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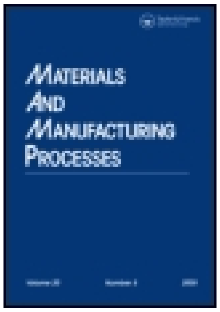

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Characterization of Copper Carbon Composites Manufactured Using Electro-less Precipitation Process

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Abstract

Copper-carbon composites with various copper nano-particulates volume fractions were manufactured using electro-less precipitation process. Composites with 10% and 20% carbon volume fractions and copper samples were sintered at two different sintering temperatures 700 and 800C. The microstructure was studied using high resolution optical scanning investigations. Experimental results indicate that the coefficients of thermal expansion and electrical resistivity intensified with the escalation of the carbon volume fraction in the composites. However, the stress-strain response was found to decrease with the increase of carbon volume fraction in the copper-carbon composites.

KEYWORDS: Electro-Less Precipitation, Nanocomposites, Failure.

1. INTRODUCTION

Metal and non-metal alloys have attracted the attention of numerous scientists aiming to develop an elite hybrid that combines the best properties of the two original materials [1-3]. The main advantage of the metal and non-metal alloys [4-6] is the superior stiffness of metals, while maintaining the structural thermal resistance and integrity of monolithic ceramics. A number of methods were proposed in the literature to fabricate metal-ceramic composites [7-11]. The general requirement is to use materials with controlled properties, such as thermal or electrical conductivity, strength, modulus of elasticity, thermal expansion and many others. This can be fulfilled by the group of metal and nonmetals composite materials [12]. Many of these controlled properties can be obtained with carbon powder reinforced copper matrix composites [13, 14]. Copper is known as a high density material with high thermal and electrical conductivity. However, it has very poor mechanical properties especially at high temperatures [15]. As a result of graphite's unique combination of properties, it is widely used for number of applications [16]. Graphite/carbon has a smooth touch, and high heat and electrical conductivities. Carbon also possesses high temperatures and corrosive environments resistance. Copper-carbon composites with high thermal conductivities can be achieved with diamond reinforcements [16].

The preparation method, the size of the used particulates, and the particulate adhesion matrix determine the properties of composites. Particulate size is a key factor influencing the final characteristics of the composites [17, 18]. Kiser et al. [19] reported that changing particulate size in the composites from micro-size to nano-size was essential to demonstrate change in the mechanical characteristics of the alloys. As reported in the literature [20], the addition of these nano particulates increases the rigidity and yield strength of the composite. Furthermore, smaller particulate sizes improve the fracture toughness [20], as studied in polyethylene matrix filled with calcium carbonate. Particulate interface adhesion with the matrix and particulate loading generally affect the mechanical properties of composites [18]. Hu et al. [22] presented various models for characterizing these mechanical characteristics.

The scope of this research is to manufacture copper-carbon composites using Electro-Less precipitation method with two copper nano-particulates volume fractions (10%C and 20%C) at different sintering temperatures (700 and 800C). The microstructure using high resolution optical investigations were thoroughly studied. The influence of copper nano-particulates on the bulk density, hardness, the thermal expansion coefficient, the electrical resistivity, and the stress-strain behavior of the manufactured composites were examined. In addition, the fracture surfaces were examined for all the tested composites.

2. MATERIALS PREPARATION

Carbon/Graphite has desirable material characteristics due to its strong ionic interatomic bonding. It exists in several hexagonal and di-hexagonal crystalline phases which are stable at elevated temperatures. Because of carbon's good dielectric properties, refractoriness, and excellent thermal properties, it is used in numerous applications [23]. Carbon is usable at high temperatures as its triple point is at 10.8 ± 0.2 MPa and 4,600 ± 300 ºK (~4,33°C) [24], as it transfers at about 3,900 ºK [25]. Carbon possesses high interaction resistance with most common reagents, other than hydrofluoric and phosphoric acids [26]. The carbon particulates with particulate sizes of 6 μm and 99.5% of purity were used in this study. Electro-less precipitation process [12, 25], was used to manufacture copper composites reinforced with carbon powder particulates. The copper-carbon composites were manufactured by coating the carbon particulates with copper nano particulates and then densification. The main advantages of this method [26] are the elimination of cross-contamination, cost reduction, and the enhancement of the materials deposited quality. In this research, the produced copper-carbon composites contained 10% and 20% carbon by volume fraction. The atomic percentages considered in this research are selected based on the large difference between the copper density (8.7 specific density), and the carbon powder density (2.25 specific density). The preparation of the composite materials was conducted through the following main three steps as presented in Figure 1[12].

