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Strengthening of Porous Alumina Ceramics Using (Cu) Metal in Nanoscale Particle and Graphite Waste as a Pore Agent

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Abstract: The preparation of porous alumina ceramics-reinforced begins with the use of (Cu) metal in nanoscale particles as a strengthening phase. At the same time, the solid state and sacrificial techniques are used for the preparation of this porous alumina ceramics-reinforced. Meanwhile, FESEM used to examine the microstructure. The main goal of this research study is to determine the effects of the (Cu) metal in nanoscale particles on the mechanical properties and porosity of porous alumina ceramics. Cu metal added to different ratios of graphite waste as a pore agent. The initial ratio of (Cu) metal added was at 3 wt.%. And this ratio was increased to 6, 9 and 12 wt.% Cu. The results of this study indicate that with an increase in the ratios of (Cu) metal, the porosity shows a decrease while the mechanical properties show an increase. The observed increase in the mechanical properties could be attributed to not only a decrease in the porosity but also to the toughening mechanism of porous alumina ceramics. The areas of potential applications for this type of ceramics include, filtration, thermal and the purging of gas.

Key words: Porous ceramic, strengthening, graphite waste, Cu metal, mechanical properties

INTRODUCTION

The unique properties of porous ceramic such as in good strain and damage tolerance, good thermal shock resistance, wear resistance, high corrosion and light weight together with a tailored microstructure have promoted the potential use of this porous ceramics for applications in structural components (Jean et al., 2014; Zhang et al., 2012) such as filtering materials that include the uses as membranes for separation, light weight structural materials (Tang et al., 2004), catalyst supports, thermal insulation, bioreactors, gas filters for high temperature (Dessai et al., 2013; Zerroug et al., 2007) medical ultrasonic imaging and underwater sonar detectors. Generally, it is known that with an increase the porosity of porous ceramic, there is a decrease in its mechanical properties. But at the same time, most applications of porous ceramics require to a good mechanical properties.

Thus, in the case of the filtration of hot gas and molten metal, any fluctuation of temperature during the process will leave the materials for filter susceptible to thermal shock. As such the mechanical properties of the filter must be high enough to bear the operation pressure and at the same time the filter properties must not show any deterioration with increase in temperature during the process (Hammel *et al.*, 2014). Also, macro porous ceramic membrane with high mechanical properties is an important support for macro porous membrane because of its ability to remain stable in harsh chemical environment and at high temperature(Li *et al.*, 2010).

Therefore, it is a requirement for the preparation of porous ceramic with high mechanical properties such as fracture toughness and high bending strength. The addition of ductile metallic phase is an effective method to overcome the negative aspects of ceramic materials, leading to an improvement in the mechanical properties such as bending strength, fracture toughness and tensile

strength. In the field of study in porous ceramic, many researchers used micro metal particle to improve the mechanical properties of porous ceramic.

Falamaki et al. (2001) reported the effects of the addition of aluminum particles on the mechanical properties of alumina membranes through the use of Reaction Bonding of Aluminum Oxide (RBAO) process. By increasing the Al wt.% content, there is an increase in flexural strength but with a decrease in porosity. Cleggl and Paterson (2004) reported the use of ammonium hexachloroplatinate (ACP) as a source of platinum particles for ductile particle toughening of hydroxyapatite. The findings revealed that by increasing the volume fraction of platinum particles, the fracture toughness of porous hydroxyapatite ceramics has shown an increase of up to twice that of the untouched hydroxyapatite. This improvement in fracture toughness may contribute to the crack bridging mechanism. Wang et al. (2007) reported that $(Si_3N_4-Al_2O_3-SiO_2)$, a type of Reticulated Porous Ceramic (RPC) can be fabricated using replication techniques with the addition of (5wt.%) aluminum particles to β-Si₂N₄. The findings showed there were improvements in fracture toughness and strength of (RPC) ceramics with the addition of aluminum powder. This enhancement in mechanical properties has attributed to the crack bridging mechanism. Li et al. (2010) studied the (RBAO) technique in the preparation for macro porous alumina ceramics with high fracture toughness with the addition of Aluminum (Al) powder in the following ratios (4, 8, 12, 16 and 20 wt.%). The findings of this study showed that Aluminum (Al) powder plays a significant role in the improvement of the mechanical properties of macro porous alumina ceramics, especially in the enhancement of bending strength and fracture toughness. Most research studies related to the field of porous ceramics have only focused on investigating the use and effects of Aluminum (Al), nickel (Ni) and platinum on the bending strength and fracture strength of porous ceramics. As such the aim of this research study is to produce porous alumina ceramics that is reinforced with (Cu) metal in nanoscale particles and with high mechanical properties. The conditions for the of porous alumina ceramics include a reinforced phase in sintering at high temperature using a new process that requires the addition of (Cu) metal in nanoscale directly through a combination of sacrificial technique and pressureless sintering methods which is a coast effected procedure.

