Investigation of the effect of SiC, TiC and TiB2 particles on the microstructure and mechanical properties of aluminum under the local laser melting influence

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ABSTRACT

Powders of Al-SiC-TiC-TiB2were mixed in two different ways: air mixing and ball milling, and the surfaces were melted using a beam of local laser melting under dif- ferent laser voltages after compaction. The microstructure, elemental distribution, and microhardness of the sample Al-SiC-TiC-TiB2 composites for each of the sam- ples prepared using different methods were studied after laser melting. In addition, the microstructure and elemental distribution of the ball-milled powders prepared at 15, 30, and 60 min were studied. A long needle-shaped phase was formed in a sample prepared by ball milling. At the high laser voltage of 300 V, the sample prepared via ball milling exhibited a high microhardness of 197 HV.

The Manuscript contains 26 pages, 32 figures, three tables, and 41 references.

INTRODUCTION

Aluminum alloys have been increasingly used owing to their advantages, such as high mechanical strength, ease of processing,good workability, ductility, good thermal conduction properties, light weight, high reflectance, and low temperature resistance. Owing to these features, aluminum alloys have received increasing attention in the construction, automobile, and aviation industries. Alternatively, aluminum matrix composites can be used owing to their low density, high specific stiffness, high wear

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resistance,hardness, and high thermal conductivity. New minimal-effort-to-low-cost samples that mimic those produced using additivemanufacturing techniques can be easily prepared and examined by laser surface melting. This could be attributed to the high melting/cooling rateand the faster solidification during the processing of the target powders, which also led to the homogeneous dispersion of the reinforcements in the matrix.

The goal of this project is to evaluate the impact of SiC, TiC, and TiB reinforce- ment particles on both the microstructure and mechanical properties of aluminum produced by local laser melting, with the aim of achieving the best results using laser techniques.

Analytical Literature Review

The literature review here focuses on classical ways to fabricating of metallic material composites, mainly highlighting metal matrix composites (MMCs) and what they consist of. Because of this, paying attention to ceramic reinforcements, mainly aluminum and its alloys, is important. The review also deals with aluminum metal composites and aluminum-silicon alloys, describing microstructure developments during their processing for better performance. The effectiveness and impact of carbide ceramic reinforcement on both the mechanical properties and overall strength of aluminum-based composites were described.

Composite Materials

Composite materials have for a long time been employed for the solution of various technological problems. Recently, with the introduction of polymer-based metal matrix composites, such materials have caught special attention of the industries. For producing a processing property which cannot be achieved by a single material, composite materials can be made from two or more materials. [1] The concept behind composites is mostly about the matrix materials. The fibers, particles, or whiskers used for the reinforcement of the composites are ceramics, metals, or polymers, and further the matrix materials are ceramics, metals, or polymers. The reinforcements can be made from metals, ceramics and/or polymers.

When compared to the feature of the component elements alone, MMC, with their unique makeup of ductile metal and nonmetallic ceramic, have much better and advanced traits [27]. How the composite behaves is mostly decided by the compatibility between the matrix and the reinforcement. The choice of reinforcement is very important in the use of MMCs because it determines the results. Rigid mixing of SiC particles and aluminum happened through microwave sintering and hot extrusion [27]. Its low weight and strong mechanical properties make sure aluminum is the most common matrix material used today. Figure 2 illustrates, a range of metals used as matrices.

Metal Matrix Composites (MMC)

Metal-matrix composites have fibers or particles added to a solid metal such as steel, copper or aluminum. Metals (especially steel) or ceramics (silicon carbide and alumina, for example) make up the secondary phase in most cases [2].

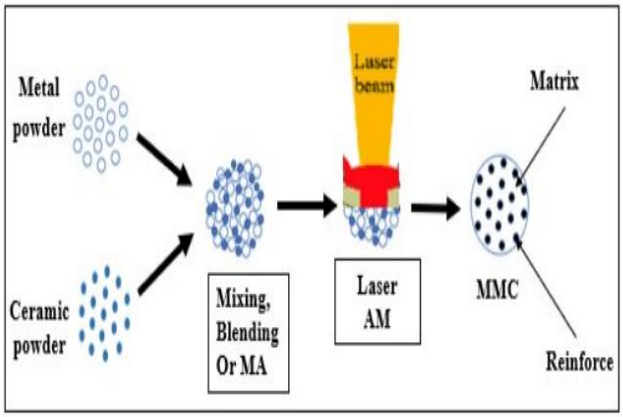

Figure 1 - A schematic of fabricated MMC and its processing steps [14]

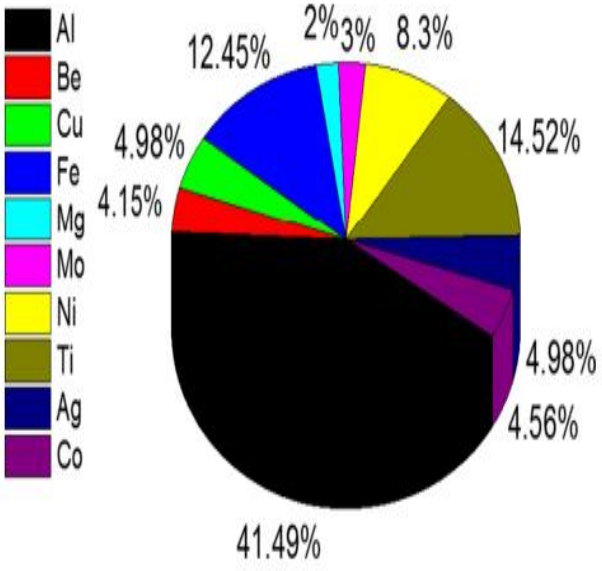

Figure 2 - Usage of various elements as a metal matrix [27]

Types of Metal Matrix Composites (MMC)

To gain a better understanding of metal

matrix composites, it is better to discuss the types of metal matrix composites. Here are the types of metal matrix composites that are divided into three categories based on the presence of reinforcement particles [2, 3, 27].

Continuous fiber reinforced metal matrix composites

These materials are made by putting continuous fibers which all point in one way, into metal matrices made from either alumina or silicon carbide. Because of their alignment, fibers obtain greater strength and stiffness in their direction which allows them to be used where extreme force and stiffness are needed.

Short fiber (Whisker) reinforced metal matrix composites

Inside the metal part, the whiskers or short fibers are usually mixed randomly or placed in one direction. Most of the time, they are built from silicon carbide and give stronger isotropic properties.

Particle reinforced metal matrix composites

When particles of silicon carbide or aluminum oxide are evenly inserted in a metal matrix, it makes these composites stronger, more capable of wear resistance and harder.

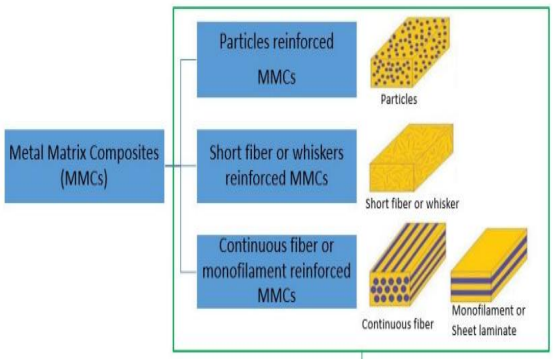

Figure 3 - Metal matrix composite reinforcement based schematic description [14]

Classical production Technologies of Metallic Composite Materials

A variety of procedures were used to make metallic material composites and these are discussed in detail in this literature review. These operations are grouped based on the temperature at which metals are processed, the stage of materials in the process, the types of processes used and particular techniques [4]. For this reason, the approved procedures are sorted into four group [6 9].

These are:

- Liquid phase fabrication method of metallic material composite;
- Solid phase fabrication method of metallic material composite;
- Deposition fabrication method of metallic material composite;
- In¬-situ processes.

Liquid phase fabrication method of metallic material composite

In this process, a dispersed phase is mixed into the liquid metal matrix which then solidifies during the process. To get high mechanical properties in a composite, the dispersed phase and liquid matrix need to bond and "wet" each other. Applying a layer on the dispersed phase's particles (the fibers) can improve the wetting. Such coating creates a separation barrier that prevents chemicals from getting in contact and reduces the energy present between the layers. Some of the methods involved in fabrication of metallic material composites with the liquid phase are discussed in in this literature.

Stir casting

The way the reinforcements are put into molten metal and then cast in the liquid form [38]. The technique is relatively easy and also the cheapest because ceramic particles or short fibers are mixed with molten metal using a chemical string, as demonstrated in figure 4. Standard casting was used to form the liquid composite into molds and after that, standard metal forming technology was applied to the item.

