

Microstructural Characterization and Mechanical Behavior of Copper Matrix Composites Reinforced by B₄C and Sea Shell Powder

Mathiazhagan Sankar^a, S. Pradeep Devaneyan^b, D. Peter Pushpanathan^b, Dawid Myszkawski^a

^a Warsaw University of Technology, Faculty of Production Engineering, Narbutta 85, 02-524 Warsaw, Poland

^b Christ College of Engineering and Technology, Department of Mechanical Engineering, Moolakulam, Puducherry-605005, India

*e-mail: myszkadawid@wp.pl

Received: 15 September 2017/Accepted: 8 March 2018/Published online: 30 March 2018

This article is published with open access at AGH University of Science and Technology

Abstract

This paper investigates the microstructural and mechanical properties of copper metal matrix composites reinforced with B₄C and crushed sea shell particles (fabricated using powder metallurgy). In powder form, copper is widely used in structural applications. Copper also possesses very good electrical and thermal conductivity, ductility, and corrosion resistance. B₄C is the third-hardest-known material that also possesses excellent toughness and wear resistance. Sea shells are readily available along coastal areas. Therefore, an attempt has been made in this work to investigate the feasibility of its utilization in powder metallurgy. Two batches of samples were prepared. In the first batch, the percentage of boron carbide and copper powder were varied, and seashell powder was not included. In the second batch, the percentages of B₄C, copper, and sea shell powder were varied in order to assess the change effected by the sea shell material. The sintered samples of both batches were subjected to microstructural characterization to ascertain the homogeneous distribution of the reinforcements. The microhardness and wear resistance of all of the fabricated samples were assessed. The results confirmed that the inclusion of 2% sea shell powder (by weight) with 10% boron carbide improved the wear resistance and hardness of the composite.

Keywords:

Cu-base MMC, Sea Shell Powder-B₄C Reinforcement, Wear Properties, Microstructure

1. INTRODUCTION

Components for switch gears were converted from wrought copper to copper P/M parts to achieve a considerable reduction in cost while still maintaining good electrical conductivity. These components are used in switch boxes with capacities of up to 600 amperes [1, 2]. Because of its excellent thermal conductivity, a P/M copper component weighing 1/2 pounds was selected for a heat sink in an electronic application. Components for 150- and 250-ampere fuse blow-outs (used in coal mining equipment) were converted from machined copper bar stock to a P/M copper part. Although drilling and tapping were still required, the conversion resulted in a cost saving of about 25% [3–5]. Materials such as silver and gold have physical properties higher than that of copper, but they are not economical when compared to the cost for utilizing them in the manufacturing sector [1]. Elemental powders like copper are more compressible, which helps produce compacted objects with good strength. Aerospace automotive drive shafts, ground vehicle brake rotors, and explosive engine components widely use copper [3]. Copper and copper alloy P/M parts can be pressed and sintered to their final shape and size, usually with the desired surface finish and with no draft

angles [6, 7]. They can also be sized to close tolerances by coining or repressing, thus eliminating much of the machining required when other metal-forming procedures are used. They can be machined, plated, and joined by brazing, and some of the alloys can be heat-treated to enhance their properties [8]. Using commercial automatic presses, copper and copper alloy P/M parts can be produced rapidly and accurately at an average rate of 1000 parts per hour. Some very simple shapes have been produced on rotary compacting presses at rates as high as 63,000 parts per hour [9]. Sizes can range from miniature parts smaller than the ball of a ball-point pen to bearings weighing over 100 pounds. The physical and mechanical properties of copper and copper alloy P/M parts are comparable with those of cast and wrought copper-based materials of a similar composition. However, the P/M process allows for a flexibility that the other processes do not possess [10, 11]. Parts can be produced that vary in density from the low-density required for self-lubricating bearings or filters to nearly the theoretical density of wrought parts. P/M parts are produced with a minimum of raw material loss and greatly reduced processing wastes, resulting in a new approach towards lowering overall costs. As a further advantage, there is no pollution of the environment in the production of P/M parts.

