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Sintering and Dimensional Analysis of Cu/CNTs via a Powder Metallurgy Route

*Nor Shamimi Shaari**
Advanced Mechanics Research Group,
Faculty of Mechanical Engineering,
Universiti Teknologi MARA,
13500 Permatang Pauh, Pulau Pinang, Malaysia
**shamimi@uitm.edu.my*

Muhammad Hussain Ismaill, Aidah Jumahat*
Industrial Metallurgy Research Group (IMReG),
Faculty of Mechanical Engineering,
Universiti Teknologi MARA, 40450, Shah Alam, Malaysia
*hussain305@uitm.edu.my

Russell Goodall
Department of Materials Science and Engineering
The University of Sheffield, Sir Robert Hadfield Building,
Mappin Street, S1 3JD, United Kingdom

ABSTRACT

Recently, carbon nanotubes (CNTs) reinforced metal matrix composites (MMCs) have attracted an increasing interest, due to their promising properties such as high Young's modulus and tensile strength. CNTs are considered to be an attractive reinforcement material for light weight and high-strength metallic matrix composites. When powder metallurgy (PM) is used to form these MMCs (such as Cu/CNTs composites), the sintering parameters are crucial in obtaining good final parts. This work attempts to investigate the effect of sintering parameters on physical properties in these MMCs. The process comprised of mixing of Cu powder with CNTs, compacting of the powder mixture to form green parts and sintering using a quartz tube furnace under argon atmosphere. In this study, four trials of heating rate were performed and evaluated before sintering process was conducted. Finally, the green body was initially heated isothermally at 100°C for 1 hour with heating rate of 1.0 °C/min and sintered at a temperature of

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900°C for 2 hours with heating rate of 0.5 °C/min. The composites contained 0 to 4 vol% of pristine CNTs (PCNTs) and acid treatment CNTs (ACNTs), respectively. It was observed that as the CNTs content increased, the density of the composites was also decreased owing to low density of the CNTs. The sintered part in this present work has undergone volume shrinkage ranging from 12.41% to 17.02% and 13.36% to 19.30%, for Cu/PCNTs and Cu/ACNTs respectively. It was found that the heating rate played an important role in producing a good sintered part. A high heating rate induced large thermal stress and possibly increased the pore volume, leading to swelling. As part of the temperature sensitivity, the heating rate was useful in controlling microstructure evolution in sintering. A correlation between the volume shrinkage, density and porosity of the good sintered part was confirmed in this work.

Keywords: *Copper, Carbon nanotubes, Powder metallurgy, Sintering, Heating Rate*

Introduction

In many industries such as sport, aerospace, automotive and others, composites are widely used. In many cases, polymers are preferred for the matrix material due to their low cost, light weight and ease of processability compared to metals. However, for some applications, the advantages of ametal matrix can offset those difficulties. Metal can maintain its strength and toughness (which are ususally significantly higher than polymers) at elevated temperature and can also conduct heat and electricity, which is important in some applications [1].

In recent years, metal matrix composites (MMCs) have been widely investigated, including aluminium-matrix composites, nickel-matrix composites and copper-matrix composites [2]–[4]. The production of MMCs based on copper (Cu) has been particularly considered in electrical applications and as bearing materials due to their excellent thermal and electrical conductivity properties [5]. As well as the matris, in any MMC there also has to be a reinforcement phase, and one of the promising is carbon nanotubes (CNTs), owing to its superior mechanical strength as well as excellent electrical and thermal conductivities. CNTs have special properties such as high elastic modulus (~1 TPa), large strength (~40 GPa), very low density (1.5-2 g/cm^3) and high thermal and electrical conductivity along their length (about twice as high as diamond, while electric current-carrying capacity is about 1000 times higher than copper wires) [6]-[7]. Their incorporation in metallic matrices often leads to the production of composites with higher mechanical, electrical and thermal properties. This hasled to an increasing belief that carbon nanotubes arethe most effective reinforcing fillers in self-lubricating metal matrix composites for structural engineering and functional devices [8].

