Mechanical Behavior of Al-SiC_{np} Nanocomposite Fabricated by Hot Extrusion Technique

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Abstract: In this paper, fabrication and characterization of Al-SiC nanocomposites is investigated. The Al matrix is reinforced with different amounts of SiC nano-particles using mechanical milling, cold pressing, and, hot extrusion techniques. To get the best quality of the samples, the extrusion process is optimized firstly. With this regard, hot extrusion parameters such as the rate of extrusion, temperature, the extrusion ratio, lubrication, and the die set dimensions are experimentally studied. Finally, the nanocomposites with relative density more than 99% could be successfully fabricated under extrusion ratio of 8.5:1. As-extruded billets were then used to prepare standard tensile test specimens based on ASTM-E8. Afterwards, relative density, tensile behaviour, and micro-hardness of the samples were determined. The results show about 50% improvement for both the tensile strength and micro-hardness and near 1% reduction of relative density as the content of SiC reinforcement increases to 3 vol%. Therefore, specimens with higher strength-to-weight ratio which is a key parameter in aerospace and automotive applications can be produced using current techniques.

Keywords: Hot extrusion, Mechanical behaviour, Nanocomposite, Powder metallurgy

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1 INTRODUCTION

Aluminium matrix composites (AMCs) have wide applications in different industries such as automotive, aerospace, military, and nuclear power due to their suitable mechanical and physical properties. These materials have attracted more attention due to their high strength, low density, good wear resistance and also different fabrication techniques [1-3]. Aluminum matrix can be reinforced using either of particles or fibers. Particles reinforcement is the most commonly used form due to easier manufacturing processes and achieving to uniform dispersion and finally to more sophisticated properties in the composites [4], [5]. The composites reinforced by ceramic particles like SiC can be fabricated by some methods such as powder metallurgy (PM) [6], [7] and stir casting [8]. The first requirement to achieve desired quality of composites is uniform dispersion of the second phase in the matrix material. PM is considered to be a suitable method due to providing uniform and isotropic distribution [9], [10].

Hot extrusion is one of the most promising PM techniques which can be used for fabrication of metal matrix composites (MMCs) or metal matrix nanocomposites (MMNCs) [4], [9], [11]. Senthilkumar et al., [12] reinforced Al with nano and micro Al_2O_3 particles using hot extrusion process and obtained low cycle fatigue behaviour of their samples. They observed higher life for nano reinforced composites compared with micron sized composites. Abdollahi et al., [9] used mechanical milling and hot extrusion under 750 °C temperature and 10:1 extrusion ratio to produce Al2024-B_4C nanocomposites. They obtained dry sliding and mechanical behaviour of their fabricated samples. El-Kady et al., [4] also fabricated Al-SiC nanocomposite using mechanical milling, cold pressing, sintering, and hot extrusion under 550 °C temperature and 9:1 extrusion ratio. They studied relative density, thermal conductivity, hardness, and compressive strength and found decrease of densification and thermal conductivity and increase of hardness and compressive strength with increasing the amount of SiC. Similar researches were also performed by Atrian et al., [2], Jafari et al., [5], and Rizaneh et al., [11].

In this research paper, Al/SiC nanocomposite is produced successfully by solid-state method using mechanical milling, cold pressing, and hot extrusion techniques. Optimization of the hot extrusion process to fabricate the best quality samples with highest level of consolidation is the first part of this study. Then, the effects of volume fraction of SiC nano reinforcement on density, micro-hardness, and tensile behaviour of the specimens are investigated as the main core of this research.

2 EXPERIMENTS

2.1. Materials

As-received materials were pure aluminium (99.6% purity, average particle size of 100 µm, and irregular morphology) as the matrix and SiC powder (average particle size of 50 nm, purity > 99%, specific surface area > 90 m^2/g, near spherical morphology) as the reinforcement. In order to ensure the purity and particle size of aluminium powder, spark emission spectroscopy (SES) and scanning electron microscopy (SEM) was used. Particles morphology is illustrated in Fig. 1.