The properties of P/M parts are influenced by the density attained. Densification can be increased by additional operations such as double pressing-double sintering or forging [12]. In the present work, 99% commercially pure copper powder of a size of 75 microns was used along with boron carbide and crushed sea shell powder for fabricating the composite. Since sea shells are readily available in coastal areas, the effect of sea shell powder on the microstructural and mechanical properties of Cu-base MMC was investigated. Two batches of samples were produced by varying the percentage weight of boron carbide alone in the first batch. Ground sea shell powder was added to the second batch of samples, and the percentage weight of both reinforcements were varied to understand the effect of sea shell powder. Metallographic characterization of the fabricated samples was done using optical microscopy. The hardness and wear resistance of the composite samples were assessed by subjecting them to Rockwell hardness and Pin on Disc tests, respectively.

2. EXPERIMENTAL

2.1. Powder and die preparation

Commercially available pure copper powder of A 75-micron mesh size was purchased from Alfa Aesar. Boron carbide powder was also procured from the same manufacturer. Sea shells available along the shores of the Bay of Bengal near Puducherry, India, were collected and subjected to a rigorous cleaning procedure to remove all impurities. The sea shells were crushed into a fine powder using a ball mill and then sieved to a size of 250 microns. These powders were then blended as per requirement to prepare to batches of samples with and without sea shell powder. The setup used for producing the PM billets was comprised of a die, bed, lower punch, upper punch, ejecting block, and ejecting rod (as shown in Figure 1). EN24T steel was used to fabricate the compacting setup. EN24T is generally used in components such as gears, shafts, studs, and bolts, and its hardness is within a range of 248/302 HB [13]. Figure 1 shows the die and its components utilized for this work.

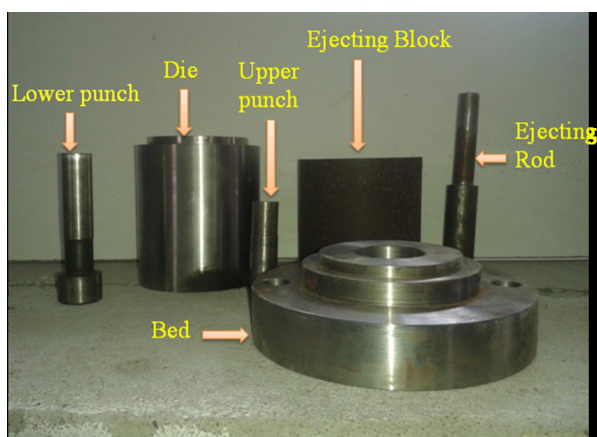


Fig. 1. Die and its components

2.2. Fabrication of B₄C-sea shell-copper metal matrix composite

The copper metal matrix composites were prepared in two batches. The first batch did not contain sea shell powder, whereas it was included in the second batch of samples. In the first batch, copper powder of 100%, 98%, 96%, 94%, 92%, and 90% compositions and boron carbide powder of 2%, 4%, 6%, 8%, and 10% (by weight) were blended. The second batch of samples contained copper in the proportions of 98%, 96%, 94%, 92%, and 90%. Boron carbide of 2%, 4%, 6%, 8%, and 10% as well as sea shell powder of 10%, 8%, 6%, 4%, and 2% (by weight) were also blended in a conical flask using a mechanical blender. Each sample was compacted in a uni-axial universal testing machine with a pressure of 260 MPa in order to obtain samples of a size of 20 mm in diameter and 10 mm in length. The green compacts were then subjected to sintering. Sintering is done to bond the metallic particles, thereby increasing the strength and hardness of the final product. This is usually carried out at temperatures between 70% and 90% of the metal's melting point [14–16]. However, the hardness of the sintered part increases remarkably when the density of the sintered part becomes smaller [9]. The compacts were then placed in silica crucibles filled with fine sand and kept in a muffle furnace for 6 hours at a temperature of 900°C and then allowed to cool down in the furnace itself [17]. The detailed composition of each sample is present in Table 1.

