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## The Influence of Sintering Time and Temperature of SiCw+Al<sub>2</sub>O<sub>3</sub>p Reinforcement on Aluminum Matrix Composite Characteristics

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### ABSTRACT

Al+(SiCw+Al<sub>2</sub>O<sub>3</sub>p) is a combination of fine aluminum powder (Al) as the matrix and Silicon Carbon whiskers together with Alumina particles as the reinforcement. This composite was formed by placing the powdered materials in a mold then pressure was exerted on it to ensure a homogenous mix. After this the composite was sintered in a Fluidized bed oven without pressure for 1 hour, 3 hours and 6 hours at temperatures of 500°C, 550°C and 600°C respectively. It was then tested for density, porosity, modulus elasticity and SEM. The test results indicated that density was had increased and porosity decreased by raising sintering temperature and extending sintering time. In addition, they showed that at a holding time of 6 hours, modulus elasticity rose at temperatures close to 550°C but fell at 600°C and that this was caused by the composite becoming more ductile. The maximum modulus elasticity occurred with a holding time of 3 hours at 550°C. Observations using the SEM showed that the composite structure became increasingly homogenous and more dense because of both particle and whisker distribution becoming even.

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## INTRODUCTION

Much research into the influence of sintering temperature on composites has already been done as it improves both their physical and mechanical properties and in the interest of developing new and better materials a great deal of effort has gone into forming new ones that have high physical and mechanical characteristics, that have many uses and that have low production costs (Gibson, 1994; Garnier, 2004). Types of composites that are increasingly being investigated are those with an aluminum matrix due to their high resistance to corrosion compared to other types, and alumina (Al<sub>2</sub>O<sub>3</sub>), as reinforcement, also imparts high anti corrosive properties (Evans, 2000). The combination of alumina and SiCw forms a composite that has high strength, is light and easily to be machined (Garnier, 2005). Composite surface strength bonding depends on treatment temperature and the formation of intersurface compound, increased temperatures together with the amount of SiC raises hardness levels, the effect of particle size, durability levels and friction coefficients causes its strength to decrease (Lim, 2001; Widyastuti, 2008). The influence of sintering temperature on density and 3% mol hardness of yttria-stable zirconia (TZ-3Y) tetragonal composite is strengthened by the addition of alumina fibers, shows that increased sintering temperature and whisker levels lowers density but increases hardness, and vice versa (Gibson, 1994; Abdullah, 2012). The characteristics Al/SiC metal-ceramic composite on the intersurface micro and chemical bonding is affected by increasing holding time and temperature, which causes increased Si separation and lowers hardness (Homeny, 1990). The effect of silicon carbide on Al-Si-Fe/SiC particulate composite demonstrates that raising the percentage of silicon carbide and aging time increases hardness (Bolaji, 2006). The composite was strengthened with SiC particles made using *Isostatic Hot Pressure (HIP)* under differing pressures and temperatures which increased ductility but caused yield strength to drop dramatically due to the lack of the defects in the composite (Jean, 1997).

The treatment of the AA.2124 hot deformation material was strengthened by SiCw and silicon carbide particles (15 vol % SiCw+SiCp) showed that composite stress rose with increased SiCw composite compared to the addition of SiCp (Ko, 1998). Experimental data obtained from the unpressured sintering of preceramic SiC in an alumina matrix in an argon atmosphere showed that there was no intergranular interstice between the

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$\text{Al}_2\text{O}_3$  particles and the SiC (Bocanegra, 2009). Both sintering temperatures and holding times influence the micro structure and mechanical characteristics of the WC- 40% vol Co composite, where increasing sintering temperature and holding time induced relative density and particle size to increase, whereas, both hardness and structural toughness levels rose at the beginning but later fell (Heng, 2013). The pressure less sintering of  $\text{ZrB}_2$ / (10 – 40 vol %) SiC composite particulate was influenced by the amount of SiC, the  $\text{Al}_2\text{O}_3$  additive and the process parameters in the density process caused hardness levels to rise over 1 hour's sintering, which, however, dropped after 2 hours because of the occurrence of grain growth (Malik, 2013). The effects of the sintering process of composite physical properties and microstructure of TiB-TiC+8wt% nano-Ni ceramic composite, especially at short holding times, improved characteristics, although longer times resulted in the formation of grains of both  $\text{TiB}_2$  and TiC, as well as pores and particle brittleness and thus degrading mechanical characteristics (Zou, 2012). The composite was made from SiC commercial powder doped with boron and carbon and treated in a two-stage sintering process an improve density. Deflection strength results were influenced by grain size, yet, hardness and fracture strength levels remained constant (Magnani, 2013). Both temperature and atmospheric pressure affected the Al-31%, SiCw sintering process, and increasing SiCw content had the effect of hampering whisker bonding, nevertheless, small particle size resulted in a more homogenous micro structure at high temperatures (Urretavizcaya, 1997). The silicon carbide ceramic was made in a heat extrusion sintering process from nano size SiC powder with nitrate as an additive. However, the addition of nanoSiC was unable to raise relative density, in contrast, nitrate has a higher density than SiC and was able to impart a higher density (Karamian, 2009). Also the effect of heat extrusion on strength and silicon carbide particle ductility was strengthened by the Al-5% Si-0.2% Mg composite, and evaluation of its mechanical properties indicated that the extrusion process induced high levels of both strength and ductility (Ok, 2002). The metal-ceramic material was made in a cold compression sintering process at differing atmospheric pressures and varying ceramic phases. Increasing oxygen levels had a significant effect on the material formed, where, at the beginning of the sintering, atmosphere, strength increased but later decreased (Chen, 2012). Two stage of experimental sintering were enacted to evaluate the influence of the addition of magnesium, zircon, and yttria to increase density and grain growth of the sub-micron alumina powder, at the end of the sintering process, the addition of Mg, Y and Zr induced a reduction or a slowing of grain growth (Ghillányová, 2012).