MATERIALS AND METHODS

A commercial aluminum oxide Al_2O_3 powder ($\rho = 3.94$ g/cm³) with a high purity of 99.9%, 0.5 μ m in particle size

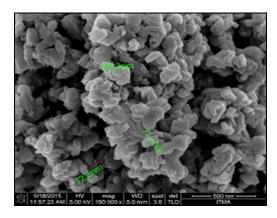


Fig. 1: FESEM image of nano-copper particles

has been selected use as the matrix material. Meanwhile, commercial copper (Cu) in nanoscale particle, of <100 nm has been identified for use as the reinforcement material. The examination of the morphology and size of nano-copper particles are conducted using the Field Emission Scanning Electron Microscope (FESEM) machine (Fig. 1). The measurement for the density of copper by the instrument, Accupyc II 1340 is recorded at 7.59 g/cm³.

For the purpose of research work of this study, commercial sucrose (sugar) was used as the binder with the addition of (10-12%) into the ceramic mixture, according to the maximum solubility of sugar in distilled water. In this research study, 60 wt.% concentration solution has been used as the binder for purpose of experiments (Mohanta et al., 2014). The binder was mixed with ceramic powder using an agate mortar for 3-5 min manually. Meanwhile, graphite waste was collected from primary batteries. The graphite rods were cleaned using acetone solvent to remove traces of oil and other wastes from the primary battery. At the start of the procedure, a drying process was carried out in air for 24 h, followed by drying process for 1 h in an electric oven at 100°C. Next, the graphite waste was crushed and ground by an electric grinder of model (RT-02A) with operations at 3000 rpm. Finally, the graphite powder was screened to a particle size of 250 µm. A particle size analyzer (Malvern, master size 2000) was used to analyse the particle size distribution of the graphite waste (Fig. 2). The true density for graphite waste was recorded at 1.77 g/cm³ as by the instrument, Accupyc II 1340.

The preparation of the ceramic mixture is separated into two process. The first process began with the mixing of (Cu) metal in nanoscale with the alumina powder (Al₂O₃). Next, the alumina powder and copper (Cu) powder were weighed in amounts, that were suitable for ratios of