Table 1
Sample chemical composition details

Sample Details	Copper, %	B ₄ C, %	Sea shell powder, %	
Without adding of sea shell	0A	100	0	
	1A	98	2	
	2A	96	4	
	3A	94	6	
	4A	92	8	
Adding of sea shell	5A	90	10	
	1B	98	2	10
	2B	96	4	8
	3B	94	6	6
	4B	92	8	4
5B	90	10	2	

The sintered samples are shown below (Fig. 2). 1A, 2A, 3A, 4A, and 5A represent the samples without sea shell powder, and samples 1B, 2B, 3B, 4B, and 5B were those prepared with sea shell powder. A pure copper sample was also prepared in order to compare the properties.

2.3. Microstructural characterization and Mechanical testing

The sintered samples were polished with emery sheets of 100, 220, 500, 800, and 1200 scales. Fine polishing was done on a disc-polishing machine using alumina paste and distilled water [18]. A microstructural analysis was performed by an Olympus optical microscope equipped with AnaliSys image processing software. The samples were analyzed under various magnifications. Macroscopic and microscopic images of all samples were captured. All samples except the pure copper sample confirmed the homogeneous distribution of the reinforcements. An in-depth analysis of the samples was done using a Hitachi S-3500 scanning electron microscope. A qualitative analysis of the samples was done using EDS. The results confirmed the presence of all elements as per the composition. Each sample was analyzed in six places in order to confirm the uniform distribution of the particulates.

The samples were subjected to a dry sand/rubber wheel abrasion measurement apparatus as per ASTM G 65-16 [19]. This method is used to determining the resistance of

metallic materials to scratching abrasion by means of the dry sand/rubber wheel test. It is the intent of this method to produce data that will reproducibly rank materials in their resistance to scratching abrasion under a specified set of conditions. Abrasion test results are reported as volume loss. The speed of the steel disc was 200 rotations/min, and a 130N load was given to the lever arm. Each sample was tested for 5 min, and control box was used to control the time and rotations. The abrasive resistance of the samples was determined by calculating the difference between the weight before the abrasion test and weight after the abrasion test given in Formula (1).

$$\Delta g = \text{weight before abrasion} - \text{weight after abrasion} \quad (1)$$

The hardness of the composites was then measured with a Rockwell hardness tester with a 1/16"-diameter steel ball. A load of 100 kg was applied, and the hardness was read on the "B" scale.

3. RESULTS AND DISCUSSION

3.1. Microstructural characterization and composition

The optical micrographs confirmed the presence of boron carbide particulates. The micrograph of the pure copper is displayed in Figure 3.



Fig. 2. Sintered samples

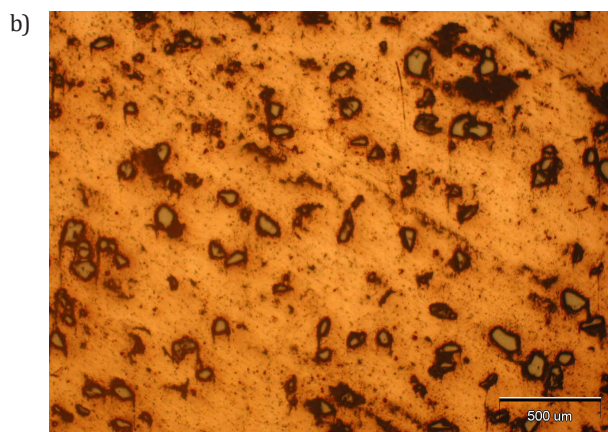
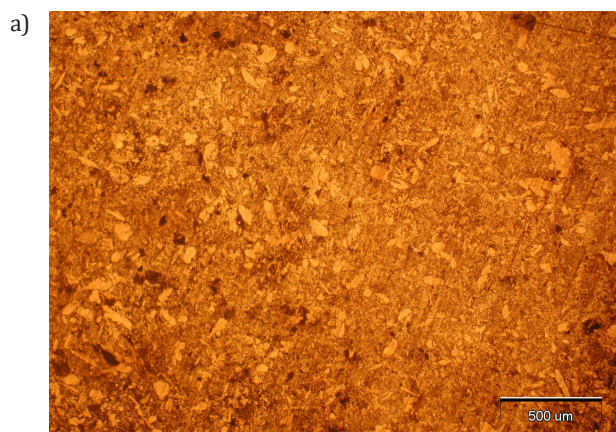


Fig. 3. Pure Copper (a) and Boron Carbide (b) in Copper Matrix

The particles are loosely packed due to the lesser weight percentage of the boron carbide. The density of the boron carbide steadily increased with the increase in weight percentage. Figure 4 shows the sample with boron carbide along with sea shell powder.

