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Research Article

Effect of Cu Metal of Nanoscale Particle Size on the Porosity and Mechanical Properties of Porous Alumina Ceramics using Yeast as a Pore Agent

[1, 5]Mohammed Sabah Ali, [1, 3]M.A. Azmah Hanim, [1]C.N.A. Jaafar, [1]S.M. Tahir
[2, 3]M. Norkhairunnisa and [4]Khamirul Amin Matori
[1]Department of Mechanical and Manufacturing Engineering,
[2]Department of Aerospace Engineering, Faculty of Engineering,
[3]Laboratory of Bio Composite Technology, Institute of Tropical Forestry and Forest Products,
[4]Department of Physics, Faculty of Science, Universiti Putra Malaysia, 43400 Serdang,
Selangor, Malaysia
[5]Department of Agriculture Machinery and Equipment Engineering Techniques, Technical College,
Al-Mussaib, Iraq

Abstract: The main goal of this study is to determine the effect of the addition of Cu metal of nanoscale particle on the mechanical properties and porosity of porous alumina ceramics. Porous alumina reinforced ceramics were prepared using nanoscale Cu metal particles as their strengthening phase. Solid state and sacrificial techniques were used to prepare the porous alumina reinforced ceramics. A FESEM was used to analyse the microstructure. Different ratios of Cu metal were added (3 wt%, 6 wt%, 9 wt% and 12 wt% Cu) at different ratios of yeast used as a pore agent. The results indicated that with increasing the ratios of Cu metal, the porosity decreased and the mechanical properties increased. The increase in the mechanical properties could be attributed to the decrease in the porosity and the toughening mechanism of porous alumina ceramics. Some potential applications include, filtration, thermal and purging of gas.

Keywords: Mechanical properties, nano-Cu metal, pore agent, porosity, porous ceramic, yeast

INTRODUCTION

The unique properties of tailored porous ceramics, such as their excellent strain and damage tolerance, good thermal shock resistance, wear resistance, high corrosion and light weight render them as potential components (Jean *et al*., 2014; Zhang *et al*., 2012) of filtering materials for separation membranes, light weight structural materials (Tang *et al*., 2004), catalyst supports, thermal insulation, bioreactors, gas filters for high temperature, (Dessai *et al*., 2013; Yu *et al*., 2011) medical ultrasonic imaging and underwater sonar detectors. Generally, increasing the porosity of porous ceramics decreases their mechanical properties, however, most applications of porous ceramics require good-to-excellent mechanical properties (Zhang and Malzbender, 2015). For the filtration of hot gas and molten metal, the temperature fluctuation during the process will leave the filter materials exposed to thermal shock. The mechanical properties of the filter must be high enough to bear operational pressure and its properties must not be affected by the increasing temperatures (Hammel *et al*., 2014). Macroporous ceramic membranes with excellent mechanical properties are important due to their stability in harsh chemical environments and high temperatures. The porous ceramic membrane that will be used needs to possess high fracture toughness and bending strength. The addition of a ductile metallic phase is an effective way of overcoming the drawbacks of ceramic materials, which results in improved bending strength, fracture toughness and tensile strength. In works on porous ceramics, many researchers have utilised micro metal particles to enhance the mechanical properties of the porous ceramic.

Li *et al*. (2010) analysed the (RBAO) technique of preparing macro-porous alumina ceramics with high fracture toughness via the addition of aluminum (Al) powder in the following ratios: 4, 8, 12, 16 and 20 wt%. Their findings showed that aluminum (Al) powder plays a significant role in improving the mechanical properties of macro-porous alumina ceramics,

Corresponding Author: Mohammed Sabah Ali, Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