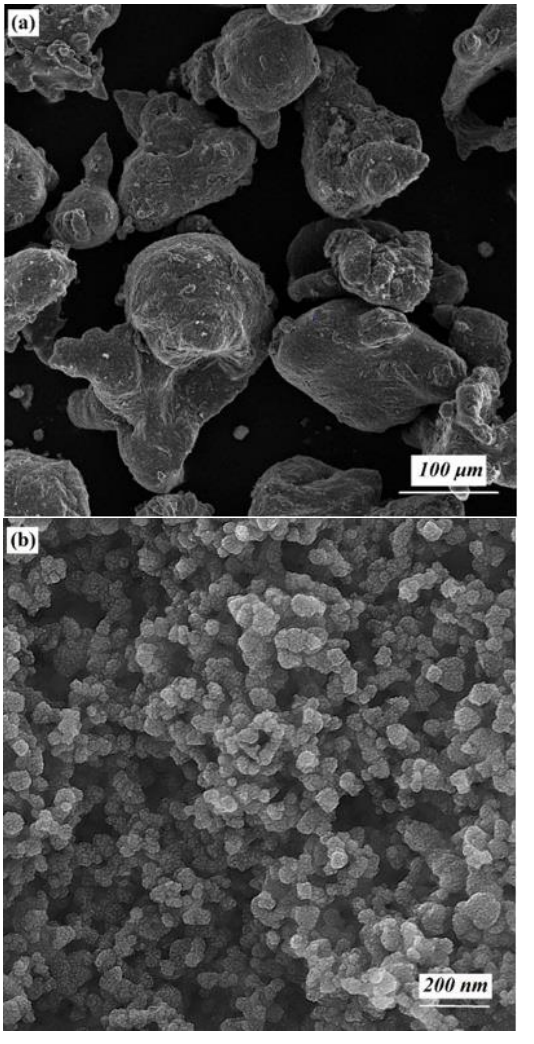


Fig. 1 SEM micrographs of (a) Al, and (b) SiC nano particles (SiC_{np})

The first stage of fabrication of nanocomposite samples is mechanical milling to have a mixture of as-received materials. This process leads to uniform dispersion of the second nano phase between micron-sized aluminium matrix and also grain refinement of

particles. In order to prevent clustering of nano particles, the mixture was suspended in ethanol and exposed to ultrasonic vibration for 30 minutes [2]. After drying, mechanical milling was performed using attrition ball milling device equipped with water cooling system. In order to create inert atmosphere and prevent oxidation of the powder, argon gas with a purity of 99.99% was used. To prevent cold welding during milling, 1 wt.% stearic acid was used as the process control agent (PCA) [9]. The aim of this procedure is grain refinement of Al matrix particles and also achievement to homogenous dispersion of SiC particles in aluminum matrix. [11], [13]. Table 1 shows effective factors in milling process. Time duration of milling was chosen in a way to reduce ductile behaviour of Al particles and make it more brittle and closer to brittle nature of SiC reinforcement [11]. After milling, the powders were degassed at 400°C for 120 minutes in order to remove any remained moisture [13].

Table 1 Milling conditions

Parameter	Value
Ball-to-powder mass ratio	10
Rotational speed (rpm)	360
Time (h)	12
Ball diameter (mm)	10

2.2. Fabrication of nanocomposite samples

Fig. 2 illustrates the drawings and dimensions of cold pressing and hot extrusion die sets. Since the punch and die sets are required to be heated up to 500ºC through the hot extrusion process, hot work tool steel 1.2344 was used for the die and punch material. It should be noted that this kind of steel is appropriate for this purpose because of some its special characteristics such as deep hardening, high toughness and excellent resistance to thermal fatigue cracks.

After mechanical milling, the powder mixture was wrapped using an aluminium foil and then cold pressed under the pressure about 400 MPa [2]. It was found that applying this pressure for some minutes caused to higher relative density and so, the cold pressing pressure was applied about 5 minutes for other samples. This stage causes the entrapped air between the powder particles to be evacuated slowly [2]. The cold pressed samples were then heated to about 500°C at a rate of 10°C per minute. Afterwards, the samples were kept at 500°C for near 30 minutes and were extruded using a 100 tones hydraulic press machine. Extrusion ratio and extrusion rate for fabrication of all samples was adjusted to 8.5:1 and 5 mm/s, respectively [11], [14]. In order to reduce frictional force and improve the extruded samples quality, internal surface of the die must be lubricated. Senthilkumar et al., [12] used zinc stearate as a lubricant between the die wall and punch. It was obtained in current research that graphite base fireproof grease, Molykote brand, had the best result and was used as the lubricant.

