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2019 The Authors. In this study a suggested model for flake powder metallurgy were implemented and its mechanism was explained. The suggested model includes dual-speed ball milling (DSBM) to take the advantage of the low-speed and high-speed ball milling (LSBM and HSBM). The modelled process was utilised to uniformly disperse SiC nanoparticles into aluminium metal matrix to produce nanocomposites. The produced mixed powder was hot isostatically pressed. The effects of processing parameters such as stearic acid content, SiC volume content, ball milling speed and time on the microstructure and consequently tensile properties of the manufactured composites have been investigated experimentally to optimise the processing parameters bringing about the enhanced tensile properties of the fabricated composites. The results showed that the implementation of LSBM and HSBM processes can be considered as a unique strategy, i.e. the dual-speed ball milling (DSBM), for uniform dispersion of SiC nanoparticles associated with perfect bonding.

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# Effect of Hot Isostatic Pressing on the Mechanical Properties of Aluminium Metal Matrix Nanocomposites Produced by Dual Speed Ball Milling

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## Abstract

In this study a suggested model for flake powder metallurgy were implemented and its mechanism was explained. The suggested model includes dual-speed ball milling (DSBM) to take the advantage of the low-speed and high-speed ball milling (LSBM and HSBM). The modelled process was utilised to uniformly disperse SiC nanoparticles into aluminium metal matrix to produce nanocomposites. The produced mixed powder was hot isostatically pressed. The effects of processing parameters such as stearic acid content, SiC volume content, ball milling speed and time on the microstructure and consequently tensile properties of the manufactured composites have been investigated experimentally to optimise the processing parameters bringing about the enhanced tensile properties of the fabricated composites. The results showed that the implementation of LSBM and HSBM processes can be considered as a unique strategy, i.e. the dual-speed ball milling (DSBM), for uniform dispersion of SiC nanoparticles associated with perfect bonding.

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**Keywords:** Hot isostatic pressing, Metal matrix composites, Mechanical properties, Powder metallurgy

## **1. Introduction**

Metal matrix composites (MMCs) can be considered as promising candidate to be utilised in aerospace, automotive and defense industries due to their superior tensile properties, high stiffness and good wear resistance [1, 2]. Aluminium metal matrix composites reinforced with ceramic reinforcements can also provide high specific strength, improved wear resistance and low thermal expansion coefficient, making them as exceptional materials for many applications [3-5].

Fabrication routes for manufacturing metal matrix nanocomposites can be classified into two different processing routes including solid-state and liquid-state processes. Liquid-state processing route such as stirring suffers from agglomeration, poor wetting of ceramic nanoparticles with molten metal [6], and chemical interfacial reaction [7], limiting their extensive application in fabrication processes. Solid-state manufacturing processes, however, mainly involve powder metallurgy (PM) process where the matrix powder and reinforcing materials are mixed together in a simple mechanical mixer followed by cold compaction and sintering [8], hot extrusion [9], or spark plasma sintering (SPS) [10] to form a bulk composite. PM process, however, cannot be considered as a perfect process for a uniform dispersion of nanoparticles within matrix under the condition of a noticeable difference between the size of reinforcement and the matrix [11].

More notably, there is a high propensity for nanoparticles to be agglomerated during solid-state process such as PM [12], aggravating the formability of the manufactured composites in the subsequent deformation process [13-15]. This, therefore, makes it necessary to optimise the parameters of the ball milling process to diminish the agglomeration tendency of nanoparticles during PM based process. Different researches have been carried out to optimise the ball milling process by changing the milling parameters such as milling time and speed [16, 17]. For instance, it has been reported that the diminished ball milling speed can result in more uniform distribution of nanoreinforcements such as carbon nanotubes (CNTs) within

aluminium flakes [18, 19]. Moreover, Kai et al [20] verified that the lower ball milling speed can facilitate the formation of flake powder during ball milling process. It has been, also, shown that aluminium flakes produced using ball milling process showed flattened surfaces and large specific surface areas which are compatible to accommodate nanoparticles and promote their uniform dispersion within aluminium matrix [21]. Based on this a new manufacturing route called flake powder metallurgy has been recently invented as a distinctive candidate to uniformly disperse nanoparticles within metal matrices [21]. The mechanism behind formation of the flake layers, however, has not been addressed clearly so far, making it vital to reveal the condition under which this microstructure is prompted during ball milling process. Nevertheless, this uniform distribution is also significantly related to the effect of other processing parameters such as milling time [22].

In this study, therefore, the effect of different milling times, speeds, volume percentages of SiC nanoparticles, and stearic acid on the formation of aluminium flakes will be studied. Additionally, this study aims to present a new model to describe the microstructural evolution of aluminium particles to be transferred into the layer by implementing a new strategy called dual speed ball milling (DSBM) process, including a combination of the low-speed ball milling (LSBM) and high-speed ball milling (HSBM) processes, and changing ball milling time during ball milling process. Furthermore, the effect of hot isostatic pressing on the microstructure and mechanical properties of flake powder will be investigated.

## **2. Manufacturing of Al-SiC Nanocomposite**

SiC nanoparticles with average size of 45 nm were mixed with aluminium powder with particle of average size 60  $\mu\text{m}$ . Three different volume fractions of reinforcement, three different volume fractions of steric acid, ten different milling periods, and low (200) and high (600) rotational speeds were used. The balls to powder ratio were fixed at 20:1. Three different processes including low speed ball milling (LSBM), dual Speed ball milling (DSBM) and high speed ball milling (HSBM) were implemented. Table 1 represents the nomination system used to identify different specimens in the rest of this study.

The samples then were hot isostatically pressed. In hot isostatic pressing the high gas pressure in the press acts uniformly in all direction, which ensures that the pressed products have isotropic properties and a density of 100%. The temperature in the press is usually about 20% lower than the solidus temperature of the alloy being processed, which keeps the alloying elements from being converted into a liquid and prevents the formation of a liquid phase.

Table 1 Nomination system of the powder and bulk specimens produced in this study associated with applied changes in manufacturing parameters.

Brief Name	SiC (Vol. %)	Stearic Acid (Vol. %)	Ball milling speed (RPM)	Ball milling Time (h)
LSBM	1, 3, 6	1, 2, 3	200	1-10
				9(200) +1(600)
				8(200) +2(600)
				7(200) +3(600)
				6(200) +4(600)
				5(200) +5(600)
				4(200) +6(600)
				3(200) +7(600)
				2(200) +8(600)
				1(200) +9(600)
DSBM	1, 3, 6	1, 2, 3	200+600	10(600)
				1-10
				9(200) +1(600)
				8(200) +2(600)
				7(200) +3(600)
				6(200) +4(600)
				5(200) +5(600)
				4(200) +6(600)
				3(200) +7(600)
				2(200) +8(600)
HSBM	1, 3, 6	1, 2, 3	600	1-10
				9(200) +1(600)
				8(200) +2(600)
				7(200) +3(600)
				6(200) +4(600)
				5(200) +5(600)
				4(200) +6(600)
				3(200) +7(600)
				2(200) +8(600)
				1(200) +9(600)

This process was utilised to take the advantage of LSBM process to make a uniform distribution of SiC nanoparticles within aluminium flakes followed by high-speed ball milling (HSBM) to achieve more dispersion of these uniformly distributed SiC nanoparticles into Al flakes due to rapid cold welding, and good bonding between the produced flakes. In this process, the ball milling process was first initiated at a low speed milling condition (200 RPM) and then followed by milling at a higher speed (600 RPM) with total milling time of 10 h.

### 3. Results and Discussions

#### 3.1. Optimisation

Due to the high number of the produced nanocomposites, and in order to select the optimised ball milling conditions such as milling time, milling speed, SiC and stearic acid volume content to achieve higher tensile properties, especially yield strength, aluminium powders that were ball milled with different percentages of SiC and stearic acid volume content under different

milling speeds and times according to Table 1, were hot isostatically pressed to produce bulk composites.

