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AN OVERVIEW ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SINTERED ALUMINUM-BASED COMPOSITES

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Abstract

Sintered composites have revolutionized as a thermal treatment to consolidate a wide range of engineering materials where the transition of powders takes place thermally in a thermodynamical equilibrium state with a decrease in free surface energy in materials owing to their specific capability. Sintering aids in providing effective bonding between the reinforced powder particles. However, the inadequate understanding of the sintering mechanism may limit the practical application of a few materials such as aluminum metal matrix composites. In addition to the rapid growth of various sintering related technologies, researchers need attention to highlight the structural barriers and forecast the emerging demands while dealing with such composites. A review report is made in this paper regarding the sintering mechanisms and sintering techniques. Common sintering techniques such as traditional, microwave assisted, hot pressing, hot isostatic sintering, and spark plasma sintering are identified and discussed here. As a result, the key challenges in sintering aluminium metal matrix composites that can affect sintering parameters are investigated. From the review, spark plasma is identified to attain densified and pore-free green composites and, microwave sintering is the best technique for achieving uniform microstructure in powder metallurgy samples.

Keywords: sintering; composites; powder metallurgy; aluminum.

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Introduction

Sintering is a final step in powder metallurgy (PM). The processes in powder metallurgy provide homogeneous particulate distribution and cause less reaction between matrix and reinforcement of aluminium based metal matrix composites (AMMC). Sintering is a thermally activated cycle for processing a ceramic particle compact into the base solid [1]. Due to the concentration gradient through particle/matrix interfaces, the driving force for sintering lies in reducing the specific surface area [2]. In the sintering, particles bond together when heated. In the sintering phenomenon, diffusion, creep, viscous flow, and plastic flow are remarkable mechanisms. Sintering, an irreversible process, consumes surface energy to bond particles together into strong, high-performance shapes. Sintering applications [3,4] range from home appliances to magnetic recording devices, automobiles, sharp cutting tools, wristwatches, wide musical instruments, sports equipment, hard bearings, hand tools, etc.

The chief phenomena in sintering are described to attain 1) increased mobility of atoms, where most of the atoms are distributed on the free surfaces in the form of pores, 2) the action of surface tension in the regions adjoining the places of inter-particle contact, there seems to be a decrease in the distances between the centers of the particles that contributes to a drop in the porosity or rise in density of the material that is sintered, 3) partial relaxation of internal stresses increase the dimensions of compacts and the change in particle size is due to compressive stresses whereas tensile stresses result in compaction of powder, 4) recrystallization due to the migration of atoms at elevated temperatures, 5) de-oxidation, i.e. the oxygen in the oxides are removed, and the surface is free from impurities and lastly 6) improved mechanical properties [5].

Sintering temperature, compaction pressure, alloy composition, heating rate, sintering time, and sintering atmosphere (dry inert gases or reducing gases are preferred to prevent further oxidation) are the parameters that control the sintering behavior of aluminium based metal matrix composites. Metal matrix composites (MMC) are highly advanced materials manufactured by combining two or more materials to achieve specific properties. In recent years, they have gained increased interest due to their superior strength quality, higher stiffness, corrosion, and wear content compared to unreinforced aluminium alloys [6-9]. AMMC's have been identified as potential candidates as typical MMCs due to their exceptional specific strength, wear resistance, and high-temperature performance when reinforced with particulates such as boron carbide, kaoline, silicon carbide, fly ash, rice hush, etc. [10-13].

Sintering parameters depend on the composition of the fabricated composite, desired properties, and the sintering method. Table 1 shows different sintering parameters incorporated while studying the sintering behavior of AMMC's. A suitable sintering methodology, the technological conditions of sintering like canning, degassing, passing inert gas during sintering or in a vacuum, the composition of the material to be sintered and the sintering parameters impact the end product. Also, the mechanical behavior of AMMC's in terms of yield strength, hardness and compressive properties concerning the sintering technique is precisely presented [14]. Proper densification with controlled grain growth is critically difficult in today's scenario, which may not be possible with traditional sintering techniques. So, the main emphasis while sintering aims to achieve optimum densification, morphology, mechanical properties, etc. Several studies have revealed that sintered composites have improved microstructural homogeneity and mechanical properties [15, 16].

Hence, sintering parameters such as temperature, pressure, and holding/dwelling time are varied till this is accomplished. Also, during sintering, the speed of particle bonding relies entirely on temperature, materials, particle size, and several other processing variables. Hence, the main issues in sintering AMMC's are the usage of proper reinforcement that can impact processing variables and sintering parameters, appropriate densification, grain growth, reduced porosity, and suitable sintering method.