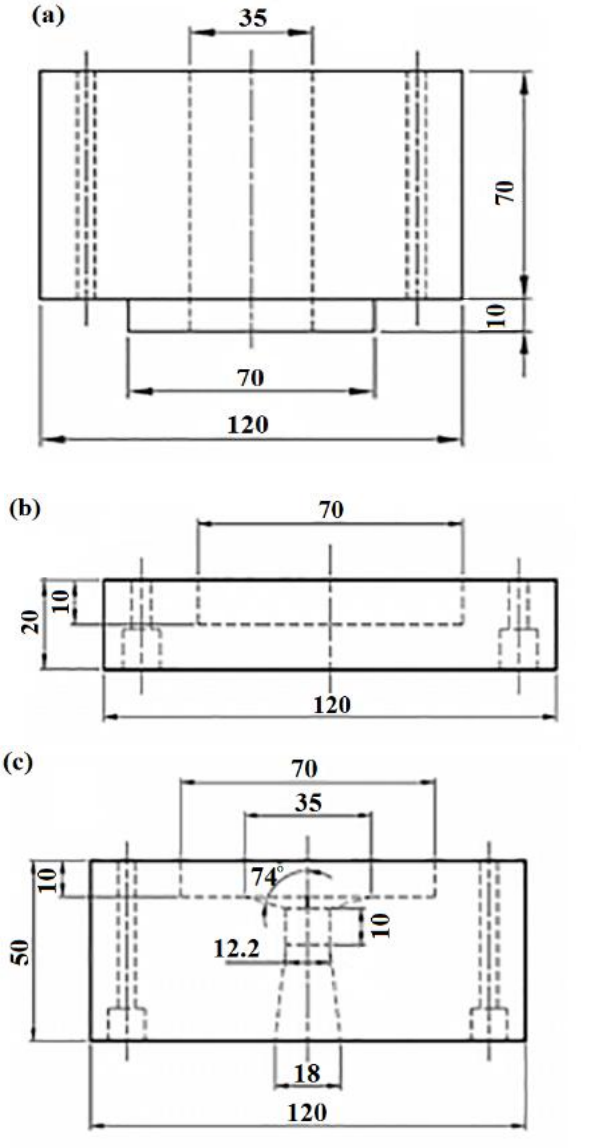


Fig. 2 (a) The common (upper) part of both dies of cold pressing and hot extrusion, (b) cold pressing die, (c) hot extrusion die (all dimension are in millimeter)

The extruded rod-shaped samples with a diameter of 12 mm and different length depending on the amount of initial powder were then successfully fabricated. In this study, the maximum amount of powder for cold pressing was about 70 g which led to the cold pressed disk-shaped samples with a diameter of 35 mm and a height of about 30 mm. These cold pressed samples were converted to rod-shaped samples with a diameter of about 12 mm and a height of about 250 mm after hot extrusion process.

2.2.1. Optimization of fabrication process

One of the most influencing parameters in cold pressing of metal powder is the magnitude and time duration of applied pressure. Applying pressure for an enough time causes the voids and entrapped air to be removed and exited from the powder compact. This leads to samples with the least porosities and the most quality. Fig. 3 shows pressed samples under different processing conditions.

Fig. 3 Cold pressed samples under 400 MPa pressure; (a) without keeping the pressure for specific time duration, (b) keeping the pressure for 5 minutes

Hot extrusion is influenced by several parameters like lubrication, extrusion rate, extrusion ratio, thermal conditions, and extrusion pressure. Generally, lubrication and frictional issues are of great importance in controlling the material flow and also the forming process. Lack of lubrication in hot extrusion process causes the samples to have inappropriate surface quality, as indicated in Fig. 4(a). Under such circumstances, the extruded sample surface is like poplar trees or snake-skin or fish-skin [15]. The main reason of such defects is the severe frictional forces at interface of extruded sample and the die, especially due to the presence of hard ceramic reinforcing particles. Fig. 4(b) shows significant improvement of surface quality after using Molykote lubricants. Efficient Lubrication reduces frictional forces and gives rise to easier material flow and more uniformity of the material structure. Surface cracks are another defect observed in the extruded samples.