Step 1 Activation and Sensitization of Carbon Particulates:

The reinforcement carbon particulates were surface activated and sensitized to enhance the copper adhesion with it is explained as follow, as presented in Fig. 1:

1- Salts of 35 gram of CuSOHO and 170 gram of KNaCHOHO, were dissolved in one liter of distilled water, where the solution pH was set to 12.5.

2- Carbon particulates were dropped into the solution, and mechanically mixed at 1400 rpm for one hour. The amounts of carbon particulates added were to obtain copper-carbon compositions of 80% and 90% of copper in volume fraction.

3- The carbon particulates were processed with 50% NaOH aqueous solution and were and mechanically mixed at 1400 rpm for about 25 minutes.

4- The sensitizing agent was prepared by mixing 10 gm/l SnCl.HO and 40 ml/l of 35 % HCl and setting the solution’s pH to (1~ 3).

5- Carbon was soaked in 100 ml sensitizing agent for each 10 gram of carbon particulates, and mechanically mixed at 1400 rpm for about 15 minutes.

6- After sensitization, carbon particulates were activated with palladium coating layer. An activating agent was added in the amount of 100 ml to 10 g of the prepared particulates for a small duration of 15 minutes. Palladium activation agent was prepared by mixing 2.5 ml/l of 35 % HCl and 0.02% PdCl. The produced solution was filtered, and the stimulated particulates were washed and dried.

Step 2 The Electro-less Precipitation Process:

The treated carbon particulates prepared in step 1 were surface coated with pure nano copper particulates as follows:

a) The copper particulates were chemically prepared from the same solution used in the copper coating process. This is done to generate copper particulates with the same properties of the Cu-coating layers on carbon particulates. Two chemical solutions A and B were used for creating the chemical bath [12].

b) 5 measures of solution A were mixed with 1 measure of solution B and the pH level of the solution was set to 12.5.

Step 3 The Compaction of Mixtures:

The produced powders were used for preparing the Cu-C samples and were shaped into standard rectangular and cylindrical shape specimens. The rectangular specimens were prepared with a 10mm x 30mm cross section, whereas the cylindrical specimens were manufactured with a radius of 5mm and pressed uniaxially with 300 MPa to form the required compacted mixtures. To eliminate moisture and any undesired substances, the green compact was then heated at a temperature level of 350°C for 3 hours. The temperature is then raised gradually to the required sintering temperatures which are 700º, and 800°C, at an increase rate of 5°C/min. To ensure homogenous heat distribution all over the samples, the furnace settings were fixed for one hour at the sintering temperatures. The furnace temperature was reduced with a slow decrease rate of 5°C/min, similar to the rising temperature rate till reaching the laboratory conditions.

The Cu-C composites were finally weighted to form two different volume fraction composites 10%C and 20%C calculated for the two different sintering temperatures 700 and 800C. The reason for selecting the sintering temperatures at 700º and 800ºC is due to experimental factors. When rising the temperature above 1100ºC, the melted copper overfills the die and the bunch losses the fraction of the designed copper within the sample irregularly. This is because the sintering process occurs simultaneously during pressing. Whereas, lowering the sintering temperature below 1000ºC, the copper in the samples do not completely melt, which leads to insufficient sintering. The density of each sample was measured by taking its weight in air and in water. The pure copper and carbon densities are 8.96 g/cm and 2.25 g/cm respectively. For Cu-C composites with 10% carbon volume fraction the densities were 7.21, and 6.73 g/cm, and for Cu-C composites with 20% carbon volume fraction the densities were 7.42, and 6.9 g/cm for the two sintering temperatures at 700º and 800ºC respectively.

3. MATERIAL EXPERIMENTATION AND CHARACTERIZATION

3.1. Sem Investigations

The investigated copper-carbon alloys were investigated using the scanning electron microscope (SEM) through the fabrication process.

