Penerbit Akademia Baru

Malaysian Journal on Composites Science and Manufacturing

Journal homepage:
https://www.akademiabaru.com/submit/index.php/mjcsm/
ISSN: 2716-6945

Characterization of the Al6061 Alloy Reinforced with SiC Nanoparticles and Prepared via Powder Metallurgy

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ARTICLE INFO

Article history:
Received 13 August 2022
Received in revised form 5 November 2022
Accepted 8 November 2022
Available online 30 November 2022

ABSTRACT

This study aimed to investigate the effect of sintering temperature on the microstructures and mechanical properties of a composite material prepared by powder metallurgy technique. The composite material was a 6061 aluminum alloy matrix reinforced with silicon carbide nanoparticles. The characterization techniques were X-ray diffraction, scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy, optical microscopy, Vickers hardness, and density measurements. The composite material was successfully prepared, as indicated by the homogeneous distribution of the silicon carbide nanoparticles in the aluminum matrix. The sintering temperature has a remarkable effect on the densification of the composite material. It was found that when the sintering temperature increases up to 600°C, the density increases. This phenomenon is also accompanied by an increase in hardness up to 32.2 Hv. We have deduced that the optimal sintering temperature is 600°C.

Keywords:
Composite material, powder metallurgy, sintering, mechanical properties, microstructure

1. Introduction

Metal matrix composites (MMCs) have rapidly replaced conventional metal alloys in many applications as their use permeates aerospace, automotive, nuclear, biotechnology, sports, automotive, and thermal applications [1]. The MMCs are combinations of two or more dissimilar metals, intermetallic compounds or second phases in which dispersed phases are incorporated into the metal matrix [2]. The properties of MMCs depend on the properties of the constituent phases, their relative quantity and the geometry of the dispersed phase, including the size, shape and orientation of the particles in the matrix [2-4].

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https://doi.org/10.37934/mjcsm.9.1.2234

Amongst MMCs, aluminium-based metal matrix composites (AMMCs) are widely used in industry, due to their low density, high specific modulus, high strength-to-weight ratio, and toughness [5]. Al-Mg-Si alloy, and more specifically AA6061, is a heat-treated aluminium alloy that is widely used for structural applications due to its good strength, weldability, corrosion resistance and toughness immunity to stress corrosion cracking [6]. The major reinforcements used in the fabrication of MMCs include silicon carbide, boron carbide, alumina, tungsten carbide, titanium dioxide, graphite, carbon nanotubes and silica. However, silicon carbide (SiC) and alumina (Al_2O_3) are the most commonly used as reinforcements [7-8].

Compared to unreinforced aluminium alloys, aluminium alloys reinforced with ceramic particles show improvement in their physical and mechanical properties, such as thermal expansion, thermal diffusivity, tensile and compressive, creep and tribological behaviors [9].

In recent years, SiC particles (SiCp) has attracted the attention of researchers, the use of SiCp as a reinforcement is due to the fact that SiC is chemically compatible with aluminum and forms an adequate bond with the matrix without developing an intermetallic phase. In addition, it has other advantages such as excellent thermal conductivity, good workability and low cost, good wear resistance with high hardness and high-temperature resistance [10].

The incorporation of ceramic particles into the metal matrix can be done in a liquid state such as stirring casting [2,11-13], infiltration [14], die casting [15], etc., or in a solid state using powder metallurgy (PM) [16]. Powder metallurgy is a processing technique in which particles are consolidated into semi-finished and finished products. It consists of mixing the powders, compacting and then sintering them at a given temperature [7]. The first advantage of this technique is its low processing temperature compared to fusion techniques, i.e., the sintering temperature in the powder metallurgy process is lower than the melting point of the major constituents of the material. The second advantage is the homogeneous distribution of the reinforcing particles. The third advantage is the ability to obtain a product with an almost clean shape [17-18].