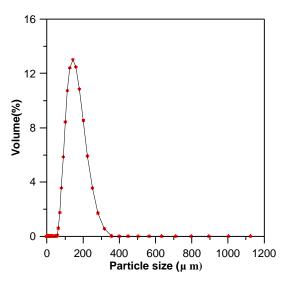


Fig. 2: Particle size distribution of graphite waste

3, 6, 9 and 12 wt.% of copper powder (Cu) to alumina powder. Nano-copper particles were mixed with alumina powder manually for around 10 min in an agate mortal. After that the ceramic mixture was milled for 3 h in planetary ball milling at (650 rpm) using 0.5 mL/g of acetone for the mixture. Then, the mixture was dried at 80°C for 24 h in an electric oven. Finally, the mixture was ball milled for 24 h to prevent agglomeration. The second process began with mixing the pore agent (graphite waste) with the (Cu) metal + Al₂O₃ mixture. The addition of graphite waste powder of ceramic slurry was performed in ratios of 10, 20, 30, 40, 50 wt%. Before ball milling, all the batches were mixed in a mortar for 5-10 min. After that, they were ball milled for 3 hrs in a plastic container to obtain a homogenous mixture in a ratio of 3:1, the weight of alumina balls to the weight of powder. The dry mixtures were pressed uniaxially in a circular steel die (diameter = 20 mm and thickness = 5 mm) using an Instron hydraulic press, at a pressure of 90 MPa. Meanwhile, the green compacts were dried in an oven at 110°C for 24 h. The organic burnout of dried samples were carried out in an ambient atmosphere in an electrically heated, programmable furnace.

The rate of heating was 1.5°C/min for each increment in the temperature. According to the TGA (thermo gravimetric analysis) of sucrose and graphite waste (Fig. 3), the samples were sintered at 200°, 300°, 500° and 900°C for a soaking time period of 1 h in an electric furnace. The rates of heating and cooling were set at 1.5°C/min for the removal of graphite waste and sucrose. After that, the ceramic samples were sintered at 1600°C for a soaking time period of 2 h and the rates of heating and cooling were set at 5°C/min. The findings revealed that all

the samples with different ratios of graphite waste and binder (sucrose) had a uniform shape without any cracks after sintering.

Characterization of ceramic samples: The compressive strength and indirect tensile strength were measured using an Instron machine for a sample of dimensions 20 mm in diameter and 5 mm in height based on Brazilian test method at a crosshead rate of 0.5 mm/min. The maximum mechanical load and cross-sectional area were used to calculate the compressive strength of the samples. The value of hardness of the samples was measured using a micro Vickers hardness machine. After, all the samples were ground and polished using a polishing media, they were etched thermally. The samples of dimensions, 20 mm in diameter and 5 mm in thickness were tested using the Brazilian test method by means of an Instron machine.

The water immersion method based on Archimedes' principle as specified in ASTM C20-00 has been used to determine the density and overall porosity of the sintered samples. The calculation for density and overall porosity of the sintered samples is based on the following equations:

$$P_{\text{overall}} = \left(1 - \frac{\rho}{\rho_{\text{theoretical}}}\right) \times 100 \tag{1}$$

$$\rho = \frac{M_{\text{dry}} \times \rho_{\text{water}}}{M_{\text{wet}} - M_{\text{suspended}} + M_{\text{wire}}} \tag{2}$$

Where:

 M_{dry} = The dry mass of the sample

 $M_{\text{suspended}}$ = The mass of the sample suspended in distilled water

 M_{wet} = The mass of the sample after soaking in water

 M_{wire} = The mass of the suspending system

P_{overall} = The volume fraction of the overall porosity (vol.%) of the sample (Hu Liangfa *et al.*, 2012; Menchavez *et al.*, 2010)

The instrument Accupyc II 1340 has been used to measure the theoretical density (true) of Alumina (Al_2O_3). The density of Al_2O_3 (3.94 g/cm³) is used the reference value. The microstructure was examined using (FESEM).

RESULTS AND DISCUSSION

Mechanical properties: Table 1 shows that the mechanical properties of the porous alumina ceramic are strongly related to the graphite waste content, the porosity ratio and (Cu) content of the porous alumina