The presence of sea shell powder can be easily figured out from this micrograph. The sea shell particles are comparatively larger than the boron carbide. Due to the presence of large sea shell particles, the boron carbide particulates were agglomerated and heterogeneously distributed. However, in Sample 5B shown in Figure 5, the percentage of boron carbide particles was the highest and sea shell powder was the lowest.

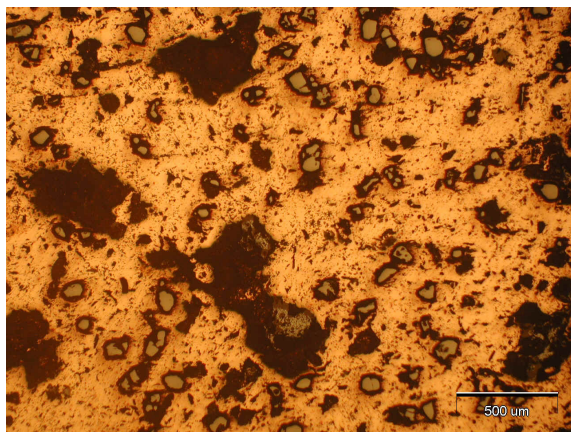


Fig. 4. Boron carbide and sea shell powder in copper matrix

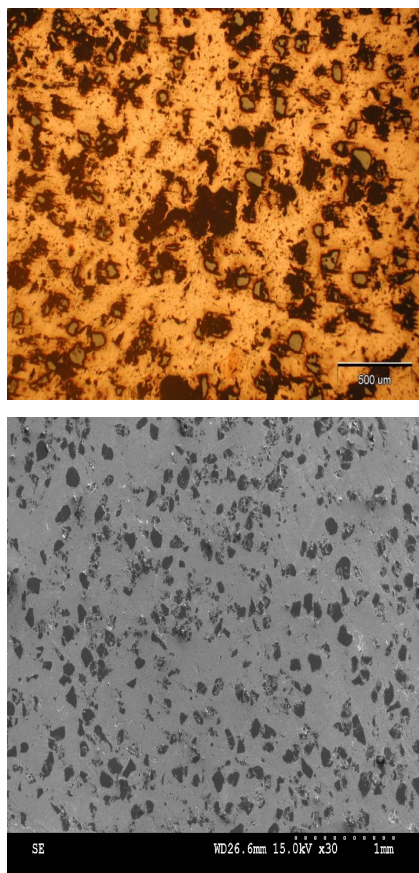


Fig. 5. Micrographs of samples with content of 10% B₄C with 2% SS

Hence, the homogeneous distribution of B₄C particulates was observed. A few larger sea shell particulates were also noted. The SEM image of this sample presented below presents a better view of the uniformly distributed fine boron carbide particles.

3.2. Hardness

From the following graph, it is clear that the hardness of the composite increases with the increase in boron carbide weight percentage. As boron carbide is the third-hardest material, it definitely improved the hardness of the metal matrix composite. Figure 6 shows that Sample 2 exhibited lowest hardness of 74 on the Rockwell B scale. However, this is four times greater than the hardness of pure copper (which was 1B). The hardness value continued to improve with the increments of the weight percentage, and a maximum hardness of 95 was achieved for Sample 5A with 10% boron carbide particles (as shown in Figure 7).

