Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Blended powder semisolid forming of Al7075/Al₂O₃ composites: Investigation of microstructure and mechanical properties



A. Javdani^a, V. Pouyafar^{a,*}, A. Ameli^{b,*}, Alex A. Volinsky^c

^a Department of Mechanical Engineering, University of Tabriz, 29 Bahman Blvd., Tabriz 5166616471, Iran

^b Advanced Composites Laboratory, School of Mechanical and Materials Engineering, Washington State University Tri-Cities, 2710 Crimson Way, Richland, WA 99354, USA

^c Department of Mechanical Engineering, University of South Florida, Tampa, FL 33620, USA

HIGHLIGHTS

GRAPHICAL ABSTRACT

- Blended powder semisolid forming was implemented to eliminate the drawbacks of conventional semisolid powder metallurgy.
- Al7075/Al₂O₃ metal matrix composites were produced for the first time using this method.
- The effects of process parameters on the mechanical and physical properties of the composites were identified.
- The highest enhancement in physical and mechanical properties was achieved when large reinforcing particles were combined with small matrix particles.

ARTICLE INFO

Article history: Received 13 June 2016 Received in revised form 30 June 2016 Accepted 9 July 2016 Available online 11 July 2016

Keywords: Elemental powder Semisolid forming Mechanical alloying Al₂O₃ Metal matrix composite



ABSTRACT

Blended powder semisolid forming was adapted to fabricate Al7075/Al₂O₃ composites. The process included powders uniform distribution, mechanical alloying, and semisolid compaction. Al7075 elemental powders (20 and 63 μ m) were incrementally added to ethanol solution under ultrasonic mixing. Al₂O₃ particles with different sizes (5 and 120 μ m) and weight fractions (5, 10, and 20 wt.%) were blended with the matrix particles using a planetary ball mill. Al7075/Al₂O₃ composites were then compacted at semisolid state under different pressures (40 and 80 MPa). The effects of Al7075 and Al₂O₃ particle size, Al₂O₃ weight fraction, and compaction pressure on the morphology, microstructure, compaction mechanism, density, hardness, compression modulus and strength, and phase formation were measured and analyzed. The highest microstructural uniformity was achieved when large Al₂O₃ particles (120 μ m) were distributed within the small matrix particles (20 μ m). The density and hardness increased as the size of the reinforcing particles and the applied pressure were increased. Therefore, the composites with 20 μ m Al7075 and 20 wt.% of 120 μ m Al₂O₃ powder compacted under 80 MPa exhibited the highest improvements in relative density (98.685%), hardness (Rockwell B of 70), and compressive strength (327 MPa). The results are of great value in developing high performance lightweight metal matrix composites.

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* Corresponding authors.

E-mail addresses: pouyafar@tabrizu.ac.ir (V. Pouyafar), a.ameli@wsu.edu (A. Ameli).



1. Introduction

The superior engineering properties of light alloys such as low density, high strength, and high toughness have made it possible for these alloys to be widely used in the automobile, aerospace and marine industries [1–3]. Aluminum alloy 7075 is a high-usage light alloy which, in specific applications, has characteristics comparable to those of soft steel [4]. These features lay the foundations for this alloy to be an outstanding substitute for steel. In contrast with high strength to weight ratio, these alloys have lower strength and hardness compared to steel [5,6]. Particulate reinforcement is a suitable method which can increase the strength and hardness of these alloys [7]. Adding particles with high strength and hardness to the soft matrix produces composites whose mechanical properties are between the matrix and the reinforcement. The processing route adopted for synthesizing aluminum matrix composites depends on the nature of the matrix alloy and reinforcing materials which also influence the final properties of the composites [8–11].

Several reports have been published addressing the problems associated with the development of AMCs, as well as their mechanical behavior, microstructure, and distribution of particulates [12–15]. The factors such as the porosity of the matrix, volume fraction of the reinforcement and their distribution, agglomeration or sedimentation of particles and particle size, dross and porosities influence the behavior of the MMC [16]. Mechanical properties of metal matrix composites (MMCs) are essentially functions of the manufacturing processes [16]. Generally, aluminum matrix composites are manufactured using three different methods: liquid state [17], solid state [17], and semisolid state [18]. In the liquid state methods, reinforcement particles are added to a molten matrix. The major problem associated with this method is the settlement of the particles, which adversely affects the uniform distribution of the filler in the final product, and consequently, lowers the quality of the produced parts [13]. Furthermore, liquid state methods are conducted in high temperatures and are highly energyconsuming [19].

Powder metallurgy is the most common solid state fabrication route, which facilitates the uniform distribution of particles in matrix phase. The separation effects and the intermetallic phase formation are less for these processes [20]. However, the final products require post-processing operations [21]. Mechanical alloying is another solid state route, which has been successfully applied in the fabrication of Al alloy composites and nanocomposites from the elemental powders [4, 22]. However, this method suffers from disadvantages such as possible contamination, from the milling media or atmosphere, and the need for the subsequent consolidation of the powder into a bulk form. The contamination problem can be minimized or eliminated by a proper choice of the milling media and by milling in a high-purity inert gas or vacuum environment [23]. There are chances of microstructural changes such as grain growth or crystallization during consolidation [23].