Previous studies (Garnier, 2004; Lim, 2001; Widyastuti, 2008; Abdullah, 2012; Homeny, 1990), reported that in order to obtain better mechanical properties, it was important to pay attention to composite parameters such as density, particle size and porosity. Furthermore, both optimum sintering temperature and holding times were vital as they influenced density and particle size and also because they directly affected composite mechanical characteristics. Until the present, little research has been implemented to study the effects of these factors in depth on their density, porosity and modulus elasticity, and this research aims to investigate these matters.

## MATERIALS AND METHODS

This study used powdered metal in the formation of the  $\text{Al}+(\text{SiCw}+\text{Al}_2\text{O}_3\text{p})$  composite, 90% pure aluminum powder of  $2.7 \text{ gram/cm}^3$  density was the matrix together with silicon carbon whiskers and alumina particles as reinforcement. The powdered alumina of 1 – 5  $\mu\text{m}$  particle size with a density of  $3.8 \text{ grams/cm}^3$ , the SiCwfibers were of commercial quality with  $d = 0.45$  to  $0.65$ , lengths of  $l = 5$  to  $80\mu\text{m}$  and  $\rho = 3.2 \text{ grams/cm}^3$  density and hardness 2400 Hv for reinforcement. An ethanol solution ( $\text{CH}_3\text{COOH}$ ) was the solvent for the wet mixing.

The weight percentages (%wt) of the  $\text{Al}_2\text{O}_3\text{p}$  reinforcement were 3% wt, 6% wt and 9% wt respectively. Table 1 shows the weight percentages of the Al composite matrix together with the  $\text{SiCw}+\text{Al}_2\text{O}_3\text{p}$  reinforcement. At this treatment stage, the weighing of the powdered Al and  $\text{SiCw}+\text{Al}_2\text{O}_3\text{p}$  was in accordance with the weight percentages of each material.

**Table 1:** The composition by percentage of the Al matrix mass and  $\text{SiCw}+\text{Al}_2\text{O}_3\text{p}$  reinforcement.

Al (gr)	Composition of composite materials	
	SiCw (gr)	$\text{Al}_2\text{O}_3\text{p}$ (gr)
1.12	0.280	0
1.12	0.238	0.042
1.12	0.196	0.084
1.12	0.154	0.126

A wet mixing method was used for the mixing process of the aluminium matrix and  $\text{SiCw}+\text{Al}_2\text{O}_3\text{p}$  reinforcement. These were placed together in a glass beaker containing the ethanol solvent. The volume proportion of the  $\text{Al}+(\text{SiCw}+\text{Al}_2\text{O}_3\text{p})$  to the ethanol was 1:2, then the mixing was done by a magnetic stirrer on a hot plate at  $50^\circ\text{C}$  for 30 minutes until the mixing was complete. After this, the wet mixture was dried in a furnace at  $100^\circ\text{C}$  for 60 minutes, in order to evaporate all the ethanol. The mixture was then put into a mould whose internal surfaces had been coated with oil.

Compacting was done with a hydraulic press at room temperature at a pressure of 15kN for 15 minutes to ensure that the pressure was exerted evenly and to prevent any oxidization of the composite matrix. The mould was cylindrical in form thus producing a cylindrical specimen of 1 cm in length and 0.8 cm in diameter for a total volume of  $0.5 \text{ cm}^3$ .

The sintering process followed in a fluidized bed furnace in an atmosphere of argon without pressure. There were 3 sintering treatments at  $500^\circ\text{C}$ ,  $550^\circ\text{C}$  and  $600^\circ\text{C}$  with holding times of 1 hour, 3 hours and 6 hours respectively. Measuring for both volume and porosity was done by employing Archimedes's principle, where the volume of water displaced is the same as the volume of an object immersed in it. An SEM was employed to test specimens for density, porosity and modulus elasticity.