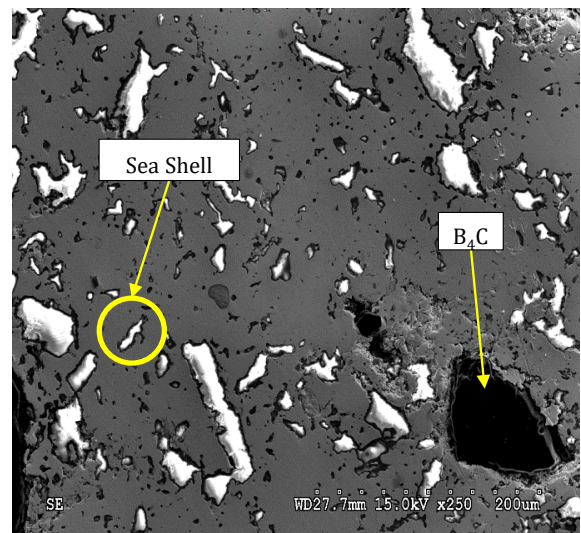


Fig. 6. Micrographs of samples with content of 4% B₄C and 8% SS

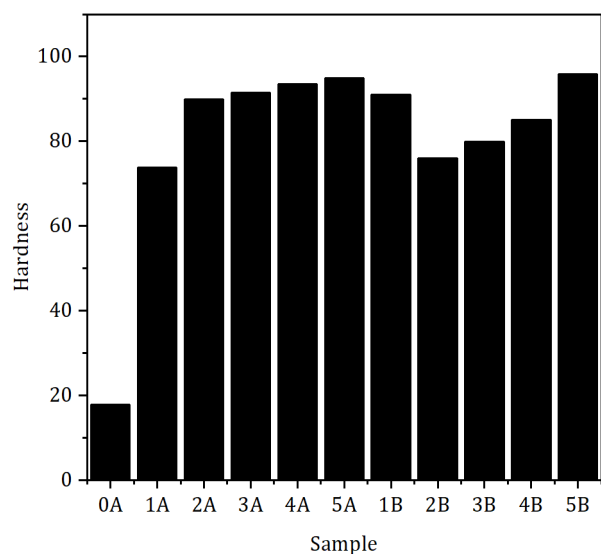


Fig. 7. Hardness vs composition of samples

This is due to the high dislocation density at the interface of the boron carbide and copper grains. Grain boundary pinning due to the homogeneously distributed B₄C particles caused the hardness to increase by restricting the movement of the copper grains. This can be confirmed by referring to the microstructure of this sample, which reveals a homogeneous distribution of the particulates. With the addition of sea shell powder, the hardness suddenly plunged to 76 for Sample 2B (which had 8% sea shell powder). This is due to the large size of the sea shell particles, which failed to pin the grain boundary dislocation. However, the hardness improved with the reduction in the sea shell powder percentage and increase in the boron carbide particles. The maximum hardness of 96 (which was 5 times higher than pure copper) was achieved for Sample 5B, which had 10% boron carbide and 2% sea shell powder. This shows that a very small inclusion of sea shell contributes to the improvement in hardness. This could be due to the Orowan strengthening mechanism initiated by the less-populated incoherent sea shell particulates. The sudden decrease in hardness could also be attributed to the voids that were formed due to inhomogeneous consolidation. The SEM image of Sample 2B presented below (with 4% B₄C and 8% sea shell powder) shows an uneven distribution of sea shell particles with voids. This is the reason for the sudden drop in the hardness of the composite. The effect of the composition of all samples on the hardness of the composite is shown in the plot.

3.3. Wear behavior

An analysis of the worn surface morphology was done in order to investigate the wear pattern of the metal matrix composites. The analysis of wear tracks revealed that both adhesive and abrasive wear mechanisms were present, and Figure 8 shows the wear morphology of Sample 2B.