Semi-solid processing is an attractive and an effective near-netshape forming process to produce components with complex geometry and in fewer forming steps [24–27]. Semisolid state methods for the fabrication of composites are similar to the liquid state routes but the mixing stage to distribute the reinforcing particles in the matrix phase is conducted at the semisolid state of the matrix. With this method, the settling problem of the particles is partially dealt with, but since the viscosity of the semisolid matrix is high, the mixing operation for high solid fractions may encounter some difficulties [19]. Therefore, in the industrial production of metal matrix composites, a method that can maintain the advantages of all mechanical alloying, semisolid processing, and powder metallurgy is of great interest.

Blended powder semisolid forming (BPSF) is a new method that meets the aforementioned requirements. It replaces elemental powder materials with prealloyed ones and extends the conventional semisolid powder metallurgy [28]. As schematically illustrated in Fig. 1, BPSF process has three major steps: a) the uniform distribution of powders using an ultrasonic homogenizer (Fig. 1.a), b) mechanical alloying which increases the temperature of powders due to collisions, and thus solid state diffusion occurs to produce the alloys (Fig. 1.b), and c) semisolid compaction which forces the liquid phase to fill the free spaces between the solid particles (Fig. 1.c). Since the initial material is in elemental powder form, it is possible to change the elemental state initial powders fraction and the size of the particles.

Several types of semisolid powder forming such as semisolid powder process [29], powder thixoforming [30], semisolid powder rolling [31,32], and powder thixoforging [33] have been introduced in the literature. The major difference between these works lies in the form of raw materials. Powders can be blended in either prealloyed state or elemental state before undergoing the semisolid forming process. The advantages of prealloyed semisolid powder metallurgy are the simplicity of the process, usage on a micro scale, and low energy cost [21]. The alteration of the constituents' weight ratios is a restriction, which can be resolved by the metallurgy of mixed powders from elemental state. If the mechanical alloying operation is carried out sufficiently, the semisolid forming of blended powders can have the benefits of prealloyed form as well as the capability of changing the elements of the compound [34]. The various techniques employed and the substantial analyses conducted on the flow behavior of semisolid powders have lead powders toward industrial applications [35].

BPSF has been successfully applied in the processing of alloy materials such as Al-Ti [34], Al-Mg [28], and composite materials, including Al-Cu reinforced with h-BN [36]. However, to the best of our knowledge, the BPSF of Al 7075/Al₂O₃ has not been reported yet. Al₂O₃ is one of the most common ceramics used for reinforcement in particulate form. Compared to SiC, another common ceramic, Al₂O₃ has higher thermal stability. It does not react with the metal matrix at the processing temperatures of BPSF and does not produce brittle phases [37]. So, it is a viable choice for the particulate reinforcement of Al. Understanding the role of various material and process parameters on the properties of the final product is the essential step in the evaluation of the suitability of such composites for industrial applications. Therefore, this work is aimed to develop Al7075/Al₂O₃ composites via BPSF.

In the present study, various Al 7075/Al₂O₃ composites were fabricated via BPSF method. The density, hardness, phase formation, compression strength and microstructure of the composites were studied. Also, the effects of reinforcement weight fraction, reinforcement and matrix particle size, and semisolid compaction pressure on the microstructure, hardness, density and mechanical properties were investigated.

2. Experimental procedure

2.1. Materials

Two different types of gas atomized Al powders (Khorasan Powder Metallurgy Company) with a particle size of 20 μ m (99.96% purity) and 63 μ m (99.94% purity) were used as the base elements for Al7075 blends. Weight percentage, purity, and particle size of Al7075 alloy elements are shown in Table 1. Al₂O₃ powders (Iranalumina Company, 94% purity), with average particle sizes of 5 μ m and 120 μ m were used as the reinforcement particles in the composites. The densities of aluminum and alumina powders were 2.80 and 3.97 g/cm³, respectively.

2.2. Preparation of Al7075/Al₂O₃ composites

In order to fabricate A17075/Al₂O₃ composites and Al7075 alloy samples, two types of Al powder, and two type Al₂O₃ powder were employed. An ultrasonic mixer (FAPAN 400R) was used to homogenize the constituents of Al7075. Powder elements of Al 7075 were incrementally added to the ethanol solution. The working condition of the ultrasonic mixer is shown in Table 2. After mixing, in order to vaporize ethanol, the solution was placed in PID-controlled resistance furnace, and the powder mixture of Al7075 was obtained. A high energy



Fig. 1. Schematic illustration of the three major steps in blended powder semisolid forming (BPSF) process.

planetary ball milling (NARYA-MPM-2*250, Amin-Asia Company) was employed to blend Al7075 mixture with Al_2O_3 reinforcement particles for 3 h. The rotation speed of the milling process was set to be 250 rpm. The vial and the balls were made of hard polymer and alumina, respectively. Using balls with 5 and 10 mm diameters, the ball to powder weight ratio was chosen as 10:1. The volume of the vial was 250 ml. To avoid agglomeration of milled particles, stearic acid (1.5 wt.%) was used as a process control agent. The powder composite samples were dried in the air after mixing.