Omyma et al. [18], studied the effect of the size of SiC nanoparticles on the physical and mechanical properties of Al (99.9% purity) matrix composite produced by PM technique followed by hot extrusion. They found that the densification and thermal conductivity of the composites decreased with the increasing amount of SiC and increased with increasing SiC particle size. Reddy et al. [19] studied the effect of adding SiC at the nanoscale on the structural, mechanical and thermal properties of the reinforced aluminum alloy. They found that adding SiC increased the strength of the composite material and decreased the ductility and also coefficient of thermal expansion. Habibur et al. [2] examined the microstructures, mechanical properties and wear characteristics of as-cast reinforced aluminium matrix composites with different SiC contents. They concluded that the introduction of SiC reinforcements into the aluminum matrix increases hardness, tensile strength and wear resistance. Moreover, they observed that extensive incorporation of SiC particles in the matrix induces a grouping and an inhomogeneous distribution of SiC particles and porosities.

Among aluminum alloys, AA6061 is widely used as a matrix material in the manufacture of MMCs. It is noted that Al6061-SiC composites have been prepared either by liquid processes [7,20-32] or solid-state processes using different techniques [4,33-41]. Previous research [4,35-37,40] on the fabrication of AMMCs, such as AA6061 aluminum alloy reinforced with SiC particles, used powder metallurgy to prepare these composites. Different routes have been applied, such as high-energy grinding, cold compaction, sintering and hot isostatic pressing (HIP). In addition, an aging treatment has also been applied to increase the hardness of the AMMCs.

In our research, a composite material based on AA6061 aluminum alloy reinforced with SiC nanoparticles was prepared using the powder metallurgy technique. The effects of the sintering temperature on the microstructure and the mechanical properties of the composite material were studied. The main characterization techniques were X-ray diffraction, optical microscopy, scanning electron microscope equipped with an energy dispersive spectroscopy (EDS) detector, Vickers hardness, porosity and density measurements.

2. Experimental Procedure

2.1 Metal Matrix and Reinforcement Powders

This study uses a powder of AA6061 with an average particle size of 63 µm and a theoretical density of 2.74 g/cm^3 as the matrix material. The chemical composition of AA6061 is shown in Table 1. SiC nanoparticles with an average particle size of 0.05 µm and a density of 3.21 g/cm^3 were added as reinforcement to prepare composites. The particle size and density of the matrix and reinforcement are shown in Table 2. The AA6061 powder and SiC nanopowders were supplied by Good Fellow Cambridge Limited Huntington PE29 WR, England.

Table 1
Chemical composition of AA6061 matrix alloy

Elements	Al	Si	Fe	Cu	Mg
(Wt.%)	97.5	0.6	0.5	0.4	1.0

Table 2
Particle size and density of the matrix and the reinforcement

	Materials	Particles size (µm)	Density (g/cm^3)
Matrix	AA6061	63	2.74
Reinforcement	SiC	0.05	3.21

2.2 Preparation Method

In this study, the composite was prepared by incorporating SiC nanoparticles in an aluminum matrix with an amount of 7.5% by weight. The high-energy ball milling was performed in a planetary ball mill (FRITSCH Planetary Ball Mills, Pulverisette 7) with tungsten carbide (WC) balls (7-7.2 mm diameter) in an argon gas atmosphere. The ball-to-powder ratio was 4:1, and the mill speed was maintained at 300 rpm for a period of 2 hours to obtain a homogeneous mixture. Argon gas was used to prevent the agglomeration of aluminum alloy powder particles. The powders were then compacted uniaxially in a cylindrical mould (diameter = 14 mm) using a HERZOG compacting machine. Zinc stearate ($Zn(C_{18}H_{35}O_2)_2$) was used during the compaction process to aid the removal of the green compact sample from the mold. The compaction load was applied gradually to obtain 10 tonnes. Then, the green samples were ejected from the dies and sintered at different temperatures (585, 600, 615 and 630°C) in a sintering oven and under a controlled atmosphere (argon and hydrogen gas). The heating and cooling rates were 4°C/min and 15°C/min, respectively.