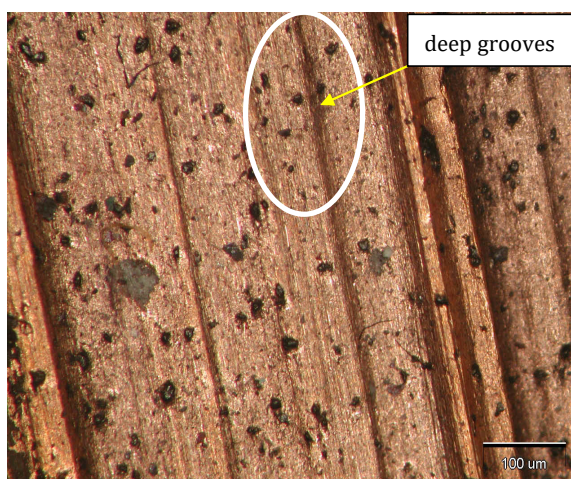


Fig. 8. Wear morphology of sample 2B

This micrograph of Sample 2B exhibits deep grooves caused by adhesion and abrasion on the surface of the composite. This sample in particular exhibited the highest

wear rate. The hardness values are in complete agreement with the wear test results. These grooves are bigger in size; hence, it is confirmed that they are formed due to the delamination of larger sea shell particles (which later paved the way for abrasive wear). This extensive delamination resulted in the highest wear rate of this sample. The wear rate continued to decrease as the percentage composition of sea shell powder decreased. The wear-loss trend could be deciphered from the plot presented in Figure 9.

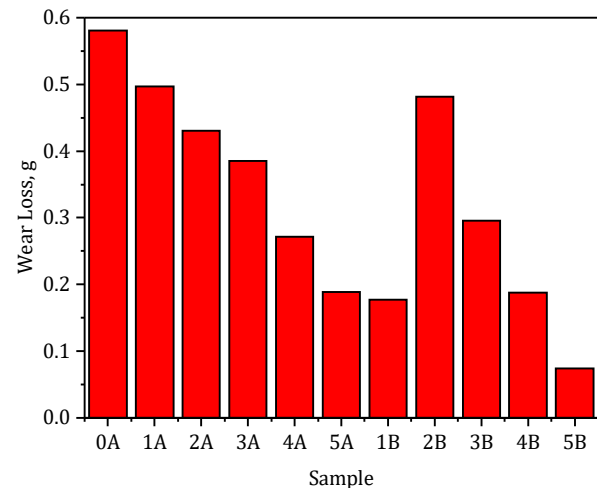


Fig. 9. Wear loss vs. composition of samples

The lowest wear rate was observed in Sample 5B (which had 10% B₄C and 2% sea shell particles). This reveals that boron carbide plays an important role in controlling the wear rate. However, the 2% of sea shell particles increased the wear resistance by 1% as compared to Sample 5A (which also contained 10% boron carbide without sea shell particles). This clearly indicates that inclusion of sea shell particles at a low percentage enhances the hardness and wear resistance of the composites. Hence, in this work, the composition of 10% B₄C and 2% sea shell powder has been deemed the optimum composition for obtaining the best hardness and wear resistance.

4. CONCLUSIONS

Copper metal matrix composites reinforced with boron carbide and sea shell powder were synthesized successfully through the powder metallurgy route. The sintered samples were free from porosity and surface defects. The hardness of the composite samples reinforced with 10% boron carbide and 2% sea shell powder exhibited the maximum hardness, which was five times higher than the pure copper sample.

The wear resistance of the composites improved with increases in the weight percentage of boron carbide but deteriorated with increases in sea shell powder composition. The best wear resistance was observed for the optimal weight percentage of 2% sea shell powder and 10% boron carbide. It is concluded that both the hardness and wear resistance of the composites improved as compared to the pure copper metal matrix composites.