Tensile tests were conducted on the hot isostatically pressed samples and the results of these tests were investigated. It has been found that HDSBM samples, manufactured using hot isostatic pressing of the powders prepared by DSBM process have shown more enhancements in the yield strength compared to other samples. The samples with high yield strength of each group were selected as best candidates to be investigated in this study. These samples with higher yield strength were called as HLSBM (230 MPa), HDSBM (355 MPa), and HHSBM (260 MPa) samples in this study. Table 2 shows the highest tensile achieved for each group associated with the processing parameters.

Table 2 The highest tensile achieved for each group associated with processing parameters

samples	SiC (Vol. %)	Stearic acid (Vol. %)	Ball milling speed (RPM)	Ball milling Time (h)	Tensile strength MPa
HLSBM	1	2	200	2	230
HDSBM	3	2	200+600	8(200) +2(600)	355
HHSBM	6	2	600	2	260

For example, HDSBM sample with SiC content of 3 Vol. % contains 2 Vol. % stearic acid represents more yield strength (355 MPa) compared to other hot isostatically pressed samples. HLSBM and HHSBM samples, in contrast, have a lower yield strength. Regarding HHSBM samples, this lower yield strength can be related to the rapid cold-welding taking place before reaching uniform distribution leading to agglomeration of SiC nanoparticles and consequently creating brittle area. The lower yield strength of the HLSBM samples, however, can be attributed to the lower number of balls to powder collision producing inadequate energy to break the bonds between agglomerated nanoparticles from one side and to bond them to the aluminum particles from the other side. These potential reasons for lower yield strength of the HLSBM and HHSBM samples will be further confirmed in the next sections dealing mainly with microstructural and mechanical analysis of the powder and bulk hot isostatically pressed samples.

Moreover, it can be said there is an optimum value (2 Vol. %) for the volume percentage of the stearic acid in the HDSBM samples, resulting in more enhancements in the yield strength compared to other samples. In fact, this is the critical stearic acid content as it was stated in previous research [22, 23], weakening the agglomeration of aluminium particles during the milling process by obstructing the cold-welding of aluminium particles. In the other words, lower values of stearic acid cannot effectively diminish the agglomeration process of aluminium particles [24], but the higher values (3 Vol. %) can produce the detrimental aluminium carbide ( $Al_4C_3$ ) [25], deteriorating the tensile properties.

### 3.2. Microstructural Analysis

Fig. 1 demonstrates the ball milled composite powders containing 1 vol. % (LSBM), 3 vol. % (DSBM) and 6 vol. % (HSBM) reinforced by SiC nanoparticles (45 nm) after 2 hours of ball milling, respectively.

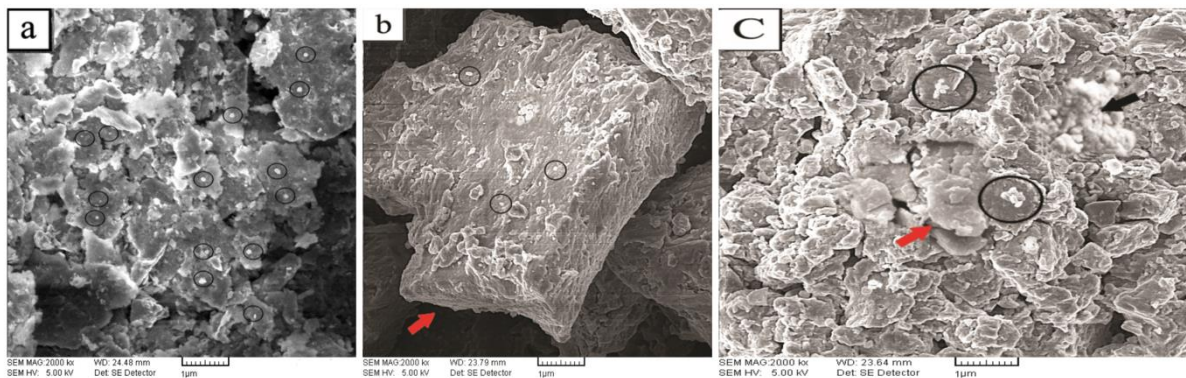


Fig. 1 FE-SEM micrograph of (a)LSBM, (b)DSBM, and (c)HSBM samples.

This figure has shown that the evolution of particles' morphologies in LSBM and HSBM samples are similar with large aluminium particles containing agglomerated SiC nanoparticles. The discrepancy shown in LSBM and HSBM samples is related to the tendency of SiC nanoparticles to be agglomerated in HSBM sample, due to rapid cold welding without achieving uniform distribution assisted by higher SiC content in this sample. As shown in **Error! Reference source not found.**(c) by black circles, there is some evidence of formation of SiC nanoparticles with clustered morphology. Moreover, there is an evidence for formation of large cluster, as shown by the black arrow in **Error! Reference source not found.**(c), which is not seen in other samples.



As can be seen in **Error! Reference source not found.** (b), aluminium matrix in the DSBM sample has been formed with a flake appearance while SiC nanoparticles settled on the surface of these flakes, as shown by black circles confirming the lower possibility of formation of SiC nanoparticles with cluster arrangement in this sample compared to the other samples. As demonstrated, the SiC nanoparticles in DSBM sample are mostly distributed uniformly on the surface of aluminium flakes. These results verify the fact that the ball milling of aluminium matrix in the initial time can be accomplished by the shearing forces, but the effectiveness of this shearing force to produce aluminium flakes significantly depends on the loading content of SiC nanoparticles. In fact, it is postulated that there is a critical amount of SiC nanoparticles, i.e. 3 Vol. %, which can accelerate the shearing of aluminium particles to form flake morphology during ball milling process. However, the effect of this critical amount cannot be isolated from other parameters. In fact, it is correlated with the employment of dual speed ball milling. This is because shifting to higher speed results in stronger ball collision accelerating cold welding of Al flakes and leading to more dispersion of the previous uniformly distributed SiC nanoparticles with better bonding.

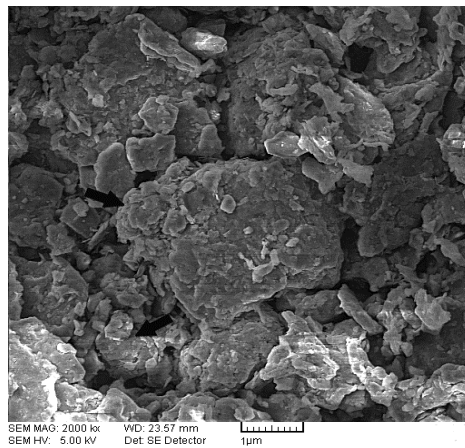


Fig. 2 FE-SEM micrograph of DSBM powder sample ball milled for 6 h at high speed regime.

Nevertheless, prolonged ball milling of DSBM sample in the milling times longer than the optimum time of 2 h after shifting to high-speed regime can result in formation of big aluminium particles with agglomerated morphology, as shown by black arrows in Fig. 2. This is due to the fact that after a length of time during HSBM, detaching of SiC nanoparticles from Al matrix could take place leading to re-agglomeration of these SiC nanoparticles.

From microstructural point of view it is good idea to have insight into the effect of hot isostatic pressing on the microstructural properties of the produced composites, microstructure of the hot isostatically pressed samples was investigated using FE-SEM as shown in Fig. 3.