CNTs-reinforced copper composite is a novel material having high potential for electrical sliding contact applications like brushes for electric motors [9]. Incorporation of CNTs as a reinforcement material in Cu should improve the properties of the current material, following the trends discussed above. However, these have not been achieved in metal-CNTs composites prepared via the powder metallurgy method. Such materials usually possess lower mechanical properties than expected due to the problem of agglomeration of the CNTs, and resulting porosity [7]. The key factor to enhance the strength of metal matrix/CNT nanocomposites is to achieve a homogenous distribution of CNTs during the blending step [10]. If the nanotubes remain as clusters or agglomerates, the composite properties will be deteriorated [11]-[12]. In order to improve the dispersion of CNTs in a metal matrix, high energy mechanical milling can be used [13-14]. Another approach used to increase the dispersion of CNTs in the matrix material is a treatment in a mixture of acid prior to consolidation with metal powder [3],[15]–[18]. By such means, improved bonding between CNTs and Cu powder was obtained, leading to improvement in mechanical properties.

Up to now, researchers have explored various aspects of these problems, including research on Cu matrix/CNTs composites containing different volume fractions (0–4 vol%) of multi-walled carbon nanotubes (MWNTs). Where CNTs are used the amount tends to be smaller, since nano-sized particles have a large surface area compared to micron-sized powder. This surface area makes the CNTs tend to agglomerate and entangle with one another, and the problem is made worse with larger quantities of CNTs. In the early contribution of Shaari and co-authors [18], the treatment of CNTs were performed in chemical solution of sulphuric acid (H_2SO_4) and nitric acid (HNO_3) which both treated and untreated CNTs were studied in order to observe the difference in terms of the dispersions and microstructures. The results show that pristine CNTs had more agglomerated and tangled up each other compared to after treatment process which had less agglomeration and well dispersed in the matrices. Besides, sintering process is the one of crucial part in order to get a good final parts of Cu/CNTs composites and at the same time to identify the best parameters during the sintering process. Thus, these factors have a significant impact and the success informulating Cu/CNTs composites. In this work**,** powder metallurgy (PM) route is employed. This is a well-known production technology, common for MMCs, which involves three main processing steps: (i) mixing the elemental or alloy powders, (ii) compacting the mixture in rigid dies and (iii) sintering the green body in a controlled atmosphere furnace to allow the particles to bond [20]. In order to produce a well sintered part, different heating rates were used and the sintered parts were analysed in terms of the volume shrinkage, density and porosity.

Experimental Procedures

There were two types of materials used in this research: (i) copper powder as the matrix and (ii) multi-wall carbon nanotubes as the reinforcing material. The copper powder was supplied by Acros Organics with 99.0% purity, and particle size of < 45 µm. It was produced by an electrolytic process which resulted in it having a dendritic shape. Meanwhile, Flotube 9000 Multi-wall carbon nanotubes (MWCNTs) were produced by a catalytic vapor deposition (CVD) process, and supplied by CNano Tech, China with > 95.0% purity, having an average diameter of 11 nm with an average length of 10 µm. In consideration for comparison and increasing the dispersion of CNTs, they were subjected to an acid treatment process using strong acid solution (H_2SO_4 : HNO_3 ; 3:1). Figure 1 shows the microstructures of the as-received Cu and CNTs as well as similar views after the acid treatment, reproduced from earlierwork by the authors [21]. The process began with mixing the matrix and reinforcement material, followed by compaction into the desired shape and finally sintering in an argon atmosphere. Figure 2 shows the schematic diagram of the powder metallurgy route for Cu/CNTs composites used in this work.

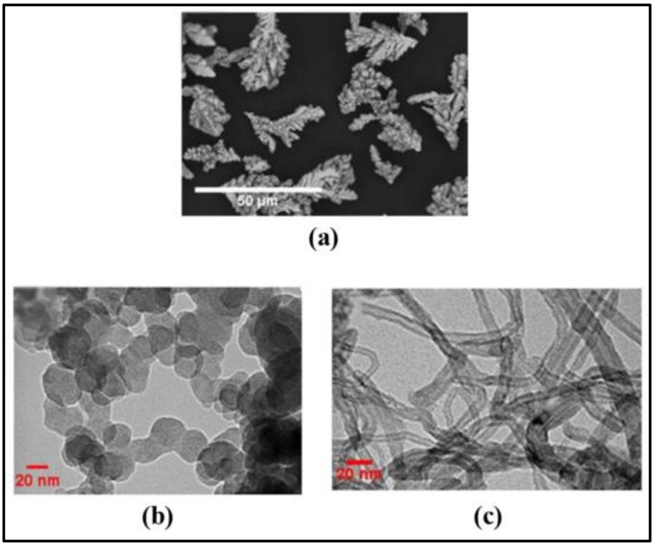

Figure 1: Microstructure of (a) Cu powder, (b) PCNTs and (c) ACNTs [21].