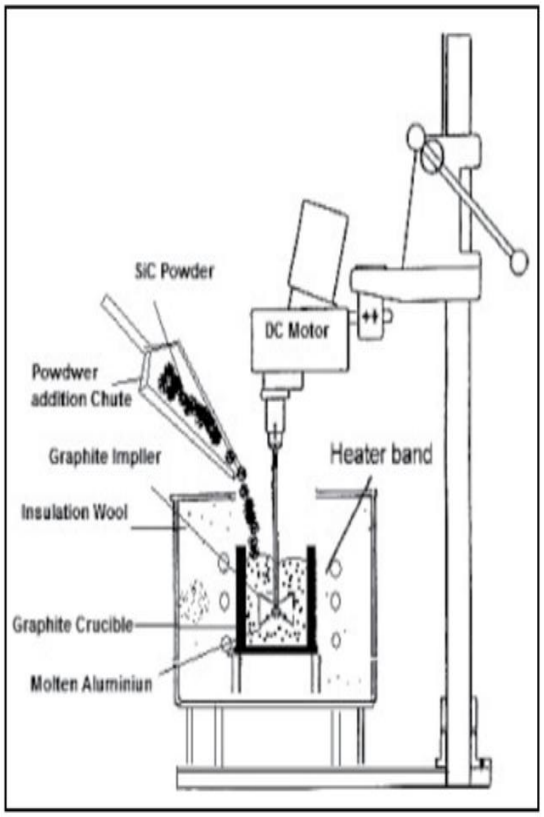

Figure 4 - Stir casting by Vortex method [4]

Infiltration

A prepared dispersion phase (ceramic particles, fibers, or woven materials) is soaked in a molten matrix metal, which fills the gaps between the dispersed phase inclusions, in this liquid state processes of creating composite materials. The capillary force of the dispersed phase (spontaneous infiltration) or an external pressure (gaseous, mechanical, electromagnetic, centrifugal, or ultrasonic) applied to the liquid matrix phase (forced infiltration) can be the driving factor behind an infiltration process.

One of the most important of this it is gas pressure infiltration. This liquid phase method of creating metal matrix composite is called gas-pressure infiltration. As illustrates in figure 5. It uses a pressurized gas to impart pressure to the molten metal forcing it to enter a prepared dispersed phase. Large composite pieces are made using the gas pressure infiltration processes.

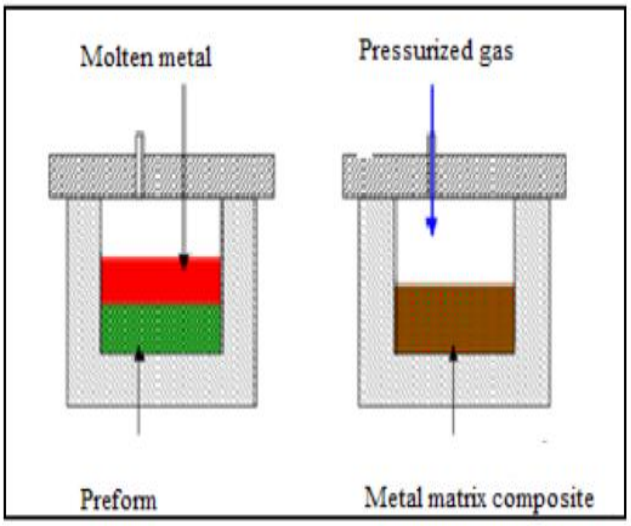

Figure 5 - Gas-pressure infiltration [5]

Squeeze casting

This involves the steps of forging and casting to apply pressure which reduces the air holes that form while the item solidifies [38]. With this process, melted metal is poured into a mold that is already filled with fibers or particles. When pressure is applied during solidification, the reinforcement and matrix bind closely which makes the metal stronger. Such a method is best for making small components like automotive engine pistons. In this case, pressure from a mobile mold makes the molten metal break into and fill, the dispersed phase which is then tucked into the stationary lower mold part, as depicted in figure 6.

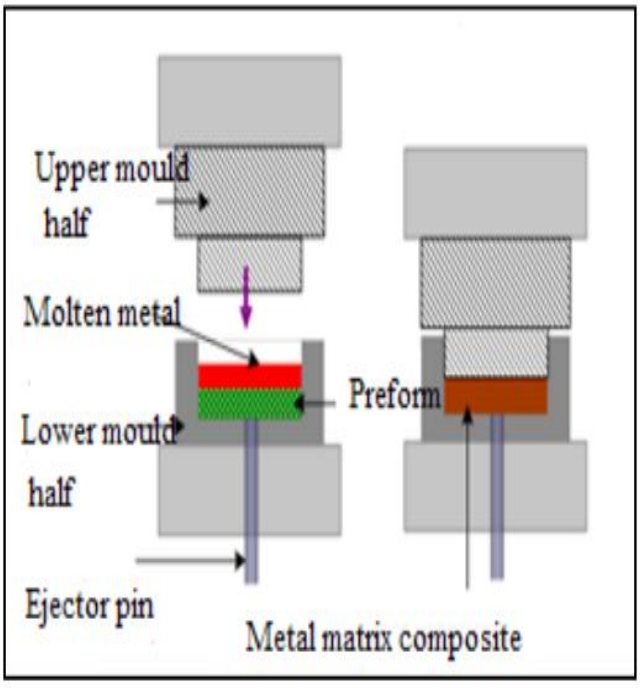

Figure 6 - Squeeze casting [5]

Solid phase fabrication method of metallic material composite

With solid phase fabrication, metal matrix composites are formed by bringing the matrix metal and dispersed phase together due to their mutual diffusion at a high pressure and temperature in solid form [6-9].

Diffusion Bonding

The technique of diffusion means stacking a layer of foils and fibers together, both in a definite way and then heating them under a great amount of pressure. The completed composite material had different layers in the final product. Basic shapes such as tubes and plates are made by using diffusion bonds. Diffusion bonding has two types that are applied differently. In order to create the first type, metal wires and continuous ceramic fibers are wrapped into a shape and pressed at a high temperature afterward. The second method is referred to as roll bonding. It requires placing two metals such as steels and aluminum alloy inside a roller to join them, either heated or not. It resulted in the formation of a composite material where both layers are bonded by metallurgy. Figure 7 illustrate diffusion bonding process.

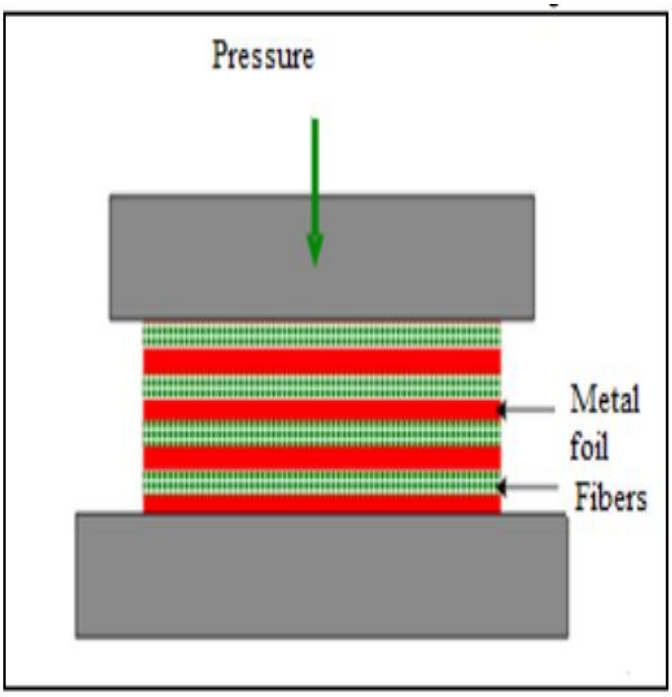

Figure 7 - Diffusion bonding [5]

Sintering

The method of sintering metal matrix composite involves combining a matrix metal powder with a dispersion phase powder in the form of particles or short fibers for later compacting and solid-state sintering.

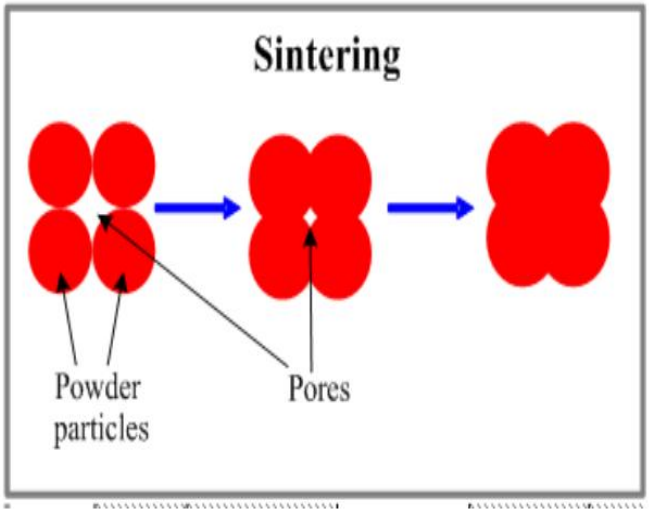

Figure 8 - sintering [5]

Powder Processing or Powder Metallurgy

Solid-state manufacturing includes blending of metal and reinforcement particles, compressing into ingots and sintering them [38]. With the help of deformation processing, particle or short fiber-reinforced composites are made using this technique. To make these composites, typical methods are hot pressing or cold pressing of sintering which uses mainly particle- or whistler-reinforcement. To make reinforcement even across the structure, the reinforcing powder was mixed with the matrix. At this point, the clay is cold pressed to make a dark green form that is about 80 % solid and very manageable. After cold-pressing, the green body was canned and degassed to make sure there was no moisture left from the particles. Through hot pressing, uniaxial pressing or astatic pressing, the material is made fully dense and then it is extruded [6]. The scheme for powder metallurgy is shown in figure 9.