Table 1. Parameters used by other researchers while sintering AMMC's.

SNo	Sample Composition	Fabrication method	Sintering technique	Sintering parameters			Ref.
				Holding/ Dwelling time [min]	Sintering Temperature [◦C]	Sintering pressure [MPa]	
1	Al–33Cu/5C	Powder metallurgy	hot-pressing	5	510	30	[17]
2	Al- B_4C	Powder metallurgy	hot-pressing	60	450	400	[18]
3	Al–Mg–Si–Cu alloy	Powder metallurgy	Conventional sintering	360	350	400	[19]
4	Al–Mg–Si–Cu alloy	Powder metallurgy	Microwave sintering	60	570	400	[19]
5	Al- nano SiC	Powder metallurgy	Microwave sintering	30	550 ±5	20	[20]
6	Al- nano SiC	Powder metallurgy	Microwave sintering	60	350	500	[21]
7	Al6061-0.1GNP-1B_4C	Powder metallurgy	Spark plasma sintering	10	450	60	[22]
8	Al-SiC	Powder metallurgy	Spark plasma sintering	5	1500-1800	50	[23]

Conventional sintering technique

Conventional sintering was one of the oldest methods to synthesize metal matrix composites by heating them using silicon rods, as shown in Fig. 1. Heat is transferred to the green compacts by conduction, convection, or radiation. Homogenous distribution and high strength are noticed [24] when Gr/Al composites are sintered at a temperature of 600 °C for 1 hour under a pressure of 25 MPa. Efficient load transfer of graphene reinforcement in the base aluminium matrix also resulted in tensile properties. The drawback with this traditional heating method is low heating rates, dissipation of volumetric heat energy from the hotter surfaces [25] and longer dwell times, which resulted in less density [26], poor densification [27], and internal stresses. A detailed study by *Manjunath and Krishnappa* [28] confined that under higher compaction pressure (280 to 350 MPa), Al-Cu alloy receives high plastic deformation as the particles experience fragmentation and exhibit 2.59, 2.68, and 2.68 g/cc of sintered density with less porosity. A comparative study is made by *Padmavathi et al.* [29] to investigate the densification of Al–7Zn–2·5Mg–1Cu (7775) alloy processed in conventional/traditional and microwave sintering. The authors revealed that 55% reduction is achieved by microwave sintering in processing time as compared to conventional sintering. Also, poor sinterability and coarser pores are exhibited by microwave sintering compacts when compared to conventional. The authors determined that this is due to the microstructural inhomogeneity. Investigations revealed that compacts sintered by conventional method at 630 °C proved to have equiaxed grains, which may be due to the liquid phase development via SLPS and high diffusion rates resulting in densification without any shape distortion.

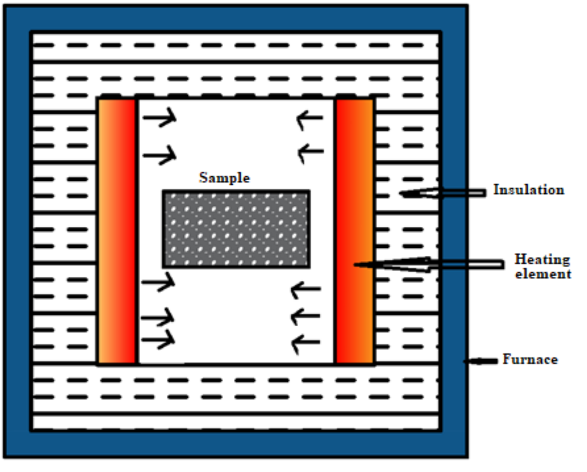

Fig. 1. Conventional heating process.

Microwave sintering technique:

When compared to conventional heating methods, microwave sintering of composites is found to be advantageous. The advantages such as energy efficiency, low manufacturing costs, energy and time saving, rapid sintering process, low soaking time, energy efficiency [30] are found in the samples sintered via microwave sintering technique. Product uniformity, high yielding, uniform volumetric heating, environmental friendliness, equiaxed pores, efficient densification, and a refine microstructure are attained due to high heating rates and slow sintering temperatures [31-33]. This volumetric heating method has been evolved as the best suitable method for powders [34] rather than metals.