Fig. 4 Effect of suitbale lubrication on surface quality of hot extruded Al-3 vol% SiC nanocomposites; (a) Without lubrication, (b) With lubrication

Fig. 5 Bent and defected sample (a), Perfect sample (b)

This deficiency can also be prevented by reducing the die wall friction. Die wall friction can be decreased by a couple of approach of proper lubrication and wrapping the cold pressed billet by an aluminium foil before placing it in the die. Aluminium foil causes to form a uniform layer on the surface of the sample and prevents the formation of deep surface cracks. It is obvious that aluminium foil prevents contacting the ceramic particles to the die internal surface and

therefore reduces the frictional forces. During extrusion process, both the internal and external material rates must be equal. Otherwise, the extruded billet will bend, as depicted in Fig. 5(a). This event is attributed to unequal frictional forces in different segments of the die wall and non-homogeneous lubrication. This fault can also be eliminated by reducing the extrusion rate and applying suitable lubrication (Fig. 5(b)) [9].

2.3. Nanocomposite characterization

Measuring the relative density is a benchmark to evaluate the quality of consolidated PM parts [16]. With this regard, Archimedes method based on ASTM C373-88 standard [17] was used to measure the density and porosity of the samples. For this purpose, extruded sample was placed in an oven for about 60 minutes under 150°C temperatures in order to evacuate any remained moisture. The sample was then dried in a desiccator and its dry weight *(D)* was recorded. Afterwards, the sample was immersed for 5 hours in boiling distilled water and then for 24 hours in distilled water at ambient temperature. The weight of suspended sample in the water *(S)* was calculated. In the last step, the weight of sample *(M)* was recorded. Having this information and using the following formulas, density and porosity of each sample can be obtained.

$$V = M - S \tag{1}$$

$$P = \left[(M - D) / V \right] \times 100 \tag{2}$$

$$B = D / V \tag{3}$$

In this equations V is the apparent volume of the sample, P is the sample porosity, and B is the density of the sample. In order to study the effects of reinforcing phase on the mechanical behaviour of the extruded samples, tensile test and hardness measurements were also carried out. To determine the tensile strength, standard specimens based on ASTM E8 [14] were prepared from extruded samples. Three tests were done for each specimen and the average values of results were reported.

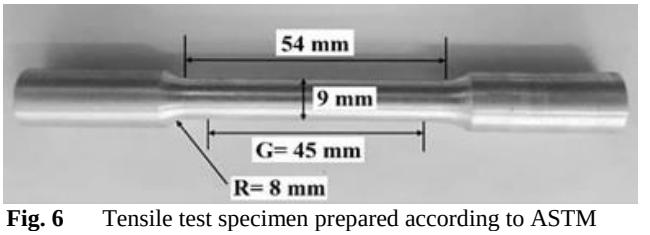
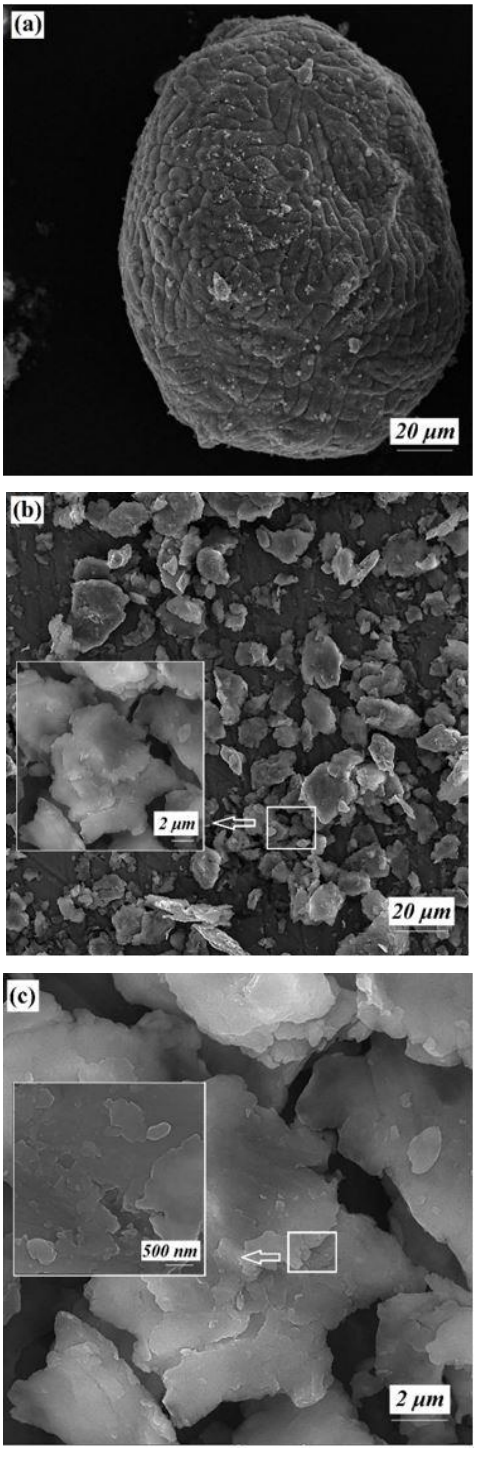