Confirmation that composite characteristics tests followed ASTM C373-88 (ASTM, 2003) standards density  $B \text{ (gm/cm}^3\text{)}$  was the comparison between the mass  $D \text{ (gm)}$  and volume  $V \text{ (cm}^3\text{)}$ , so composite density can be calculated by the following equation:

$$B = \frac{D}{V} \quad (1)$$

Actual porosity ( $P$ ) was the percentage of volume of the visible pores in the specimen to its exterior volume. In this calculation it is assumed that  $1 \text{ cm}^3$  of water weighs 1 gram, thus the equation for computing this was:

$$P \% = [(M - D) / V] \times 100 \quad (2)$$

Where: exterior volume ( $V$ ) is calculated by deducting the volume of saturated mass from the mass in the water ( $S$ ).  $V \text{ cm}^3 = (M - S)$ . The dry mass can be found by drying the mass in an oven. Suspended mass  $S$  can be measured by suspending it on the arm of a scale using a wire hanger with the scale calibrated to zero. The length of the hanger must be the same for all specimens. The saturated mass ( $M$ ) is the weight of the object after all surface water has been wiped off with a damp cloth and rolling over on a wet cloth.

Modulus elasticity was measured using the following equation:

$$E_k = \sigma / \epsilon \quad (3)$$

Where:  $E_k$  is composite modulus elasticity,  $\sigma$  is the tension and  $\epsilon$  is strain. Modulus elasticity levels were obtained by slope values of the material's elastic areas and these values can be analyzed based on the correlation between stress and strain of the elastic area obtained from a tensile strength test.

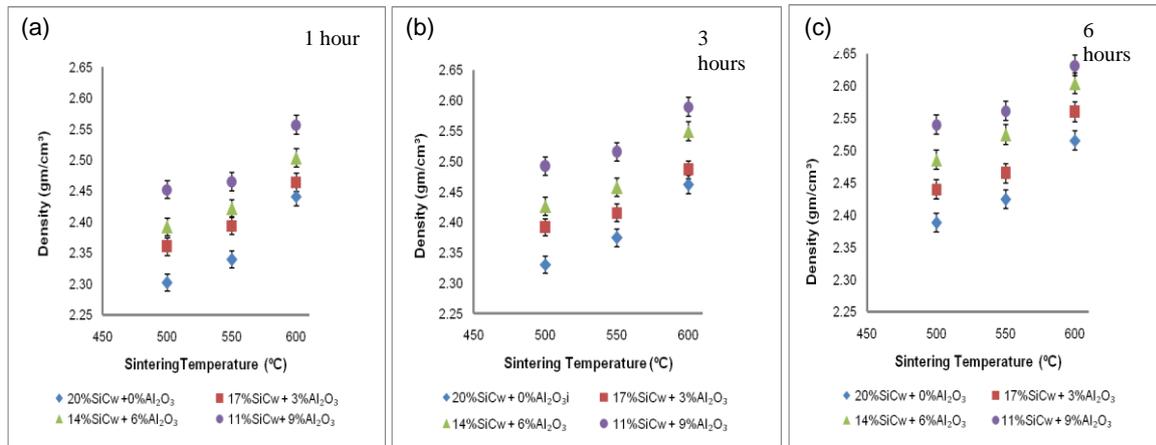
The difference value of green density with sinter density explain percentage of shrinkage because of gas evaporate or lubricant which trapped between particle surfaces. The pore shrinkage happened during sintering process. The different between green density and sintered density are the pore shrinkage that happened. The testing of scanning electron microscope (*SEM*) with testing device "Neo Space JCM-5000" used to detecting microstructure interface composite material. Mechanism taking of "SEM" data is electron bundle who radiated by electron source and then that electron bundle will get contact with specimen. Another electron get contact with specimen and other electron will bounce back. The electron who bounced will be caught with "Secondary electron" detector and other bounced electron will be caught with *Back Scatter Electron*. The observation did in the area around composite interface  $\text{Al} - [\text{SiCw} + \text{Al}_2\text{O}_3]$

## RESULTS AND DISCUSSION

### *The influence sintering temperatures on composite density:*

Figures 1 (a), (b) and (c), illustrate the relationship between composite density and sintering temperature at holding times of 1 hour, 3 hours, and 6 hours respectively. Increasing sintering temperatures together with the weight percentage of the  $\text{Al}_2\text{O}_3$  in the composite aluminum matrix resulted in an increase in composite density, but density was lowered on the introduction of the SiCw. The lowering of density was related to the pores formed in the composite where both strength and sintering temperatures raised density in each  $\text{Al}_2\text{O}_3$  weight percentage. Previous research (Heng, 2013; Karamian, 2009) reported the longer the sintering process, the greater the density. In connection with this, and also, as a result of the process of grain growth and thus the shrinking of the pores that depends on sintering temperature and the holding times length. Grain growth significantly affects pore elimination, the greater grain growth, the greater pore elimination and thus increasing density. Figure 1 (a) shows the influence of sintering temperature on density at  $500^\circ\text{C}$ , where raising the temperature of the composite reinforced with SiCw +  $\text{Al}_2\text{O}_3$ , density rose at holding times of 1 hour, 3 hours, and 6 hours respectively, this was also so where a reduction in the SiCw caused density to increase. Research results demonstrate that the composite compositions of 80% Al + (20% SiCw + 0%  $\text{Al}_2\text{O}_3$ ), 80% Al + (17% SiCw + 3%  $\text{Al}_2\text{O}_3$ ), 80% Al + (14% SiCw + 6%  $\text{Al}_2\text{O}_3$ ) and 80% Al + (1% SiCw + 11%  $\text{Al}_2\text{O}_3$ ) the density was