Microwave energy is electromagnetic energy that functions within the 300 MHz to 300 GHz frequency range. Microwave heating is a method that absorbs microwaves and converts electromagnetic energy into heat, and the pictorial representation is shown in Fig. 2. Microwave heating, however, first produces heat inside the material and then heats the whole volume [35]. Because of this fact, the heating process is advantageous due to increased diffusion processes, reduced energy consumption, faster heating rates and slower processing times, reduced sintering temperatures, enhanced physical and mechanical characteristics, simplicity, distinctive properties, and reduced environmental hazards.

Previously this sintering method was applied only to ceramics, but from a review made by *Morteza Oghbaei et al.* [36], it is recognized that microwave sintering is applied to powdered metals too. At 2.45 GHz, the skin depth into bulk metals is noted to be very small, and therefore, very slight microwave penetration in depth occurs. Hence, quick heating may happen for fine powders. Their investigation is confined that microwaves absorb nearly 2.45 GHz if the base grain size is less than 100 µm. The degree of microwave absorption also relies on the parameters such as conductivity, temperature, and frequency. In microwave sintering, the energy is directly deposited onto the samples leading to the uniform temperature distribution.

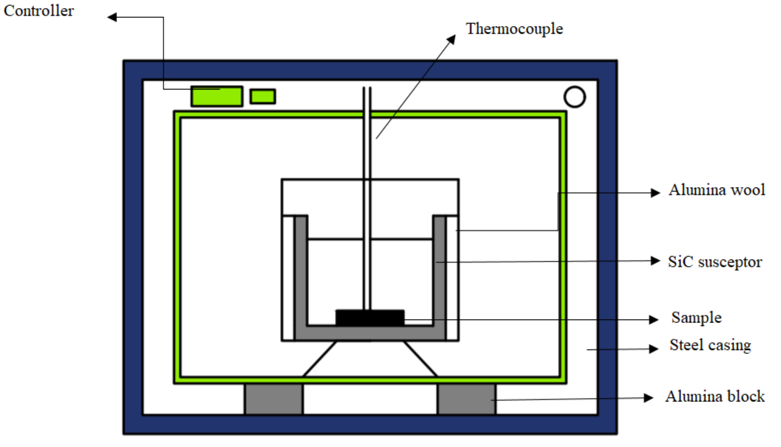

Fig. 2. Microwave sintering furnace.

Microwave sintering frequencies used most frequently are 915 MHz and 2.45 GHz. In addition, the absorbed power and depth of microwave penetration are the two significant parameters for the dielectric interaction of any material. They are found to record the evenness of heating across the material. The absorbed power, which is the volumetric absorption of microwave energy (W/m^3) in any material, is given by Eq. (1). The loss factor (tan δ) measures the material's capacity to convert microwave energy into heat, and the dielectric constant estimates the material's capacity to polarize. Another significant parameter is the penetration depth (D), which specifies that the incident power is lowered by one half showing the consistency of heating across the material. The depth of penetration is possibly expressed by Eq. (2)

$$P = |E|^2 = 2\pi f \varepsilon_0 \varepsilon_{eff}'' |E|^2 = 2\pi f \varepsilon_0 \varepsilon_r' \tan\delta |E|^2 \quad (1)$$

$$D = \frac{3\pi_0}{8.686\pi \tan\delta \left(\frac{\varepsilon_r'}{\varepsilon_0}\right)^{½}} = \frac{C}{2\pi f \sqrt{2\varepsilon' \left(\sqrt{(1+\tan^2\delta - 1)}\right)}^{½}} \quad (2)$$