Fig. 6 Tensile test specimen prepared according to ASTM E8

Fig. 6 shows the prepared standard sample for tensile test. The tensile test was carried out under room temperature and loading rate of 3 mm/min (strain rate of about 1×10^{-3} S^{-1}). Vickers micro-hardness test was also done applying 100 grams force to the specimen for 15 seconds using a tetragonal indenter (according to ASTM E384 [2]). The hardness test was performed at least for 6 points on the cross section of each sample. In this research, KOOPA machine MH1 model was used for measuring micro-hardness.



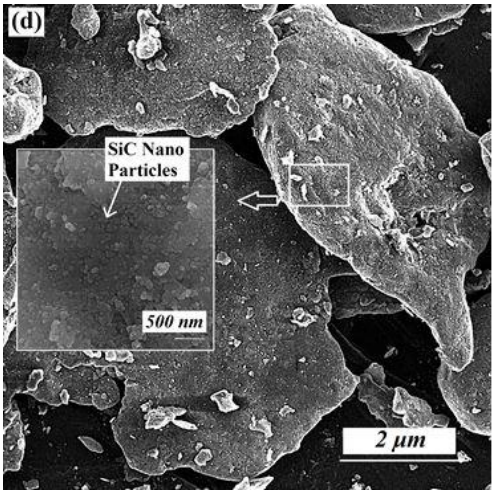


Fig. 7 SEM micrographs of (a) Al, (b) Al after 12 h milling, (c) Al after 12 h milling with higher magnification (d) Al-3 vol% SiC_{np} after 12 h milling

3 RESULTS AND DISCUSSION

3.1. Nanocomposite powder characteristics

Aluminium particles before and after 12 h milling are shown in Fig. 7 (a) and 7 (b), respectively. As these figures suggest, particle size of Al powder has decreased significantly after 12 hours of mechanical milling. Comparing Fig. 7 (c) and 7 (d) also reveals that the SiC nano particles have fully covered the surface of Al particles after 12 h mechanical milling as obtained similarly by Atrian et al., [2]. The XRD patterns of nanocomposite powder with different amounts of SiC reinforcement are shown in Fig. 8. As this figure indicates, the milled powder includes only Al and SiC and no new interaction phases are produced as a result of milling.

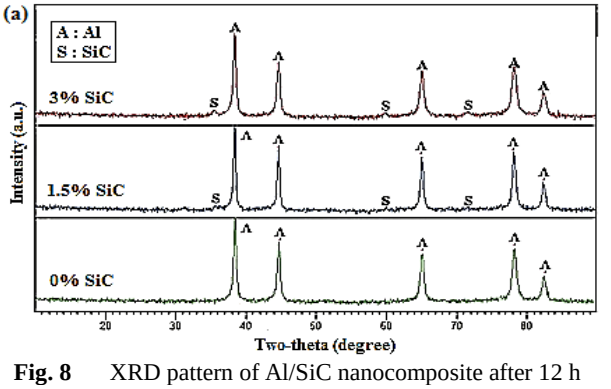


Fig. 8 XRD pattern of Al/SiC nanocomposite after 12 h milling

SEM micrograph of a typical extruded nanocomposite sample and its X-ray map for Si particles are depicted in Fig. 9 (a) and 9 (b), respectively. As these figures demonstrate, uniform dispersion of SiC nano particles over the Al particles is clear. This uniform dispersion of SiC nano particles after 12 h milling was expected and is similar to result obtained by Abdollahi et al. [9]. They reported quite uniformly distribution of B_4C particles in aluminium matrix and also Al particle size reduction after 50 hours of mechanical milling.