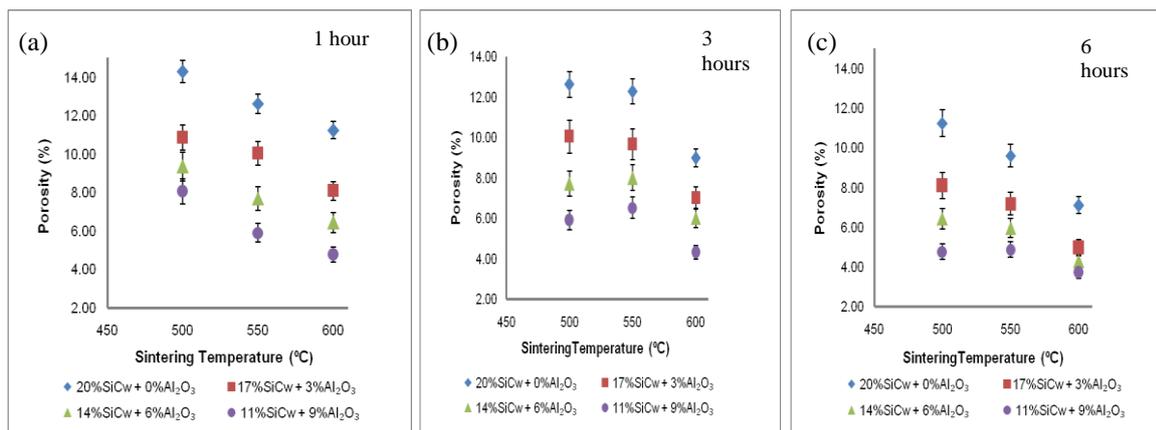
2.3023 gm/cm<sup>3</sup>, 2.3601 gm/cm<sup>3</sup>, 2.3918 gm/cm<sup>3</sup> and 2.4522 gm/cm<sup>3</sup> respectively. The same was true for the same compositions at a holding time of 6 hours and a temperature of 600°C, where the density was 2.515 gm/cm<sup>3</sup>, 2.559 gm/cm<sup>3</sup>, 2.604 gm/cm<sup>3</sup> and 2.631 gm/cm<sup>3</sup> respectively.



**Fig. 1:** The relationship between the composite matrix aluminum density and the sintering temperature at a holding time of (a) 1 hour, (b) 3 hours and (c) 6 hours.

#### *The sintering time influence on composite porosity:*

Figures 2 (a), (b) and (c) show the relationship between composite porosity and sintering holding times of 1 hour, 3 hours and 6 hours respectively and demonstrates that porosity falls at higher temperatures, which is supported by previous studies (Abdullah, 2012), this was so because the particles between composite surfaces were able to form easily during the sintering process as well as the fact that the mixing process produced an almost homogenous material. Pores were able to form due to gases trapped during the material production process and/or the vaporization of any lubricant trapped between particles between the matrix and reinforcement. Porosity is defined as the difference between the total volume of cavities in a dense object and the actual volume of the object itself and can be calculated with a standard equation (2). The porosity levels of the aluminum composite matrix and the various compositions were : 80%Al + (20%SiCw + 0%Al<sub>2</sub>O<sub>3</sub>p), 80%Al + (17%SiCw + 3% Al<sub>2</sub>O<sub>3</sub>p), 80%Al + (14%SiCw+6% Al<sub>2</sub>O<sub>3</sub>p) and 80%Al + (1%SiCw + 11% Al<sub>2</sub>O<sub>3</sub>p) at a holding time of 1 hour and a temperature of 500°C, were 14.2896%, 10.8514%, 9.3567% and 8.0491% respectively. For the same compositions at a holding time of 6 hours and a temperature of 600°C they were 7.103%, 4.291% and 3.706% respectively.

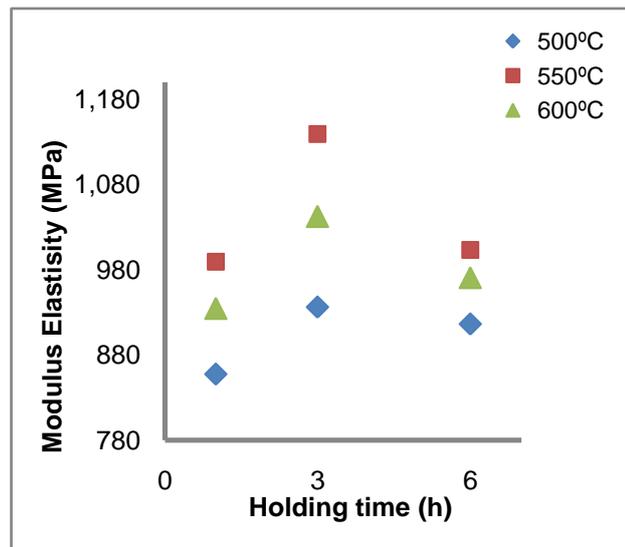


**Fig. 2:** The relationship between the composite aluminum matrix porosity and sintering temperature at a holding time of (a) 1 hour, (b) 3 hours and (c) 6 hours.