2.3 Characterization

The theoretical density was calculated by applying the rule of mixtures as a function of the weight fraction of reinforcement of Eq. (1) [18]. The final density was measured using the Archimedes method (ASTM: B962-13), according to Eq. (2) [18], and the percent porosity was determined using Eq. (3) [42].

$$\frac{100}{\rho_t} = \frac{m}{\rho_m} + \frac{r}{\rho_r} \quad (1)$$

$$\rho_e = \frac{w_a}{w_a - w_w} \quad (2)$$

$$P = \left(1 - \frac{\rho_e}{\rho_t}\right) \times 100 \quad (3)$$

where ρ_t and ρ_e are respectively the theoretical and final (measured) densities of the composite; ρ_m and ρ_r are the densities of the matrix alloy and of the reinforcements, respectively; *m* and *r* are wt.% of matrix alloy and reinforcements, respectively; w_a is the mass of the cylindrical sample in air and w_w is the mass of the sample in water. The sample was weighed using a digital balance with an accuracy of 0.1 mg. *P* is the percentage of porosity of the composites. The sintered samples were polished in an automatic polishing machine (STRUERS), using emery papers of different grades from P600 to P4000, followed by finishing with colloidal particles of 3 um, 2 um, and OPS solution, respectively. Then, the samples were etched with HF reagent and examined under a HIROX Kh-8700 digital microscope and a scanning electron microscope (HITACHI SU8020) equipped with an energy dispersive spectroscopy (EDS) detector. The phase analysis was carried out using an X-ray diffractometer, Siemens model (BRUKER D-5000) with CuKα radiation, in a 2θ range from 10° to 90°. The mechanical characterization tests consisted of hardness measurements using a Vickers tester (EMCO-M4U025) under an applied load of 5 kg. An average of six hardness measurements was taken per sample.

3. Results and Discussion

3.1. Microstructural Observation of AA6061/SiC Composites

Figure 1 shows the microstructures of AA6061 reinforced composites with 7.5 wt.% SiC, and sintered for 2 hours at 585, 600, 615 and 630°C. According to these optical observations, most of the SiC particles are localized between the Al particles, and an interface has formed between the SiC and Al particles. Moreover, increasing the sintering temperature did not affect the particle size, as there is no significant change in particle size. However, the densification phenomenon can be observed above the sintering temperature of 600°C (Figure 1b).

A selected area from a sample sintered at 600°C revealed a bonding process between the aluminum particles (Figure 2), as observed by other study [43]. Figure 2 shows a growth reaction between two adjacent aluminum grains (particle 1 with particle 2, and particle 3 with particle 4). This welding process is carried out by the semi-fusion process, because the liquidus of the AA6061 matrix is about 588°C [44]. However, increasing the sintering temperature to more than 600°C causing a morphological transformation of the composite material, because at 615 and 630°C (Figure 1(c) and (d)), the fusion effect of the particles of aluminum is more visible and the Al particles were surrounded by SiC. At this high temperature, the interaction of SiC with Al particles is more intense, as clearly shown in Figure 3. Muttharasan et al. [44] reported that when the processing temperature increases, a chemical reaction taking place between the liquid Al and the solid SiC. At high temperatures, solid SiC separates into Si and C. In addition to this segregation, another reaction between Al and C can form this Al_4C_3, and Si dissolves faster in liquid aluminum than carbon [45].