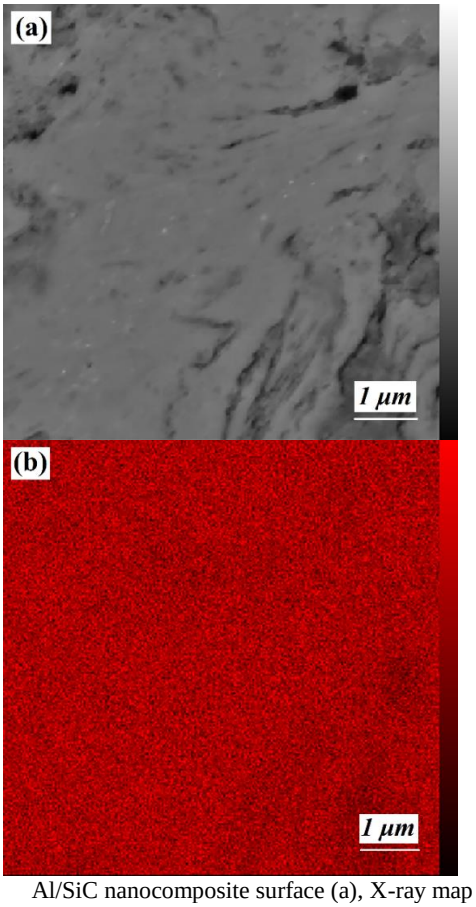


Fig. 9 Al/SiC nanocomposite surface (a), X-ray map of Si particles (b)

3.2. Density

As stated before, density is one of the most important parameter in measuring the quality of consolidation of powder materials. Fig. 10 shows the variations of relative density against volume fraction of SiC. It can be observed that relative density of Al extruded sample reduces slightly after reinforcing with SiC nano particles. The main reason of the density reduction is presence of hard and non-deformable ceramic nano particles between Al particles which hinder more densifications [1], [2]. Mohanty et al., [18] also obtained that the relative density of Al 1100 matrix has been decreased after reinforcing with B_4C particles and reported same reasons. In spite of small reduction of relative density in current research, all the samples have high level of relative density (>99%) with the least amount porosities (Fig. 9 (a)). The high level of

relative density may be attributed to high values of pressure and temperature during hot extrusion process and uniform dispersion of SiC nano particles as well (see Fig. 9 (b) [9], [19]. Uniform distribution of reinforcing phase plays an important role in fabrication of composite components and improvement of their properties. Otherwise, expected improvements cannot be achieved [4].

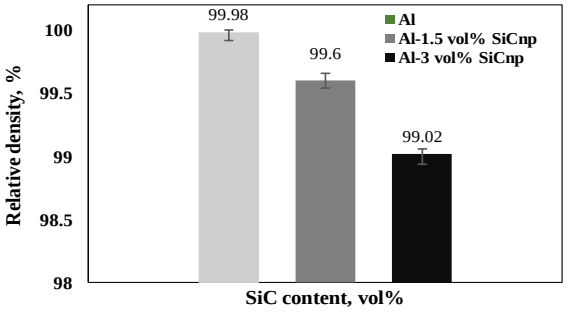


Fig. 10 Variations of relative densities versus SiC content

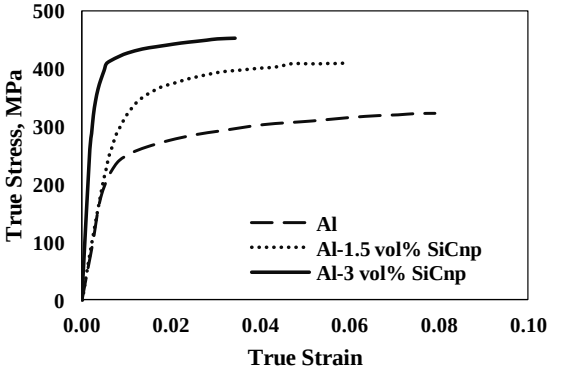


Fig. 11 Effect of SiC nano particles amount on tensile behavior of nanocomposite samples

3.3. Tensile properties

Improvement of tensile strength of Al after reinforcing with SiC nano particles is clearly shown in Fig. 11. Table 2 also depicts the result of these tensile tests. Reinforcing Al with SiC nano particles led to improvement of tensile strength by about 50%, as can be observed in Fig. 11. This enhancement may be attributed to some strengthening mechanisms such as Orowan [2], [9] and thermal mismatch [2], [20]. In Orowan or direct hardening mechanism, the dislocation movement is hindered by Orowan bowing mechanism, as depicted in Fig. 12. The former leads to increase in the dislocation density and consequently accelerating the grain refining progress. Finally, this phenomenon causes to higher flow stress and or higher strength [2]. Onoro et al., [21] could improve tensile strength of Al alloys using B_4C reinforcement. They attributed this improvement to load bearing effects arisen from good bonding between the matrix and the reinforced particles.