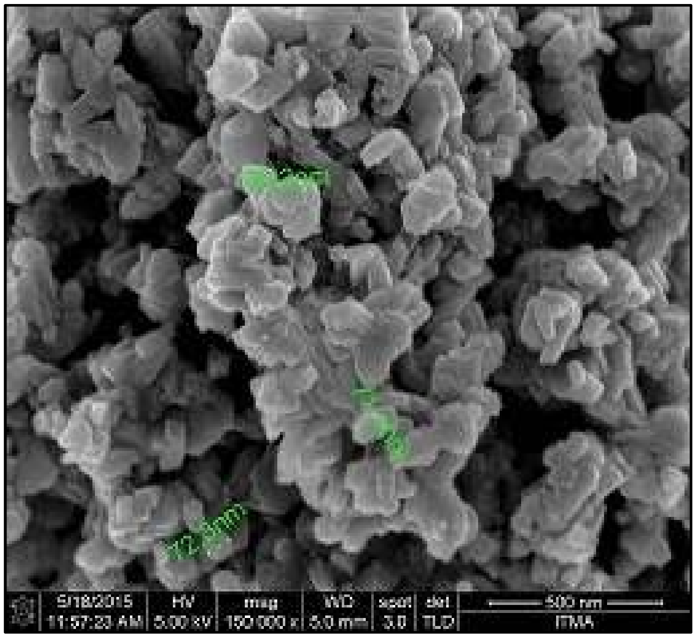


Fig. 1: FESEM image of nano-Cu particles

especially in terms of their bending strength and fracture toughness. Clegg and Paterson (2004) reported the use of ammonium hexachloroplatinate (ACP) as a source of platinum particles for the ductile particle toughening of hydroxyapatite. The results showed that by increasing the volume fraction of the platinum particles, the fracture toughness of the porous hydroxyapatite ceramics increased by up to twice that of pristine hydroxyapatite. Improved fracture toughness might contribute to the crack-bridging mechanism. Wang *et al*. (2007) reported that Si_3N_4-Al_2O_3-SiO_2, which is a type of reticulated porous ceramic RPC, can be fabricated using replication techniques via the addition of 5wt % aluminum particles to the β-Si_3N_4. The results showed improvements in the fracture toughness and the strength of the RPC ceramics due to the addition of the aluminum powder. This enhanced mechanical property results in an improved crack-bridging mechanism. Falamaki *et al*. (2001) reported the effects of the addition of aluminum particles on the mechanical properties of alumina membranes via the use of the Reaction Bonding of the Aluminum Oxide (RBAO) process. By increasing the Al wt% content, the flexural strength increased while the porosity decreased. Most studies on porous ceramics have focused on investigating the utilisation and effects of Aluminum (Al), Nickel (Ni) and Platinum (Pt) on the bending strength and fracture strength of porous ceramics. As such, the objective of this study is to produce porous alumina ceramics, which are reinforced with Cu metal of nanoscale particle size with excellent mechanical properties. The conditions for the porous alumina ceramics include a reinforced phase when sintering at high temperatures using a new process that requires the addition of Cu metal in the nanoscale directly through a combination of the sacrificial technique and pressure-less sintering methods: this is a cost-effective procedure.

MATERIALS AND METHODS

A commercial aluminum oxide (Al_2O_3) powder ($\rho = 3.94 g/cm^3$), with a purity of 99.9% and particle size of 0.5 μm was used as the matrix material. Commercial copper (Cu) of nanoscale particle size at < 100 nm was used as the reinforcement material. The morphology and the size of the nano-copper particles were examined using the FESEM technique (Fig. 1). The density of copper is 7.59 g/cm^3, as determined by the Accupyc II 1340.

Commercial sucrose (sugar) was used as a binder (10% -12%) in the ceramic mixture based on the maximum solubility of sugar in water (distilled water); 60 wt.% concentration solution was the concentration used in this study (Mohanta *et al*., 2014). The binder was manually mixed with ceramic powder using an agate mortar for 3-5 min. Yeast was purchased from a supermarket. The yeast was ground using an electrical grinder (RT-02A, 3000 rpm) for a minute and sieved using an electrical sieve (Retsch, As 200) to gather particles that measured 250 μm. A particle size analyser (Malvern, master size 2000) was used to determine the particle size distribution of the yeast (Fig. 2).