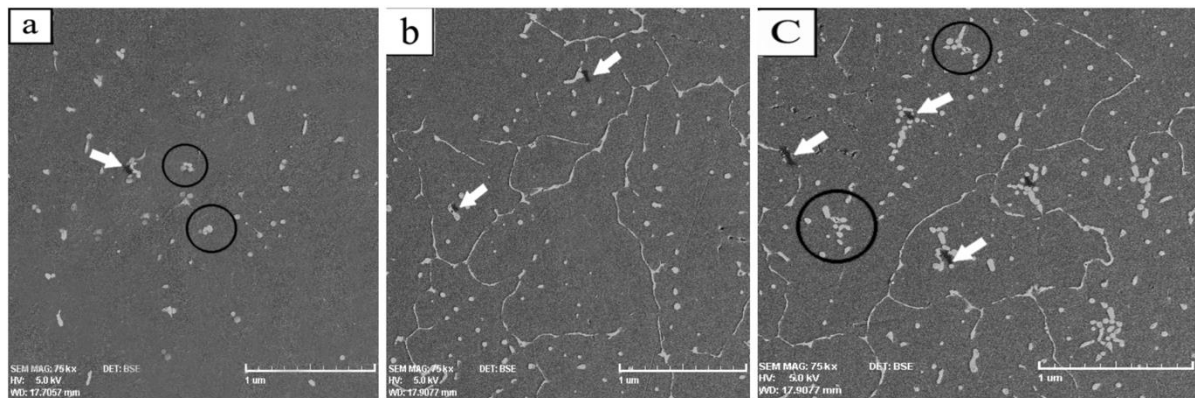


Fig. 3 FE-SEM micrograph of (a) HLSBM, (b) HDSBM and (c) HHSBM samples after hot isostatic pressing at 530 °C.

Analysing the microstructure of the hot isostatically pressed samples using image tool software has shown that SiC volume fractions of HLSBM, HDSBM and HHSBM samples are around 0.93 vol. %, 2.95 vol. % and 5.85 vol. %, respectively, demonstrating the effectiveness of the hot isostatic pressing process on uniform distribution of SiC nanoparticles.

There are two major discrepancies making the microstructure of the HDSBM sample (Fig. 3 (b)) different from the ones seen for the HLSBM (Fig. 3 (a)) and HHSBM samples (Fig. 3 (c)). Firstly, the SiC nanoparticles have been uniformly distributed within the aluminium matrix in the HDSBM sample, but they tend to be agglomerated in the HHSBM sample (black circles in Fig. 3 (c)). This agglomeration behavior of SiC nanoparticles can also be seen in HLSBM sample, but it is less severe in this sample due to the lower SiC content of this sample compared to the HHSBM sample. Secondly, formation of microvoids in HDSBM sample is not predominant, but HHSBM sample suffers from large amount of microvoids formed in the aluminium matrix (white arrows in Fig. 3 (c)) in the vicinity of agglomerated SiC nanoparticles. The severity of microvoids formation in HLSBM sample, however, is less considerable, as

shown by white arrows in Fig. 3 (a) compared to the HHSBM sample, related to the lower SiC content in this sample compared to other hot isostatically pressed samples.

### 3.3. Process Mechanism

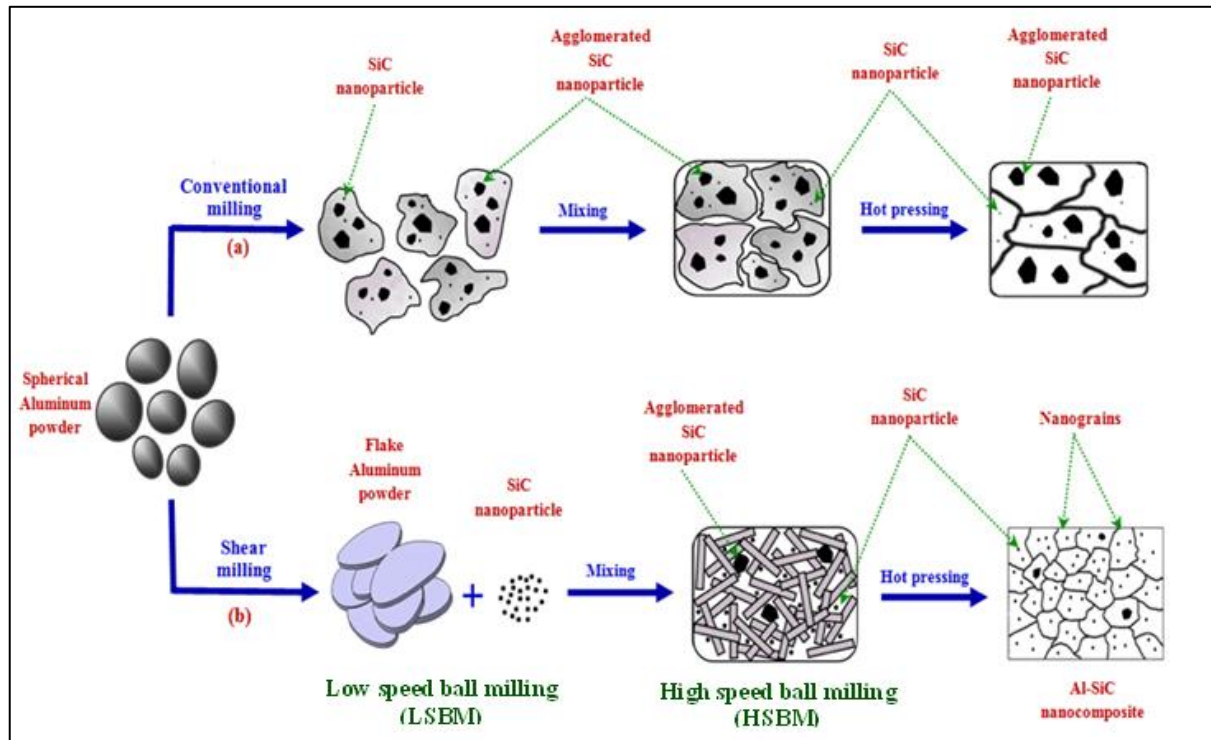


Fig. 4 Schematic demonstration of the fabrication process of aluminium matrix composite reinforced with SiC nanoparticles using two different approaches of (a) conventional milling and (b) shear milling.

Fig. 4 represents the mechanism suggested in this study for formation of different morphologies shown in the composite powders studied in Fig. 1. As shown in Fig. 4 (a), conventional milling process such as LSBM and HSBM resulted in formation of large aluminium particles due to cold welding [26] with agglomerated SiC nanoparticles, as seen in Fig. 1 (a) and (c). This, however, is in contrast with the structure seen in the shear milling process (Fig. 4 (b)). This can be related to implementing the combination of LSBM and HSBM processes, prompting shear milling process. Dual-speed ball milling (DSBM) process includes two major steps encompassing low-speed ball milling (LSBM) followed by the high-speed ball

milling (HSBM). LSBM process has the capability to make the globular aluminium powders more flattened and distribute the SiC nanoparticles more evenly between these aluminium layers. These flattened aluminium powder can play a building block role in the next step of the process (HSBM) and be rapidly welded together to form large aluminium particles with stronger dispersion of SiC nanoparticles between welded aluminum flakes.

Indeed, this study demonstrates that SiC nanoparticles and low-speed ball milling have a significant effect on the shearing ability of ball milling process to produce aluminium flakes by which SiC nanoparticles are also trapped during the milling process, thereby prompting uniform distribution of these particles in the produced composite powders. In general, ball milling process has the capability to diminish the size of the aluminium particles, and this can be accelerated by adding SiC nanoparticles as reinforcements. In fact, as shown in Fig. 4, formation of flake aluminium layer is responsible for uniform distribution of SiC during shear milling, i.e. milling at low speed ball milling, while shifting to higher speed lead to confinement of SiC nanoparticles. On the other hand, conventional milling normally, conducted at constant speed ball milling, is resulted in formation of large aluminium particles containing agglomerated SiC nanoparticles.