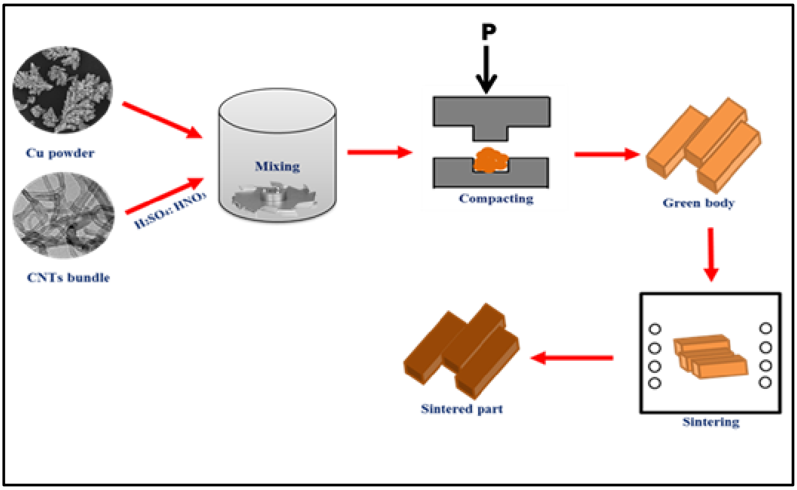

Figure 2: Schematic diagram of PM route for Cu/CNTs composites.

The pristine CNTs are labelled as PCNTs while CNTs after acid treatment process are labelled as ACNTs. The powder was weighed using an analytical balance (resolution 0.0001 g) and mixed using a planetary ball mill (Fritsch pulverisette). A stainless steel jar was used with steel balls as the milling media,with aball to powder ratio (BPR) of 10:1 at 250 rpm for 5 hours with 15 minutes interval (in reverse mode) to avoid heat generation due to friction between the particles during the mixing process. The mixtures of copper and CNT powder were pressed into steel dies under a compression force of 8 tonnes, with 3 minutes holding time. After that, the sample was ejected gently from the die and the green part was produced. Then, the part was heated in a quartz tube furnace under argon atmosphere. Table 1 shows the parameters used in the study and the profile for the sintering process.

Table 1: Parameter study and profile for the sintering process

Parameter	HR 1	S.Temp 1	ST 2	HR 2	S.Temp 2	ST 2
Trial 1	5	900	90	-	-	-
Trial 2	3	100	60	2	900	120
Trial 3	2	100	60	1	900	120
Trial 4	1	100	60	0.5	900	120

*HR-Heating Rate;S.Temp-Sintering Temperature; ST- Soaking Time

For analysis purposes, well sintered parts of Cu/Pristine CNTs (PCNTs) and Cu/acid treated CNTs (ACNTs) were examined in terms of volume shrinkage, density and porosity. The volume shrinkage was calculated by measuring the dimensions of the samples before and after the sintering process. Then, the density of the sample was calculated using the following Equation (1):

$$\rho = \frac{A}{A-B} x(\rho_0 - d) + d \qquad (1)$$

where ρ is the density of the sample, A is the weight of the sample in air, B is the weight of the sample in liquid, ρ_0 is the density of liquid (approximately 0.99730 g/cm^3) and d is the density of air (approximately 0.001 g/cm^3). The porosity (ε) of the samples was calculated using the following Equation (2):

$$\varepsilon = \left(1 - \frac{\rho}{\rho_0}\right) x100 \qquad (2)$$

where ρ is the measured density by the Archimedes principle and the theoretical density ρ_0 was measured using pycnometer density.

Results and Discussion

Sintering of Cu reinforced with CNTs powders

The sintering process and the parameters used during the sintering process are the most crucial steps in order to get good final parts of Cu/CNT composites. For the first trial, samples with cylindrical shape (Ø = 13 mm; *h* = 10 mm) were sintered at temperatures up to 900 °C with a soaking time of 90 minutes and a heating rate of 5 °C/min in an argon atmosphere. This trial used similar conditions to the study conducted by Goudah and co-workers [22], except for the gas flow rate, which was here set to 1.5 l/min. In their study, copper particles with a spherical shape were used and sintered at various temperatures for comparison purposes. They concluded that performing sintering at 900 °C for 90 minutes was sufficient to produce a good product. The parameters were given in Table 1.