The true density of the yeast was determined to be 1.77 g/cm^3, as measured by the Accupyc II 1340. The preparation of the ceramic mixture involves two processes. The first step is to mix the nano-Cu metal with Al_2O_3 powder. The Cu powder was added at ratios

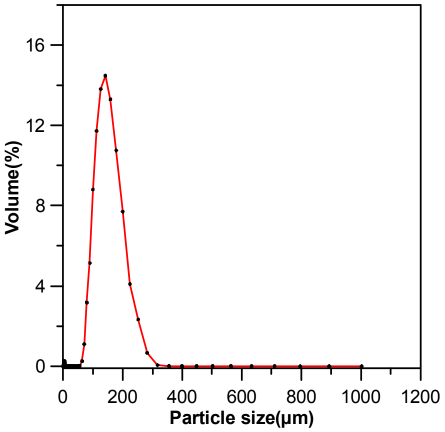


Fig. 2: Particle size distribution of yeast

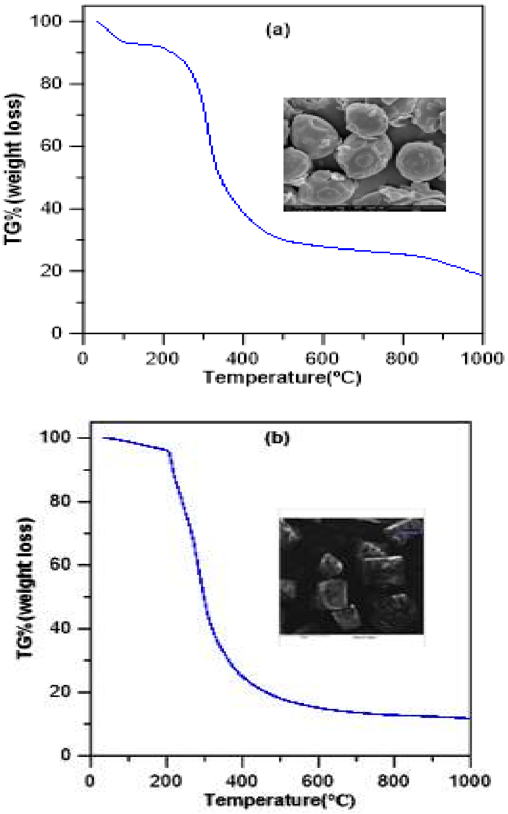


Fig. 3: TGA and FESEM images; a: yeast; b: sucrose (sugar)

of 3 wt%, 6 wt%, 9 wt% and 12 wt. % to the Al_2O_3 powder and mixed manually for 10 min in an agate mortar. The ceramic mixture was then milled for 3 h using planetary ball milling (650 r.p.m) using acetone at 0.5 ml/gram for the mixture, then dried at 80°C for 24 h in an electric oven. Finally, the mixture was ball-milled for 24 h to avoid agglomeration. The second step involved mixing the pore agent (yeast) with the previous mixture. The yeast powder was added at ratios of 10, 20, 30, 40, 50 wt. % to the ceramic slurry. Prior to ball milling, the batches were mixed in a mortar for ~5-10, min and then ball-milled for 3 h in a plastic container to render the mixture homogenous at a ratio of weight of alumina balls to weight of powder of 3:1. 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. The green compacts were dried in an oven at 110°C for 24 h. The organic burnout of the dried samples was conducted out in an ambient atmosphere in an electrically heated and programmable furnace. The rate of heating was set to 1.5°C/min for each increment in temperature. Based on the Thermo-Gravimetric Analysis (TGA) of sucrose and yeast (Fig. 3), the samples were sintered at 200°C, 325°C, 500°C and 900°C for a soaking time of 1 hr in an electric furnace. The rates of heating/cooling were 1.5°C/min for the removal of the yeast and sucrose. The ceramic samples were then sintered at 1600°C for a soaking time of 2 h and the rates of heating/cooling were set to 5°C/min.

