was suitable in producing hybrid composite materials with improved 126 127 microstructure, good distribution of reinforcement and mechanical

characteristics [36]. Adrian Goldstein et al. conducted microwave 128 sintering on ceramic mixtures. Results showed a dense composite 129 with high hardness [27]. Flora Molinari et al. synthesised M type 130 hexaferrite in a single mode microwave cavity and reported that 131 the precursors couple well with microwave mode without any sus-132 ceptor. The magnetic properties of samples heated in microwave 133 cavity for 30 min were similar to those exhibited by conventionally 134 sintered samples for a time period of 12 h [37]. Yoon et al. made 135 comparisons between microwave and conventional pressure-less 136 sintering. Relative density of microwave sintered samples crossed 137 99% and phase transformation rate achieved was 100% at a temper-138 ature of 1600 °C whereas for conventional pressure less sintering 139 such high density and phase transformation rate could not be 140 achieved even at sintering temperature of 1850 °C [38]. Xu et al. 141 studied the characteristics of microwave melting of copper pow-142 der. Changes in microstructure and densification were analyzed. 143 Results showed that copper powder could be quickly heated to 144 melting. Microwave heating efficiency and the heating rate were 145 found to be higher with the decrease in particle size and increase 146 in microwave power. Heating rate had a linear relationship with 147 the reciprocal of the particle size. Microstructure and density indi-148 cates that the densification process accelerates when the tempera-149 ture is above 900 °C. At lower temperature the migration of the 150 matter for copper particles is mainly internal diffusion [28]. Xu 151 studied the effect of kinds and contents of additives on mechanical 152 properties, phase transformation and microstructure behavior of 153 Si_3N_4 ceramic materials processed by microwave sintering [39]. 154

Literature points out that addition of even a small amount of graphene to the monolithic material can improve the properties to a considerable extent [40,41]. In this work composites of copper

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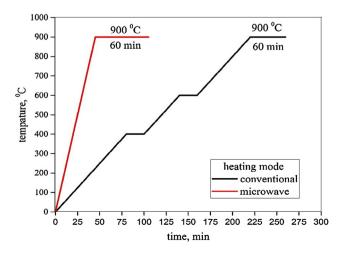


Fig. 2. Comparison of the temperature profile for conventional and microwave sintering modes.

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