Table 2 Variations of yield strength (YS), ultimate strength (UTS), and elongation (ε) of extruded samples in terms of volume fraction of reinforcing phase

SiC (vol%)	YS (MPa)	UTS (MPa)	ε (%)
0	236	320	7.90
1.5	344	410	5.79
3	402	450	3.42

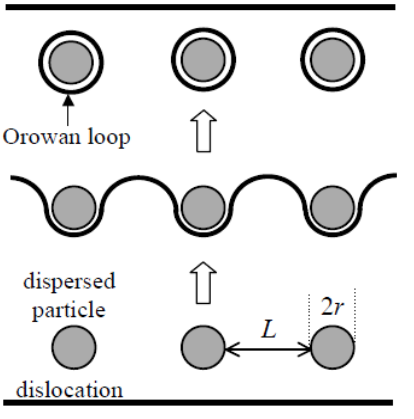


Fig. 12 Orowan mechanism for dispersion hardening [22]

3.4. Micro-hardness

Hardness of aluminium and its alloys are influenced by some factors that affect the movement of dislocations. The most important factors leading to increase hardness are reduction of grain size and formation of inter-metallic compounds. Hardness of mechanically alloyed metal matrix composites is affected by two factors: matrix hardness and the role of second added phase [1], [2], [4], [6]. Work hardening effects of added nano phase and its intrinsic hardness is thought to be the main reasons for the hardness improvement [2]. As can be observed in Fig. 13, it is obvious that the Vickers micro-hardness of reinforced nanocomposite sample is enhanced about 50% with respect to the monolithic material. This finding is consistent with the results obtained by Dong et al., [23] and El-Daly et al., [24]. El-Daly et al., [24] attributed hardness improvement in their Al/SiC nanocomposite samples to grain refinement strengthening mechanism. While, other researchers like Dong et al., [23] and Shirvanimoghaddam et al., [25] reported that improved

hardness is probably due to uniform dispersion of secondary hard phase in Al_2O_3/SiC and Al/B_4C composites, respectively. Behaviour of Vickers micro-hardness in current study is in accordance with variation of yield strength (see table 2). This verifies the Tabor equation that says the material Vickers micro-hardness (HV) (in terms of *Pa*) is near 3 times of its yield strength (YS) and is as follows [2], [26]:

$$YS = HV/3 \tag{4}$$

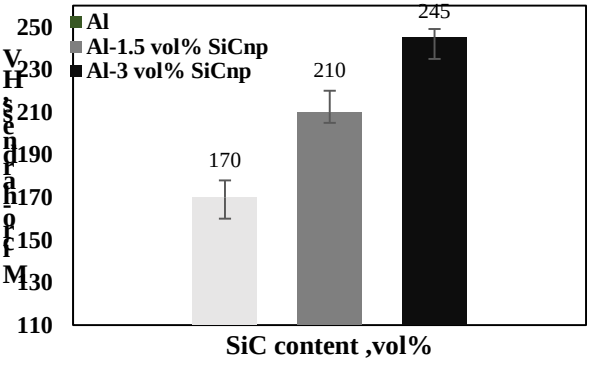


Fig. 13 Variations of Vickers micro-hardness of the cross section surface of extruded samples versus SiC content

4 CONCLUDING REMARKS

The following conclusions may be derived from this research work:

1. Nanocomposite samples with relative density more than 99% are fabricated successfully using mechanical milling, cold pressing, and hot extrusion processes.
2. Relative density of the samples decreases less than 1% as the content of SiC nano reinforcements increases to 3 vol%.
3. Both the tensile strength and Vickers micro-hardness of the samples improve by about 50% after adding 3 vol% SiC reinforcement.
4. The improvement of tensile strength may be attributed to uniform dispersion of SiC nano particles between Al micro particles and also some strengthening mechanisms like the Orowan and load bearing effects.
5. Variation of Vickers micro-hardness agrees well with variation of the yield strength. This agreement verifies also the Tabor equation.

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