German [37] has observed the behaviors of sintered Al 2124 and concluded that, with the rise in sintering temperature, the pores are reduced, resulting in improved micro hardness and density. However, a slight increase of sintering temperature to 500 °C resulted in reducing microhardness. Microwave sintering offers an improved diffusion process, high heating rates, shorter processing times (which contributes to reduced grain growth), and a uniform microstructure with smaller grains and greater mechanical strength when compared to traditional methods [38]. *Adnan Khan et al.* [39] synthesized $Al/SiC/ZrO_2$ nanocomposites at a sintering temperature of 1600 °C with high-grade alumina insulation using microwave sintering techniques. The authors mentioned that insulation in the microwave furnace helps in a more efficient heating rate and serves against thermal shock heating cycles. The sintering temperatures are maintained at 650 °C, 750 °C, 850 °C, and 950 °C while investigating microstructural refinement and mechanical characteristics of B_4C/Al matrix composites in microwave sintering [40]. *Penchal Reddy et al.* [41] synthesized Al–Cu matrix composites using both vacuum and microwave related sintering techniques. The authors inferred that sintering duration affects the sintered composite powder. Microwave sintering which is carried out for 30 min has emerged as an efficient technique compared to vacuum sintering (2 hours) as retention of alloy structure of the ceramic powder is obtained by microwave sintering treatment, thereby preventing intermetallic phase. Al/SiC samples sintered in a microwave sintering furnace at 550 °C ± 5 °C exhibited improved mechanical properties compared to pure aluminum [42]. From the investigations, it is clear that the samples' ultimate tensile strength and yield strength increase with an increment in SiC content. This feature can be revealed from the tensile stress-strain plot (Fig. 3a). A similar observation was made by *Penchal Reddy et al.* [43] while working with composites with different reinforcements sintered using a microwave furnace. As seen from Fig. 3b, it is clear that all composites reinforced with SiC, Al_2O_3, and Si_3N_4 exhibit high tensile strength. Also, from the tensile stress-strain plot, it could be observed that the elongation decreased when compared with aluminum. Fig. 3c [44] shows compressive engineering stress-strain curves for Al/TiB_2 composites sintered using a microwave furnace. The authors noticed that the sample's strength first increased with the TiB_2 level, then began to fall; nonetheless, altogether, both strength and elongation were significantly improved [44]. The compressive stress-strain behavior of Al/Cu_p composites sintered using a microwave furnace is presented in Fig. 3d [45]. The authors demonstrated that as the Cu content in the Al matrix increases, the yield strength and compressive strength increase as well. The authors also made a comparative study of the mechanical properties of the composites fabricated through vacuum and microwave sintering. They observed that the composites fabricated through microwave sintering exhibit high tensile and yield strength [45]. Hence, it is understood that enhanced physical and mechanical characteristics are obtained by microwave sintering technique due to high heating rates, slow sintering temperatures, product uniformity, high yielding, homogeneous volumetric heating, minimal soaking time equiaxed pores, effectual densification and refined microstructure. The authors have compared microstructures of microwave and vacuum sintered Al/Cu_pcomposites and found that microwave sintering results in good bonding and hence exhibits higher strengths. This feature was presented in Fig. 4.

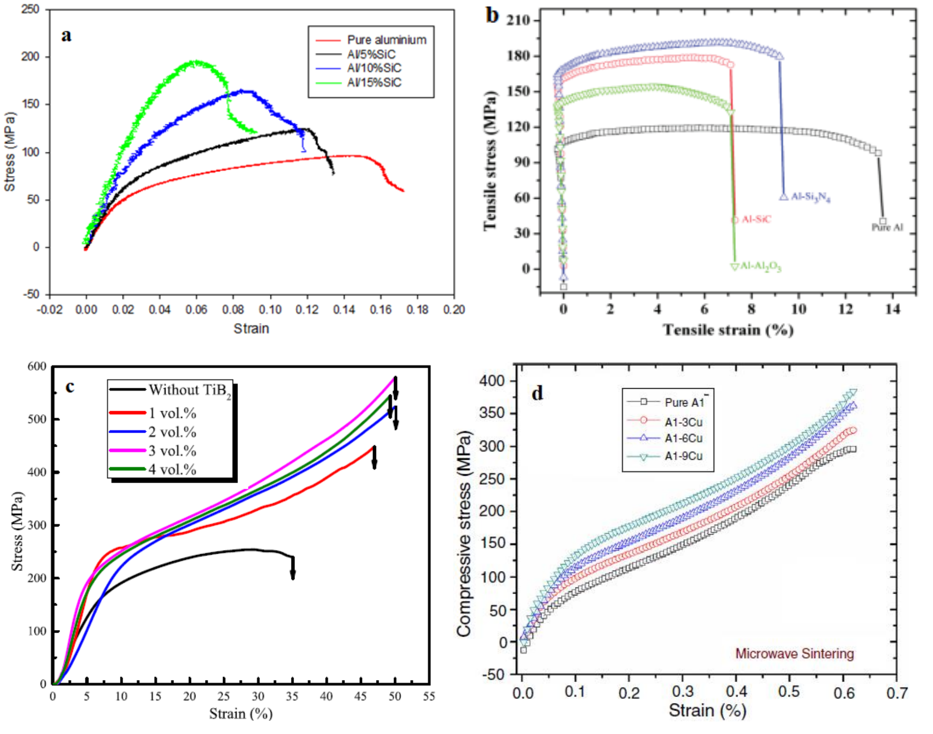

Fig. 3. Stress –strain plot for a) Al/SiC composite [42] b) Al-SiC/TiC composites [43] c) Al/TiB_2 composites [44] d) Al/Cu_p composites [45].