2.3. Compaction procedure

Fig. 2 shows the die set used in the BPSF process. Die material was L 316 stainless steel. Powder samples were weighed and poured into the die cavity and the die set was placed into the furnace. The cold compaction pressure was 5 MPa for all the compacted samples. Then, the die was placed in the furnace and the temperature of the furnace was raised up to the temperature range of semisolid Al7075. The semisolid temperature was about 610 °C in which the liquid fraction is about 25% (Fig. 3.a). Liquid fraction versus temperature gradient for Al7075 powders was measured by diffraction scanning calorimeter (Setaram, France). When the temperature of the powder sample reached to the semisolid temperature, pressure was gradually applied to the semisolid powder samples; the applied pressure was kept for 45 min in all the samples. Then, the pressure was removed and the samples were cooled down in air.

A separate experiment was conducted to determine the actual temperature of the powder sample during heating from initial conditions to semisolid state. One thermocouple was placed on the die surface while another was placed within the powder. As it is illustrated in Fig. 3.b, the furnace temperature is at first set to 700 °C and kept at this temperature for about 35 min. When the powder temperature reached 610 °C, the temperature of the furnace was set to 630 °C. After that, temperature difference between the die and the furnace was stable. This heating profile was used in all forging experiments.

In addition to the furnace heating cycle used for the powders (Fig. 3.b), in all of the experiments, a thermocouple was placed within the hole embedded on the die (Fig. 2.a). The depth of the hole was equal to the depth of the mold cavity. This thermocouple started to work as

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Al7075 constituents and their content, purity, and particle	size.

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Element	Zn	Mg	Cu	Cr	Mn	Fe	Si
Content (%)	5.5	2.5	1.5	0.23	trace	trace	trace
Purity (%)	99.98	99.98	99.96	99.90	99.94	99.90	99.98
Particle size (µm)	20	63	45	63	63	45	5

soon as the furnace started to heat the die to measure the temperature of the sample. As illustrated in Fig. 3.a, the temperature of the sample can be used to control the liquid phase fraction during semisolid sintering.

2.4. Design of experiments

Experiments were conducted to investigate the effects of the size and amount of the reinforcement particles, matrix particle size and the applied compaction pressure on the microstructure, and the mechanical and the physical properties of the samples. The experimental arrays are shown in Table 3.

2.5. Characterization

Cross sectional areas for metallography and subsequent mechanical property evaluation were selected as normal to the forging direction. Polishing was completed on a standard series of grit papers and diamond polishes. The morphology of the ball milled powders and the microstructure of the forged samples were analyzed using a scanning electron microscope (SEM) (MIRA3 FEG-SEM of Tescan company). Alloying behavior during milling and semisolid forming were determined by X-ray diffraction (XRD) analysis using a Philips PW1730 diffractometer (40 kV) with Cu Ka radiation (k = 1.54060 nm). Macro hardness tester (TRS, SR. No. 2011/224) based on Rockwell B standard was also used to determine the strength of composites against indentation. The reported values represent the average of 5 measurements. Compression tests were performed on samples with 10 mm diameter and 20 mm height using universal hydraulic machine (800 KN) with a speed of 1 mm/min. The experimental density of the forged samples was measured using Archimedes principles coupled with water immersion. The theoretical density was calculated using the mixture rule according to the weight fraction of the Al₂O₃ particles. The absolute porosity of the sintered specimens was calculated according to Eq. (1):

$$p = \frac{\rho_t - \rho_e}{\rho_t} .100\% \tag{1}$$

where *p* is the absolute porosity [%], and ρ_t and ρ_e are the theoretical and experimental densities [g/cm³], respectively.

 Table 2

 Ultrasonic homogenization process parameters set points.

Parameter	Power	Frequency	Pulse duration	Temperature
Set point	750 watt	25 KHz	2 s	28 °C



Fig. 2. (a) The compaction die set of BPSF process, and (b) The schematic of the assembled die.

3. Results and discussion

3.1. Powder morphology

The initial characteristics of the powders, i.e., the size, the morphology, and the size ratio of the matrix and the filler all affect the consolidation behavior of the samples [38]. Identifying the morphology of the particles before and after the mechanical mixing is helpful to describe the mechanical properties of the final bulk materials. Fig. 4 shows the morphology of the employed matrix and filler powders. Based on their initial size and shape, the powders underwent different deformation mechanisms through the mixing process. As illustrated in Fig. 4.a and .b, 120 µm Al₂O₃ particles and 20 µm Al7075 particles have a nearly spherical initial shape. These particles have also maintained their initial shape after ball milling (Fig. 4.c). The distribution of the reinforcement particles in the matrix phase is uniform for this combination of the particle sizes. 5 µm Al₂O₃ particles can be verified as sharp-edged and relatively irregular agglomerated particles (Fig. 4.d). In their mixture with 20 µm Al7075 (Fig. 4.e), tiny particles of Al₂O₃ adhered to and covered the surface of the matrix phase particles. As seen in Fig. 4.f, 63 µm Al7075 had particles with soft outer surfaces and elliptic shape. In the process of mixing these particles with 5 μ m Al₂O₃ particles (Fig. 4.g), some of the agglomerated reinforcement particles, existing from initial state, were divided into smaller pieces and surrounded the matrix phase particles. However, as illustrated by white arrows on Fig. 4.g. some agglomerates of the reinforcement particles were still present after mixing.