Figure 3 (a) and (b) show the images of the sintered samples where two out of four of the samples (Figure 3 (a)) unexpectedly swelled instead of shrinking and the formation of a pink layer was also observed covering the outside of the samples. It was suggested that the samples were exposed more to the argon gas and crucially, the trace amounts of oxygen that it contained since they were located near to the inlet gas (refer to Figure 4). Meanwhile, Figure 3(b) showed a cracked dark brown layer was observed. It was observed that the effects of the sample position and different gas flow pattern near the sintering surface resulted in variations of oxygen content from the incoming gas flow in the local sintering atmosphere, which affected the self-gettering process of the parts during sintering [23]. Besides thatthe swelling of the sample was possiblly due to gas trapped in closed pores; this would lead to an increase in internal pressure and an increase in pore size, and thus sample volume [23].

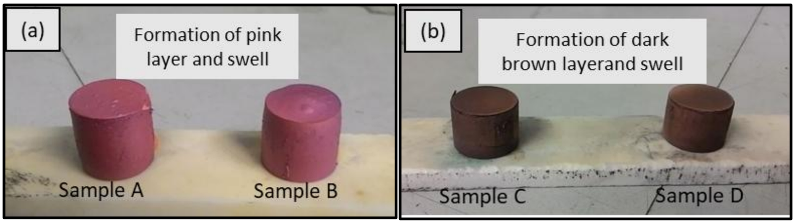

Figure 3*:* Samples of Cu/CNTs after sintering with sintering temperature of 900 °C with heating rate of 5.0°C/min.

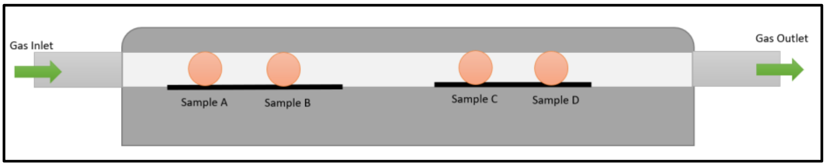

Figure 4: Samples position inside the quartz tube furnace under an argon gas flow.

Figure 5 shows the samples for second and third trial which were sintered in two stages (refer Table 1). Figure 5 (a) shows an example image of acracked sample A and swollen sample B. For the third trials, Figure 5 (b) shows that sample A had swollen and sample B had cracked. From the above results, it was inferred that the defective samples from trial 1 to trial 3 had occurred due to the rapid heating rate used during the sintering process. The high heating rate induced large thermal stress between different areas of the part leading to cracking, and possibly increased trapped gas pressure and pore volume, thus leading to swelling [24].

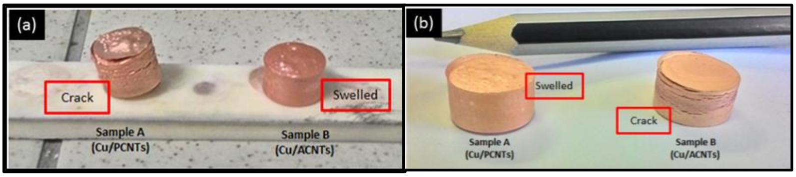

Figure 5*:* Samples of Cu/CNTs after sintering with sintering temperature of 900 °C with (a) heating rate 1 (3.0 °C/min) and heating rate 2 (2.0 °C/min) and (b) heating rate 1 (2.0 °C/min) and heating rate 2 (1.0 °C/min).

A final trial was done with different heating profile, and also with different sample shapes. In this trial, the rectangular samples (*l* = 13 mm; *w* = 37 mm; *h* = 10 mm) were produced. The samples were finally sintered in an argon atmosphere in two stages. In the first stage, the samples were heated from room temperature (RT) to 100 °C at a heating rate of 1.0 °C/min and held at that temperature for one hour to remove any moisture or air trapped in the samples. During second stage, the temperature was slowly raised up to 900 °C/min with the heating rate of 0.5 °C/min and held at that temperature for 2 hours before they were cooled down to room temperature. Figure 6 compares the images of green and sintered parts for this trial. The result shows that, after the sintering process, the sample had shrunk in most directions. It was found that the shrinkage observed was inversely proportional to the CNT content.