CHARACTERISATION OF CERAMIC SAMPLES

The compressive strength and indirect tensile strength were determined using an Instron machine and samples measuring 20 mm in diameter and 5 mm in height were determined using the 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 hardness values of the samples were measured using a micro Vickers hardness machine. All the samples were ground and polished using a polishing media, then thermally etched. A Brazilian test was performed using samples 20 mm in diameter and 5 mm thick via the Instron machine. The microstructure and ceramic phases were examined using a Field Emission Scanning Electron Microscope (FESEM).

The density and overall porosity of the sintered samples were determined by the water immersion method based on the Archimedes' principle, as specified in ASTM C20-00 using the following equations:

$$P_{overall} = (1-\frac{\rho}{\rho theoritical})X100 \tag{1}$$

$$\rho = \frac{Mdry X \rho water}{Mwet-Msuspended+Mwire} \tag{2}$$

where,

M_{dry} = The dry mass of the sample,
$M_{suspended}$ = 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 *et al*., 2012; Menchavez and Intong, 2010).

Table 1: Mechanical properties of porous alumina ceramic

Yeast content (wt%)	Alumina (Al_2O_3) +Cu ratio (wt %) (g)	Porosity (vol %)	Hardness (HV_1)	Compressive strength (σ_{comp}). (MPa) (Brazilian test)	Tensile Strength ($\sigma_{tensile}$) (MPa)
10	90%Al_2O_3	30.21	160.64	41.20	26.24
10	(87%Al_2O_3+3%Cu)	23.45	240.24	49.09	31.89
10	(84%Al_2O_3+6%Cu)	22.39	273.19	52.67	33.53
10	(81%Al_2O_3+9%Cu)	21.60	222.72	48.77	33.68
10	(78%Al_2O_3+12%Cu)	21.01	282.68	58.55	37.27
20	80% Al_2O_3	37.53	98.12	38.01	24.21
20	(77%Al_2O_3+3%Cu)	36.24	154.57	42.26	28.27
20	(74%Al_2O_3+6%Cu)	32.83	160.68	46.38	29.53
20	(71%Al_2O_3+9%Cu)	31.94	200.77	44.30	31.20
20	(68%Al_2O_3+12%Cu)	31.60	207.43	46.91	32.86
30	70% Al_2O_3	47.26	51.01	18.28	11.64
30	(67%Al_2O_3+3%Cu)	45.18	77.20	18.31	13.57
30	(64%Al_2O_3+6%Cu)	44.56	97.22	21.27	14.01
30	(61%Al_2O_3+9%Cu)	43.88	103.29	22.20	15.04
30	(58%Al_2O_3+12%Cu)	40.08	106.92	26.19	16.67
40	60% Al_2O_3	55.65	38.31	13.70	8.72
40	(57%Al_2O_3+3%Cu)	51.63	52.23	17.28	11.00
40	(54%Al_2O_3+6%Cu)	50.05	54.84	17.08	11.87
40	(51%Al_2O_3+9%Cu)	47.90	56.43	19.78	12.59
40	(48%Al_2O_3+12%Cu)	47.64	61.32	21.45	13.65
50	50% Al_2O_3	63.79	15.01	8.51	5.42
50	(47%Al_2O_3+3%Cu)	56.95	20.17	9.23	5.88
50	(44%Al_2O_3+6%Cu)	55.72	21.92	9.33	5.94
50	(41%Al_2O_3+9%Cu)	52.32	23.24	14.23	9.06
50	(38%Al_2O_3+12%Cu)	53.47	23.10	12.86	8.55

The theoretical density (true) of Alumina (Al_2O_3) (3.94 g/cm^3) was used as a reference and measured by the Accupyc II 1340.