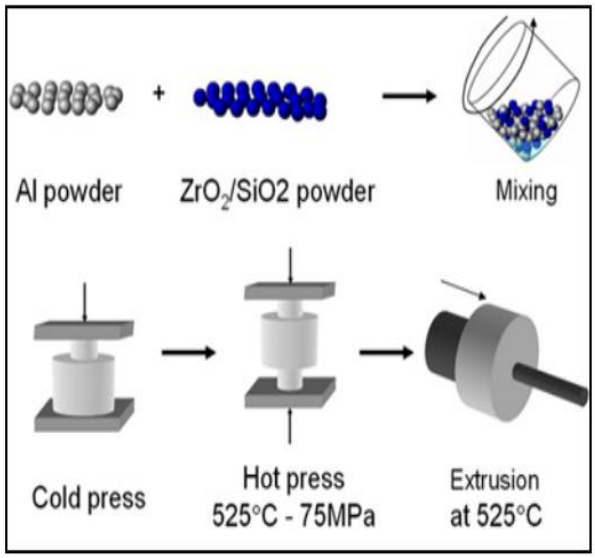

Figure 9 - powder Metallurgy [5]

Mechanical alloying methods

Metal matrix composites (MMCs) and alloys are created with a powerful solid-state powder process called mechanical alloying (MA). The reinforcement was evenly blended into the polymer matrix by using ball milling at a high energy. When a powerful ball mill is used, the powder particles go through a cycle of deforming, fracturing and bonding with each other [38, 39]. Applying this process may produce alloys and composites that look the same and have special microstructures and properties.

Deposition Techniques

Molten metal and reinforcing materials are delivered to a substrate as a spray and they harden there. You can produce products with special microstructures and tiny grains by using this technology [7]. Nonetheless, it suffers from some issues, for example, areas that do not have the same strength, reduced possibilities for designs and expensive building materials.

This technique includes:

a. Physical vapor deposition (PVD);

b. Chemical vapor deposition (CVC);

c. Spray deposition (SP).

Physical vapor deposition (PVD)

Physical vapor deposition is a special process used to deposit thin films and coatings. To use this method, the metal is vaporized at a high temperature, then plasma is created in a high vacuum and finally the surface you want to coat is exposed to the metal in low pressure [8]. Because the process requires a lot of time, this method is not suitable when you want to apply a coating with very small sizes to MMCs.

Chemical vapor deposition

Solid materials with high performance and quality are obtained through the Chemical vapor deposition process by having their substrate placed in vacuum and affected by the breakdown of several volatile chemicals [9]. By interacting chemical sprays with gases, Chemical vapor deposition results in non-volatile solid nanomaterials on the chosen surface. The way material can be deposited from different angles is a feature that makes Chemical vapor deposition special.

Spray deposition

The method sprays tiny, strong and hot drops of molten material onto a fixed metal surface with the help of an inert gas jet [10]. Short fibers, particles or whiskers are added into the spray mixture.

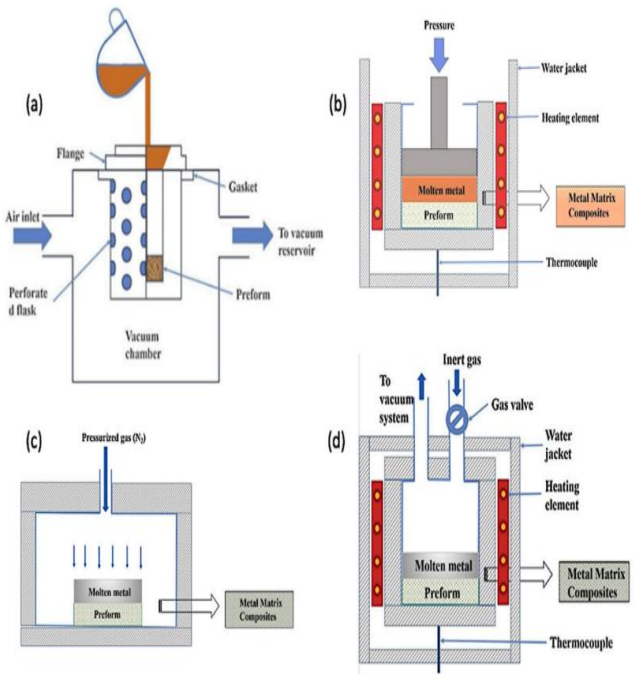

Figure 10 - Schematics of infiltration variants depending on the driving force; (a) Melt infiltration, (b) Pressure infiltration, (c) Gas pressure infiltration and (d) Vacuum pressure infiltration [8]

In-situ Processes of metal matrix composites

Since Metal Matrix Composites (MMCs) are manufactured directly in the metal matrix, there is a clean bond between the reinforcement and the matrix which increases the strength of the material. The process grinds powder particles together in a ball mill which helps to weld, divide and weld them [11]. In comparison to the traditional way of making composites, in-situ composites have better overall strength and structure. In situ metal matrix composites get their advantages from being manufactured with the additives inside the melt. Because of this, their ceramic-metal surfaces are clean and pure, their ceramic particles have a stable size and the total process is less costly [12]. In situ MMCs have multiphase materials, where the reinforcing phase is produced in the matrix during the reaction between the reinforcement and the matrix [13]. The process of in-situ synthesis of MMCs makes it easier for both types of materials to react and become well distributed throughout the matrix [15].

Particulate based in-situ metal matrix composites

These composites have fine particles uniformly spread inside the metal matrix. For example, aluminum matrices reinforced with titanium boride (TiB_2) particles or magnesium matrices reinforced with Mg_2Si particles [22, 24].

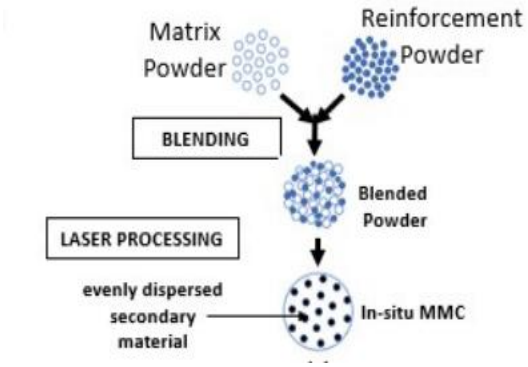

Figure 11 - A schematic representation of in-situ MMCs [13]

Short fiber based in-situ metal matrix composites

In short, short-fiber reinforced in-situ MMCs are formed by adding fibers during the making of the metal matrix. With this method, materials may bond strongly at the interface and could have improved mechanical properties when compared to other forms of metal matrix composites. This is a case where reinforcement comes from whiskers grown on site. Some examples are titanium matrices that contain TiB_2 and aluminum matrices that have titanium aluminide ($TiAl_3$) [22, 24].

Long fiber based in-situ metal matrix composites

Long fiber-reinforced in-situ metal matrix composites are produced by including reinforcement fibers that are built during the making of the composite. This means that, with ex-situ forms, fibers are made beforehand and then built into the matrix. Some of the benefits of in-situ MMCs are improved contact at the material interfaces, a uniform spread of reinforcing materials and a reduced cost for construction or manufacturing. These composites contain continuously produced fibers because the NiAl matrix in this example is reinforced by continuous Molybdenum fibers [22, 24].

Reactive process

Using chemical interactions between liquid and gas, liquid and liquid, liquid and solid, and mixed salts, reactive methods create small ceramic particles inside the metal matrix without the need for external reinforcements. In-situ processing means adding reinforcements by making them or new phases form through chemical reactions between the key components while the material is being processed. When making metal matrix composites (MMCs), small particles of titanium carbide (TiC) are made in the metal by adding a carbon source and mixing it with titanium before pressing into shape. The implanted particles are uniform and well attached to the surrounding matrix because this approach relies on reactions. The processes do away with the need to add reinforcement particles, and this allows for more straightforward and better working composites. For example, silicon dissolved in copper may be converted into a dispersion of silica particles by

reacting with oxygen that diffuses from the environment []. The two components comprise the reactive-type process, which forms the reinforcing phase through an exothermic reaction [12,13].

Non-reactive process

When Metal Matrix Composites (MMC) are made, there is no chemical reaction between the metal and reinforcement materials and these materials are smoothly mixed in or added via transforming into a phase. In this kind of process, nothing is broken into chemicals or combined with other chemicals. Alternatively, the way new phases or structures are formed is through phase transformation, precipitation, or segregation. Precipitation of Al_3Sc particles during purposeful heating of aluminum alloys is an example of this. These particles appear when supersaturation happens, and the mixture separates into parts without forming new substances. Precipitation-strengthened alloys frequently use in-situ non-reactive processes, trying to achieve greater strength by adding finely distributed secondary phases. [12, 23].

Table 1 Summary of some fabricated MMCs through various processes with their matrix and reinforcement [38]

MMC Type	Matrix Material	Reinforcement	Fabrication Process
Al–SiC	Aluminum	Silicon Carbide (SiC)	Stir Casting, Powder Metallurgy, Squeeze Casting
Al–TiC	Aluminum	Titanium Carbide (TiC)	Stir Casting, In-situ Reaction, Powder Metallurgy
Al–TiB_2	Aluminum	Titanium Diboride (TiB_2)	In-situ Reaction, Stir Casting, Powder Metallurgy
Al–B_4C	Aluminum	Boron Carbide (B_4C)	Powder Metallurgy, Hot Pressing
Mg–SiC	Magnesium	Silicon Carbide (SiC)	Stir Casting, Squeeze Casting
Cu–Graphite	Copper	Graphite	Powder Metallurgy, Sintering
Ti–SiC	Titanium	Silicon Carbide (SiC)	Powder Metallurgy, Hot Isostatic Pressing
Al–SiC–Graphite (Hybrid MMC)	Aluminum	SiC and Graphite	Stir Casting, Powder Metallurgy
Al–Al_2O_3	Aluminum	Aluminum Oxide (Al_2O_3)	Stir Casting, Powder Metallurgy
Al–CNT (Carbon Nanotube)	Aluminum	Carbon Nanotubes (CNTs)	Powder Metallurgy, Mechanical Alloying

Ceramic reinforcement for Al/Al-alloys

The reason SiC, TiC and B4C were picked for Al-MMCs is that they are very hard substances and resistant to chemical changes which increases the resistance of the MMCs [18]. Nevertheless, silicon carbide dissolves gradually into liquid aluminum to form different intermediate compounds that depend on the temperature [13]. Al4SiC4 changes to Ternary Al4SiC4 at 1670 K,and the formation of Al4SiC4 takes place between 940 K and 1620 K [18]. The fact that the final particles can be easily broken and react with water is the reason it is undesirable for Al4C3 to appear [13, 14, 18]. This means that high temperatures such as 1670 K [19], are needed for processing SiC-reinforced Al-MMCs. When nano-TiC particles are added to AlSi10Mg MMCs, TiC and Al9Si form, as well as Mg2Si which lowers the metal matrix composite's coefficient of friction and wear rate [19]. Even though carbides of tungsten normally dissolve in Al, metals such as SiC and TiC often lead to the development of Al4C3 which is toxic.