### **3.4. XRD Analysis**

Fig. 5 shows the X-ray diffraction (XRD) spectra of the LSBM, DSBM and HSBM samples in addition to the as-received pure aluminium powder for comparison. It can be seen from Fig. 5 that there is no extra peak in the XRD pattern of aluminium powder used in this study, attributed to the formation of solid solution of most elements within aluminium powder. As shown in Fig. 5, incorporation of SiC nanoparticles within aluminium powder has resulted in broadening of the aluminium peaks in LSBM, DSBM and HSBM samples. This broadening can be ascribed to the accumulative effects of ball milling and SiC nanoparticles on diminishing the crystallite size and enhancing the lattice strain of the aluminium matrix.

The investigation of the XRD peaks shown in Fig. 5, approves that there is no detected peak representing formation of the aluminium carbides ( $Al_4C_3$ ) in the different samples. This can be ascribed to the manufacturing process using ball milling which does not involve formation of any liquid phase during processing [10], indicating formation of small percentage of  $Al_4C_3$  which cannot be characterised by the XRD or may be either no formation at all.

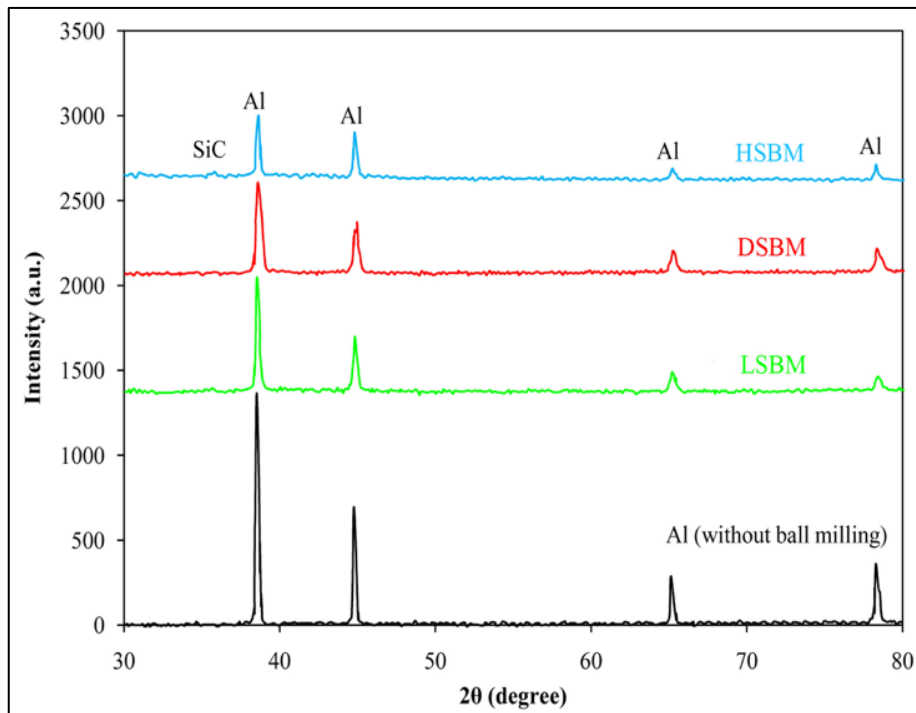


Fig. 5 X-ray diffraction pattern of aluminium powder without application of ball milling associated with patterns of the LSBM, DSBM and HSBM samples.

Another discrepancy between the XRD patterns of the produced composite powder is related to the formation of XRD peak of SiC nanoparticles only in HSBM sample. This can be attributed to the fact that most SiC nanoparticles in HSBM sample are agglomerated as indicated by black circles in Fig. 1(c), attributed to the higher SiC content in this sample. It should also be noted that the elimination of the SiC peak in LSBM sample can be related to the lower content of SiC nanoparticles, making it difficult for XRD spectroscopy to detect these particles. The absence of this peak in DSBM sample, however, is ascribed to the low-sized crystallite formed in this sample, resulting in broadening of the SiC peak in the XRD pattern of this sample.

To go further with XRD analysis, Fig. 6 demonstrates the X-ray diffraction pattern of composites powder hot isostatically pressed at 530 °C. It can be seen that there is no evidence for formation of  $Al_4C_3$  in the produced composites. Additionally, it can be seen that the hot isostatic pressing of the HDSBM powder has resulted in more broadening of the aluminium peaks compared to other hot isostatically pressed samples. This can be related to the fact that

SiC nanoparticles have a more uniform distribution in HDSBM sample, restricting the grain growth during hot isostatic pressing process. SiC nanoparticles in HLSBM and HHSBM samples, however, are formed with agglomeration structure, diminishing their capability in restricting the grain growth of aluminium matrix during hot isostatic pressing process. Furthermore, as shown in Fig. 6, broadening of the XRD peaks of SiC nanoparticles in HDSBM sample is more predominant compared to HLSBM and HHSBM samples. This substantiates the formation of SiC nanoparticles without any severe agglomeration such as the ones seen in the HLSBM and HHSBM samples, resulting in broadening of the XRD peaks of SiC nanoparticles in these samples.

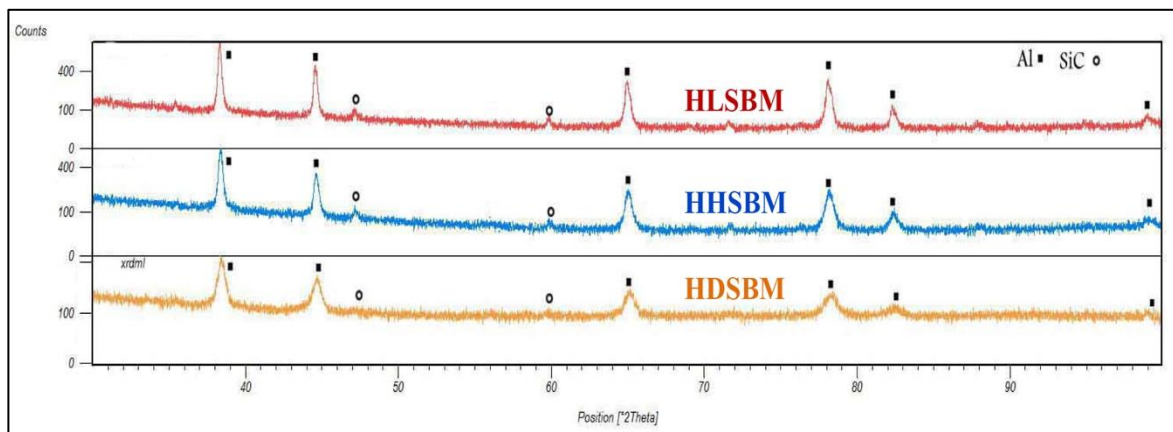


Fig. 6 X-ray diffraction pattern of HLSBM, HDSBM and HHSBM samples hot isostatically pressed at 530 °C.

### 3.5. Crystallite Size and Lattice Strain

Fig. 7 represents the changes of crystallite size for LSBM, DSBM and HSBM powder samples as a function of the milling times to demonstrate the effect of ball milling on the crystallite size of these samples. Time of ball milling for DSBM sample is related to the time of ball milling at the high-speed regime.

As shown in Fig. 7, the ball milling process has resulted in diminishing the crystallite size of ball milled samples, but the degree of this reduction is more obvious in DSBM samples compared with other samples. In fact, these samples have shown reduction of crystallite size to the values lower than 100 nm after 2 h of shifting to high speed attributed to the effect of

milling process on producing aluminium flakes and confining SiC nanoparticles between the produced flakes.