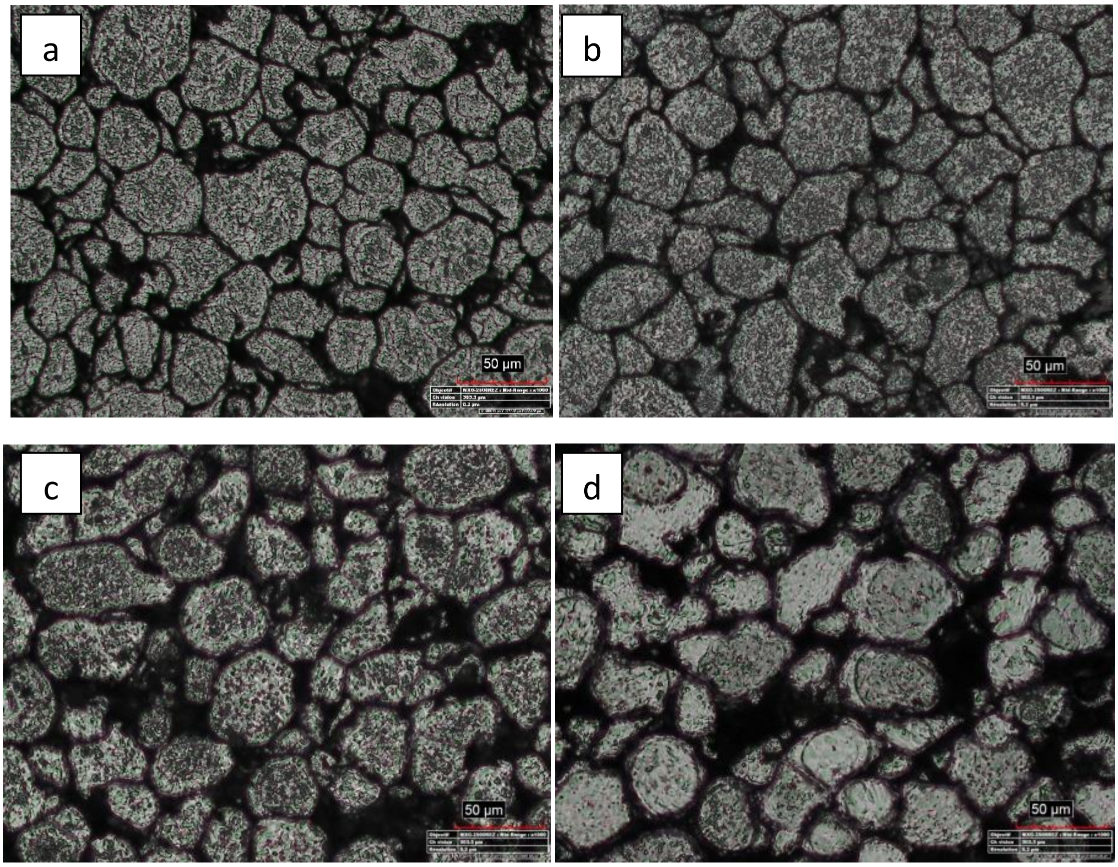

Fig. 1. Optical observations of reinforced AMMC with 7.5 wt.% SiC, sintered for 2 hours at (a) 585°C, (b) 600°C, (c) 615°C, and (d) 630 °C

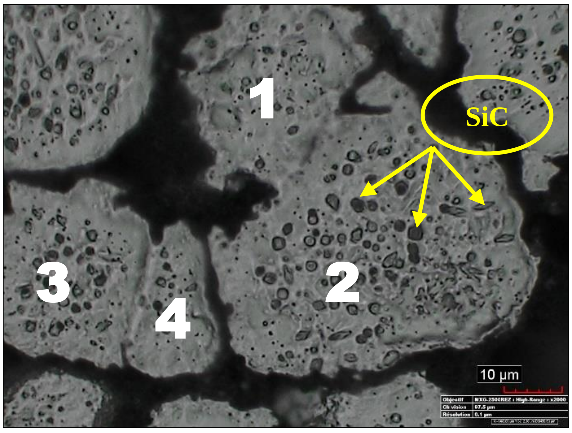

Fig. 2. Microstructure observation of 7.5 wt.% SiC reinforced AMMC sintered for 2 h at 600°C

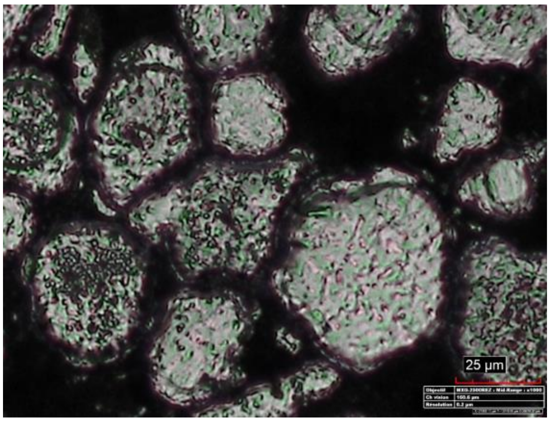

Fig. 3. Microstructure observation of 7.5 wt.% SiC reinforced AMMC sintered for 2 h at 630°C