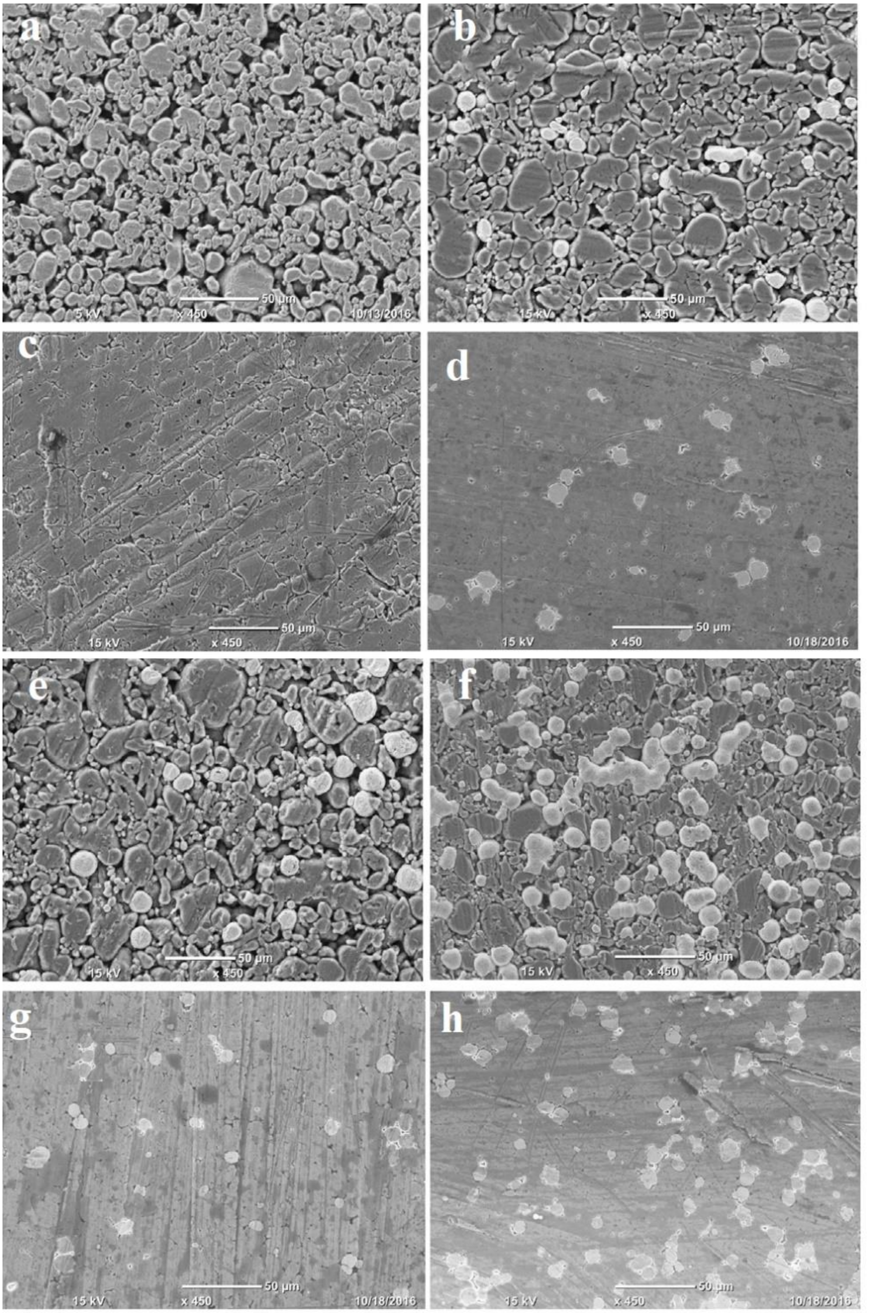

Fig. 4. SEM micrographs of Al/Cu_p composites (a–d) vacuum sintered and (e–h) microwave sintered [45].