Table 3

Variable experimental parameters in the fabrication of Al7075/Al₂O₃ composites.

Parameter	Settings
Al matrix particle size (μm)	20, 63
Al ₂ O ₃ particle size (μm)	120, 5
Al ₂ O ₃ weight percentage (%)	5, 10, 20
Semisolid compaction pressure (MPa)	40, 80

3.2. Microstructure

Fig. 5 shows representative microstructures of Al7075/Al₂O₃ composites prepared at various experimental conditions. In samples that the size of reinforcement particles was larger than that of the matrix particles (i.e., $120 \mu m Al_2O_3$ and $20 \mu m Al7075$), the distribution of the reinforcement particles was relatively uniform (Fig. 5.a). As shown by white arrows in Fig. 5.a, micro-sized pores existed within the matrix phase particles. This can be attributed to the low compressibility of the spherical matrix phase which resulted in the insufficient bond between the soft phase particles at this level of pressure (40 MPa) [39, 40]. Increasing the process pressure forced the liquid phase to flow better and fill the gaps between the matrix phase particles and hence a reduced number of voids existed when the high pressure (80 MPa) was applied (Fig. 5.b).

In the case of 5 μ m Al₂O₃ and 20 μ m Al7075 (when the sizes of the matrix and the reinforcement particles were relatively close), the reinforcement particles were dispersed uniformly along the grain boundaries, as indicated by white arrows in Fig. 5.c and .d. Since the reinforcement particles had stuck to the surface of the matrix particles in the ball milling process (Fig. 4.e), their microstructural arrangement after compaction were influenced by such a pattern from the blending procedure. In this case also, a relatively uniform distribution of the reinforcement particles was observed. Barrier action of the surface oxide of the matrix particles as well as the barrier action of the Al₂O₃ particles lowered the inter-diffusion of Al7075 particles, and consequently, diffusion bonding between the matrix particles was attenuated [41]. The gaps shown by black arrows on Fig. 5.c and .d affirm weak bonding between the matrix particles. Increasing pressure from 40 MPa to 80 MPa increased the flowability and the surface contact areas, and consequently, microstructures with a less number of inter-particle gaps were obtained (Fig. 5.d).

Microstructures obtained from the semisolid forming of 63 μ m Al7075 reinforced with 5 μ m Al₂O₃ (Fig. 5.e) showed micro-sized pores at low compaction pressure of 40 MPa. As Fig. 5.f depicts, the increase of pressure to 80 MPa filled the gaps between Al7075 matrix particles. However, the agglomeration of the reinforcement particles was still evident. Even though the higher pressure applied in this condition



Fig. 3. (a) The Al7075 liquid percentage as a function of temperature obtained from DSC, and (b) powder samples heating cycle during semisolid compaction.



Fig. 4. SEM micrographs showing the morphology of (a) as received 120 μ m Al₂O₃, (b) as received 20 μ m Al7075, (c) milled 120 μ m Al₂O₃/20 μ m Al7075, (d) as received 5 μ m Al₂O₃, (e) milled 5 μ m Al₂O₃/20 μ m Al7075, (f) as received 63 μ m Al7075, (g) milled 5 μ m Al₂O₃/63 μ m Al7075.

was able to close the pores and gaps, its effect on the degree of densification was low (Fig. 5.f). Decreasing the particle size will considerably increase the number of alumina particles, thus leading to their agglomeration and clustering [42]. Apparently, the agglomerated structural network of the reinforcement particles formed at the blending stage was so resistant to the diffusion of Al7075 that even a pressure of 80 MPa was insufficient to push the liquid Al into the free spaces within the network of the reinforcement particles.

Another observation that is worth noting is the formation of microcracks in some cases. Mechanical milling and solidification shrinkage causes the formation of micro-cracks in the process of BPSF. As shown in Fig. 6.a, micro-cracks were not observed on the surfaces of the large Al₂O₃ particles after ball milling process. At 610 °C, the liquid fraction of the Al7075 is about 25% (Fig. 3.a). As shown in Fig. 6.a and .b, the solidification shrinkage occurred during cooling from semisolid state to room temperature, exerted tensile stresses on the matrix and the reinforcement particles. Since Al₂O₃ large particles were much stronger than the matrix phase, the cracks were generated within the reinforcing phase. These micro-cracks were not observed in the case of small reinforcement particles. It seems that the small reinforcement particles could rearrange and relax the stresses caused by thermal shrinkage.

3.3. Compaction mechanism

The semisolid compaction behavior of Al alloy powders is categorized into three main types [43]. When the liquid phase is less than 10%, an isolated liquid phase formation is observed. When the liquid phase is between 10% and 20%, some of the liquid phase within the particle may form a network. In the third type where the liquid phase is higher than 20%, compaction causes the liquid phase to squeeze out and form solid arms with irregular shape. In the present study, liquid phase is about 25% and the compaction mechanism is considered to be the squeezing of the liquid phase out of the solid phase.