3.2 EDS Analysis

Figure 4 shows the result of the chemical analysis in the matrix (area 1) and in the connection joint between the aluminum particles (area 2) of the composite material sintered at 630°C (Figure 4a). The purpose of this analysis was to determine the distribution of the main elements likely to be found in the composite material prepared. The chemical analysis spectra (Figure 4b) show us that the bonding joints are rich in silicon and carbon, which represents the proof of the existence of SiC in this bonding zone and confirmed the microstructural observation. However, the presence of oxygen in the two zones analyzed is due to the conditions of production of the composite material before the sintering treatment.

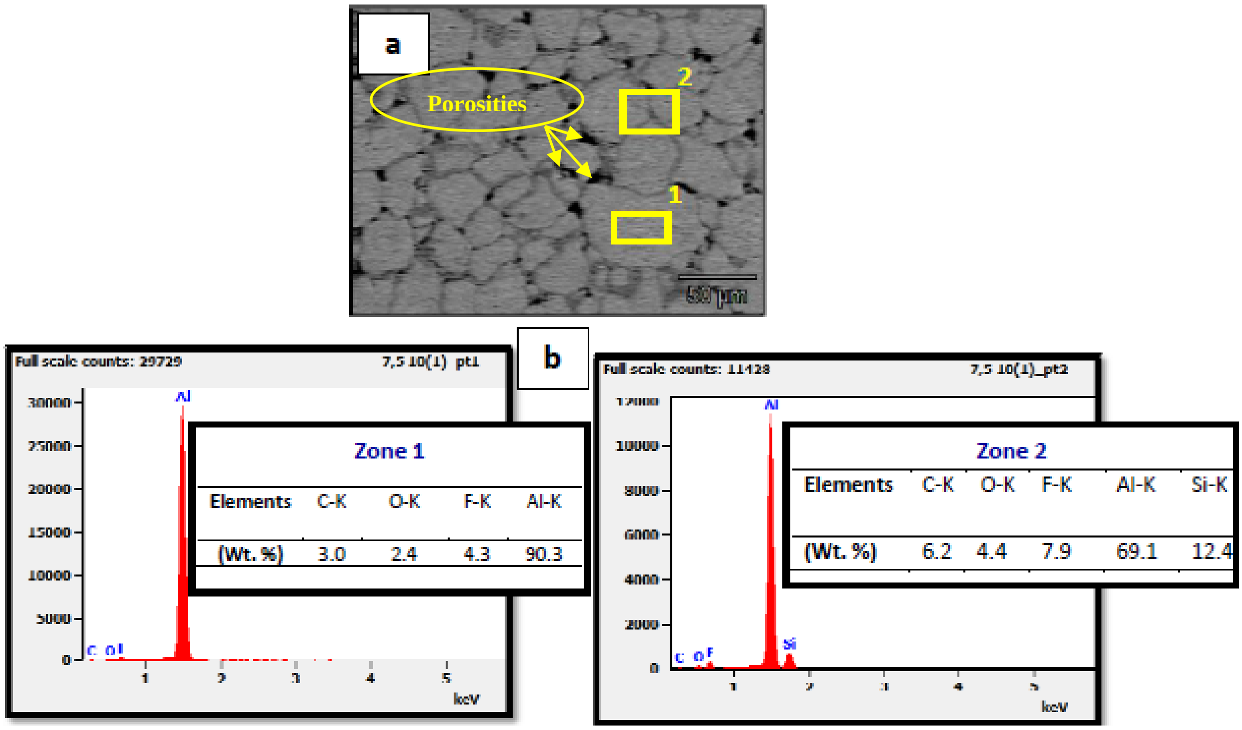

Fig. 4. (a) SEM image with (b) EDS spectra with the chemical composition of the two selected zones in the Al6061-SiCp composites (reinforced with 7.5 wt.% SiC) and sintered for 2 h at 630°C