Table 1: Mechanical properties of alumina porous ceramic

			Compressive strength		
Graphite waste	Alumina (Al ₂ O ₃) +	Porosity	Hardness	$(\sigma_{comp.})$ (Mpa)	Tensile strength
content (wt %)	Cu ratio (wt.%) (g)	(vol.%)	(HV_1)	(Brazilian test)	($\sigma_{tensile}$) (MPa)
10	90% Al ₂ O ₃	37.27	172.64	39.195	24.97
10	(87% Al ₂ O ₃ +3% Cu)	26.94	249.90	78.91	40.24
10	(84%Al ₂ O ₃ +6%Cu)	24.80	292.19	79.41	40.55
10	(81%Al ₂ O ₃ +9%Cu)	24.71	346.63	72.17	44.94
10	(78%Al ₂ O ₃ +12%Cu)	24.00	390.18	85.42	46.38
20	$80\% \text{ Al}_2\text{O}_3$	45.71	96.67	31.31	19.63
20	(77% Al ₂ O ₃ +3% Cu)	34.03	172.91	96.40	33.37
20	(74% Al ₂ O ₃ +6% Cu)	33.44	201.40	75.85	38.28
20	(71% Al ₂ O ₃ +9% Cu)	31.07	372.06	65.76	41.86
20	(68% Al ₂ O ₃ +12% Cu)	28.95	386.04	73.17	44.58
30	$70\% \text{ Al}_2\text{O}_3$	46.19	66.72	29.25	18.63
30	(67% Al ₂ O ₃ +3% Cu)	37.24	134.03	68.23	33.44
30	(64% Al ₂ O ₃ +6% Cu)	31.56	240.51	62.74	36.94
30	(61% Al ₂ O ₃ +9% Cu)	33.02	235.23	65.33	34.59
30	(58% Al ₂ O ₃ +12% Cu)	34.37	229.97	69.23	34.07
40	$60\% \text{ Al}_2\text{O}_3$	53.60	47.97	28.08	17.88
40	(57% Al ₂ O ₃ +3% Cu)	44.26	145.09	56.30	33.84
40	(54% Al ₂ O ₃ +6% Cu)	40.07	174.90	57.24	34.44
40	(51% Al ₂ O ₃ +9% Cu)	37.31	241.94	63.14	36.20
40	$(48\% \text{Al}_2\text{O}_3 + 12\% \text{Cu})$	38.09	230.43	50.32	34.04
50	$50\% \text{ Al}_2\text{O}_3$	61.08	38.11	22.412	14.28
50	(47% Al ₂ O ₃ +3% Cu)	51.43	74.80	36.71	23.37
50	(44% Al ₂ O ₃ +6% Cu)	44.93	170.29	48.05	28.59
50	(41% Al ₂ O ₃ +9% Cu)	41.19	213.72	46.81	30.80
50	$(38\% Al_2O_3 + 12\% Cu)$	42.00	200.68	34.60	27.02

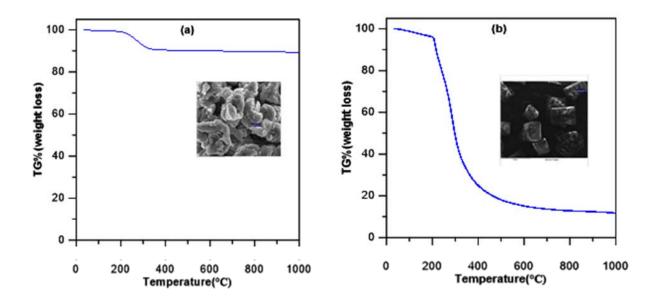


Fig. 3: TGA and FESEM images; a) graphite waste, b) sucrose (sugar)

ceramic (ASTM standard C 1327-03, 2004). In general, it is known that with an increase in the porosity of porous ceramic, there is a decline in the mechanical properties. Unfortunately, most applications using porous ceramics

require good mechanical properties (Zhang, Jurgen, 2015) such as the filtration of hot gas and molten metals. In recent years, several reports have shown that the dispersion of metal particles (Ni, Cu, W, Co, Ti, Mo) of