Using differential scanning calorimetric Perkin-Elmer DSC7, the thermal propoerties of the samples was analysed under argon atmosphere and at a rate of 20 Kmin. Figure 2a presents SEM of the carbon powder particulates after surface activation treatment and before precipitation. Noticeable image distortion was reported and could be explained due to the carbon surface treatment with palladium coating layer. Figure 2b shows SEM after the electro-less precipitation where the uniformity of the shape of the carbon powder particulates can be clearly observed. Figure 3a presents the Cu-C composite after the electro-less precipitation process. As shown in the Fig. 3b the nano copper (matrix part) particulates are coating the different size carbon particulates. The homogeneity of the particulates coated distributed over the composites were clearly scrutinized. Fig. 3b is a higher magnification of the Cu-C composites with nano size copper particulates. SEM analysis of these copper particulates reveals that most of the nucleation during crystallizations was nearly homogenous nucleations because of the former oxygen elimination. Homogenous copper particulates with approximate size of 50 nm nano-crystallites were observed.

The change in the carbon particulates before and after the treatment was observed. The high resolution scan imaging of the carbon powder particulates was a challenging process. This was due to the light distraction associated with the particulates morphology [29]. It also could be explained due to the activation coat surface covering the carbon powder after treatment, where the surface is shinier. The reason for this homogenous dissemination is justified by the images. Carbon particulate was completely coated allowing minimal interaction among the carbon particulates. This results in a decrease of the debonding zones throughout the materials. This also could be explained through the macro hardness measurements. The average micro hardness readings (The MPa Brinell micro hardness type Shimadzu at 31.25 kg load) for Cu-C composites with 10% carbon volume fraction were 25.8, and 24.2, but for Cu-C composites with 20% carbon volume fraction were 26, and 24.8 for the two sintering temperatures at 700º and 800ºC respectively. This was consistent with the homogenous uniform distribution of the copper particulates through and surrounding the carbon particulates observed through the SEM investigations.

3.4. Electrical Resistivity Measurements And Coefficient Of Thermal Expansion

The measured electrical resistivity was converted into the international annealed copper standard conductivity (IACS) units as detailed in [12]. The high precision Micro-Ohmmeter, Omega CL8400 was utilized to measure the electrical resistivity (*ρ with units of $\mu\Omega.cm$*) of the copper–carbon alloys. Table 1 presents electrical conductivity measurements in $\mu\Omega.cm$ (which is $1/\rho$), for the Cu-C composites with two volume fractions of copper at two different sintering temperatures.

There is a strong correspondence between the thermal and the electrical conductivities of any metal [12]. Metals that are the good electrical conductors also possess good thermal conducting properties. However, increasing the temperature raises the thermal conductivity and reduces the electrical conductivity. Thus, the electrical resistivity can be calculated in terms of (*W*/*mK*). The coefficient of thermal expansion (CTE) was measured every 50ºC, over the temperature span between 50º and 300˚C, with an increasing rate of 10ºC per minute. The CTE results were calculated [12] as presented in Table 2.

Therefore copper composites reinforced with copper-carbon powder particulates with the highest copper volume fraction have higher electrical conductivity. Furthermore, the cu-c alloys with high fractions of copper have the highest CTE values. This is a result of the high densification breaking the continuous network of the copper matrix. Copper alloys reinforced with carbon powder have excellent electrical and thermal conductivities, great thermal expansion rates and high wear resistance properties.

3.5. Tensile Mechanical Measurements

Compression tests were conducted at room temperature with an Instron 8562 universal mechanical tester under quasi-static loading (strain rate of 8×10 to 1.3×10 s). Cylindrical specimens were prepared with a diameter of 3mm and a height of 5.5mm from cast rods. The samples were deformed until failure. To ensure consistency and homogeneity, three identical samples were prepared for each test case and exposed to the same loading conditions. The mean test value of all the three samples was reported in the results. The results indicated a measurement variation less than 9% and a cross-head rate of 1 mm/min. Figure 4 presents the stress-strain responses as presented with error bars for these composites.