3.3 Density and Porosity

Figure 5 shows the sintering temperature's effect on the composite material's densification. It can be noted that the highest density (2.75 g/cm³) is reached after the sintering temperature of 600°C, of which there is a slight difference between this value and the theoretical density of a composite, which is 2.77 g/cm³. This result confirms the success of the fabrication of this composite by the powder metallurgy technique. Remember that the density of SiC (3.21 g/cm³) is greater than that of aluminum (2.7 g/cm³), which favours this densification of the composite material. Additionally, this might have been associated with stronger or denser interface bonding between matrix and reinforcement, which is affected by increasing sintering temperature [46].

In addition, the calculated porosity of the reinforced composites is shown in Figure 6. It is observed that the porosity is also affected by the sintering temperature. The porosity of the composite material increases with the increase in sintering temperature. This is due to high-temperature transformations such as the segregation of SiC and the fusion of Al particles. The lowest porosity value was found to be 0.72% for the sample sintered at 600°C, due to the densification phenomenon at this sintering temperature. However, the highest porosity (5.05%) is calculated for the composite material sintered at 630°C. This is an obvious result because the interfacial interaction between SiC and aluminum is more intense at this temperature. Bonding at the metal-ceramic interface is a significant phenomenon in metal matrix composites. The properties of the composite mainly depend on the behavior of the interface [45]. As reported in previous works [47-48], this interfacial reaction is mainly influenced by some factors, such as the free energy at the interface, the convection properties and the temperature gradient that exists between the particles and the matrix during the sintering process. It is also found from Figures 5 and 6 that there is a relationship between density and porosity, i.e., as the density of the composite increases, the porosity decreases and vice versa.

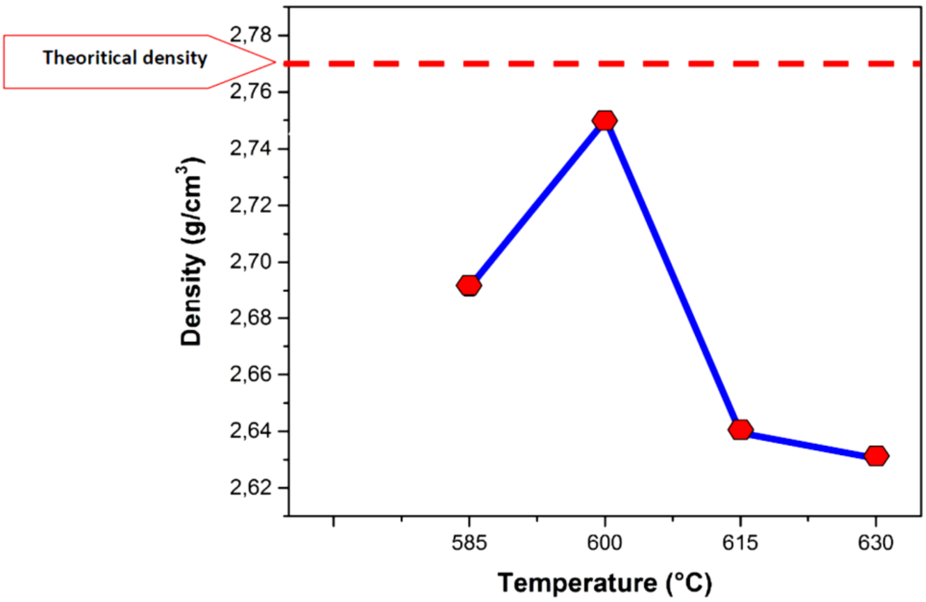

Fig. 5. Theoretical density and average bulk density of reinforced composites fabricated using powder metallurgy method at various sintering temperatures from 585 to 630°C