It is clear that WC is more inert than TiC, but TiC has a greater inertia than SiC when dealing with aluminum. All the same, TiC has the least tendency to react with Al out of the three carbides and dissolves in Al much more slowly than SiC and WC [13, 20]. Carbides are commonly used reinforcements for various kinds of metals in laser processing when it comes to the behavior of ceramic reinforcements [13]. At the same time, reaching successful LAM processing usually means paying close attention to the development of Al4C3 in Al-MCs (which is prone to breaking and becoming irritable in moist environments). Also, even using oxides for reinforcement, the formation of an oxide layer can be an issue that often calls for extra attention in choosing the fabrication process [18, 21]. Ideas to replace carbides with nitrides and borides have come up due to these two being more chemically and mechanically stable. Moreover, adding rare-earth oxide nanoparticles is said to enhance the metal matrix composites (MMCs) quality by boosting both mechanical and corrosion features [18].

Aluminum-Silicon alloys

Aluminum–silicon alloys which are called Silumin, have a main composition of aluminum and silicon. Without copper or magnesium, pure AlSi alloys cannot be hardened, but their inclusion makes the alloy respond the same as do AlCu and AlMgSi alloys [30]. AlSi alloys are especially valued among aluminum casting materials since they have strong casting properties and work well in different casting methods [30, 31].

For example, Catatonic systems are widely applied in car parts, in engine blocks and pistons. Experts are studying thermochemical as useful materials that allow hot storage in electric vehicles.

Carbide ceramic reinforced Al-based alloys

Since aluminum material with carbide ceramics enhances their properties and reduced weight, a lot of attention in research has been given to these alloys. Adding ceramic particles to aluminum makes them more resistant to wear, strong and tougher which is why they are used for many types of structural, automotive and aerospace parts. Scientists have carried out many investigations on using SiC particles in Al alloys. Due to low density, extreme hardness and good thermal stability, SiC supports the overall improvements of the composite. It has been found that adding SiC to an aluminum composite notably increases its strength [35]. For example when SiC particles were added to aluminum, an effective load transmission between the reinforcement and matrix resulted in a finer microstructure and improved mechanical properties. Also, the presence of TiC particles in aluminum alloys has been appreciated for making the alloys more durable and improved for mechanical performance [35]. One reason TiC is used as reinforcement is because of its great melting point and durability. Basing on research results, incorporating TiC in aluminum improves its heat resistance which is very useful in laser melting [32]. Having TiC particles makes the microstructure more similar and increases the performance of the material. Similarly, TiB2 has shown it may be a useful addition to aluminum composites. Adding TiB2 particles makes the reinforcement and aluminum matrix stick together well and improves how well they mix [33]. Adding TiB2 to aluminum composites boosts their hardness and tensile strength. The reason was that the load was carried well and there was an advanced microstructure. How the microstructure of carbide ceramic reinforced aluminum alloys changes as a result of local laser melting is important to study in order to grasp the alloys' final mechanical behavior. Using laser melting, the fast-cooling process helps create small grains and equal distribution of enhancing particles. It is very important for the microstructure to improve so that the finished composite material has proper mechanical properties. All in all, reinforcing aluminum-based alloys with ceramic carbides such as SiC, TiC and TiB2 results in much better mechanical properties. If we add laser melting to the picture, metal reinforcements offer exciting chances to develop aluminum matrix composites that excel in tough circumstances [32, 33, 35].

Microstructure Development

Laser Melting effect on microstructure

The repeated heating and cooling of localized laser melting can improve the quality of the material's microstructure. Laser cooling at a high rate may create small grains and help

reinforcement particles spread evenly in aluminum. At the same time, getting the right mechanical properties is important, as stronger materials are normally co-subjected [34].

Role of reinforcing particles

The structure of the resulting material can be changed a lot by using reinforcing particles during laser melting. Because the particles in the metal are stressful to its structure, new grains may appear between them. Adding SiC to aluminum may cause it to resist heat better which should result in more effective mechanical features. In addition, adding TiC and TiB2 particles improves the bond between the matrix and the reinforcement and makes it more wettable which alters the microstructural properties [32].

Impact on mechanical properties

The addition of strengthening particles and melting the laser with the laser affects the overall mechanical properties of the composite in a direct way. Evidence reveals that advanced microstructured composites are harder and more resistant to tensile forces than their simple structured counterparts. Better connections formed by the matrix with the strong particles also lead to better load bearing capabilities and performance [34, 35].

Solidification Mode

As a phase transition during solidification, the solid-liquid interface is responsible for organizing the solute that appears in the form of crystals when the temperature is reduced [37].The process of cooling and solidifying aluminum matrix composites (AMCs) has a strong impact on their final structure and mechanical properties. How aluminum forms after local laser melting is mainly influenced by how fast it cools, the temperature differences within it and the process of melting the particles. Most of the time, aluminum alloys are solidified by either equilibrium or non-equilibrium solidification. When a material cools gradually while in equilibrium solidification, the structure often grows stable with obvious grain boundaries. When melted with a laser, the material's fast cooling leads to non-equilibrium solidification and creates smaller structures as well as may help make metastable phases [36].

The process of solidifying aluminum composites has a major impact on their microstructure. Laser melting is a technique that cools very quickly which helps to limit the development of bigger dendrite structures and produce a fine-grained end product. Many studies have linked how strong and hard some materials become to their small microstructures [34]. Adding SiC, TiC or TiB2 particles may influence the solidification of the material, increase the number of ultrafine grains and raise the overall strength [35]. The presence of SiC, TiC and TiB2 particles in the matrix helps change its solidification mode and its thermal and physical features. Researchers have confirmed that including TiB2 particles in alloys can make aluminum grains more uniform while they solidify. In the same way, SiC and TiC can make the composite more heat conductive which may impact solidification and the amount of reduction [32].

Aluminum matrix composites get their microstructure from the way they are cooled down during manufacturing. Because of rapid solidification, the resulting grains are finer, there are less pores and the bars of reinforcement are more evenly spread. Strength and reduced wear are made possible in steel by having certain microstructural features in the metal [32-36].

MAX phase

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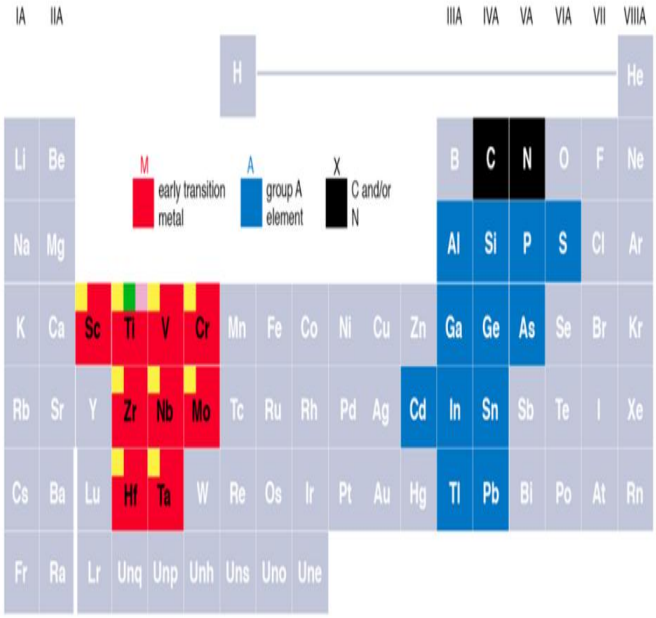


Figure 12 - shows periodic table of elements that are suitable to form MAX phase

The fact that MAX phases are a combination of primary and early secondary is a very interesting trait. They are easy to machine, conduct electricity and heat and can bend or deform under stress, as is usual for metals and not most ceramics. They still have several qualities that are like those of ceramics such as extreme stiffness, great thermal stability, resistance to oxidation and hardness. Since MAX phases can store energy and hinder cracks from spreading, they can be used successfully in challenging mechanical and thermal conditions [41]. It is the crystal unit cells which define the individual layers that give MAX phases their special structure. These phases consist of transition metal carbides and nitrides with hexagonal structure and layer after layer of these elements as shown in figure 14 [41]. The known MAX phases in Ti-Al-C and Ti-Si-C system are Ti2AlC, Ti3AlC2, Ti3SiC2 and Ti4SiC3 with $M_{n+1}AX_n$ value (211, 312, 312 and 413) respectively, where the n value indicates the number of M layers between two A layers [41]. This means there are two layers in M2AX, three layers M3AX and four layers in M4AX as shown in figure 14 [41].