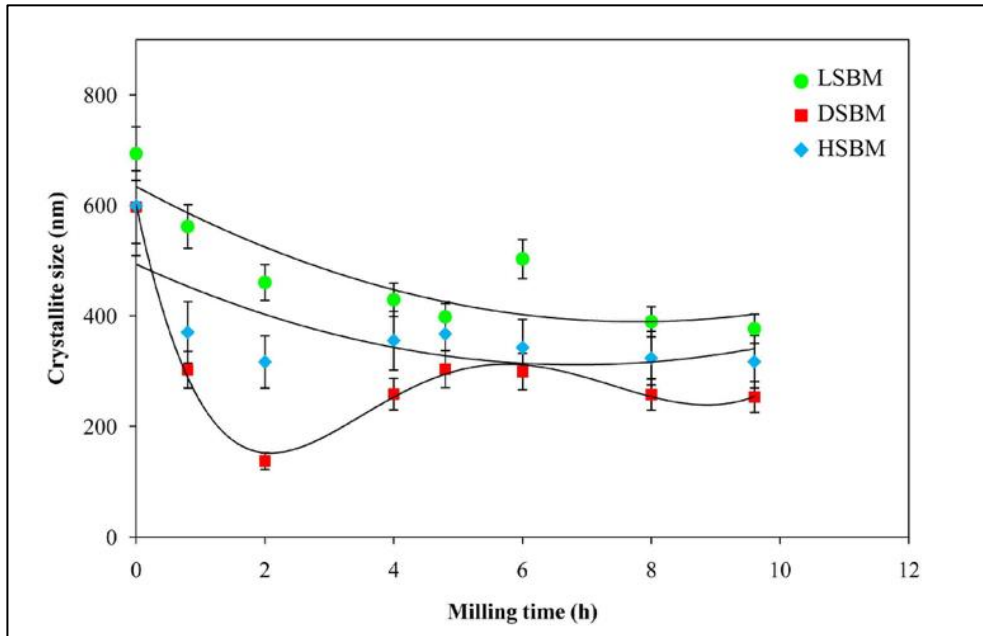


Fig. 7 Effect of different milling times on the crystallite size of LSBM, DSBM and HSBM powder samples.

As shown in Fig. 7, LSBM and HSBM samples, however, have demonstrated a moderate decline in the crystallite size over the different times of the ball milling, which is more significant for HSBM samples compared to the LSBM samples. This can be attributed to the higher volume content of SiC nanoparticles in HSBM samples, which is more predominant at the beginning of milling process.

As can be seen from Fig. 7, although a significant drop in the crystallite size of aluminium matrix in DSBM sample has been achieved after 2 h of shifting to high-speed ball milling, extended time of high-speed ball milling in this sample has not diminished the crystallite size to the values lower than 100 nm. This can be attributed to the fact that the after a length of time during HSBM, the flake shapes of aluminium matrix obtained in the initial times of the milling process can be lost, in addition to the possibility of detaching SiC nanoparticles from Al matrix leading to re-agglomeration of these SiC nanoparticles.

Fig. 8 represents the variation of the lattice strain of composites powder as a function of the milling time. It is obvious that the ball milling process has increased the strain stored in the grains of the aluminium matrix to values around 0.6 for the LSBM and DSBM samples after 2 h of milling, but this enhancement is more significant for HSBM sample, registering value around 1.2. This enhancement in the strain of the grains can be related to the fact that the mechanical alloying can produce nonuniform strain within the grains during milling process, as a result of the plastic strains accumulated within the microstructure.

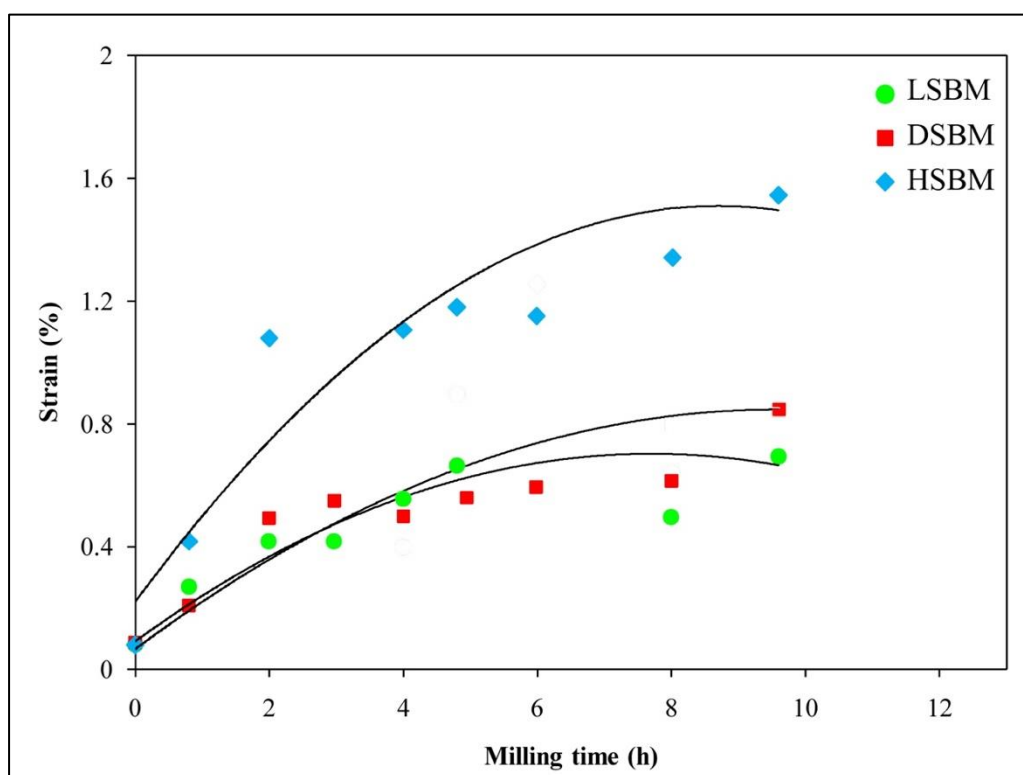


Fig. 8 Effect of different milling times on the strain stored in the aluminium powders during different ball milling processes.

It can be seen from Figs. 7 and 8 that the crystallite size and lattice strain of the powder sample is close to the steady state after 2 h of ball milling, and there are no considerable changes in the lattice strain and crystallite size of the powder samples. This can be attributed to the fact that after initial time of the milling the rate of grain refining and particle fracturing are going to be levelled off to values that are very close to the rate of the grain growth and recovery.



Indeed, by increasing milling time, the average of crystal size decreases (Fig. 7), and the maximum lattice strain increases (Fig. 8). It is believed that in the mechanical milling process, the hit applied by the milling balls to the powder causes plastic deformation and consequently enhances the strain stored in the grains.

It has been approved that the reduction of the crystallite size of the alloys during ball milling is a process of three different stages [27]. The first stage can be defined due to the formation of shear bands accompanied by high density of dislocations. The process then proceeds by annihilation and recombination of these dislocations, bringing about generation of grain boundaries specified by their small angle resulting in formation of grains with small size. The generated small grains are finally subjected to random orientation compared to their adjacent grains, resulting in formation of crystallite with small size represented by broadening of diffraction peaks in the XRD pattern.

According to the previous study conducted by Witkin and Lavernia [28] in the explanation mechanisms involved in the formation of the nanostructure during mechanical alloying, it has been demonstrated that shear bands were created in the initial step of mechanical alloying due to large plastic deformation. Fig. 9 represents the schematic view of the mechanism working during ball milling to produce nano-sized grains.