The evolution of the composites during the compaction of the soft matrix and the hard reinforcement particles is schematically shown in Fig. 7. In the case of $20 \,\mu m \, Al7075/120 \,\mu m \, Al_2O_3$ (Fig. 7.a), the reinforcement particles were distributed uniformly into the matrix phase. The uniformly distributed reinforcing particles in larger sizes and symmetric shapes, formed during the preceding ball milling stage, produced well-densified compacts [44]. Large reinforcement particles with rough surface morphology were in contact with a sufficient number of matrix particles and the liquid phase squeezed out from the surrounding Al7075 particles sufficiently wetted the interfaces of the matrix and



Fig. 5. SEM micrographs of composites with 5 and 20 wt.% Al₂O₃. Experimental conditions are: (a) 40 MPa, 20 μm Al7075/120 μm Al₂O₃; (b) 80 MPa, 20 μm Al7075/120 μm Al₂O₃; (c) 40 MPa, 20 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 20 μm Al7075/5 μm Al₂O₃; (e) 40 MPa, 63 μm Al7075/5 μm Al₂O₃; (d) 80 MPa, 63

the reinforcement particles. The liquid phase flow filled the empty spaces between the particles and increased the wet area between the matrix and the reinforcement phases [36].

When small spherical matrix powders ($20 \mu m$) reinforced with tiny particles ($5 \mu m$) experienced the hydrostatic pressure (Fig. 7.b), a fraction of liquid, squeezed from the matrix phase, dissolved the reinforcing particles in itself. Tiny reinforcement particles could easily rotate and rearrange to fill the empty spaces between the matrix particles [37]. Since the reinforcement particles had stuck to the surfaces of the matrix particles in the mechanical alloying process (Fig. 4.e), the matrix phase was surrounded by tiny reinforcement particles.

The third case was the mixture of irregularly-shaped and large $(63 \ \mu m)$ matrix particles with the tiny reinforcing particles $(5 \ \mu m)$. It can be confirmed from Fig. 4.g that the resultant compound had two different arrangement of the filler particles, i.e., the uniformly distributed individual particles and the agglomerates of the particles. The compaction mechanism for this case is depicted in Fig. 7.c. Depending on the amount of agglomerated reinforcement particles and their arrangement within the composite, the final product can be either dense or porous.

The wetting behavior of A1 on $A1_2O_3$ investigated by the sessile drop method has been reported to strongly depend on temperature [45,46]. Good wetting leads to strong bonds at the interface of the matrix and reinforcement [47]. Investigation on the Al/Al₂O₃ composites produced by powder metallurgy at different temperatures (500, 550 and 600 °C) showed the temperature of 600 °C as an optimized condition for sintering and the stronger bonding between the particles was correlated to the elevated sintering temperature, leading to greater strength [48]. In the present study, blended powder semisolid forming of Al7075/Al₂O₃ composites were carried out at 610 °C which is higher than conventional powder metallurgical route. So, mechanical properties of the composites could be affected by sintering temperature. All of the large reinforcement particles with uniform distribution in small matrix could be wetted at semisolid temperature. In the case of 5 µm Al₂O₃ and 20 µm Al7075, the reinforcing particles were wetted sufficiently by matrix (Fig. 5.c) but barrier action of the reinforcing particles prevented the sticking of the matrix particles to each other. Nonuniform distribution of 5 µm Al₂O₃ particles within 63 µm Al7075 particles seemed to lower the effect of sintering temperature.



Fig. 6. SEM micrographs of (a) ball milled 120 µm Al₂O₃/20 µm Al7075, (b) 20 µm Al7075/5 wt.% 120 µm Al₂O₃ composites compacted at 80 MPa and (c) 20 µm Al7075/20 wt.% 120 µm Al₂O₃ composites compacted at 40 MPa.



Fig. 7. Compaction mechanisms during the semisolid formation of Al 7075/Al₂O₃: a) 20 µm Al7075/120 µm Al₂O₃, b) 20 µm Al7075/5 µm Al₂O₃, and c) 63 µm Al7075/5 µm Al₂O₃.

3.4. Density and porosity

The theoretical and experimental densities (Table 4) of the Al7075/ Al₂O₃ composites as a function of Al₂O₃ weight fraction are shown in Fig. 8.a. The experimental values are the densities of the composites prepared under a compaction pressure of 80 MPa. As expected from the rule of mixtures, the theoretical density of the samples increased linearly with the increase of the Al₂O₃ weight fraction [37]. Although a relatively linear relationship was observed between the experimental densities and the Al₂O₃ weight fraction, the experimentally measured density values were lower than those of the theoretical densities. This indicates the existence of some porosity in the microstructure of the composites. Also, as seen in Fig. 8.a, the density of the composites increased as the size of Al_2O_3 reinforcing particles was increased from 5 µm to 120 µm. This trend is well correlated with the microstructure of the composites (Fig. 5) and the proposed compaction mechanism for each of these composites (Fig. 7). In the case of 20 µm Al7075/120 µm Al_2O_3 composites, the large reinforcing particles were uniformly distributed within the small Al matrix particles (Fig. 5.a and .b) and a sufficient degree of wetting was achieved by the liquid phase of the matrix, and consequently, the composite was compacted with less number of pores and gaps, resulting in the highest density.