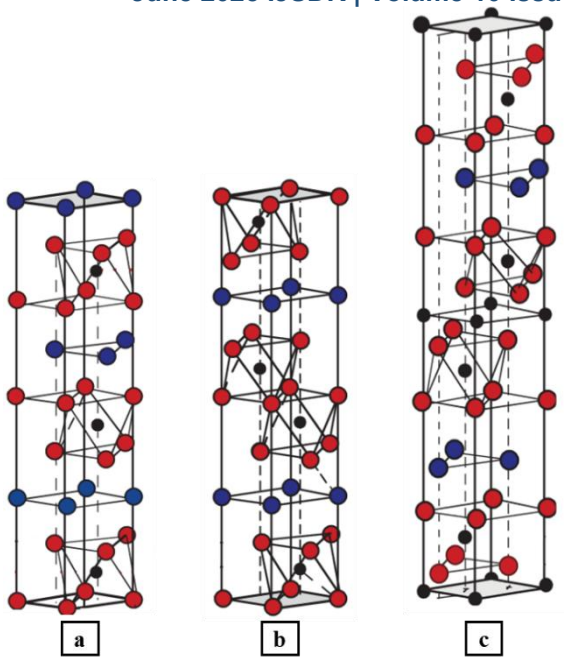


Figure 13 - Unit cells of (a) 211, (b) 312, (c 413 MAX phases. Red balls: M, blue balls: A, black balls: X

Materials and methods

Materials

Table 2 shows the chemical composition of elements that was used for aluminum composite production in weight percentage.

Table 2 Chemical composition of the used aluminum composite at weight percentage (%)

Al Alloy	TiC	SiC	TiB_2
72.5	9.1	10.5	7.9

Figure 14 illustrates the SEM images of reinforcement used powders. Light gray SiC powder quite irregular in shape and black granular shape TiC powder with powders average particle sizes of 4.56 and 3.07 μm, as shown in Fig. 14 (a) and 14 (b), respectively. The powder of TiB2 is relatively large compared to the other two, and quite blocky, and has a bimodal distribution with an average particle size of 317.4 μm, as illustrated in figure 14 (c).

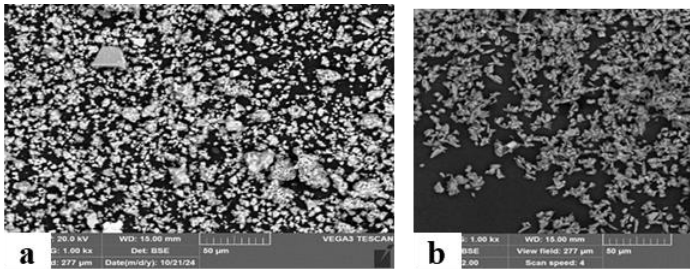


Figure 14 - SEM images of reinforcement used powders a) SiC, b) TiC and c) TiB_2

Air Mixing

Samples with same composition was prepared after the weight of the powders was measured. Figure 15 shows how to use a mixing tube set as well as the parts it includes. The tube is made of air mixing, stands vertically and has filtering papers sealing both its tops and bottoms, about 50 cm long. The air is pushed into the tube with the help of a (Smart Dari 50) compressor. A mixture of powders was formed in the mortar by air mixing with the tube for about 10 minutes until they were mixed well.

Ball Milling

The jars and balls were made tougher than the powders to prevent contamination and damage during the milling process. Such parts are made with hardened steel, keeping them safe from contamination and making them hard. Granules of powdered composite materials were obtained using a planetary ball mill at a rotational speed of 500 rpm of the planetary disk and a rotational speed of 1175 rpm of the drums. The ratio of ball grinding media with a diameter of 5.5 and 11 mm was 1:1 for 350 g each. The processing time of the powder compositions was 60 minutes.

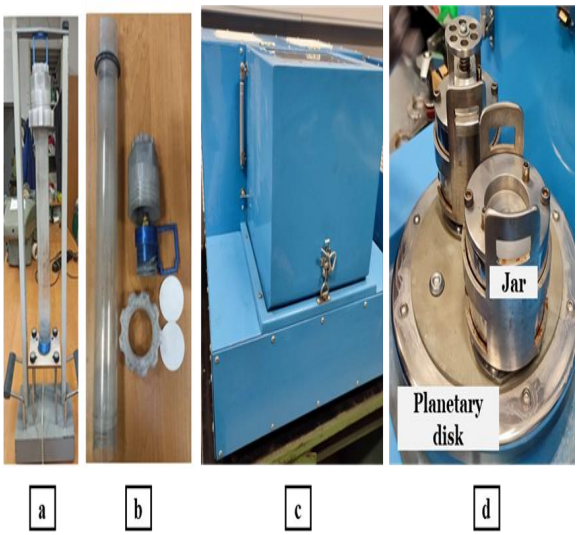


Figure 15 - shows (a) The mixing tube set and (b) the components of the mixing set which was made locally at NUST, MISIS University, to the left (c) is planetary ball milling equipment and (d) are a pair of jars and planetary disk which are located inside the equipment.

Compacting

After cleaning the walls of a 25 mm diameter die, 5 grams of each mixture was added. The powder was then compressed uniaxial at 40 metric tons, or 5 tons per second on average. Ten seconds was the dwell period at the highest pressure before the pressure abruptly plummeted or suddenly dropped to zero. According to [40], a quick reduction in pressure on these kinds of powder combinations prevents sample cracking during ejection by applying a small amount of pressure to the green compact. To get the satisfactory samples, with the sufficient strength for handling and further processes and uses of the samples, the green compact and die was then transferred to furnace (SNOL 1, 6. 2, 5. 1/9-I5, Russia) at 400 °C for 20

minutes.

Following the brief sintering, the top punch was taken out. Following the removal of top punch, the sample was ejected by downward movement of the floating die body. For this purpose of compacting, a 5-ton electric hydraulic press (Nordberg N3650E, Russia) was utilized.

Polishing

The samples were polished following to compacting, using a metallic sample polishing machine (SDEKON PG-2DA) Shandong Province, China for about 15 minutes each using water proof SiC polishing papers. Those papers were 320 (46μm), 800 (22μm), 1200 (15μm) and 2400 (8μm) respectively.

Laser Melting

Laser melting was carried out with an Nd:YAG laser which had a wavelength of 1064 nm, a (MUL-1-M-200 system from Russia), a laser power between 250 and 350 V, a pulse duration of 12 milliseconds, a scanning speed of 0.25 millimeters per second and an overlap of 0.2 millimeters. High-purity argon (grade 5.5) was used to protect from oxidation formation. From the striking, tiny tracks were created according to the desired parameters as summarized in table 5.

Table 3 Laser melting parameters

Parameters	**Values**
Voltage	250V, 300 V, 350V
Pulse duration	12 ms
Pulse frequency	5 HZ
Shielding gas	Argon
Preheating temperature	200 ^{0}C,400 ^{0}C
scanning speed	0.25 mm/s
Feed overlapping	0.2 mm

After the surface of the samples were melted by laser melting process and cooled down, the created tracks were polished for 5 minutes using papers 800 (22μm), 1200 (15μm) and 2400 (8μm), 4000 (5 μm) and in a 20 % water solution of colloidal silica-based suspension for further investigations of microhardness and microstructure.

Investigation of Microstructure

Optical Microscope(OM): In this work, the tracks and microstructures of the polished samples were examined under polarized light at different magnifications using Optical Microscope (OM), Axiovert 200MMAT (Carl Zeiss, Obesrkochen, Germany).

The Scanning Electron Microscope (SEM) uses focused electrons to look at a sample's surface, producing clear images that show structure, different phases and the material ingredients used. In this work, Scanning Electron Microscope (SEM) TESCAN VEGA 3LMH (Tescan, Kohoutovice, Czech Republic), equipped with an energy-dispersive X-ray spectrometer, X-Max 80 (EDS) (Oxford Instruments plc, Abingdon, UK) was performed to examine the microstructure and elemental distribution in the tracks as shown in figure 16.

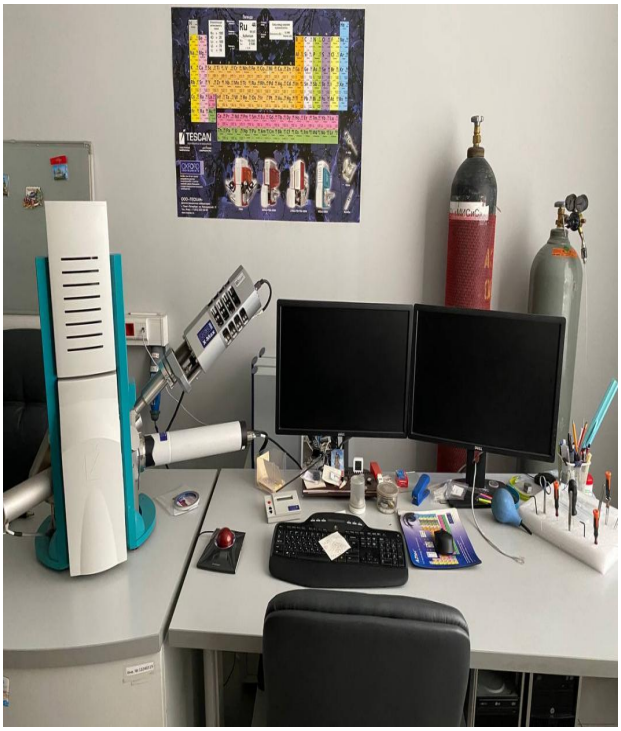


Figure 16 - Scanning Electron Microscope (SEM)

Microhardness Measurement

Microhardness is done by measuring the dimensions of small indents made on a material after applying very little force. It helps to find the hardness of thin, small or delicate items with precision. In the research, a square-shaped indenter with a 136° apex angle and a 50-gf load

was used to measure microhardness on the tracks with a micro-Vickers tester (Wolpert Wilson Instruments, Aachen, Germany). At least 20 locations were tested for microhardness and the average value for every track was found.