RESULTS AND DISCUSSION

Mechanical properties: Generally, increasing the porosity results in decreased mechanical properties, however, the majority of applications involving porous ceramics require excellent mechanical properties (Zhang and Malzbender, 2015) for the filtration of hot gas and molten metal. Recently, it was reported that the dispersion of nanoscale metallic particles (< 100 nm) (Ni, Cu, W, Co, Ti and Mo) in ceramics such as ZrO_2 and Al_2O_3 notably improves the mechanical properties (hardness and/or toughness, mechanical strength) of the ceramic body (Liu *et al.*, 2013). Also, sintering at higher temperature results in higher mechanical properties and densities for the metal-reinforced materials (Lieberthal and Kaplan, 2001). Table 1 shows that the mechanical properties of the porous alumina ceramic are strongly correlated to the content of yeast, the porosity ratio and the Cu content of the porous alumina ceramic.

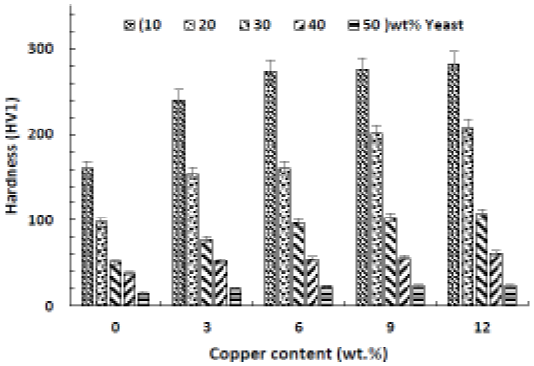
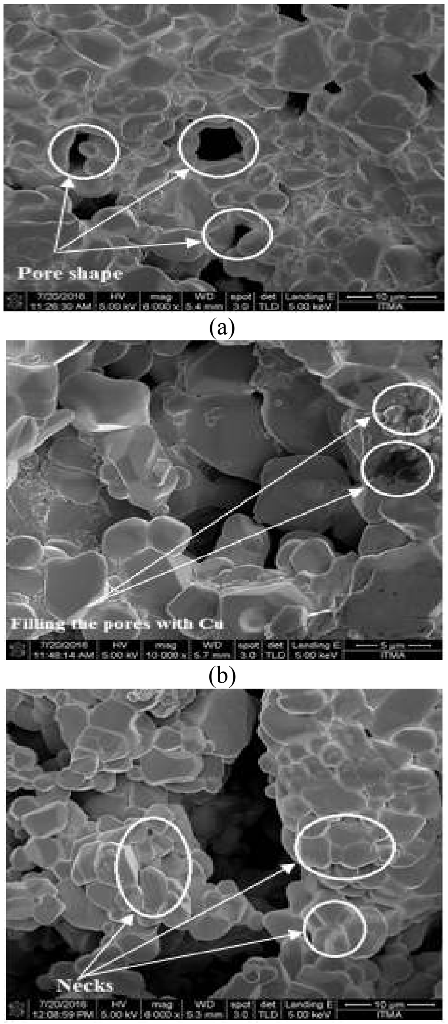


Fig. 4: Variation of the hardness of porous alumina ceramic samples sintered at 1600°C for 2 h with Cu content for different ratios of yeast



(a)

(b)

(c)

Fig. 5: Microstructure, filling the pores with molten copper, the irregular shape of the pores and the necks in the porous alumina ceramic body, for porous alumina ceramic samples sintered at 1600°C for 2 h for different ratios of yeast; (a):10% yeast + 12wt% Cu+ Al_2O_3; (b): 30% yeast + 12wt% Cu+ Al_2O_3; (c): 50% yeast + 12wt% Cu+ Al_2O_3