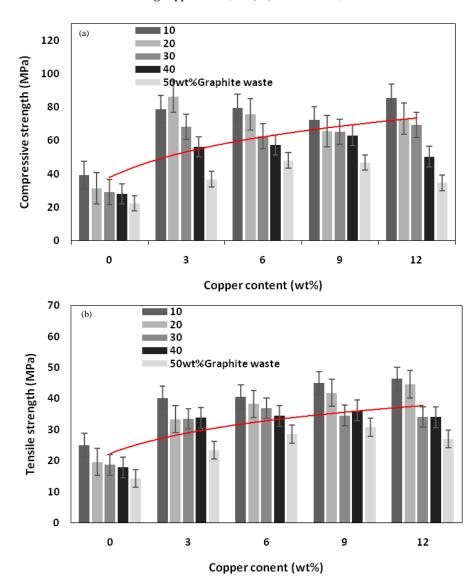


Fig. 4: Variations of: a) compressive and b) tensile strengths of porous alumina ceramic samples sintered at 1600°C for 2 h with Cu metal content for different ratios of graphite waste

nanoscale (<100 nm) in ceramic bodies such as $\rm ZrO_2$ and $\rm Al_2O_3$ has shown notable improvements in their mechanical properties (hardness and/or toughness, mechanical strength) (Liu *et al.*, 2013). Also, conducting sintering at higher temperature has resulted in higher mechanical properties and densities for the metal reinforced materials (Lieberthal and Kaplan, 2001).

Compressive strength and tensile strength: The compressive strengths of porous alumina ceramic samples were calculated according to $(\sigma_{\text{(compressive MPa)}} = (P/A)$ where $\sigma_{\text{(compressive)}}$ is in MPa, p = the applied force (N) and A = the

original area of the ceramic samples (mm²) (Seeber *et al.*, 2013). The diametrical tensile strengths of the porous alumina ceramics were calculated according to the equation of tensile strength of the Brazilian test ($\sigma_{\text{(tensile MPa)}} = (2 \times P/\pi \times t \times d)$ where $\sigma_{\text{(compressive)}}$ is in MPa, P = the applied force (N), d = the diameter of the samples (mm) and t = the thickness of the sample (mm) (Seeber *et al.*, 2013).

Figure 4a, b show that a decrease in the compressive strength and the tensile strength occurs with an increase in the graphite waste ratio, before addition of nano-copper particles at 0 wt.% Cu. The decrease in the compressive strength and the tensile strength was attributed to the

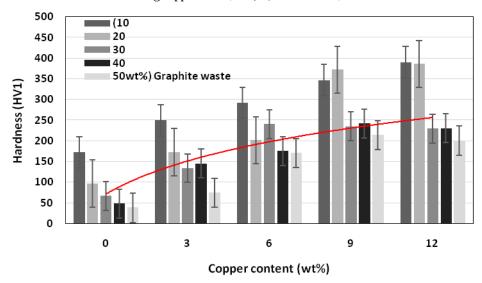


Fig. 5: Variation of hardness of porous alumina ceramic samples sintered at 1600 °C for 2 h with Cu content for different ratios of graphite waste

increase of the porosity in the porous alumina samples which acts as a function of the increasing ratio in graphite waste according to Rice's formula ($\sigma = \sigma^{\circ} \exp(-bp)$) where σ and σ° are the strengths of the porous and non-porous materials, p is the porosity of porous ceramics while b is the constant related to the pore characteristics (Kennedy et al., 2011; Liu, 1997). This occurs because the pores reduce the cross-sectional area-across where the load has been applied. Also, the pores act as stress concentrators (William and David, 2010). The values of the compressive strength and the tensile strength are in the range of 112.25-34.28 Mpa and 24.97-14.28 MPa, respectively with a graphite waste ratio of 10-50% wt. After the addition of nano-copper particles in different ratios, ranging from 3, 6, 9 and 12 wt.% of (Cu) metal to porous alumina ceramic samples, the compressive strength of porous alumina ceramics show an increase with increases in the ratio of nano-copper (Fig. 4a, b). The increase in compressive and tensile strengths are attributed to the decrease of porosity as a result of the filling of pores by (Cu) metal and the existence of more bonds in the initial green compact due to the higher content of ductile Cu particles, may attribute to the crack bridging mechanism (Clegg and Paterson, 2004; Falamaki et al., 2001). There are several ways to improve the toughening mechanism of ceramic composites. One way to increase the strength and fracture toughness of ceramic material is by mixing the metallic of second phases into the ceramic matrix (Kafkaslioglu and Tur, 2016; Liu and Tuan, 1997; Sbaizero and Pezzotti, 2001). The most effective toughening mechanism of this phase is crack bridging (Zuo et al., 2007). The addition of the metal