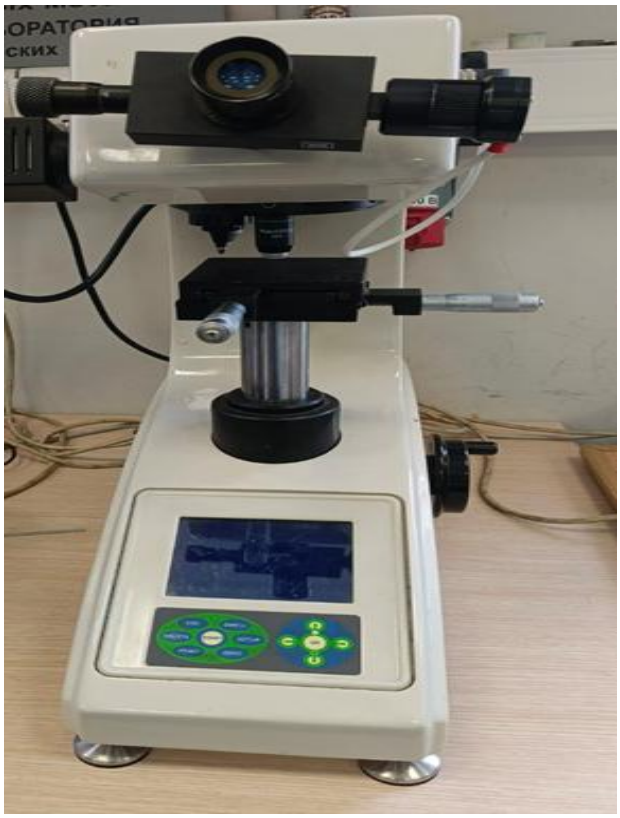


Figure 17 - Microhardness test machine

Results and Discussion

Figure 18 illustrates the schemes for laser melting of sample's surface at different energy inputs represents single dot and tracks for this work.

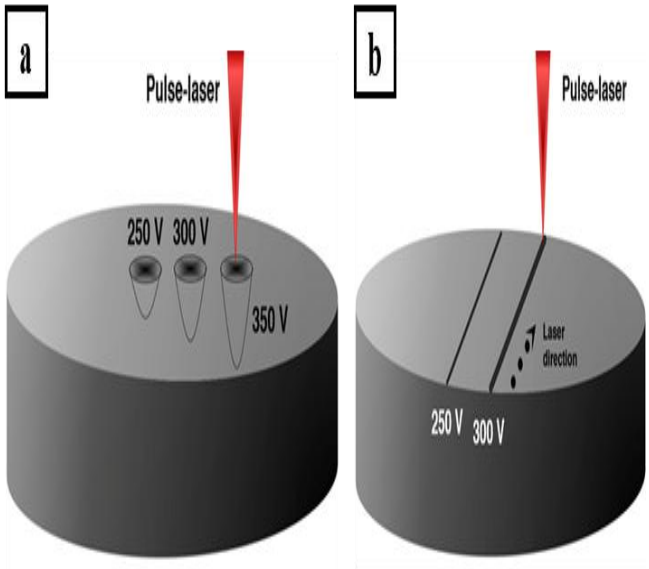


Figure 18 - scheme for (a) sample prepared by air mixing process and melted as single dot and (b) Tracks that formed during laser melting of air mixed and ball milled samples

The result and discussion part of the microstructure for this thesis is divided and discussed in to four parts. These are:

a. Microstructures and elemental distribution of laser melted surface as single dot
b. Microstructures and elemental distribution of air mixed sample and melted at energy input of 250 V, 300 V and 300 V after preheating at 400 ^{0}C
c. Microstructures and elemental distribution of powder during ball milling process at 15, 30 and 60 minutes
d. Microstructures and elemental distribution of ball milled sample and melted at energy input of 250 V, 300 V and 300 V after preheating

Microstructures and Elemental distribution for laser melted surface as single dot

Microstructures of laser melted surface as single dot melted at 250 V

Figure 19 represents the SEM images of a single dot Al–SiC–TiC–TiB_2 alloy prepared by air mixing process and melted at 250 V. As clearly shown on the microstructure, very limited amount of SiC and oxides were observed with no any reactions and phase formation due to the poor preparation method and insufficient input energy that can enhance melting and distributions of the reinforcement particles to initiates in-situ chemical reaction.

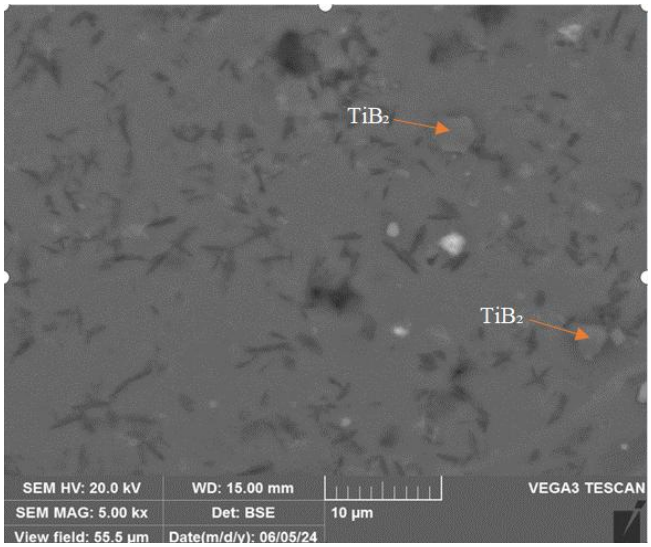


Figure 19 - represents the SEM images of a single dot Al–SiC–TiC–TiB_2 composite prepared by air mixing process and melted at 250 V.

Microstructures and elemental distribution of laser melted surface as single dot melted at 350 V

Figure 20 (a) shows an overview of the microstructure of a single dot of air mixed Al-SiC-TiC-TiB2 composite and melted at 350 V. SEM images reveal the presence of TiC and some oxides. This is due to the poor preparation of air mixing method which may lead to inadequate particle dispersion of reinforcements and the formation of oxidation reaction during laser

melting respectively. According to EDS maps in figure 20 (b), Ti and Si elements were observed in a very limited amount due to the lack of effective integration reinforcement particles in aluminum matrix.

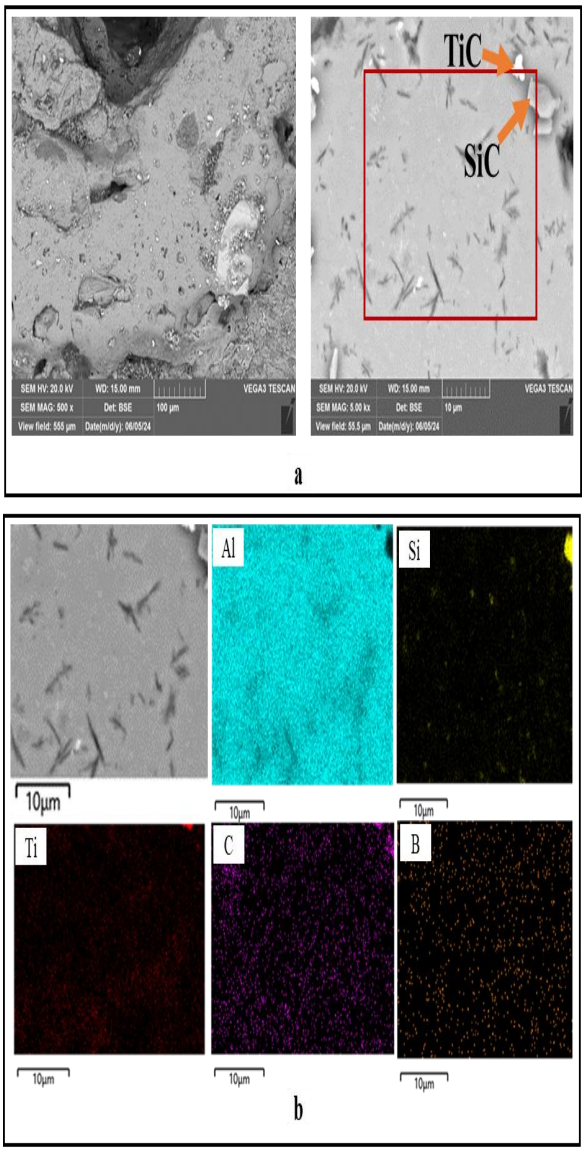


Figure 20 - (a) shows an overview of the microstructure and (b) EDS maps of a single dot of the air mixed Al-SiC-TiC-TiB2 composite melted at 350 V.

Microstructures and elemental distribution of laser melted surface as single dot melted at 350 V after preheating at 200 ^{0}C

Figure 21 (a) shows an overview of the microstructure of a single dot of the air mixed Al-SiC-TiC-TiB2 alloy and melted at 350 V after undergo preheating at 200 ^{0}C. It clearly shows the visibility of TiC and SiC particles were increased due to the preheating enhancement of particle integration. But the particles are very far apart from each other. It is also clearly seen that; the formation of oxides was decreased due to preheating support to reduce thermal gradient between melting pool and sample. However, there is no in-situ reaction observed due to insufficient particle integration to initiate in-situ reaction between the matrix and reinforcement. As shown in figure 21 (b), EDS maps indicate the increment in concentration of Ti and Si elements.