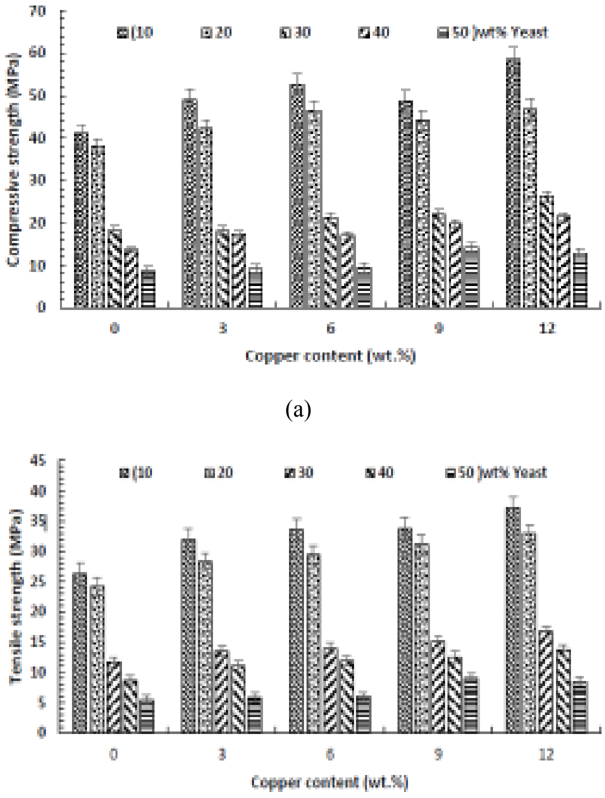
Hardness was measured based on the equation $(HV_1 = 1.8544*(\frac{p}{d_2})$, where P is the applied load and d is the average length of the two diagonals of the indentation. The applied force was 9.81 N for 15 secs at full load. Figure 4 shows that hardness is inversely correlated with yeast content prior to the addition of Cu particles at a ratio 0 wt % Cu. This is due to the increase of the volume fraction of the overall porosity for porous alumina ceramics. Generally, the mechanical properties of porous ceramics are inversely correlated with porosity (Eom *et al*., 2013; Zhou *et al*., 2015). The value of the hardness is in the range of 160.64 -15.01 HV_1 for porosities of 30.21- 63.79%. After the addition of Cu at different ratios (3 wt %, 6 wt %, 9 wt % and 12 wt %), a significant increase in the hardness was observed in porous alumina ceramics sintered at 1600°C for 2 h (Fig. 5 for ratios (3 wt %, 6 wt %, 9 wt % and 12 wt %) of Cu). The results are likely to be related to the decrease in the porosity and increase in the density. However, the hardness of the porous alumina ceramic has indicated a decrease at 12 wt % Cu for a 50 wt % ratio of yeast due to the increase in porosity. In recent years, many researchers have been able to increase the reliability of ceramic materials by reducing flaws (Ekström, 1993). Another way to improve in the mechanical properties of ceramic materials lies in the addition of metal powders for distribution within the brittle matrix (Rong *et al*., 2007; Rubinstein and Wang, 1998). Therefore, in comparision with recent studies, Rong *et al*. (2007) reported on the influence copper particle addition on the alumina ceramics' mechanical properties, when sintered at 1550°C for 1 h using the hot-press method. The study showed that the mechanical properties and density improved significantly for the alumina ceramics. Increasing the Cu content results in an improvement in the density and mechanical properties. Due to the low melting of Cu metal, the transfer of solute will accelerate during the sintering process which leads to an increase in density and an improvement in the mechanical properties.

Compressive strength and tensile strength: The compressive strengths of porous alumina ceramic samples were calculated using the following formula:

$$\sigma_{\text{(compressive MPa)}} = \left(\frac{P}{A}\right)$$

where $\sigma_{\text{(compressive)}}$ is in MPa, P is the applied force (N) and A is the original area of the ceramic samples (mm^2) (Seeber *et al*., 2013). The diametrical tensile strengths of the porous alumina ceramics were calculated using the equation of tensile strength of the Brazilian test:

$$\sigma_{\text{(tensile MPa)}} = \left(\frac{(2*p}{\pi*t*d}\right)$$



(a)

(b)

Fig. 6: Variations of (a) compressive and (b) tensile strengths of the porous alumina ceramic samples sintered at 1600°C for 2 h, with Cu metal content for different ratios of yeast

where,

σ (compressive) = In MPa
P = The applied force (N)
d = The diameter of the sample (mm)
t = The thickness of the sample (mm) (Seeber *et al*., 2013)