From Figure 7, the data show that by increasing the CNT content, the shrinkage process is inhibited, owing to the highly rigid, connected web-like distribution of the CNTs within the material [25]. The sintered part in this present work has undergone volume shrinkage ranging from 12.41% to 17.02% and 13.36% to 19.30%, for Cu/PCNTs and Cu/ACNTs respectively. A study conducted by Peigney et al. proposed that the CNT web-like structure becomes very well interconnected, very rigid, thus inhibiting the rearrangement process which decreases the volume shrinkage as the CNT content is increased [25]. It was found that the heating rate was the important aspect to produce a good sintered part. As part of the temperature sensitivity, changing the heating rate was useful in controlling microstructure evolution in sintering [24]. A slow heating rate was used to prevent cracks and thermal stress in the samples. From observation during the experimental process, the unique aspects such as extremely high temperatures, fast heating rates, and short reaction times, may control the behavior the of the sample and microstructure [26].

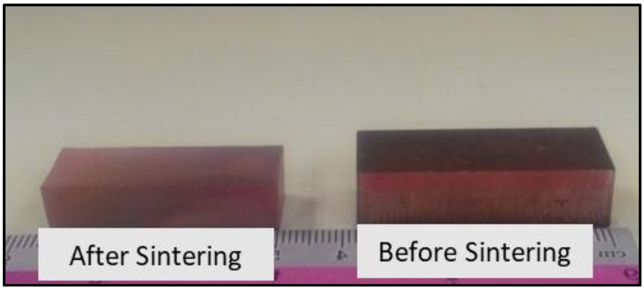

Figure 6: Images of green part and sintered part of Cu/CNTs composites.

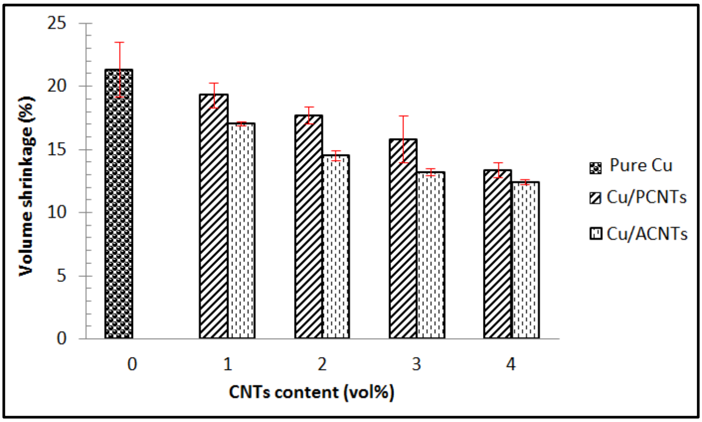

Figure 7*:* Volume shrinkage of sintered samples with different CNTs contents of PCNT and ACNTs.

Density and porosity

In this work, the sintered parts were initially measured in terms of their dimensions followed bya density test using the liquid weighing (Archimedes) method and compared to the theoretical value in which the results were obtained using the pycnometer density. Relative density was calculated by dividing the measured density with the theoretical density. While for porosity, the value was calculated based on the value of measured and theoretical density. Table 2 shows the measured density, theoretical density and porosity of sintered samples with different vol.% of CNTs

Figure 8 and Figure 9 show the variation in relative density and porosity of the Cu/CNTs nanocomposites with different vol% of CNTs. The graphs show that by increasing the CNTs contents, the density of the composites decreased. It was due to the lower density of the CNTs in the copper matrix which, as per rule of mixtures, theoretical density reduces with the increase in volume fraction of CNTs in copper [9]. The results also showed that porosity increases as the fraction of CNTs increased. It was because the CNTs are gathered at grain boundaries of the copper matrix which is the origin of the pore formation [27]. It was also noticed that Cu/ACNTs composites had a lower density value and high porosity compared to Cu/PCNTs, because of the density of acid treated CNTs is itself lower compared to pristine CNTs, due to the elimination of the impurities during the treatment process. Furthermore, the CNTs have a hollow structure that contributed to pore formation. This result was consistent with many previous reports [3][9][28-31].