In the case of 20 μm Al7075/5 μm Al_2O_3 composites, however, a different type of microstructural arrangement was observed. Tiny Al_2O_3

Table 4

The physical properties of the Al7075/Al₂O₃ composites fabricated using various experimental conditions.

Exp. No.	Al ₂ O ₃ weight percentage (%)	Al_2O_3 particle size (µm)	Al matrix particle size (µm)	Compaction pressure (MPa)	Hardness (Rockwell B)	Theoretical density (g/cm ³)	Experimental density (g/cm ³)	Relative density (%)
1	0	-	20	40	37.1	2.810	2.690	95.720
2	0	-	20	80	44.0	2.810	2.743	97.610
3	0	-	63	40	41.4	2.810	2.710	96.441
4	0	-	63	80	46.0	2.810	2.764	98.362
5	5	120	20	40	57.3	2.867	2.738	95.521
6	10	120	20	40	62.1	2.926	2.801	95.756
7	20	120	20	40	65.6	3.042	2.924	96.124
8	5	120	20	80	63.2	2.867	2.820	98.360
9	10	120	20	80	64.5	2.926	2.880	98.427
10	20	120	20	80	70.0	3.042	3.002	98.685
11	5	5	20	40	44.6	2.867	2.698	94.140
12	10	5	20	40	50.0	2.926	2.786	95.218
13	20	5	20	40	62.0	3.042	2.902	95.419
14	5	5	20	80	55.3	2.867	2.805	97.837
15	10	5	20	80	57.0	2.926	2.860	97.744
16	20	5	20	80	64.0	3.042	2.960	97.304
17	5	5	63	40	41.6	2.867	2.740	95.587
18	10	5	63	40	40.0	2.926	2.792	95.423
19	20	5	63	40	55.0	3.042	2.944	96.781
20	5	5	63	80	55.8	2.867	2.780	96.965
21	10	5	63	80	49.8	2.926	2.820	96.377
22	20	5	63	80	68.2	3.042	2.940	96.646



Fig. 8. (a) Theoretical and experimental densities of various Al 7075/Al₂O₃ composites, prepared under 80 MPa, as a function of Al₂O₃ weight fraction; and (b) Comparison of porosity versus reinforcement addition at different experimental conditions.

particles adhered to the surface of and covered the surrounding of the Al large particles. This acted as a barrier for the effective inter-diffusion of the matrix particles during compaction. Therefore, separated matrix phase laminates were created as was observed in the microstructure (small black arrows in Fig. 5.c). Diffusion barrier action of Al₂O₃ ceramic particles to the Al matrix composites have been previously reported in the literature [41]. This ineffective compaction and insufficient inter-diffusion resulted in the free spaces between the matrix phase particles and consequently the density was lowered.

The lowest density was obtained for 63 μ m Al7075/5 μ m Al₂O₃ composites. In this case, other than the inter-diffusion barrier, an additional mechanism was active for the formation of free spaces in the microstructure. As discussed before (Figs. 5.e, .f, and 7.c), the agglomerated reinforcement particles acted as a barrier against the movement of semisolid phase to fill gaps within the networks of the agglomerated Al₂O₃ particles and this resulted in additional free volume. Therefore, the lowest density was obtained at this composite configuration.

Comparison of the unreinforced Al7075 compacted parts density which is shown in Fig. 8.a, revealed that in spherical particles ($20 \mu m$), symmetrical opposite forces appears in the contact points, which promote only compressive deformation of particles. An irregular morphology of the particles ($63 \mu m$) propitiates the formation of asymmetrically opposite forces in the contact points between particles, which results in shear deformation and, consequently, cold-welding of the powder particles. As a consequence, spherical powder is very hard to compact [40,49]. Low compressibility of the spherical particles lowered relative density of the samples.

The porosity of the composites prepared at different experimental conditions is shown in Fig. 8.b. As expected, a raise in the compaction pressure lowered the porosity values in all the conditions. Increasing the compaction pressure further filled the free spaces at the interfaces of the reinforcement and the matrix phase, and consequently, the bonding force between the matrix and the reinforcement phase increased.

High-strength bonds in the metal matrix composites raise the densification degree of the final product [50,51].

Porosity change as a function of pressure was not the same for all the composites. Composite samples produced with 20 μ m Al7075/120 μ m Al₂O₃ blends showed the maximum porosity decrease and 63 μ m Al7075/5 μ m Al₂O₃ samples had the minimum decrease as the pressure increased. As observed in the microstructure and compaction mechanism of the composites with irregular shape matrix particles, the agglomerated structural network of the reinforcement particles were strong and applying more compaction pressure could not fragment the structural network. Consequently, the effect of compaction pressure on the porosity was less. On the other hand, uniformly distributed reinforcement particles (120 μ m) received liquid phase in all composite locations and this facilitated a higher degree of compaction.