Hot press (HP) sintering technique

The initial stage in hot pressing is cold compaction of the powders. This method of heating and the applied pressure are suitable to compact powders to attain a theoretical density (TD) of about 60-70% in the composites. At the interface of the grain contact, the authors [46, 47] revealed that densification is performed by pressure directed diffusion resulting in sliding, powder rearrangement, and plastic flow. In general, hot pressing is performed in a hydraulic press fitted with an oven [48] and heating is done by electrical resistance heating, induction, or radiation. The whole process is actually carried out in a vacuum to avoid oxidation, as shown in Fig. 5. The implication of temperature and pressure helps in easy deformation through creep and material transfer, and the variables tend to achieve high densities in the specimen via the hot pressing method of sintering. In this process, holding time plays a prominent role as coarse grains occur after a long period (almost after 60 min) [49]. The composites manufactured with the HP technique were subjected to a high temperature for a longer dwell time period for grain growth to occur. A process such as a vacuum hot pressing has emerged as an efficient process to attain high thermal conductivity in diamond/aluminum composites. The high thermal conductivity in vacuum hot pressing is due to interfacial bonding and interface reaction's controllability by optimizing the sintering parameters like temperature, pressure, and time [50]. The sintering procedure is mentioned to have five stages, as shown in Fig 6. First stage A, in which the powders are heated to 400 °C by 10 °C/min, and stage B comprises of holding them for 30 min at 400 °C, later, heating up to 650 °C again and waiting for 90 min, i.e. stage C and then stage D, where a uniaxial pressure of 67 MPa was applied to the compacts and finally, the sintered compacts with desired shape and size were obtained in stage E.

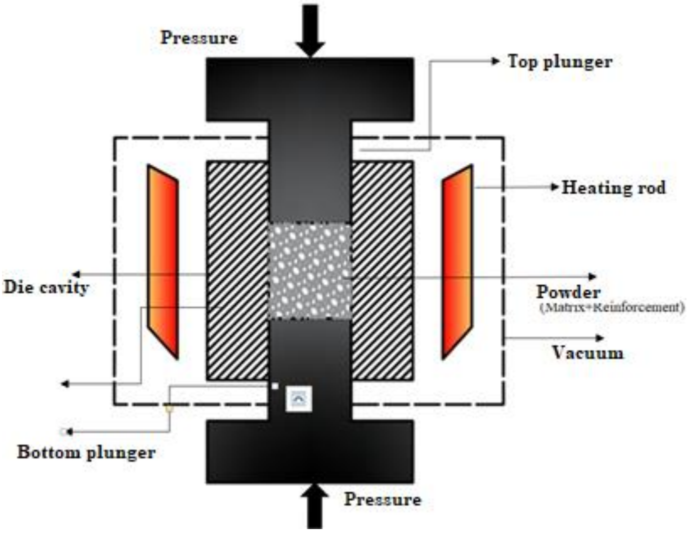

Fig. 5. Hot press sintering furnace schematic diagram.

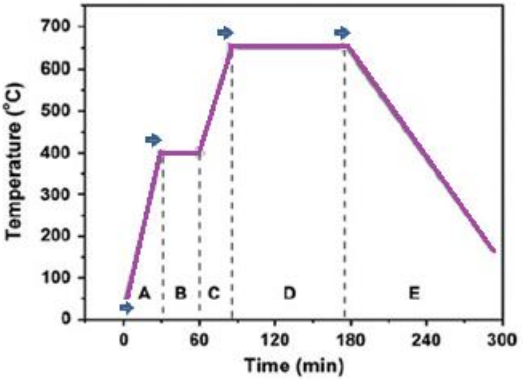

Fig. 6. The five stages of the sintering procedure in HP method.

Hot isostatic pressing (HIP)

Hot isostatic pressing is a method in which high pressure is applied isostatically [51] to one or more parts to fabricate 100% dense materials, as shown in Fig 7. The pressure acts uniformly in all directions, and the method is observed to be convenient to attain uniform densification, isotropic mechanical properties with complex shapes, improved microstructure and uniform grain growth [52]. It is a combination of pressing and sintering, thereby avoiding impurity and pollution hazards caused by the binder. Several authors inferred that the HIP process achieved reduced porosity and pore size in A356-T6 cast aluminum alloy [53]. The authors pointed out that this reduction is due to the improvement in the grain size and growth, which is caused by high temperatures during HIP sintering process. The methodology in this sintering method involves the application of pressure in all directions by using a gaseous or molten salt medium. A remarkable increase in yield strength and tensile strength is noticed [54] when Al alloy is reinforced with graphene nano-flakes and sintered using hot isostatic pressing at 480°C and 110 MPa for 2h. *Essa et al.* [55] declared that encapsulated powders are prone to isostatic pressure at a higher temperature for several hours in hot isostatic pressing. *Saud M. Almotairy et al.* [56] concluded that the density of the hot isostatically pressed samples depends upon several factors such as deformation, sintering driving force, crystallite grain size, strain energy stored in the powder, and the morphology of the fabricated composites.