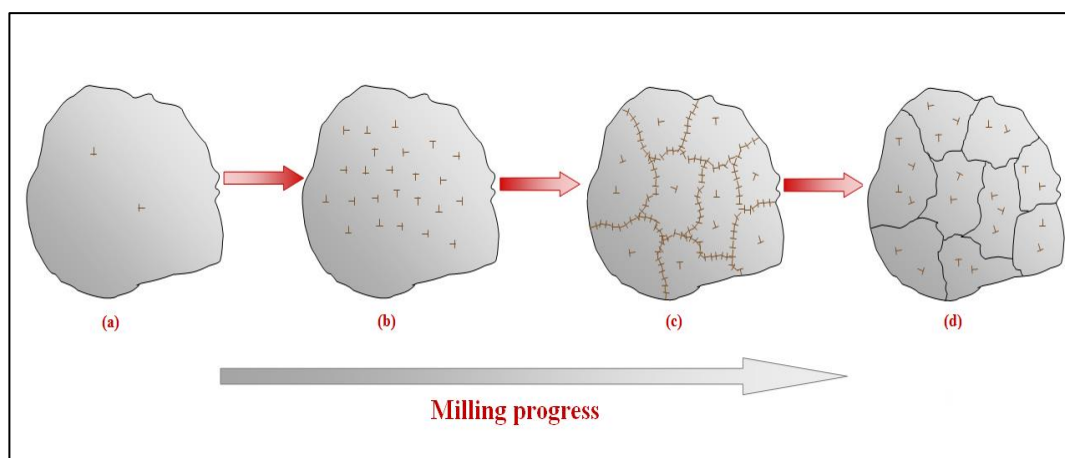


Fig. 9 Schematic picture of the process for formation of nano-sized- grains during ball milling process.

As shown in Fig. 9 (a), the created shear bands contain high density of dislocations with 0.5 to 1 micrometer of width. As the ball milling process proceeds, the strain of crystal lattice increases based on increasing the milling time, resulting in enhancing the dislocation density, as shown in Fig. 9 (b). The created dislocations are combined in high strain areas which can be converted into low angle boundaries, as can be seen in Fig. 9 (c).

It has shown by many researchers that increasing milling time reduces the crystallite size [28, 29] and the lattice strain diminishes gradually in the extended times of ball milling process. The mechanism expressed for this phenomenon is reduction of the crystal size while the ball milling process proceeds, and when the reduction of crystal size reaches to the saturation point, the density of dislocations cannot be increased using the impact imposed by the milling balls. In this step, dislocations can be sorted and some of them disappeared. Generally, the milling process has three phenomena, hardening due to create dislocations, recovery due to rearrangement of the dislocations and annihilation due to the heat generated during milling process. Accordingly, the activation energy for recovery is increased in the progress of the milling process and the rates of these two phenomena equalise. It should be noted that the increase of the temperature is also an important factor on the reduction of hardening phenomenon and reduction of lattice strain in the final steps of milling process, resulting in manifestation of the recovery processes due to increasing the temperature.

Fig. 10 shows changes of crystallite size for samples hot isostatically pressed at 530 °C, as a function of the milling time. Fig. 10 has been depicted by investigation of the results achieved from implementing the XRD analysis on the bulk samples hot isostatically pressed at different milling times. It can be clearly affirmed the effectiveness of flake powder morphology prompted by DSBM process in keeping the crystallite size of aluminium matrix in the nano scale regime compared to other samples such as HLSBM and HHSBM samples, suffering from abnormal grain growth.

As can be seen from Fig. 10, the hot isostatic pressing process has resulted in enhancing the crystallite size of samples, but the degree of this enhancement is more considerable in HLSBM and HHSBM samples compared with other HDSBM samples. Having considered the variation

of crystallite size for different ball milling times presented in Fig. 7, it can be concluded that the hot isostatic pressing process has increased the crystallite size of the powder samples from the value lower than 600 nm to the values higher than 600 nm for HLSBM and HHSBM samples.

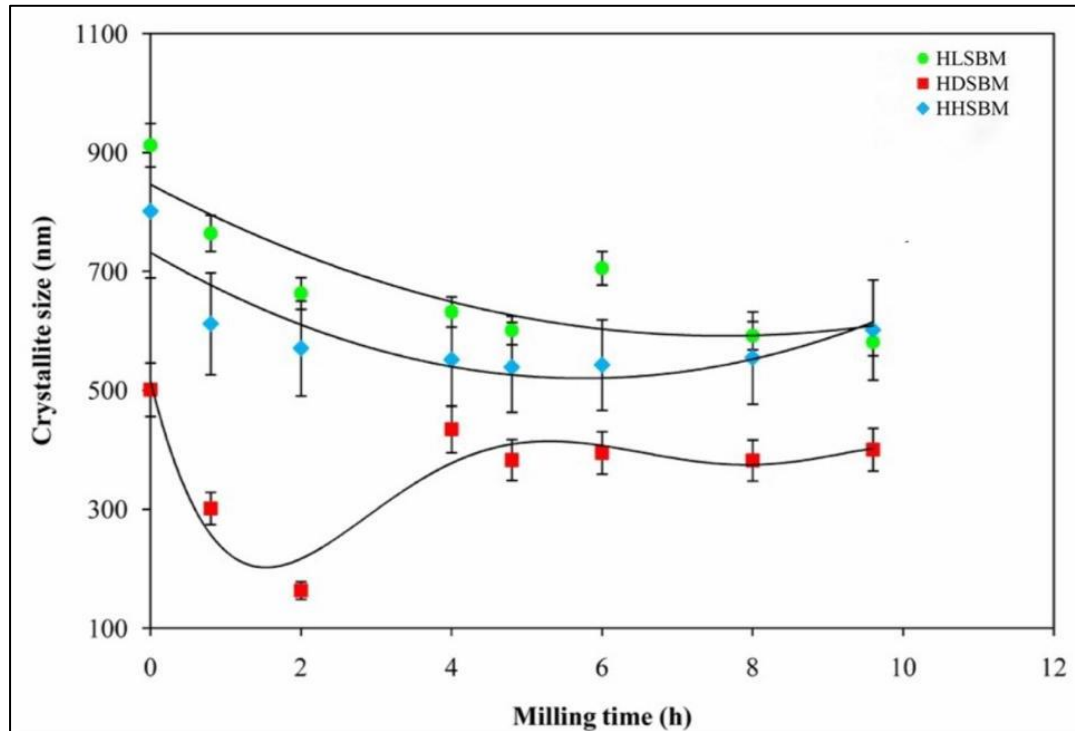


Fig. 10 Effect of hot isostatic pressing on the crystallite size of powder samples ball milled at different ball milling times and hot pressed at 530 °C.

Regarding HLSBM sample, the main reason behind this could be the lower SiC content in the HLSBM sample, imposing lower resection on the grain growth during hot isostatic pressing. HHSBM sample, however, suffers from abnormal grain growth due to the high propensity of agglomeration of SiC nanoparticles in this sample, attributed to the higher volume content of these particles. HDSBM sample, however, has shown a different behavior, as the crystallite size of all powder samples after hot isostatic pressing is lower than 500 nm, which contrasts with the trend observed for HLSBM and HHSBM samples. This behavior can be ascribed to the uniform distribution of SiC nanoparticles in the HDSBM sample compared to agglomeration morphology predominant in HHSBM sample. More importantly, the maximum diminution in crystallite size observed for LSBM sample as shown in Fig. 7. In fact, the extended times of ball milling will result in deteriorating the flake morphology of aluminium

powder, alleviating their capacity to confine the SiC nanoparticles by the mechanism demonstrated in Fig. 4.

### 3.6. Relative Density

To quantify the effect of hot isostatic pressing on the porosity content of the produced composites, the relative density of the samples hot isostatically pressed at 530 °C was calculated and compared with powder samples before implementation of ball milling process, i.e. mixed samples, as shown in Fig. 11. It can be seen that increasing the SiC volume content results in diminishing the density of the produced composites. This is ascribed to the higher resistance of these particles to deformation, restricting the amount of deformation and consequently reducing the density.