3.5. Hardness

The results of hardness testing indicate that the size of blended particles and the amount of reinforcement particles have a significant effect on the hardness of the resultant composite samples. The macro-hardness of the composite samples with 5 wt.% Al₂O₃ content is illustrated in Fig. 9.a. As seen, the composite samples with larger reinforcement particles were harder than the other two composites with smaller sizes of the filler particle. This can be attributed to the uniform distribution of large reinforcement particles, formed during the mechanical blending stage. This facilitates the load transformation capability from the matrix phase to the hard reinforcement phase and thus results in an increased hardness.

When the compaction pressure of the semisolid forming was increased from 40 MPa to 80 MPa, the hardness of all the samples increased. Higher pressure forced the liquid phase to move into pore spaces between reinforcement and matrix particles and samples with higher relative density was obtained. So, higher hardness is simply due to the higher relative density obtained with higher pressure [44].

Fig. 9.b shows the relationship between the hardness and alumina content for the composites having various combinations of particle



Fig. 9. Rockwell B scale macro-hardness test results: (a) hardness of the composite samples with 5 wt.% Al₂O₃ at different experimental conditions; and (b) hardness versus Al₂O₃ content at various combinations of the particle sizes and at the pressure of 80 MPa.

sizes and prepared under 80 MPa compaction pressure. As expected, the hardness proportionally increased with the increase of alumina content, in all the cases, and a relatively linear relationship was observed. Alumina is harder than the Al matrix and thus the increase of the reinforcement content increases the hardness of the entire composite. Based on the rule of mixtures, the hardness of a composite depends on the volume faction of the reinforcement and the matrix phases as:

$$H_c = H_r * F_r + H_m * F_m \tag{2}$$

where H_c , H_r and H_m are the hardness of the composite, the reinforcement, and the matrix phase, respectively. Also, F_r and F_m are the volume fractions of the reinforcement and the matrix phase, respectively [52].

As shown in Fig. 9.b, the hardness of the unreinforced Al7075 powders ($20 \mu m$) with spherical shape was lower than that of Al7075 powders ($63 \mu m$) with irregular shape. As previously mentioned in Section 3.4, low compressibility of the spherical powders lowered the relative density of the composite samples, resulting in the reduced strength of the composites against indentation.

3.6. Compression modulus and strength

Poor adhesion, pores or the formation of brittle intermetallic phases at the interface between matrix and reinforcements may result in premature and catastrophic failure of the composites [53]. The compression strength of Al7075/20 wt.% Al₂O₃ composites with different combinations of reinforcement and matrix particle size prepared under 80 MPa is illustrated in Table 5. As seen, 20 µm Al7075/120 µm Al₂O₃ showed the highest value of 327 MPa. Uniform distribution of the reinforcing particles within matrix phase (Fig. 5.b) and the high compaction pressure affirms that. This value is nearly twice as the strength of the Al/ Al_2O_3 compacted parts produced with conventional powder metallurgy techniques [54]. In the case of 5 µm reinforcement particle size and 20 µm matrix particle size, Al₂O₃ particles had appeared along grain boundaries (Fig. 5.d). Barrier action of the tiny particles caused poor bonding of the reinforcement and matrix particles [51] and lowered bonding strength of the samples. The samples with 63 µm matrix and 5 µm reinforcing sizes had two different mechanisms that controlled their plastic deformation during compression. As it was mentioned in Section 3.4, compressibility of the irregular shape particles is better than spherical ones. This improves sticking ability of the irregular powders to each other. Matrix particles with irregular shape played an enhancement role for the good bonding of matrix and reinforcement interfaces; however, agglomerated reinforcement networks as shown in the microstructure and proposed compaction model (Figs. 5.e and 7.c) had a stronger negative effect, resulting in pores. Existence of the pores in these samples caused lower strength [55].

As a sample for analyzing the effect of reinforcement addition on compression behavior of 20 μ m matrix and 120 μ m filler phase composite, the samples with 0, 5, 10 and 20 wt.% reinforcement underwent compression test. The compression curves are shown in Fig. 10. When the reinforcement content increased, the compression strength increased. This is due to the higher value of hard reinforcement particles. Lower elongation was observed that is related to the brittle characteristic of Al₂O₃.

Table 5

Compression strength and elastic modulus of the pure Al7075 and Al7075/20 wt.% $Al_2O_3.$

Material	Elastic modulus (GPa)	Compression strength (MPa)
Al 7075 20 μm Al 7075 63 μm	72 76	173 187
Al 7075 20 µm–Al ₂ O ₃ 120	97	327
μm Al 7075 20 μm–Al ₂ O ₃ 5 μm Al 7075 63 μm–Al ₂ O ₃ 5 μm	82 78	285 264

-5 wt. % Al-O 20 wt. % Al-O 350 -Pure Al 7075 10 wt. % 300 250 Stress (MPa) 200 150 100 50 0 0 5 10 15 20 25 30 35 40 45 50 55 Strain (%)

Fig. 10. Compression curves of the 20 μm Al 7075/120 μm Al_2O_3 composites prepared under 80 MPa semisolid compaction pressure.