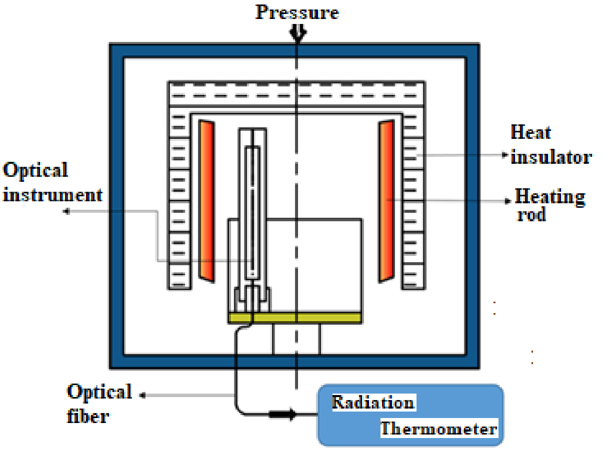

Fig. 7. Hot isostatic pressing furnace schematic diagram.

Spark plasma sintering (SPS)

A high degree of densely sintered composites is challenging to obtain employing conventional and other hot pressure sintering methods. The SPS method is primarily used to densify the composites with different melting temperatures here in between the base and the strengthened phase. SPS technique is also based on the generation of spark and an electrical discharge. It was revealed that by this method, fine refinement in the microstructure is obtained even at high sintering temperatures with reduced porosity. *Aliyu et al.* [57] studied the mechanical behaviour of Al/nano SiC composites synthesized by SPS process. The authors fabricated the samples at different parameters like pressure, sintering time and sintering temperature and heating rate. Their findings witnessed that the increase in sintering pressure and heating rate resulted in a considerable rise in microhardness at constant sintering temperature and duration. The authors also noticed that increasing the sintering pressure from 20 to 50 MPa greatly reduced the number and size of pores (Fig. 8a & 8b), and the microstructure did not change much when the heating rate was increased from 200 to 300 °C/min (Fig. 8c).

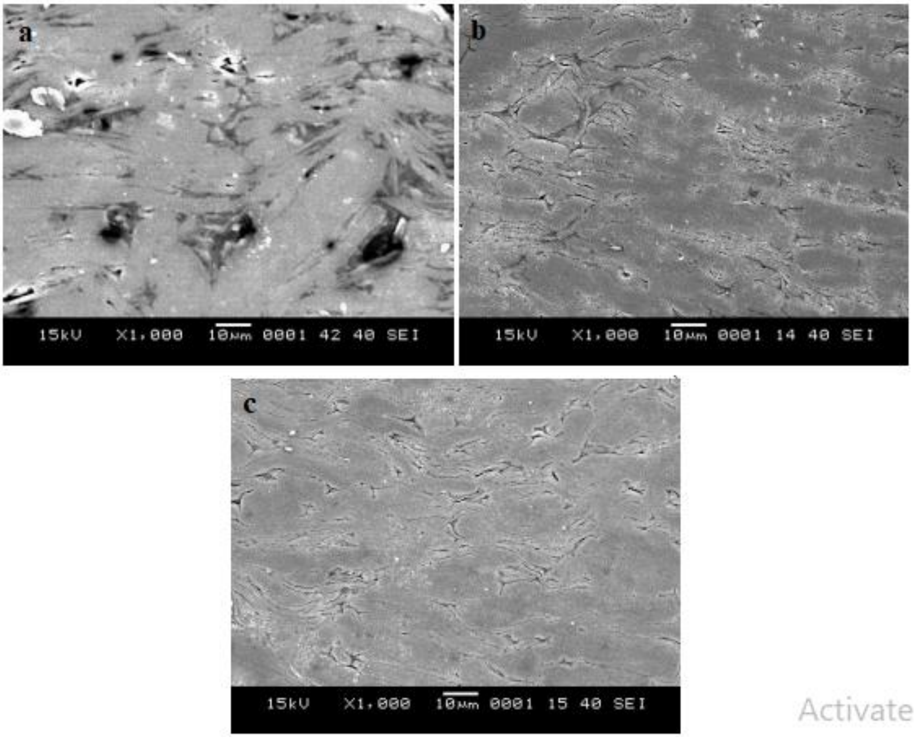

Fig. 8. SEM micrograph of SPS sintered Al–SiC composites sintered at 550 °C (a) 5 min, 200 °C/min, 20 MPa (b) 5 min, 200 °C/min, 50 MPa, (c) 5 min, 300 °C/min, 50 MPa [57].