Figure 6a and 6b shows that a decrease in the compressive and tensile strengths takes place with increased yeast ratio prior to the addition of nano-Cu particles at 0 wt%. The decrease in the compressive and tensile strengths was attributed to the increased porosity in the porous alumina samples, which is a function of the increasing ratio of yeast, based on 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 the porous ceramics and b is the constant related to the pore characteristics (Ali *et al*., 2017; Kennedy *et al*., 2011; Liu, 1997). This takes place because the pores reduce the cross-sectional area across where the load has been applied and also act as stress concentrators (Callister Jr, 1997). The values of the compressive and tensile strengths are within 41.2 -8.51 MPa and 26.24 - 5.42 MPa, respectively, with a yeast ratio of 10-50 % wt. After the addition of nano-Cu particles at different ratios (3 wt %, 6 wt %, 9 wt % and 12 wt %) of Cu metal to the porous alumina ceramic samples, the compressive strength of the porous alumina ceramics increased with the increased ratio of nano-Cu (Fig. 6a and 6b). Increases in the compressive and tensile strengths are attributed to the decrease of porosity due to the pores being filled with Cu particles and the presence of more bonds in the initial green compact is due to the higher content of ductile Cu particles, which will improve the crack-bridging mechanism (Clegg and Paterson, 2004; Falamaki *et al*., 2001). Mashhadi *et al*. (2011) studied the effect of Al additions from 0 to 5 wt% on B_4C- TiB_2 composites sintered at 2050°C and 2150°C using the pressure less sintering process. It is found that with increase in Al content, the mechanical properties of B_4C- TiB_2composites have a significant enhancement. The maximum values of the fracture toughness, hardness, elastic modulus and fracture strength were measured to be about 6.2 $MPa.m^{1/2}$, 35 GPa, 500 GPa and 450 MPa respectively. This improvement in the mechanical properties was attributed to the increase in the relative density with less grain growth.

There are several ways to improve the toughening mechanism of ceramic composites. One is by mixing the metallic component of the second phases into the ceramic matrix (Kafkaslıoğlu and Tür, 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 the deflection of the crack (Smirnov and Bartolomé, 2014). Some of the other toughening mechanisms include plastic deformation and crack deflection (Ji and Yeomans, 2002). These mechanisms are regarded as capable of enhancing toughness (Chou and Tuan, 1995), as the crack driving force is reduced at the crack tip (Gu *et al*., 2006). Common ways of enhancing the fracture resistance of the ceramic matrix are by a combination of mechanisms such as:

- Bridging the crack using ductile phases 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; Rösler *et al*., 2007). However, the compressive and tensile strengths of the porous alumina ceramic indicated a decrease at 12 wt% Cu ratio for 50 wt % ratio of yeast due to the increased porosity.

EFFECTS OF CU METAL ON THE MICROSTRUCTURE OF POROUS ALUMINA CERAMICS

Figure 5 shows the microstructure of porous alumina ceramics with pores that are filled with molten Cu. Features include the irregular shape of the pores and the necks in porous alumina ceramics. Figure 5a to 5c shows the irregular shaped pores, which could be attributed to the shape of the yeast particles after high temperature sintering. Sintering at high temperature results in well-developed necks and improved mechanical properties (Veljović *et al*., 2011).