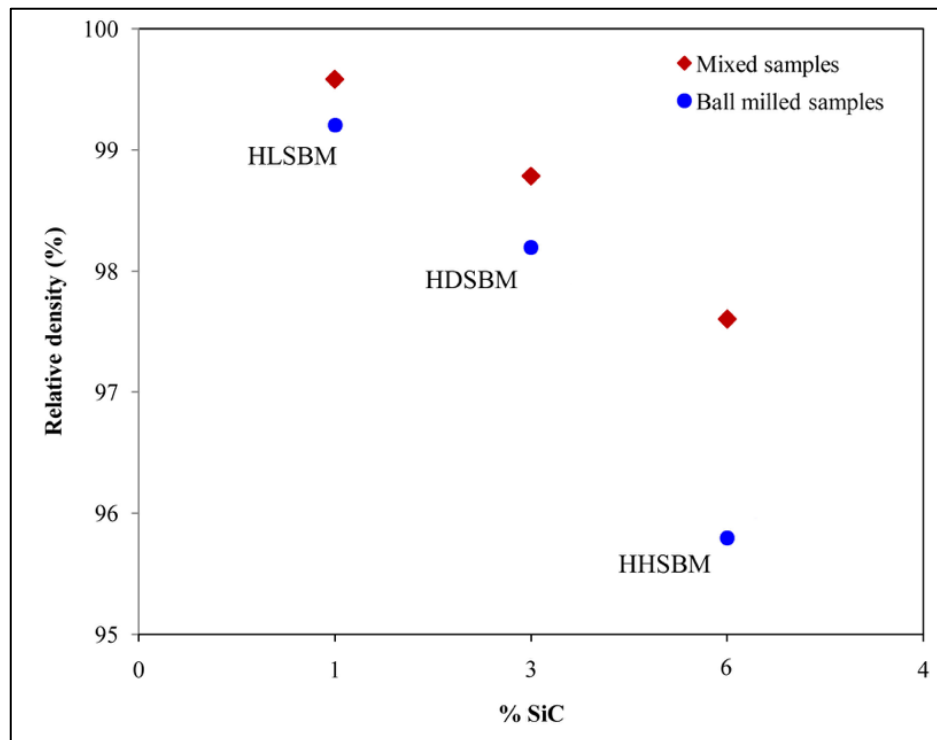


Fig. 11 Effect of content of SiC nanoparticles on the relative density of the hot isostatically pressed samples at 530 °C for powder samples which are not ball milled and those which are ball milled before hot isostatic pressing.

As demonstrated in Fig. 11, the density of the samples produced by hot isostatic pressing of the ball milled powder, when the SiC volume content is the same, is lower than those produced

using powder which is not ball milled, i.e. mixed powder. This can be attributed to the parameters controlling the final density of the hot isostatically pressed samples such as resistance in front of deformation, sintering deriving force, crystallite size, strain stored in the powder and the morphology of the powder. It has been approved before that the ball milled powder containing high density of defects, lower crystallite size and higher stored strain resulted in enhancing the sintering rate [30, 31]. The higher strain hardening of these powders, on the other hand, can enhance the resistance to deformation bringing about diminishing the density of the powder after hot isostatic pressing [32], as shown in Fig. 11. The competition between these parameters is the one determining the final density of the hot isostatically pressed samples. However, it seems that in the hot isostatically pressed samples produced from ball milled powder, there is a high amount of strain hardening, confirmed by high strain energy stored in the crystallites as shown in Fig. 8, enhancing the resistance to the deformation. So, the higher sintering rate of these powders cannot compensate this enhanced resistance to the deformation, thereby diminishing the final density of the hot isostatically pressed sample produced by composite powders compared to those produced using the mixed ones.

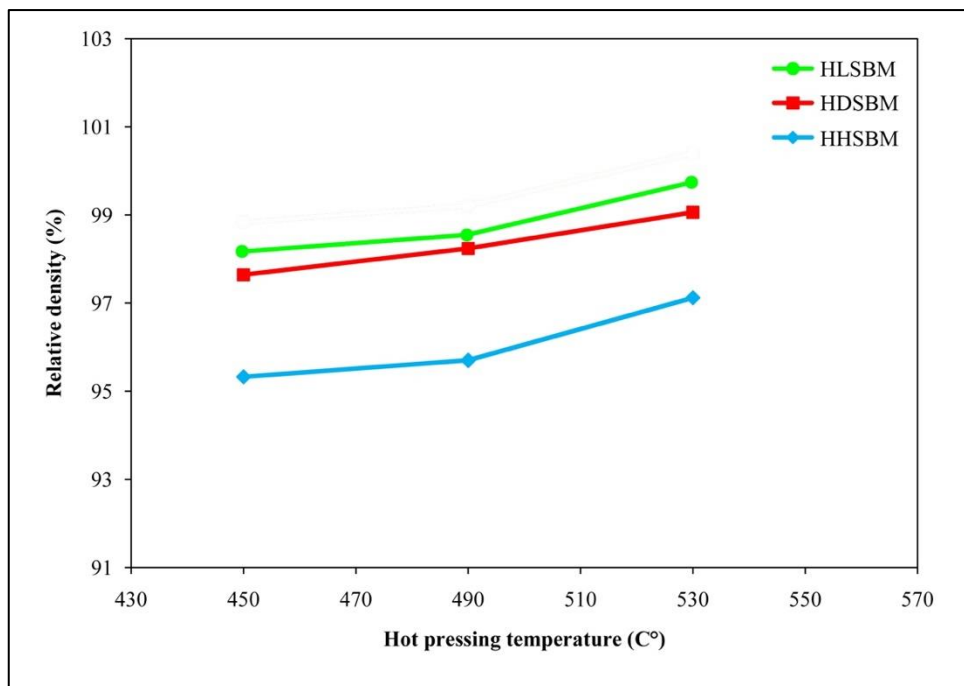


Fig. 12 Effect of content of SiC nanoparticles and hot isostatic pressing temperature on the relative density of the samples hot isostatically pressed at different hot isostatic pressing temperatures.

Fig. 12 represents the relative density of samples hot isostatically pressed at different temperatures to show the effect of hot isostatic pressing temperature on the density of produced composites. It is obvious that increasing the hot isostatic pressing temperature can enhance the density of samples, but as shown in Fig. 12, increasing the SiC volume content diminishes the density of samples attributed to the resistance of these particles to the deformation force.

As shown in Fig. 12, HLSBM sample has a higher density for samples hot isostatically pressed at different hot isostatic pressing temperatures, ascribed to the lower SiC content of this sample compared to other samples. HHSBM sample, however, shows lower density at different hot isostatic pressing temperatures due to the higher SiC content of this sample, resulting in formation of agglomerated SiC nanoparticles. These agglomerated SiC nanoparticles contain microvoids, as shown in Fig. 3, which cannot be filled easily during hot isostatic pressing treatment, thereby diminishing the density of this sample. HDSBM sample, on the other hand, represents unique densification behavior during hot isostatic pressing treatment, which is similar to the behavior shown in the HLSBM sample.

In fact, HDSBM sample has about 3 Vol. % SiC nanoparticles and it is expected that there is a big divergence between the density of this sample and the one seen for HLSBM sample. However, as shown in Fig. 12, the HDSBM hot isostatically pressed at 530 °C demonstrates a high relative density of about 99%, attributed to the diminished agglomeration of SiC nanoparticles in this sample.

### **3.7. Mechanical Properties**

According to Fig. 13, the yield strength (355 MPa) and UTS (430 MPa) of HDSBM sample are also significantly higher than HHSBM sample by 37% and 20%, respectively. This is attributed to the uniform distribution of SiC nanoparticles and consequently higher relative density of this sample (99%) compared to the HHSBM sample (97%) hot isostatically pressed at 530 °C.

Indeed, this can facilitate higher strain hardening within HDSBM sample. The uniformly dispersed SiC nanoparticles can also block the dislocation movement within the aluminium

grains, thereby diminishing the dislocation density at grain boundaries resulted in higher ductility.