Elastic modulus of the metal matrix composites is dependent on several factors such as reinforcing particles weight percentage, elastic modulus, size and shape [56]. Among them, the elastic modulus and reinforcing phase ratio can primarily enhance the elastic modulus of the composite, while the effects of particle shape, size and tilt angle on composite's modulus is relatively minor [57]. As shown in Fig. 10, the slope of the initial linear portion of the curves increased with increasing the reinforcing weight percentage. Similar trend has been reported for the extruded aluminum matrix reinforced with SiC particles [58].

3.7. X-ray diffraction analysis

Due to the existence of Al, Zn, Mg, and Cu elements in the composition of the blended Al7075, identification of the metallic and intermetallic phases is essential for the evaluation of the mechanical and physical properties of the sintered samples [59]. Semisolid temperature for all the experiments was 610 °C, where 25 wt.% of Al7075 is liquid. To determine different phases in the ball milled and the semisolid sintered samples, XRD was performed. XRD spectrum obtained from the ball milled 20 μ m Al7075 reinforced with 10 wt.% 120 μ m Al₂O₃ shows only Al peaks, as illustrated in Fig. 11.a. Apparently, this is referred to the amorphization of Al₂O₃ micro particles in ball milling process [60]. However, three different peaks of Al, Al₂O₃, and Al₂MgO₄ were identified after the semisolid compaction process, as seen in Fig. 11.b. Aluminum oxide, Al₂O₃ is stable in pure aluminum but reacts with Mg in Al-Mg alloys as [61]:

$$3 Mg + Al_2O_3 \rightarrow 3 MgO + 2 Al \tag{3}$$

and

$$3Mg + 4Al_2O_3 \rightarrow 3Al_2MgO_4 + 2Al \tag{4}$$

The magnesium equilibria for Eqs. (3) and (4) have been calculated and show that MgO may form at high magnesium levels and low temperatures, whereas spinel will form down to very low magnesium levels [62]. It was reported that magnesium plays an important role to break up the stable alumina layers and make a liquid aluminum. Magnesium can help to break up the alumina layers on the surface of the aluminum powders through the formation of a spinel phase Al_2MgO_4 [63]. It is concluded form the reactions occurred during semisolid compaction of 20 μ m Al 7075 reinforced with 10 wt.% 120 μ m Al₂O₃ that Al₂MgO₄ spinel phase broke the aluminum oxide surfaces on the surrounding of Al powders and more liquid phase extracted from the matrix phase and consequently, bonds between matrix and reinforcement phase were stronger. Good mechanical properties of the 20 μ m Al 7075 reinforced with 10 wt.% 120 μ m Al₂O₃ could be related to the spinel phase formation during semisolid compaction.



Fig. 11. X-ray diffraction spectra of (a) ball-milled 20 μ m Al7075/10 wt.% 120 μ m Al₂O₃ and (b) semisolid compacted 20 μ m Al7075/10 wt.% 120 μ m Al₂O₃ under 40 MPa.

4. Conclusions

The blended powder semisolid forming of Al7075/Al₂O₃ composites was studied. The effects of various experimental conditions, i.e., particle size, reinforcing phase percentage, and compaction pressure on the microstructure, morphology, and physical and mechanical properties of the composites were thoroughly investigated. Some of the conclusions are listed below:

- Three compact mechanisms were identified for the semisolid forging of Al 7075/Al₂O₃ composites. In the compaction of 20 μm Al7075/ 120 μm Al₂O₃, Al₂O₃ particles were uniformly distributed within Al7075 particles. In the case of 63 μm Al7075/5 μm Al₂O₃, two different regimes were observed: uniformly distributed Al₂O₃ particles and agglomerated Al₂O₃ areas.
- Large reinforcing particles resisted the solidification shrinkage, and resulted in some micro cracks in their microstructure. Smaller reinforcing particles appeared to easily adjust and form new arrangement, preventing micro cracks.
- 3. The highest hardness was achieved when the large reinforcement particles (120 μ m) were added to the small matrix particles (20 μ m). The hardness decreased when the particle size ratio was decreased (i.e., 20 μ m Al 7075 and 5 μ m Al₂O₃) or the matrix particle size was larger than that of the reinforcement (i.e., 63 μ m Al 7075 and 5 μ m Al₂O₃). This behavior was attributed to the uniform distribution of large reinforcing particles in the microstructure.
- 4. In 20 μm Al7075/5 μm Al₂O₃ composites, tiny Al₂O₃ particles acted as a barrier, preventing sufficient inter-diffusion of the matrix particles. In 63 μm Al7075/5 μm Al₂O₃ composites, the agglomeration of reinforcing particles appeared between the matrix particles and resisted the liquid phase infiltrating into the free spaces; consequently, the porosity increased.

- 5. Applying more compaction pressure resulted in better liquid phase filling of the empty spaces and thus improved both the density and hardness of the composites.
- 6. After 3 h of mechanical mixing, no new phase was created within the samples. Al₂MgO₄, in semisolid form, was formed through the reaction of magnesium with aluminum oxide.
- 7. Highest compressive strength improvement of about 89% was achieved for 20 μm Al7075 matrix particles with the incorporation of 20 wt.% 120 μm Al₂O₃ particles.

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