Table 2: Measured density, theoretical density and porosity of sintered samples with different vol.% of CNTs

Vol.% CNTS	1	2	3	Average density (g/cm^3)	Theoretical density (g/cm^3)	Relative density (g/cm^3)	Porosity (%)
0	8.6231	8.6015	8.6363	8.6203	8.8718	97.17	2.83
1PCNTs	8.3156	8.3658	8.4649	8.3821	8.6364	97.06	2.94
2PCNTs	8.2918	8.3215	8.3092	8.3075	8.6203	96.37	3.63
3PCNTs	8.1905	8.2569	8.4268	8.2914	8.7435	94.83	5.17
4PCNTs	8.0961	7.8526	7.8602	7.9363	8.7729	90.46	9.54
1ACNTs	7.3475	7.1452	7.1898	7.2275	7.6445	94.55	5.45
2ACNTs	6.9578	7.1596	7.2663	7.1279	7.6934	92.65	7.35
3ACNTs	7.005	6.9281	6.784	6.9057	7.576	91.15	8.85
4ACNTs	6.5236	6.6618	6.7667	6.6507	7.4967	88.72	11.90

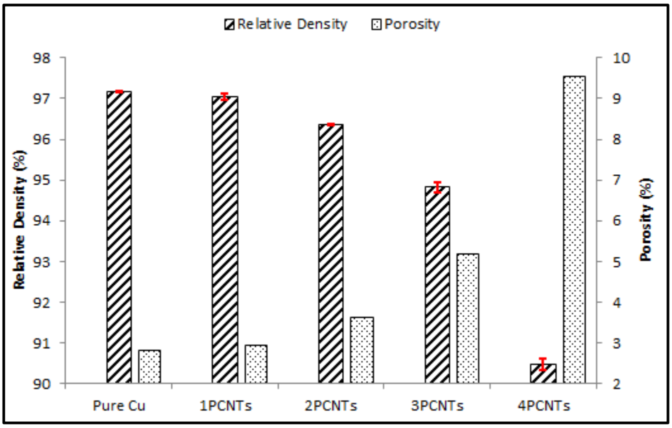

Figure 8: Relative density and porosity of Cu/PCNTs composites with different percentages of carbon nanotubes.

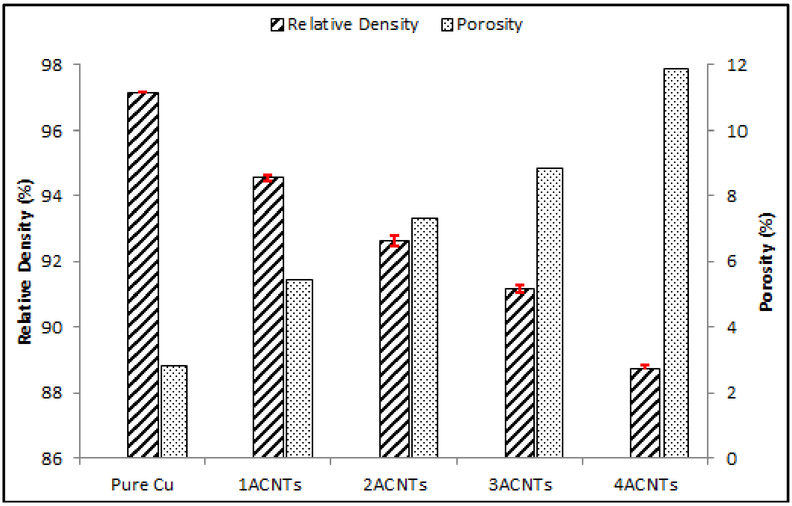

Figure 9: Relative density and porosity of Cu/ACNTs composites with different percentages of carbon nanotubes.

Conclusions

Copper matrix composites reinforced with carbon nanotubes have been successfully fabricated using a conventional powder metallurgy route. Regarding the physical properties, the Cu/CNTs composites were successfully formulated after four attempts by varying the heating rate. In this work, the samples were heated in an argon atmosphere at a heating rate of 1.0 °C/min from room temperature to 100 °C, held at that temperature for one hour and then were heated at a heating rate of 0.5 °C/min to 900 °C/min and finally held at that temperature for 2 hours before letting it to cool down toroom temperature. The slow heating rate was selected to prevent cracks and thermal stress in the samples. The composites contained 0 to 4 vol% of either PCNTs or ACNTs. It shows that by increasing the CNT content, the shrinkage process was inhibited owing to their highly rigid connected web-like distribution within the material. It was also observed that as the CNT content increased, the density of the composites was also decreased owing to low density of the CNTs. Besides, the shrinkage volume of the composites were also inversely proportional with the CNT content ranging from 12.41% to 17.02% (Cu/PCNTs) and 13.36% to 19.30% (Cu/ACNTs).

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