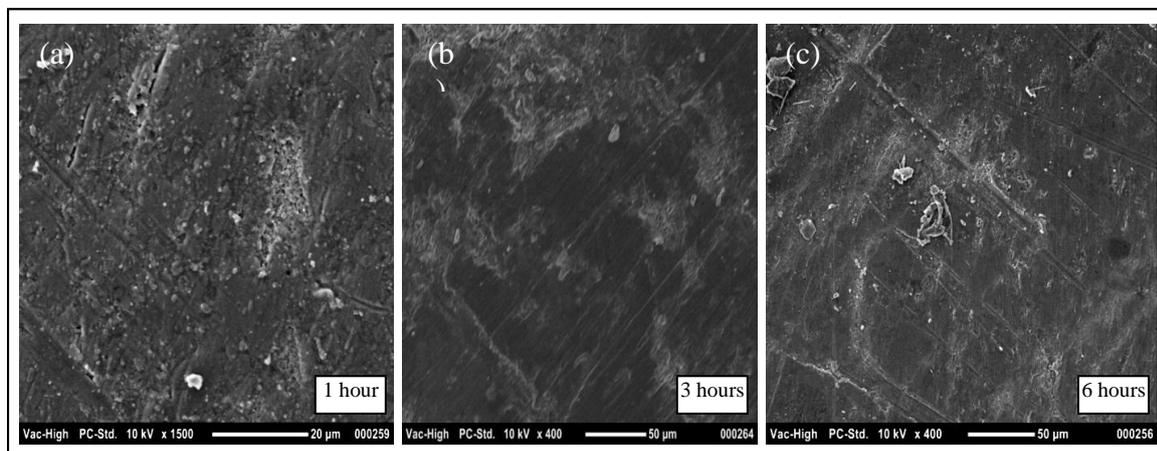
#### *The sintering holding time effect on composite modulus elasticity:*

The modulus elasticity of a composite material is an indication of whether its mechanical properties are good or not, and can be calculated by an equation (3) based on the concept of elasticity. Figure 3, indicates the relationship between sintering holding time and temperature on composite modulus elasticity. At first, modulus elasticity rose at a holding time of 1 hour and a temperature of 500°C but fell at a 6-hour holding time and a

temperature of 600°C. This was due to composite strength dropping because of the intrinsic properties and structure of the aluminum matrix itself. Holding times of over 3 hours at temperatures higher than 550°C induced the formation of fine grains causing the Al + (SiCw+Al<sub>2</sub>O<sub>3</sub>p) composite to become more ductile and also to lessen stiffness. The maximum modulus elasticity came from a holding time of 3 hours and a temperature of 550°C. Figures 4 (a), (b) and (c) of SEM photograph show that holding times caused composite structure to become ever more homogenous and the aluminum matrix to become ever more evenly distributed. The 6-hour holding time indicates the higher the percentage of Al<sub>2</sub>O<sub>3</sub>p reinforcement by weight, the lower the composite modulus elasticity levels. This was caused by the fact that composite particles became increasingly finer and became ever more evenly and homogeneously distributed and thus increasing ductile, whereas composite stiffness became lower. Apart from this, composite density after sintering at longer holding times and higher temperatures can also reduce modulus elasticity and give rise to fracturing.