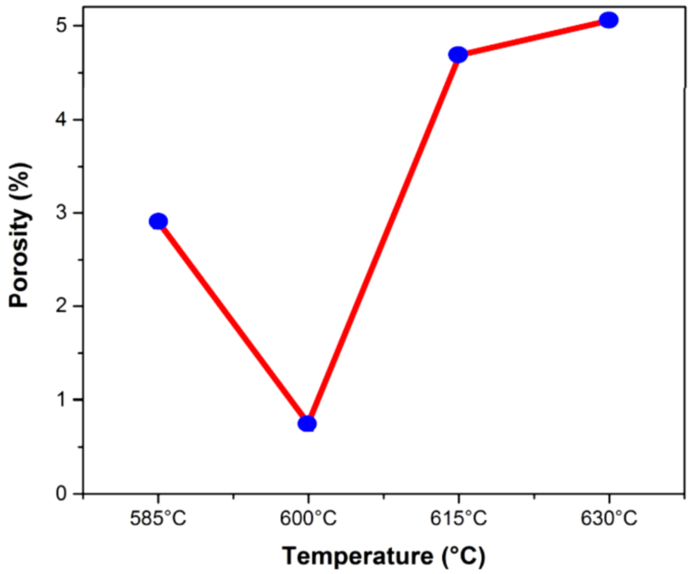

Fig. 6. Variation of porosity at various sintering temperatures of 585, 600, 615, and 630°C

3.4 XRD Diffraction

Figure 7 shows the X-ray diffraction (XRD) patterns of SiC powder, AA6061 powder, and aluminum composites reinforced with 7.5 wt% SiC sintered at 600°C. The XRD pattern of SiC powder reveals major SiC peaks. The same observation can be made for the XRD diagram of the AA6061 powder. The XRD patterns of the unsintered and sintered aluminum composite at 600°C only show the peaks corresponding to the diffraction of Al and SiC particles. It can be inferred that during the sintering process, no solid state reaction took place between the matrix and the reinforcement to form other undesirable phases at all sintering temperatures, since the XRD spectra show no sign of the presence of secondary phases. Similar observations were reported by Omyma et al. [18] for conventionally extruded Al-SiC composites.

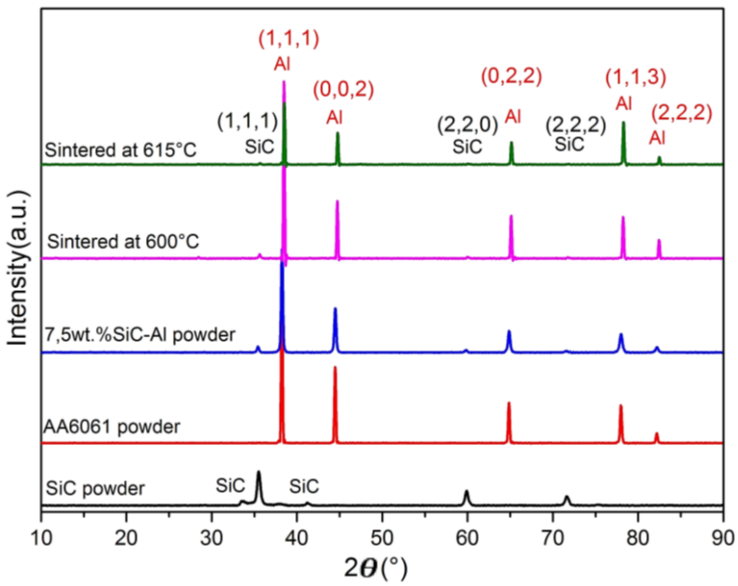

Fig. 7. XRD patterns of SiC powder, AA6061 powder, and aluminum composites reinforced with 7.5 wt.% SiC sintered at 600°C

3.5 Hardness

Figure 8 shows the histogram of the hardness variation of AMCs at different sintering temperatures. The hardness reaches its maximum value at the sintering temperature of 600°C, and then it decreases with the increase of sintering temperature. Thus, the maximum hardness value (32.2 HV) is observed in the AMMC sintered at 600°C.