REFERENCES

- [1] Celebiefi G., Zeytin S. & Bindal C. (2012). The effect of SiC particle size on the properties of Cu-SiC composites. *Materials and Design*, 36, 633–639.
- [2] Moustafa S.F., Abdel-Hamid Z. & Abd-Elhay A.M. (2002). Copper matrix SiC and Al₂O₃ particulate composites by powder metallurgy technique. *Material Letters*, 53, 244–249.
- [3] Wang H., Zhang R., Hu X., Wang C.-A. & Huang Y. (2008). Characterization of a powder metallurgy SiC/Cu-Al composites. *Journal of Materials Processing Technology*, 197(1–3), 43–48.
- [4] Adelstein S.J., & Vallee B.L. (1961). Copper Recommended Dietary Allowances. Washington, D.C.: National Research Council, Food Nutrition Board, NRC/NAS. 1980, 151–154.
- [5] Walker W.R. & Keats D.M. (1976). An investigation of the therapeutic value of the ‘copper bracelet’ – dermal assimilation of copper in arthritic/rheumatoid conditions. *Agents and Actions*, 6(4), 454–459.
- [6] Biurrun A., Caballero L., Pelaz C. & Gago A. (1999). Treatment of a Legionella pneumophila – Colonized Water Distribution System Using Copper-Silver Ionization and Continuous Chlorination. *Infection Control and Hospital Epidemiology*, 20(6), 426–428.
- [7] Lin Y.C., Li H.C., Liou S.S. & Shie M.T. (2004). Mechanism of plastic deformation of powder metallurgy metal-matrix composites of Cu-Sn/SiC and 6061/SiC under compressive stress. *Material Science and Engineering A*, 373(1–2), 363–369.
- [8] Pleger T.C. (2002). A Brief Introduction to the Old Copper Complex of the Western Great Lakes: 4000–1000 BC. Proceedings of the Twenty-seventh Annual Meeting of the Forest History Association of Wisconsin, Oconto, WI, 5 October, 10–18.
- [9] Fuchsloch J. & Brush E.F., (2007). Systematic Design Approach for a New Series of Ultra-NEMA Premium Copper Rotor Motors. *EEMODS 2007 Conference Proceedings*, 10–15 June, Beijing.
- [10] Olivia M., Mifshella A.A. & Darmayanti L. (2015). Mechanical properties of seashell concrete. *Procedia Engineering*, 125, 760–764.
- [11] Stelter M. & Bombach H. (2004). Process Optimization in Copper Electrorefining. *Advanced Engineering Materials*, 6(7), 558–562.
- [12] Celebi Efe G.F., Altinsoy I., Ipek M. & Bindal C. (2011). Some properties of Cu-SiC composites produced by powder metallurgy method. *Kovove Materialy – Metallic Materials*, 49(2), 131–136.
- [13] Günter J. (1999). Copper: Its Trade, Manufacture, Use and Environmental Status, edited by Kundig, Konrad J.A., ASM International, Ohio, Chapter 3: Products, 141–192, Chapter 5: Applications, 331–375.
- [14] Rickard T.A. (1932). The Nomenclature of Copper and Its Alloys. *Journal of the Royal Anthropological Institute of Great Britain and Ireland*, 62, 281–290.
- [15] Weißgärber T., Lefranc, G., Schulz-Harder, J., Meyer, A. & Stoecker, O. (2003). Cu-SiC composites for thermal management application. *Advances in Powder Metallurgy & Particulate Materials*, part 6, Metal Powder Industries Federation, Princeton, NJ, 33–40.
- [16] Celebi Efe G.F., Yener T., Altinsoy I., Ipek M., Zeytin S. & Bindal C. (2011). The effect of sintering temperature on some properties of Cu-SiC composites. *Journal of Alloys and Compounds*, 509(20), 6036–6042.
- [17] Celebi Efi G.F. (2010). Development of Conductive Copper Composites Reinforced with SiC. Ph.D. thesis, Sakarya University, Institute of Science and Technology, June 2010.
- [18] Schubert T., Brendel A., Schmid K., Koeck T., Ciupiński Ł., Zieliński W., Weißgärber T. & Kieback B. (2007). Interfacial design of Cu/SiC composites prepared by powder metallurgy for heat sink applications. *Composites: part A: Applied Science and Manufacturing*, 38(12), 2398–2403.
- [19] Schubert T., Trindade B., Weißgärber T. & Kieback B. (2008). Interfacial design of Cu-based composites prepared by powder metallurgy for heat sink application. *Material Science and Engineering A*, 475(1–2), 39–44.