As expected, the stress and temperature are the two most important factors influencing the atomic movements and therefore phase alterations. For pure copper, the elastic modulus value is 131 GPa [8]. The ultimate compression stress for copper composites with reinforced 10% volume fractions carbon particulates sintered at 800ºC reaches about 281.6 MPa with 5.8% fracture strain and Elastic Modulus of 14571.69 MPa. The ultimate compression stress for copper composites with reinforced 10% volume fractions carbon particulates sintered at 700ºC reaches about 262.4 MPa with 5.42% fracture strain and Elastic Modulus of 14928.47 MPa. On the other hand, the ultimate compression stress for copper composites with reinforced 20% volume fractions carbon particulates sintered at 800ºC reaches about 252.5 MPa with 4.24% fracture strain, and Elastic Modulus of 16292.53 MPa. Finally, the ultimate compression stress for copper composites with reinforced 20% volume fractions carbon particulates sintered at 700ºC reaches about 243.2 MPa with 3.78% fracture strain, and Elastic Modulus of 16677.54 MPa. For composites with carbon volume fraction 10%C and 20%C at the same sintering temperature an approximately a 10% reduction in the elastic modulus between is evident. The fracture surface visibly shows that the fracture occurs in the copper as the carbon particulates show high resistance to the fracture. SEM images of the fracture surfaces show a vein shaped structures presented in Figure 5a and 5b for the copper-carbon composites with two volume fractions 10%C and 20%C respectively at 800ºC sintering temperature. Higher SEM magnification for the copper-carbon composite with two volume fraction of 10%C is presented in Fig. 5c. Further examination of the fractured composites displays veins and undistinguished area distortion at various rates and layers. Moreover, Fig. 5c shows evident of delamination that reflects high fracture resistance.

4. CONCLUSION

This research focuses on the manufacture of copper-carbon composites using the Electro-Less Precipitation method with various copper nano-particulates volume fractions (10%C and 20%C) at different sintering temperatures (700º and 800ºC). Evaluation and determination of the effect of different carbon volume fraction and sintering temperature on the electrical, mechanical, and fracture characteristics of the alloys were successfully achieved. HR-SEM investigations revealed homogenous uniform distribution of the copper particulates surrounding the carbon particulates. The hardness of the alloys monotonically decreased with increasing the carbon in the composites and increased with increasing the sintering temperature. That is a clear indication of the strong bonding between the particulates at high sintering temperatures, which indicates high bulk density. Similarly, the electrical conductivity is decreased with increasing the carbon in the composites and is increased with increasing the sintering temperature.

For the copper-carbon composite with lower volume fraction of carbon the stress-strain response was the highest, and it decreases with increasing the carbon volume fraction in the composites. This is due to its nanostructure with high fraction of copper and the inter-granular surface that influences the stress resistance of the composites. This explains the fact that composites with higher carbon volume fraction show high fracture strain values compared to composites with lower carbon volume fraction. The decrease in the elastic modulus among alloys with various carbon fractions 10%C and 20%C at the same sintering temperature is evident, approximately 10% reduction. The modifications in the mechanical properties are due to the rise in the volume fraction of the carbon particulates in the copper-carbon composites.

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Table 1. The Electrical conductivity $\mu\Omega.cm$ of the copper composites reinforced with carbon powder particulates at the two sintering temperatures.

Sintering temp	10% C Vol. Fraction	20% C Vol. Fraction
700ºC	0.482	0.456
800ºC	0.51	0.466

Table 2. The Coefficient of Thermal Expansion (CTE) 10K of the copper composites reinforced with carbon powder particulates at the two sintering temperatures measured every 50ºC, over the temperature range from 50º to 300˚C.

Sintering temp	10% C Vol. Fraction		20% C Vol. Fraction	
700ºC	50	10.5	50	9.8
	100	10.9	100	10.3
	150	11.1	150	10.6
	200	11.3	200	11.0
	250	11.6	250	11.2
	300	12.0	300	11.6
800ºC	50	10.9	50	10.1
	100	11.2	100	10.6
	150	11.3	150	10.9
	200	11.8	200	11.3
	250	12.1	250	11.5
	300	12.5	300	11.9

Figure1. Schematic of the main electro-less precipitation dissolution process for manufacturing copper-carbon composites.