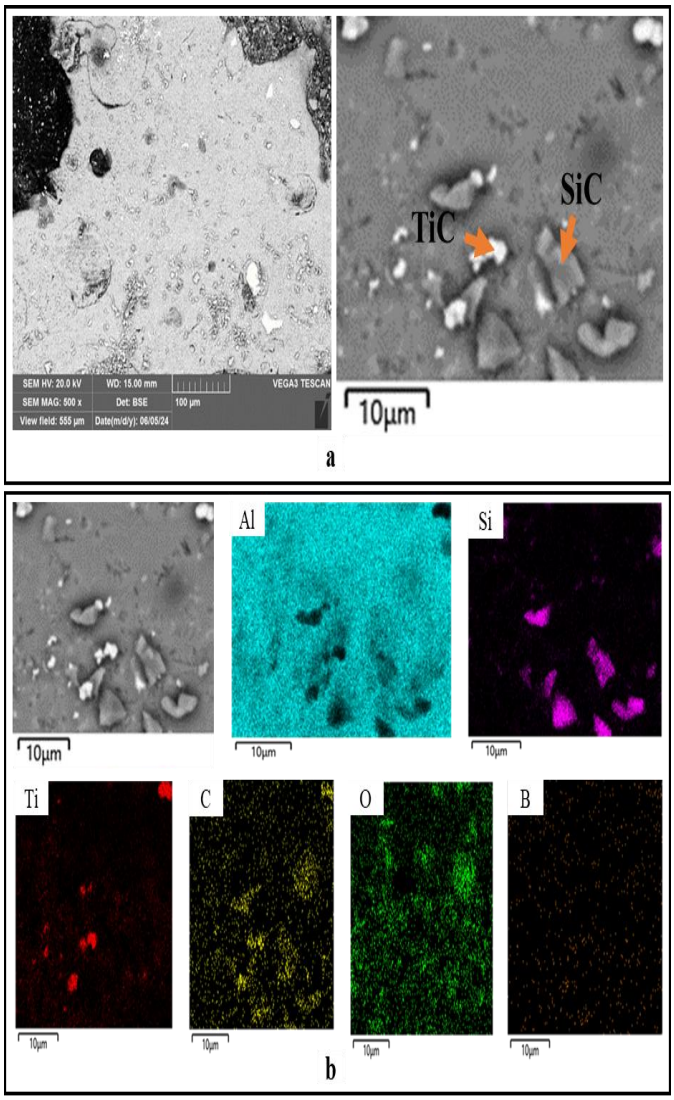


Figure 21 - (a) shows an overview of the microstructure and (b) EDS maps of a single dot of the air mixed Al–SiC–TiC–TiB_2 alloy melted at 350 V after undergo preheating at 200^{0}C.

Microstructures and elemental distribution of laser melted surface as single dot melted at 350 V after preheating at 400 ^{0}C

Figure 22 shows images from SEM and the corresponding EDS maps of elemental distribution after preheating an Al–SiC–TiC–TiB_2 composite sample preheated at 400 °C and then melted at 350 V. In figure 22 (a), it is clearly seen that the reinforcement particles are now more

tightly packed and their amount has increased which is likely because of the elevated temperature at which they were initially preheated.

Still, it was clear that there were no in-situ chemical reactions happening in the aluminum with the reinforcement in the composites. According to Figure 22 (b), the EDS maps that titanium (Ti) and silicon (Si) have a more uniformly distribution than in previous samples.

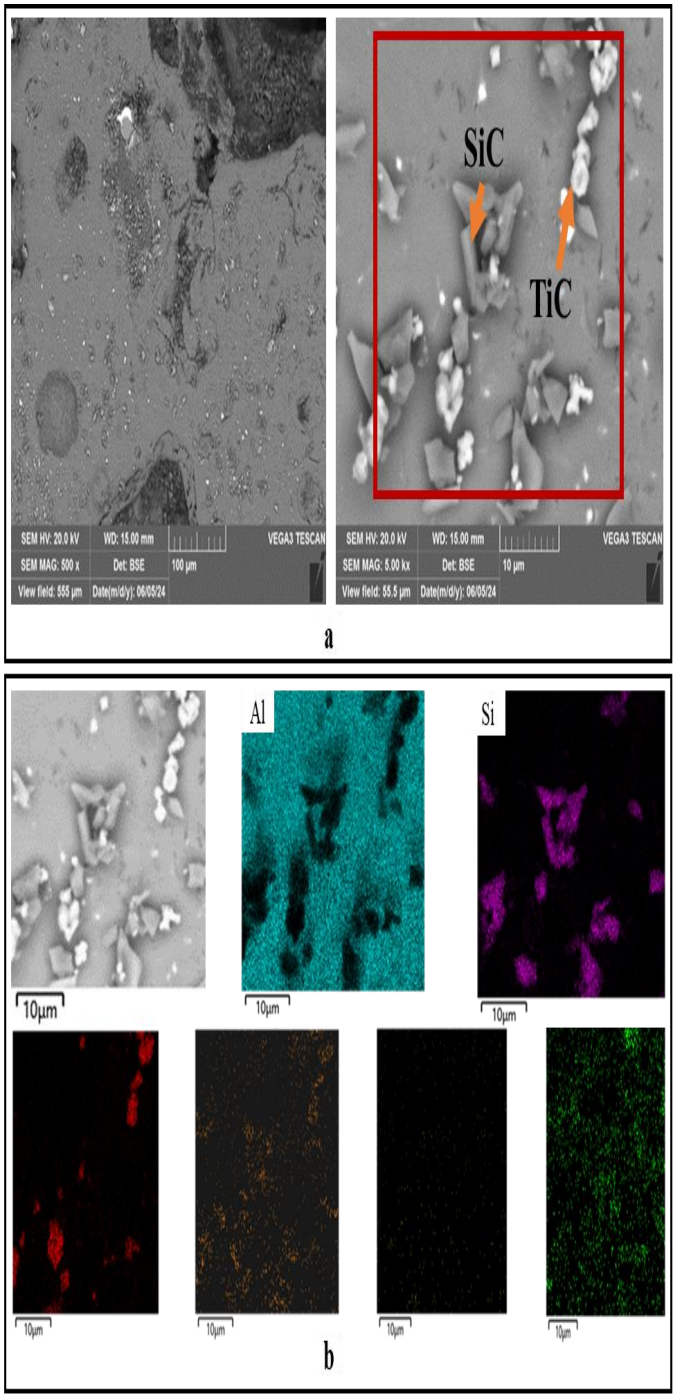


Figure 22 - (a) shows an overview of the microstructure and (b) EDS maps of a single dot of the air mixed Al–SiC–TiC–TiB_2 alloy melted at 350 V after undergo preheating at 400 ^{0}C

Microstructures and elemental distribution of air mixed sample

Microstructures and elemental distribution of air mixed sample and melted at energy input of 250 V

Figure 23 (a) illustrates the SEM microstructure of air mixed Al–SiC–TiC–TiB_2 alloy (sample AM) under 250 laser voltage. It is clearly indicated that, the large and irregular particles were observed in heterogonous form and only aluminum particles were melted while reinforcements were neither melted nor mixed to form reaction and bonds. The EDS mapping on figure 23 (b) shows the localized concentrations of titanium and boron.

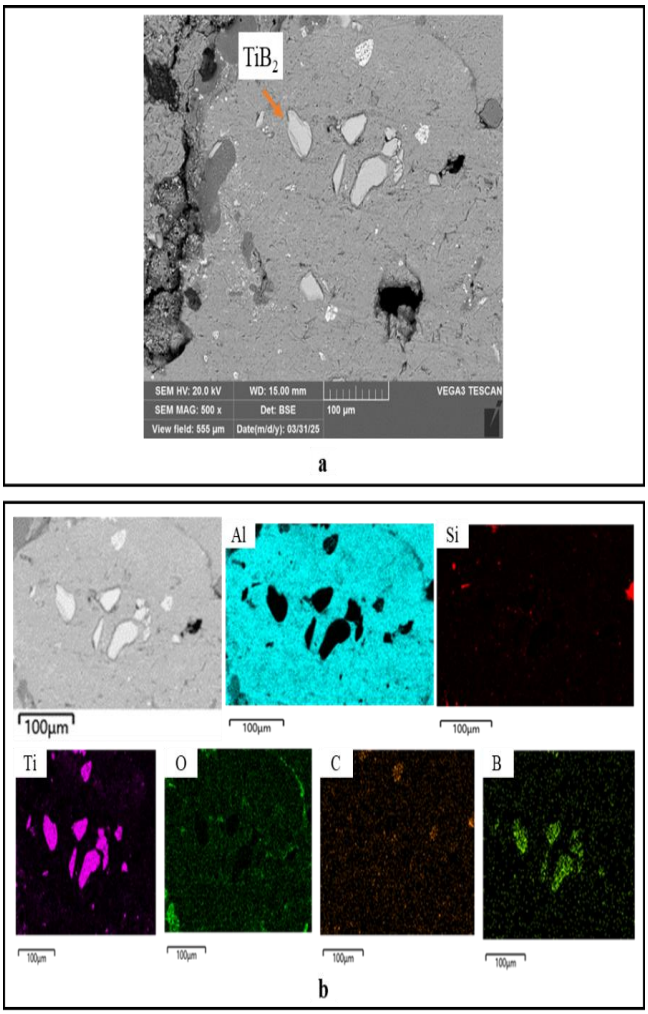


Figure 23 - (a) shows an overview of the microstructure and (b) EDS mapping of a track of the air mixed Al–SiC–TiC–TiB_2 alloy melted at 250 V.