particles into the ceramic matrix, causes bridging or deflection of the crack (Smirnov and Bartolome, 2014). The other toughening mechanisms are plastic deformation and crack deflection (Ji and Yeomans, 2002). These two are considered as mechanisms that can enhance the toughness (Chou and Tuan, 1995) because the crack driving force is reduced at the crack tip (Gu et al., 2006). The common ways to enhance the fracture resistance of the ceramic matrix are by a combination of mechanisms including bridging of the crack by the ductile phase (Clegg and Paterson, 2004) such as fibers or circular grains behind the crack face (Boch and Niepce, 2010; Lalande et al., 2002), deflection of the crack by the ductile phase, ductile rupture of the metallic phase at the crack tip (Alman and Hawk, 2001; Chen and Tuan, 2001; Liu et al., 2013; Rosler et al., 2007). However, the compressive strength of porous alumina ceramic has indicated a decrease at 12 wt.% (Cu) ratio for 30, 40 and 50 wt.% ratios of graphite waste because the porosity has increased.

Hardness: The value of hardness has been measured according to the equation $(HV_1 = 1.8544*(p/d^2))$ where P = the applied load and d = the average length of the two diagonals of the indentation. The applied force was 9.81 N for 15 secs at full load.

Figure 5 shows that the value of hardness decreases with increasing graphite waste content before the addition of (Cu) particles at ratio 0 wt.% Cu. This is because of the increase of the volume fraction of the overall porosity for porous alumina ceramics. In general, the mechanical properties of porous ceramics decrease with an increase in the porosity ratio (Eom *et al.*, 2013; Zhou *et al.*, 2015).

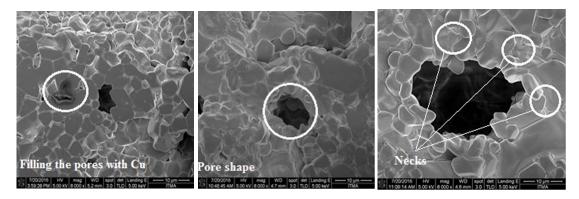


Fig. 6: Microstructure, filling the pores with copper molten, the irregular shaped of pores and necks in porous alumina ceramics body of porous alumina ceramics samples sintered at 1600 °C for 2 h for different ratios of graphite waste

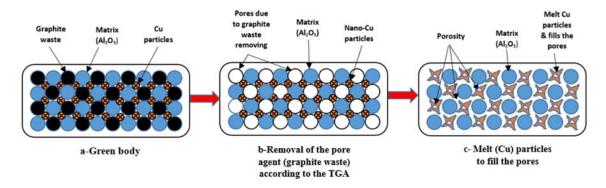


Fig. 7: a) green body of alumina ceramic composite at room temperature; b) removal of the pore agent (graphite waste) according to the TGA; c) melt (Cu) particles to fill the pores of porous alumina samples which leads to reduction of porosity

The value of hardness is in the range of 172.25 HV₁-38.11 HV₁ for porosity that ranges from 37.27-61.08%. After the addition of (Cu) metal with different ratios (3, 6, 9 and 12 wt.%), a significant increase in the hardness value was observed in porous alumina ceramics sintered at 1600°C for 2 h (Fig. 6 for ratios of (3, 6, 9 and 12 wt.%) of Cu). These results are likely to be related to the decrease in porosity. Meanwhile, at the ratio of 12 %wt copper content for 30, 40 and 50 wt.% graphite waste, the value of hardness indicates a decrease. It can be concluded that an increase in porosity can result in a decreasing the hardness of porous alumina ceramics.