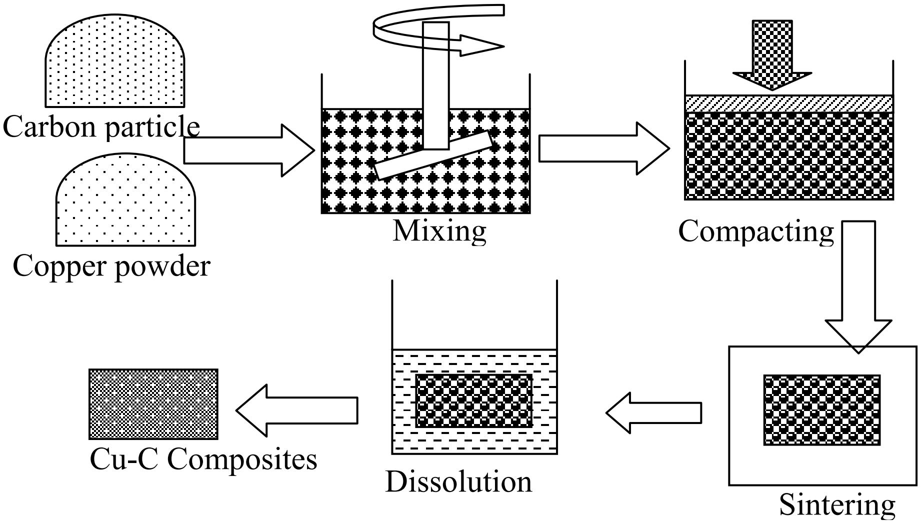

Figure 2. SEM Cu-C composites during electro-less precipitation process (a) carbon particulate after surface activation and before any copper precipitation, (b) the fully coated carbon particulate after the electro-less precipitation.

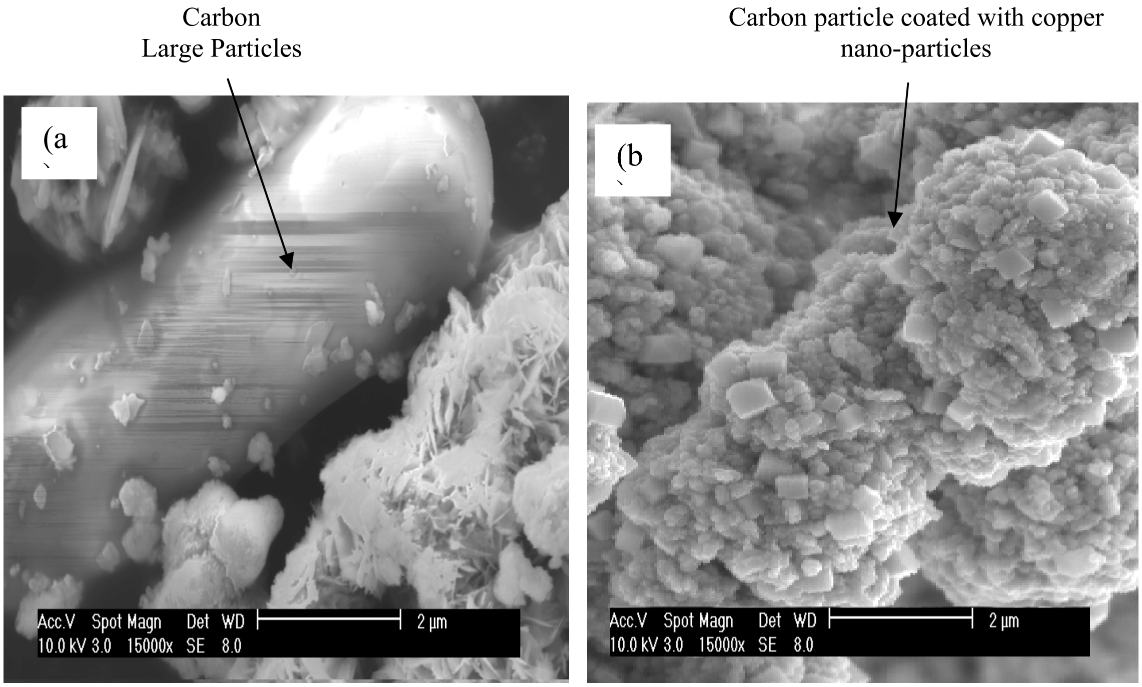

Figure 3. (a) SEM Cu-C composites after the electro-less precipitation process with fully coated carbon particulates with the nano copper particulates, and (b) SEM magnification of 500nm of the Cu-C composites with nano range copper particulates.