A 2-stage process mechanism can be used to explained the behaviour of the Cu metal and the pore agent ratio (yeast) in porous alumina ceramics. The first stage involves the removal of yeast, according to TGA analysis. The removal of yeast from the green body takes place at a temperature below the melting point of Cu. In this case, the pores of the alumina matrix are filled with Cu particles. The second stage is the melting of the Cu. At a sintering temperature of 1600°C, the melting of the Cu becomes the focus. This leads to improved mechanical properties due to Cu uniformity within the composite. The molten Cu starts to fill the pores of the matrix at an increasing ratio throughout the porous alumina body. Meanwhile, at a ratio of pore agent (yeast) of 50 wt%, the maximum mechanical properties are reported to be 9 wt % Cu. However, the mechanical properties decrease after 9 wt % Cu, because there is not enough molten Cu to fill the pores due to the increased yeast ratio and the pore size. There are several possible explanations for this. At 10 wt%-40 wt% yeast, the mechanical properties show an increase when the Cu metal ratio increases. This is due to the size of the pores being smaller than the size of the grains. As a result of this, the pores accumulate between the grains and they become wet in the liquid phase of the Cu metal (Shaw, 1993). Also, capillarity drives the liquid phase of Cu to fill in the smaller pores. As these pores become filled, the number of pores and the corresponding porosity decreases, while the mean pore size increases, which leads to improved mechanical properties (German *et al*., 2009). As for yeast at 50 wt %, the mechanical properties increase when the Cu metal ratio increases, but for Cu metal of 12 wt %, the mechanical properties decrease for all ratios of yeast. When the size of the pores exceeds the size of the grains, it is difficult to eliminate the pores because the liquid phase is inadequate for filling up the large pores. Unfortunately, the large pores work against densification, leading to decreased mechanical properties (German *et al*., 2009; Oh *et al*., 1998; Oh *et al*., 1988). The toughening mechanism significantly influences thepreviously mentioned mechanical properties. Furthermore, the addition of Cu metal particles inhibited the growth of grains in the porous alumina (Al_2O_3) matrix. Therefore, when increasing the Cu metal ratio, the porous alumina ceramics showed improvements in their mechanical properties due to the agglomeration of Cu metal particles in the grain boundaries and this decreased the porosity (Fig. 7) (Oh *et al*., 1998). In addition, researchers in recent studies have reported the effect of adding metal particles on the mechanical properties of cermic materials. Latifi *et al*. (2014) reported that the influence of Ni and Fe metals particles on Boron Carbide (B_4C) and 10 vol.% nano-titanium diboride (TiB_2)ceramic composite properties using the cold pressing method. It was found that the addition of Ni and Fe enhanced the values of the Young' modulus, the hardness and the fracture toughness. This improvement in the mechanical properties was attributed to the increase in the density which comes from the improvement of the condensation procedure due to the addition of the metal phase to the ceramic composites during the sintering process. The formation of liquid phases, such as Ni_3B and FeB, with a low melting point helps to reduce the excessive grain growth, the porosity value and the boundary mobility.

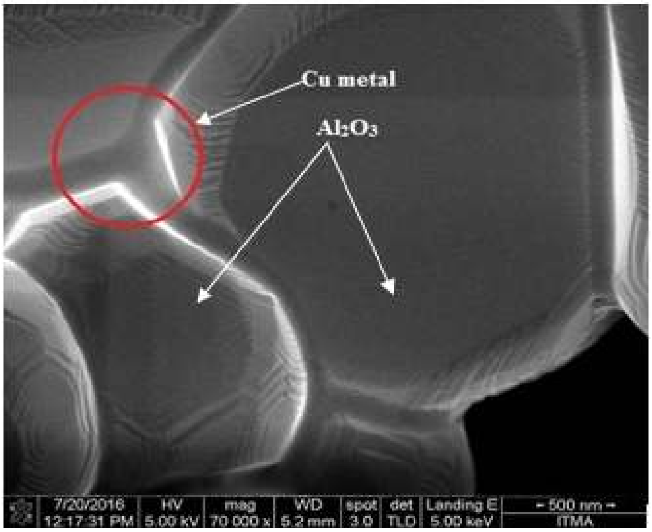


Fig. 7: Agglomeration of Cu metal in the grain boundaries of the porous alumina matrix

CONCLUSION

Porous alumina ceramic composites reinforced with Cu metal at the nanoscale have been successfully manufactured. This study was designed to determine the effect of Cu metal at the nanoscale and the pore agent ratio on the mechanical properties of porous alumina ceramics. The results of this study showed a significant improvement in the mechanical properties of porous alumina ceramics, such as the hardness and the compressive and tensile strengths. The results support the idea that Cu metal at the nanoscale can be used as a secondary phase to enhance the mechanical properties of porous ceramics and control their porosity. The results have several practical applications, such as hot gas filters, molten metal filters and membranes.

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