Effects of (Cu) metal on the microstructure of porous alumina ceramics: Figure 6 illustrates the microstructure of porous alumina ceramics with pores that are filled with molten copper including irregular shape of pores and necks in porous alumina ceramics body. Figure 6a-c show the irregular shaped pores which may be attributed to the shape of graphite waste particles after burning in high sintering temperature. The sintering at high temperature

bears positive impact as the necks are well-developed, us leading to improvements the mechanical properties (Veljovic *et al.*, 2011).

There are 2-stages in the process mechanism that allow for the explanation of the behavior of (Cu) metal and pore agent ratio (graphite waste) in porous alumina ceramic samples. The first stage is the removal of graphite waste according to the TGA analysis. The removal of graphite waste from green body takes place at a temperature below the melting point of (Cu) metal. In this case, the alumina matrix has a porosity with (Cu) particles (Fig. 7b). The second stage is melting the (Cu) metal. When the sintering temperature is at 1600°C, the main focus is the melting of (Cu) metal. This stage leads to an improvement in the mechanical properties as (Cu) metal phase is in uniformity. At this stage, the molten (Cu) metal fills the pores to reduce porosity. With an increase in the ratio of (Cu) metal, the molten (Cu) is sufficient to fill the porous alumina body (Fig. 7c). The maximum mechanical properties is recorded at 9 wt.% Cu. However, the mechanical properties experience a drop after 9 wt.% Cu

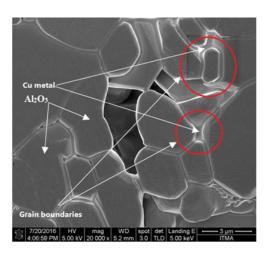


Fig. 8: Agglomeration of (Cu) metal in the grain boundaries of porous alumina matrix

because there is not enough molten (Cu) to fill the pores, due to increases in the graphite waste ratio and pore size. There are several possible explanations for this occurrence. At 10 and 20 wt.% graphite waste, the mechanical properties shown an increase when the (Cu) metal ratio increases. This is because the size of the pores is smaller than the size of the grains. As a result, the pores accumulate between the grains and they become wet in the liquid phase of (Cu) metal (Shaw, 1993). Also, capillarity drives the liquid phase of (Cu) metal to fill the smaller pores. As the smaller pores are filled, the number of pores and porosity decrease while the mean pore size increases. This leads to an improvement in the mechanical properties (German et al., 2009). As for graphite waste of 30, 40 and 50 wt.%, the mechanical properties shown an increase when the (Cu) metal ratio increases but for (Cu) metal of 12 wt.%, there is a drop in the mechanical properties for all the ratios of graphite waste. When the size of the pores is larger than the size of the grains, it is difficult to eliminate the pores because the liquid phase is not enough to fill the large pores. Unfortunately, the large pores work against densification leading to a decline in the mechanical properties (German et al., 2009; Oh et al., 1998a).

The toughening mechanism has significant effects on the mechanical properties mentioned above. Furthermore, the addition of (Cu) metal particles has inhibed in the growth of grains in the porous alumina (Al₂O₃) matrix. Therefore, increasing the (Cu) metal ratio, the porous alumina ceramics showed improvements in the mechanical properties due to the aglomeration of (Cu) metal particles in the grain boundareis (Fig. 8) (Oh *et al.*, 1998b).

CONCLUSION

Since, porous alumina ceramic composites reinforced with (Cu) metal in nanoscale have been successfully manufactured, this research study was designed to determine the effects of (Cu) metal in nanoscale and pore agent ratio on the mechanical properties of porous alumina ceramics. The results of this investigation showed that significant improvements in the mechanical properties of porous alumina ceramics such as hardness, compressive strength and tensile strength. The results of this research study support the idea that (Cu) metal in nanoscale can be used as a secondary phase to enhance the mechanical properties of porous ceramics and to control the porosity with mechanical properties. This research study has promoted several practical applications such as hot gas filters and molten metal filters.

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