Researchers also concluded that there is strong bonding between the base and the strengthened phase with negligible intermetallic phase with this technique [58-60]. SPS effectively densifies SiCp/Al composites [61] at a reduced temperature of 510 °C, with relative densities above 99% and below 2.65 g/cm^3. A noticeable grain growth, high densification and better control over microstructure during the consolidation of powders and nano powders are obtained with a nonconventional technique such as spark plasma sintering. The technique in SPS is dependent on the pulsed DC electric current that is passed through the die or the consolidated powders [62, 63]. A high heating rate of about 1000 K s^{-1} is involved in SPS method, which produces spark discharges at the interface. Researchers mentioned that the heating process is carried out by spark impact pressure or joule heating or electrical field diffusion. High relative density [64] strength and hardness with negligible interfacial reactions are recorded when Al-GNP (graphene nanoplatelets) composites) are sintered using SPS route and the heating mechanisms during the sintering by SPS route are observed to be due to (i) pulsed current activation, (ii) resistance sintering (heating by an electrical current passage), and (iii) pressure distribution. The fundamental variables of SPS methodology involve the rate of heating, sintered temperature, rate of cooling, rate of pressure, peak load hold, and rate of removal of load [65]. Sintering temperature has an impact role in the densification of the powder, which involves the mass transfer and is therefore estimated dependent on temperature. The schematic view of the SPS apparatus is shown in Fig. 9. Uniaxial hydraulic press, punch electrodes, vacuum chamber, regulated atmosphere, DC pulse generator and position, temperature, and pressure measuring units are the devices that support the sintering machine [66]. This technique consists of pressing and sintering, which were performed simultaneously in the vicinity of argon gas atmosphere (to avoid oxidation of aluminum) with the following conditions: at 560 ºC, Pressure of 5 MPa, heating rate as 112 ºC [67].

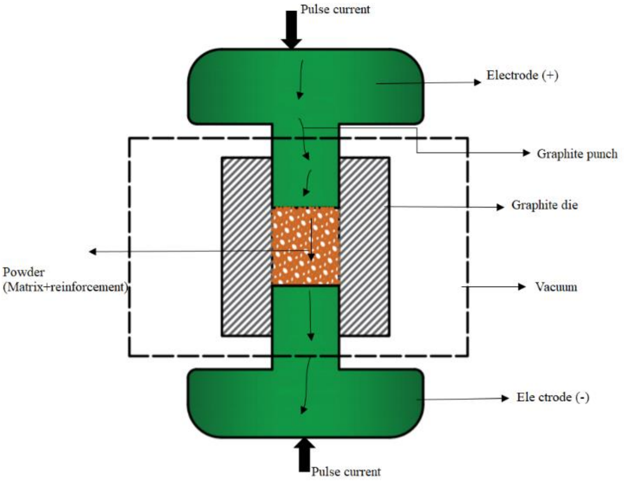

Fig. 9. The schematic view of SPS apparatus.

Conclusions

The experimental evidence related to the methodology, sintering parameters and the behavior of sintered aluminium metal matrix composites is presented. This review also included an overview of several sintering processes and their respective benefits over one another in terms of mechanical characteristics and densification. Microwave and spark-plasma assisted sintering are highly known technologies that are widely employed to sinter AMMCs, according to published studies. From the study, it is understood that the sintering technique has a significant impact on the microstructure and mechanical properties of the composite. Parameters such as compaction pressure, sintering temperature, sintering time, sintering atmosphere also play a crucial role in composite's microstructure and characteristics. Densified and pore free green composites are achieved when the AMMC's are sintered through the spark-plasma sintering technique. The microwave sintering technique allows faster production time and lowers energy use.

Each sintering technique has significant advantages over the other methods with different mechanisms and methodologies involved in it. It may be stated that this review paper provides a complete overview of various sintering procedures, their respective advantages, and how characteristics vary with different sintering techniques, which aids in the selection of a suitable sintering approach for multiple applications.

Acknowledgments

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