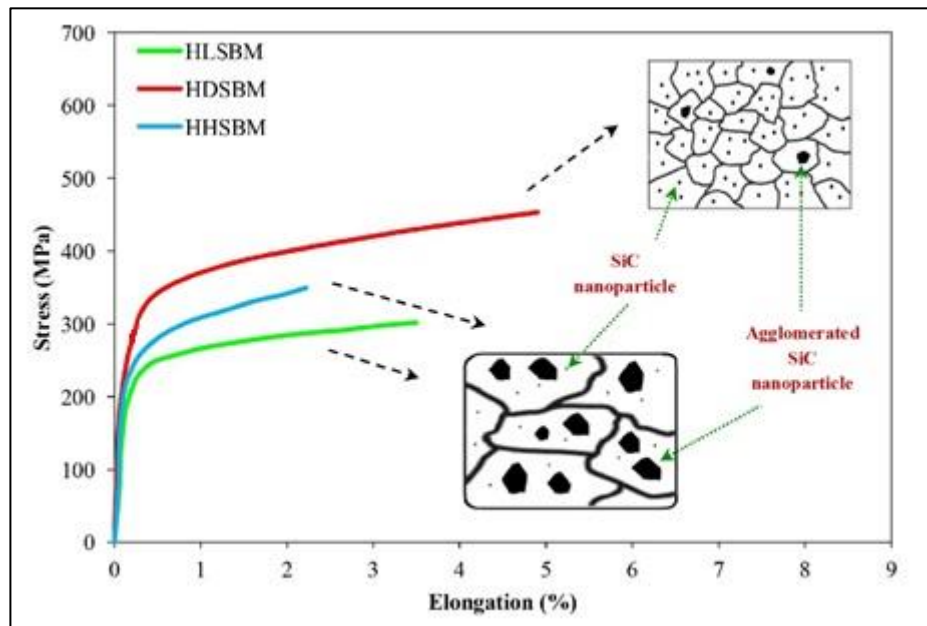


Fig. 13 Stress-strain curve of HLSBM, HDSBM and HHSBM samples hot isostatically pressed at 530 °C samples accompanied with schematic illustration of their structures.

Fig. 14 represents the microhardness of hot isostatically pressed samples associated with pure aluminium hot isostatically pressed under the same condition. As shown in Fig. 14, although the unreinforced aluminium hot isostatically pressed at the same hot isostatic pressing conditions that other SiC reinforced aluminium samples subjected to the hot isostatic pressing, it demonstrates the hardness is lower than other composites. This higher hardness of other samples can be attributed to the higher possibility of enhancement in the crystallite size of unreinforced aluminium at high hot isostatic pressing temperature like 530 °C used in this study. As shown in Fig. 14, incorporation of SiC nanoparticles within aluminium matrix can enhance the hardness of the produced composites, attributed to the higher density of dislocation produced within aluminium matrix as a result of these particles. It can also be seen in Fig. 14, that the hardness of HDSBM sample is around 114 HV which is higher than that of HHSBM sample showing hardness value around 94 HV, which is not in an agreement with the fact that adding the SiC content of the composites can enhance the hardness. This can be attributed to the fact that uniform distribution of SiC nanoparticles plays more profound role in enhancing the hardness of the produced composites compared to the enhancing the SiC content of the

composites. This, in turn, is related to the enhanced dislocation density of the HDSBM sample compared to the HHSBM sample due to the lower crystallite size of this sample compared to the HHSBM sample, as shown in Fig. 10.

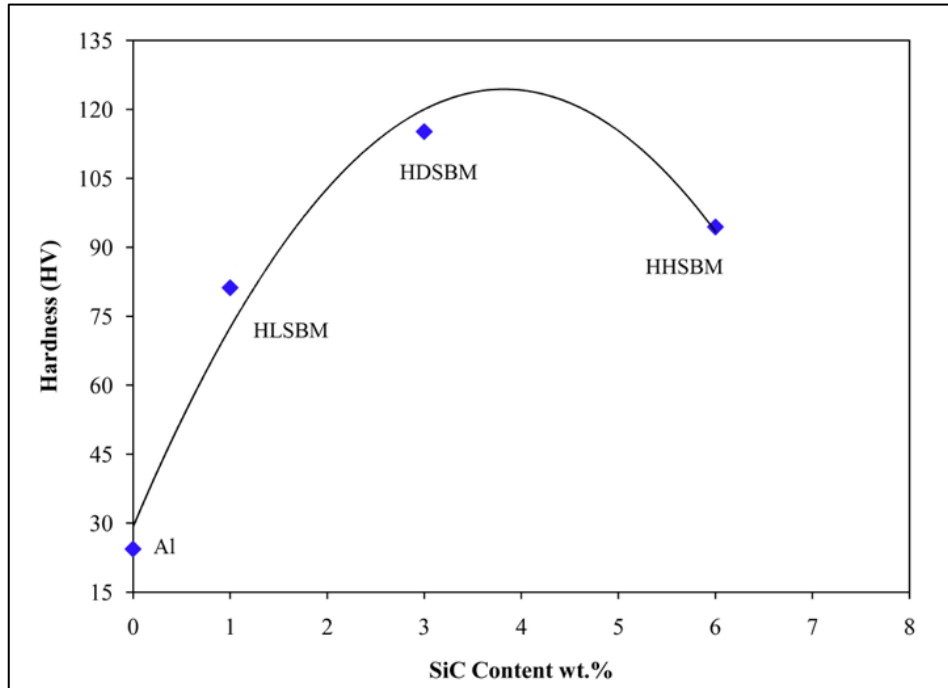


Fig. 14 Results of the Vickers hardness test conducted on HLSBM, HDSBM and HHSBM samples hot isostatically pressed at 530°C.

#### 4. Conclusions

This study reveals the significant effect of dual-speed ball milling in conferring uniform distribution on SiC nanoparticles dispersed within aluminium matrix compared to those produced using ball milling process. The produced composite powders in this study using the flake powder metallurgy process can be used significantly as a precursor for fabrication of bulk composite materials with superior mechanical properties. The effect of hot isostatic pressing process on the crystallite size of the composites was investigated using XRD, demonstrating the profound effect of well-dispersed SiC nanoparticles in restricting the grain growth of the hot isostatically pressed samples to the values lower than 140 nm. This was lower than crystallite size of the hot isostatically pressed samples (510 nm) with 6 Vol. % SiC, representing the fact that the uniform distribution of nanoparticles in HDSBM sample has a considerable effect in restricting the grain growth during hot isostatic pressing compared to the samples with high SiC volume content such as HHSBM sample.



This study also uncovers that sample with a uniform distribution of SiC nanoparticles can also bring about a significant enhancement in microhardness compared to the unreinforced matrix and samples with agglomerated SiC nanoparticles. This enhanced microhardness of the samples with uniform distribution of SiC nanoparticles compared to the samples with agglomerated SiC nanoparticles is attributed to the lower crystallite size and higher density of the former compared to the latter.

Tensile tests have also represented the profound effect of hot isostatically pressed samples produced using flake powder metallurgy under DSBM in enhancing the tensile strength (19%) and elongation (113%) compared to the ones reported for composites samples, containing 6 Vol.% SiC nanoparticles, supposed to represent higher tensile properties due to higher SiC content. These lower tensile properties are attributed to the higher agglomeration of SiC nanoparticles produced using ball milling process as a result of high SiC loading content (6 Vol. %), diminishing the relative density of these composites (97%).

It is found in this study, the dual speed ball milling (DSBM) process, prompting formation of flake powder during ball milling process, can significantly restrict the grain growth during the hot isostatic pressing process, bringing about enhanced mechanical properties for hot isostatically pressed samples.

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