**Fig. 3:** The relationship between the composite aluminum matrix modulus elasticity and the sintering holding time.

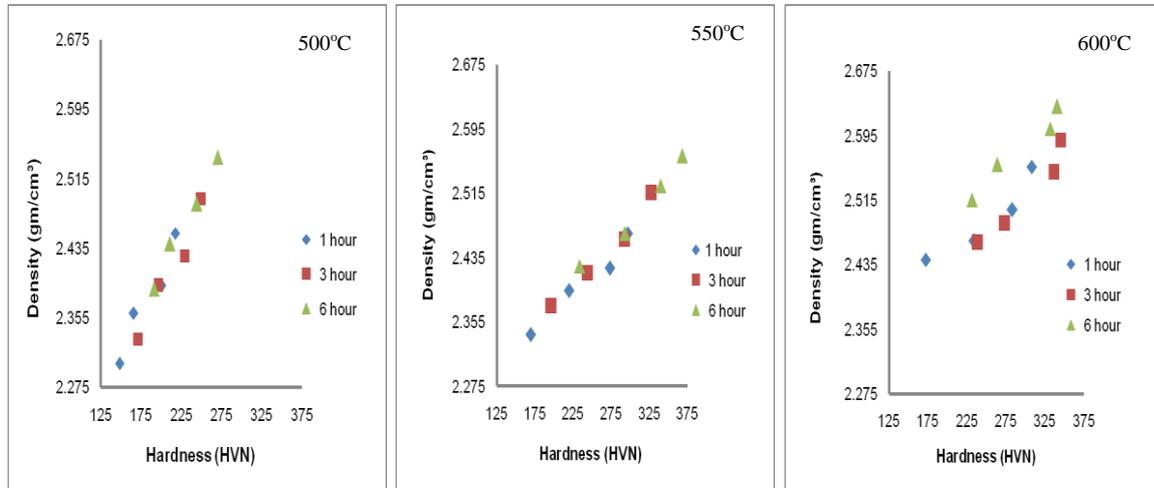


**Fig. 4:** SEM photographs of the Al+(SiCw+Al<sub>2</sub>O<sub>3</sub>p) composite interface at 600°C and holding time of (a) 1 hour, (b) 3 hours and (c) 6 hours.

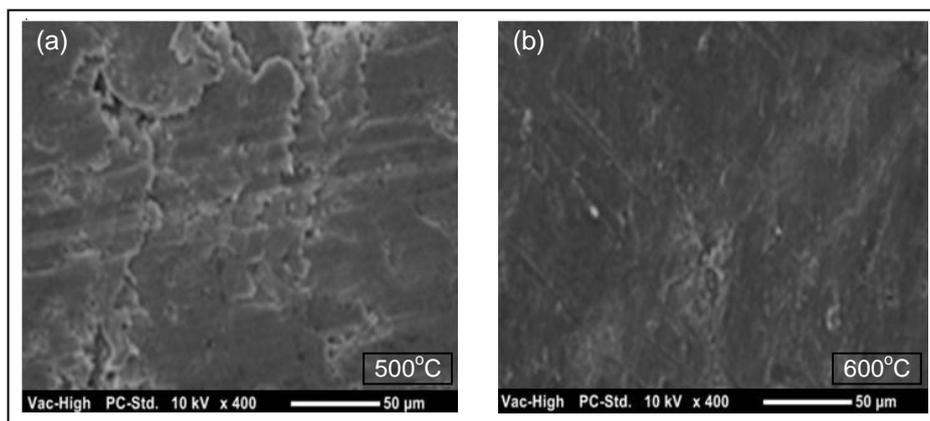
#### *The sintering density influence on composite hardness:*

Figures 5 (a), (b) and (c) illustrate the relationship between density and hardness in the Al + (SiCw + Al<sub>2</sub>O<sub>3</sub>p) composite as well as heat and sintering time treatments. Increased amounts of the Al<sub>2</sub>O<sub>3</sub>p reinforcement together with a longer holding time would raise the material density. The temperature increased with a longer holding time would raise the material hardness. Figure 5(a) shows that the longer holding time given would increase both the material hardness and density levels as shown by the following results : 148.1632VHN, 165.9591 VHN, 217.2774 VHN and 2.3029 gm/cm<sup>3</sup>, 2.360 gm/cm<sup>3</sup>, 2.391 gm/cm<sup>3</sup> and 2.452 gm/cm<sup>3</sup> at 500°C. The same increase in hardness and density was obtained in the sintering treatments at 550°C and 600°C, figures

5 (b) and 5(c). The higher the sintering temperature, the higher both the density and hardness levels obtained, this was due to the successful grain growth and the reduction in pores depending on treatment stage, Figures 6 (a) and (b).



**Fig. 5:** The composite aluminum density influence on hardness at the temperatures of (a) 500°C, (b) 550°C and (c) 600°C.



**Fig. 6:** SEM photographs of the Al+(SiCw+Al<sub>2</sub>O<sub>3</sub>p) composite interface at (a) 500°C with holding times 1 hour, and (b) 600°C with holding times 6 hours.

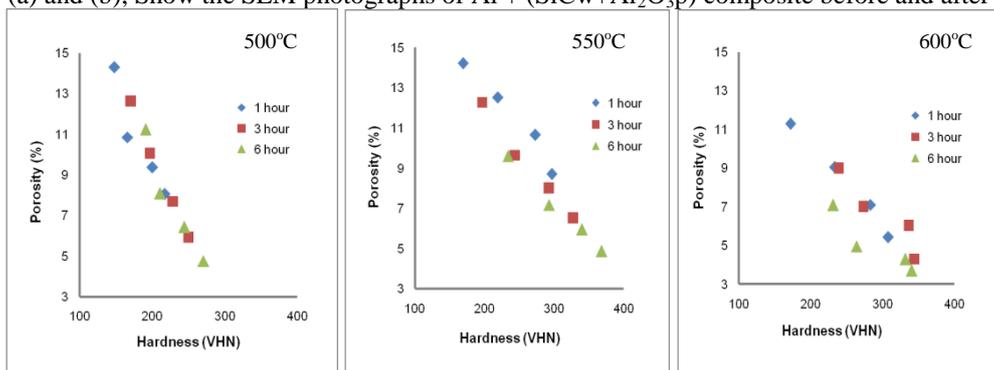
#### ***The porosity effects on composite hardness:***