From the results of density and porosity, it was obvious that with the increase of the sintering temperature, the hardness and density of composites increased at the beginning, reached the highest values at 600°C, but decreased as the temperature increased beyond 600°C. Meanwhile, the porosity decreased and increased inversely proportional to the density and hardness.

This highest hardness value of the composite is due to the highest density after this sintering temperature. It is important to mention that the high hardness values obtained by Knowles [4] in AA6061 reinforced with SiC nanoparticles were due to three factors. The first factor was the incorporation of fly ash particles with SiC nanoparticles. The second factor was related to the method of preparation. The third factor is the aging treatment applied after the preparation of the AMMCs and which was a critical factor on the hardening of the composite material.

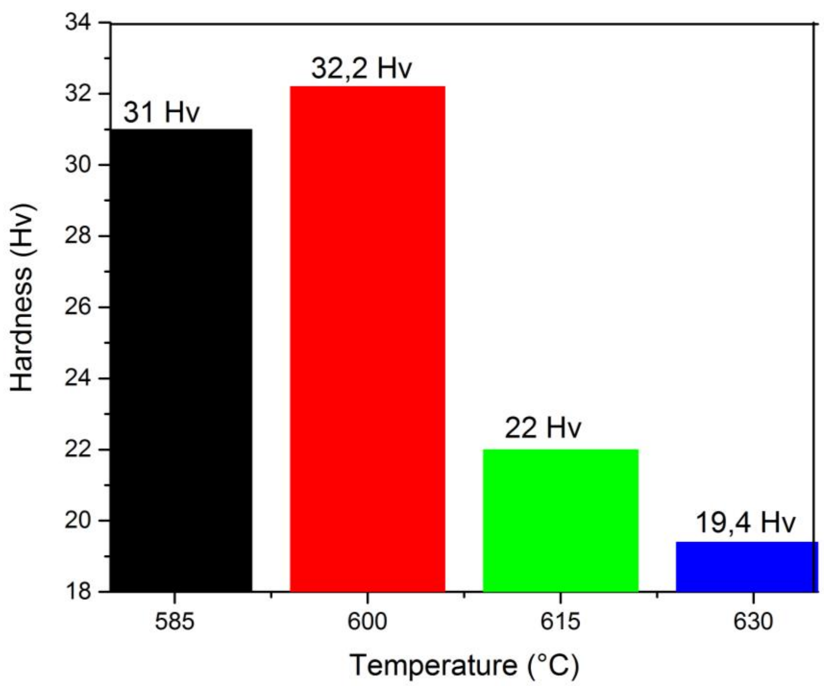

Fig. 8. The Vickers HV5 hardness of composite samples reinforced with 7.5 wt.% SiC, and sintered for 2 hours at 585, 600, 615, and 630 °C

4. Conclusions

In this study, the effect of sintering temperature (585, 600, 615 and 630°C) on an AA6061 aluminum matrix reinforced with 7.5 wt% SiC, prepared by powder metallurgy technique, has been carried out. The microstructural aspects, the hardness and the density of the prepared composites were studied. Based on the experimental results, the following conclusions can be derived:

(1) Microstructural observations revealed a typical composite material with the distribution of SiC between Al particles.
(2) The segregation of SiC particles in AA6061 aluminum matrix increased with the increase of sintering temperature. At high temperatures, the Al particles were surrounded by SiC.
(3) The XRD spectra of the composites revealed the presence of SiC nanoparticles in the aluminum matrix of the composite material.
(4) Density increased, and porosity decreased with increasing sintering temperature up to 600°C and vice versa up to 630°C.
(5) The hardness increased from 31.0 Hv to 32.2 Hv with increasing sintering temperature up to 600°C, then decreased to 19.4 Hv as the temperature increased to 630 °C.

Finally, based on all the results, the composite material, sintered at 600°C, showed the maximum density and hardness.

Acknowledgment

The authors would like to thank WBI and MESRS of Algeria for their financial support. We would also like to thank the entire team of the metallurgy department of the University of Mons for their technical support and encouragement.

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