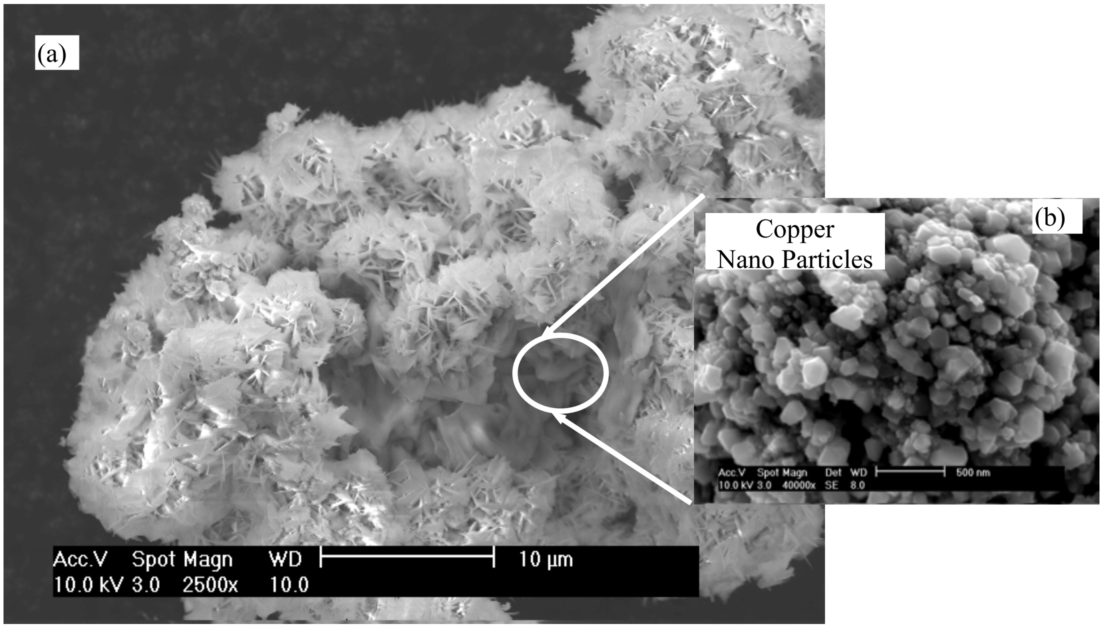

Figure 4. Stress-strain responses for the copper composites with reinforced 10%C, and 20%C volume fractions carbon particulates at the two sintering temperatures 700ºC and 800ºC.

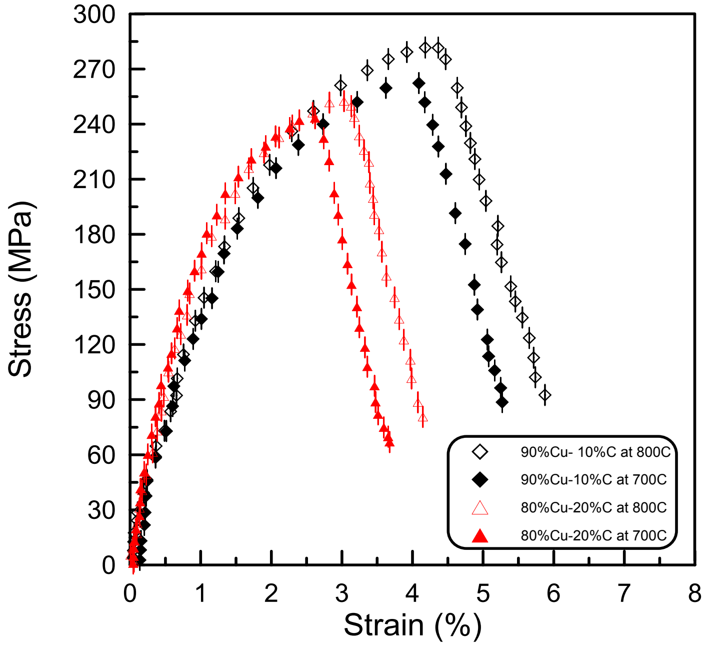

Figure 5. The SEM Fracture surfaces for the Cu-C composites at 800ºC sintering temperature with two volume fractions (a) 10%C, (b) 20%C (c) higher magnifications at the top the fractured surface for (a).