During the sintering process, the number pores falls, the higher the temperature and the longer the sintering process of the Al + (SiCw + Al<sub>2</sub>O<sub>3</sub>p) composite, the lower the porosity and the higher the hardness. Figures 7(a), (b) and (c) demonstrate the relationship of porosity to composite hardness as well as the effects of longer holding times in reducing the number of pores and thereby increasing composite density. Hardness levels were also increased by the rapid development of a relative grain hardness which occurred towards the end of the sintering process. However, at a sintering temperature of 600°C, hardness fell, Fig. 7(c), this was a result of the intrinsic properties of the aluminum matrix as well as temperature and holding time nearing aluminum's melting point which reduced both ductile and stiffness and thereby lowered hardness.

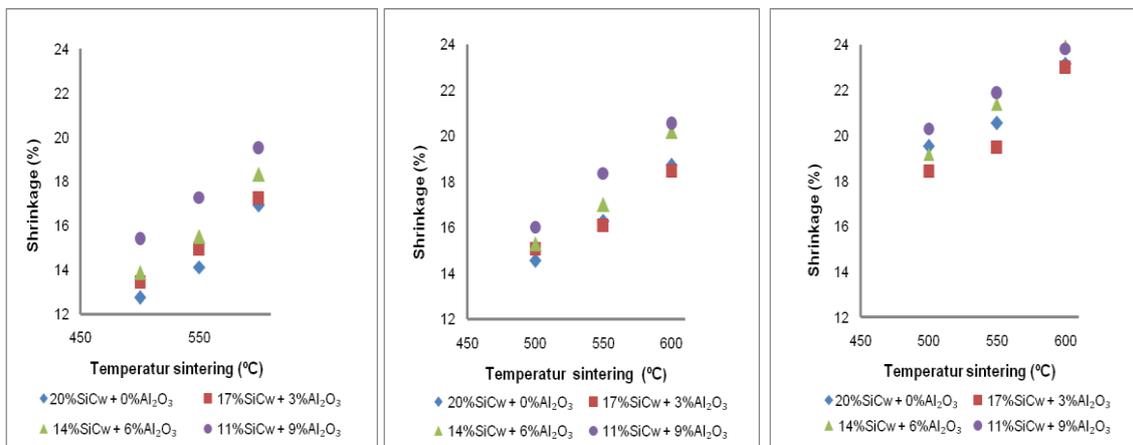
#### ***The sintering temperature and holding time influence on composite shrinkage:***

During sintering, pore shrinkage takes place which is dependent on mid to intermediate stages of this process. The longer the holding time, the greater the pore shrinkage took place. This shrinkage is defined as the difference between density before and density after sintering as throughout this process, bonding between particle surfaces becomes ever more even and homogenous. Figures 8 (a), (b) and (c) show the relationship between sintering temperatures and shrinkage, and the results of this research indicate the effect of pore shrinkage percentage rises with increased temperatures at holding times of 1 hour and 3 hours. Yet, at a holding time of 6 hours and a temperature 550°C shrinkage slowed, but increased again at 600°C where density rose for each Al + (SiCw+Al<sub>2</sub>O<sub>3</sub>p) composite treatment due to increased density from rising pore reduction and a more

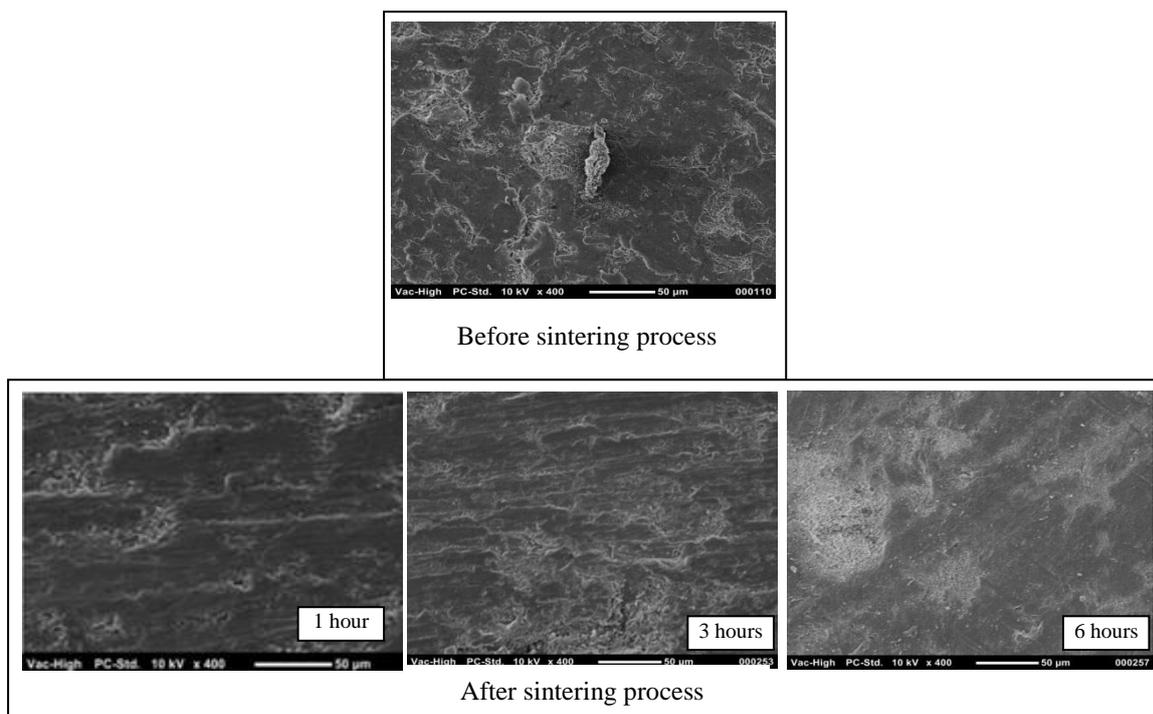
homogenous structure. Greater homogeneity affected both matrix grain size and composite reinforcement. Figures 9 (a) and (b), Show the SEM photographs of Al + (SiCw+Al<sub>2</sub>O<sub>3</sub>p) composite before and after sintering.

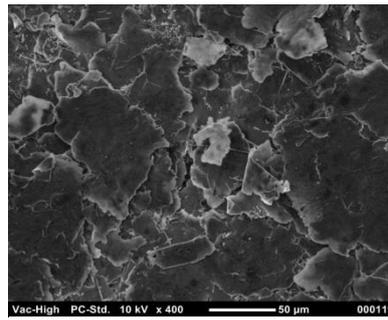


**Fig. 7:** The influence of composite aluminum porosity on hardness at the temperatures of (a) 500°C, (b) 550°C and (c) 600°C.

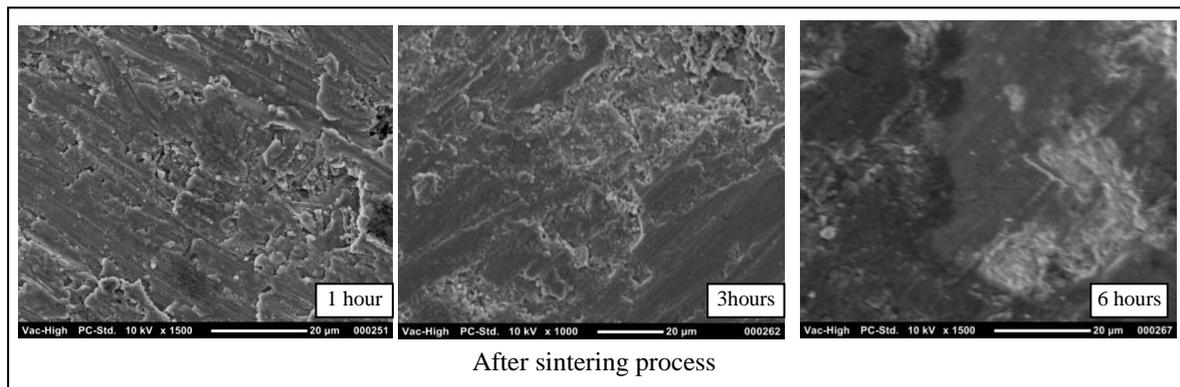


**Fig. 8:** The effects of the relationship between aluminum matrixes pore reduction and sintering temperatures (a) 1 hour, (b) 3 hours and (c) 6 hours.



**Fig. 9 (a):** An SEM photograph of the 80%Al + (20%SiCw+0%Al<sub>2</sub>O<sub>3</sub>p) interface composite.

Before sintering process



After sintering process

**Fig. 9 (b):** SEM photograph of the 80%Al+(11%SiCw+9%Al<sub>2</sub>O<sub>3</sub>p) interface composite.**Conclusion:**

1. The influence of temperatures and holding times on both composite density and composite porosity demonstrated that raising the sintering temperature and lengthening the holding time would increase the composite density and reduced the composite porosity due to rising grain growth. At a sintering temperature of 600°C and a holding time of 6 hours eliminated all pores. The same situation was true for the composite modulus elasticity which was increased during the 1-hour holding time at 500°C treatment, but fell during the 6-hour holding time at 600°C treatment. That was the result of a drop in stiffness from the structure and ductile properties intrinsic to the aluminum matrix itself.
2. The effects of density and porosity on hardness, hardness rose due to the increase in density and the reduction in porosity from higher temperatures and longer sintering holding times.
3. The research results shows that the effects of pore reduction percentages rose with a higher sintering temperatures at 1-hour and 3-hour holding times, whereas, the 6-hour holding time at 550°C produced lower *shrinkage* rates but would rise again at 600°C as a result of the sintering process eliminating pores and greater composite homogeneity.

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