Microstructures and elemental distribution of air mixed sample and melted at energy input of 300 V

Figure 24 illustrates SEM images and EDS maps of the 300 V laser voltage track of the air mixed Al–SiC–TiC–TiB_2 alloy sample. As shown in figure 24 (a) the SEM image displays a somewhat more refined structure with a minor improvement in particle dispersion with slight decrease the pores. Nonetheless, EDS mapping figure 24 (b) continue to show distinct elemental boundaries much like 250 V regime. There is no diffusion of silicon, titanium, carbon and boron elements rather they are still tightly packed. These all evidences show that increased energy input to 300 V by itself without mechanical alloying or enough mixing energy is still insufficient to initiate interfacial bond to assist in-situ reactions.

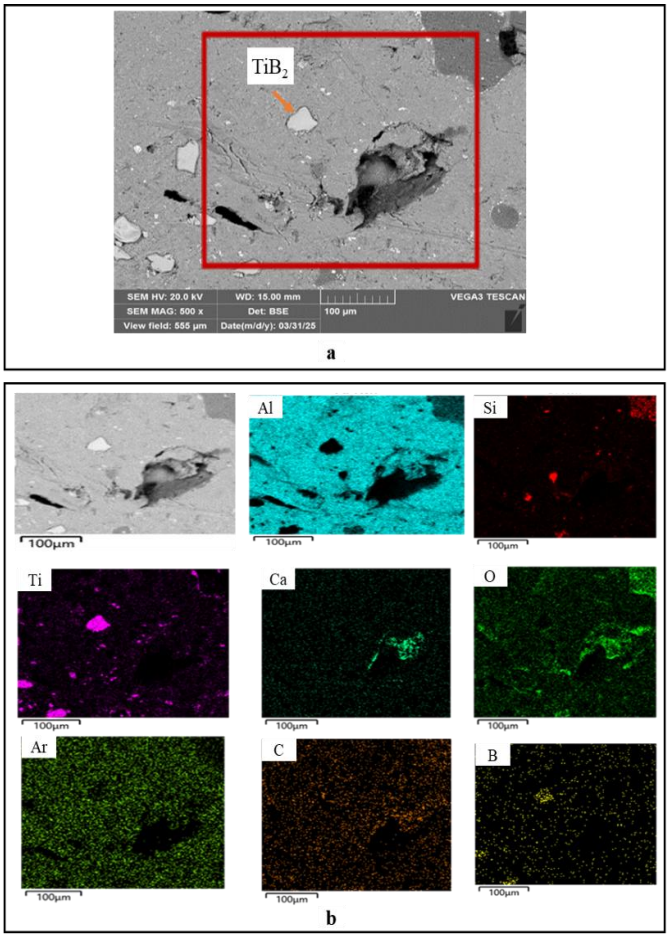


Figure 24 - (a) SEM images and (b) EDS maps of the 300 V laser voltage track on the air mixed Al–SiC–TiC–TiB_2 alloy sample.

Microstructures and elemental distribution of air mixed sample and melted at energy input of 300 V after preheating at 400 ^{0}C

Figure 25 represents the SEM images and EDS maps of the 300 V laser voltage track of the air mixed Al–SiC–TiC–TiB_2 alloy sample after preheated at 400 °C. As shown in figure 25 (a) preheating the 300V regime to 400 °C has a little effect on homogeneity and on the matrix flow. But it could not assist the particles of reinforcements to react with aluminum particles. That means the particles of reinforcements are still inert but they are somewhat close to each other when compared to other two regimes. Figure 25 (b) clearly shows the EDS maps of the 300 V laser voltage track on the air mixed 400 °C preheated Al-SiC-TiC-TiB_2 alloy sample.

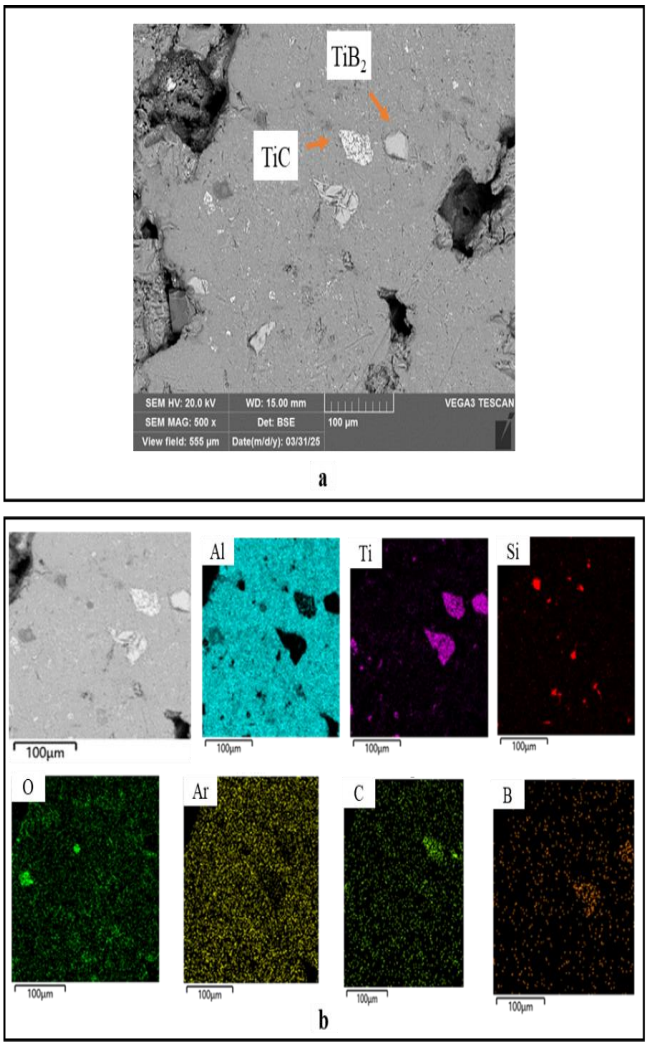


Figure 25 - (a) SEM images and b) EDS maps of the 300 V laser voltage track of the air mixed Al-Al–SiC–TiC–TiB_2 alloy sample after preheated at 400 °C

Microstructures and elemental distribution of powders during ball milling process

Microstructures and elemental distribution of powder during ball milling process at 15 minutes

Figure 26 illustrates the SEM images and EDS maps of the ball-milled powder at 15 minutes Al–SiC–TiC–TiB_2 sample. As clearly seen on figure 26 (a), the aluminum particles were relatively coarse and hardener particles are embedded in the outer surface of matrix alloy particles because of insufficient time to hit the particles by high-energy ball milling to reduce the particle size. Owing to the insufficient duration of ball milling, the reinforcement particles were not uniformly distributed across the aluminum particles, which resulted in the formation of a larger granular structure leading to potential agglomerations or clusters, which greatly affected the final mechanical properties of the sample.

The mapping from the SEM-EDS as stated in figure 26 (b) Si and Ti elements from reinforcements were distributed in an uneven manner, and aluminum was dominant over other elements.

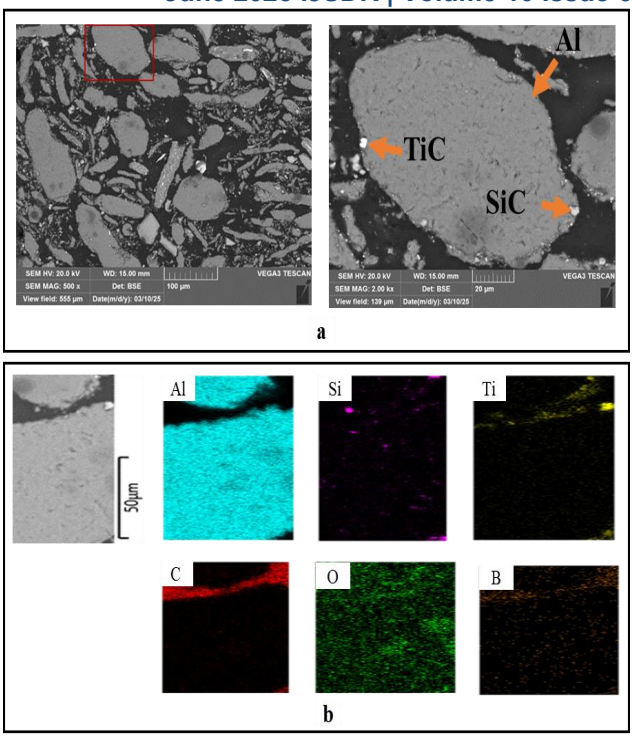


Figure 26 - (a) SEM images and (b) EDS maps of ball milled powder at 15 minutes Al-SiC-TiC-TiB2 sample.

Microstructures and elemental distribution of powder during ball milling process at 30 minutes

Figure 27 shows the SEM images and EDS maps of the ball-milled powder of the Al-SiC-TiC-TiB2 sample after 30 min. Figure 27 (a) clearly shows that as the milling duration increased, the particles collided more frequently, which caused more fragmentation. As a result, the particle size was noticeably smaller than that of the sample milled for 15 min. By dispersing the reinforcement particles more evenly throughout the aluminum matrix, the composite became more homogenous and a reduction in the size of the granules was observed. As illustrated in figure 27 (b), the increment in ball milling duration clearly shows changes to a more refined and somewhat enhanced distribution of Ti and Si.

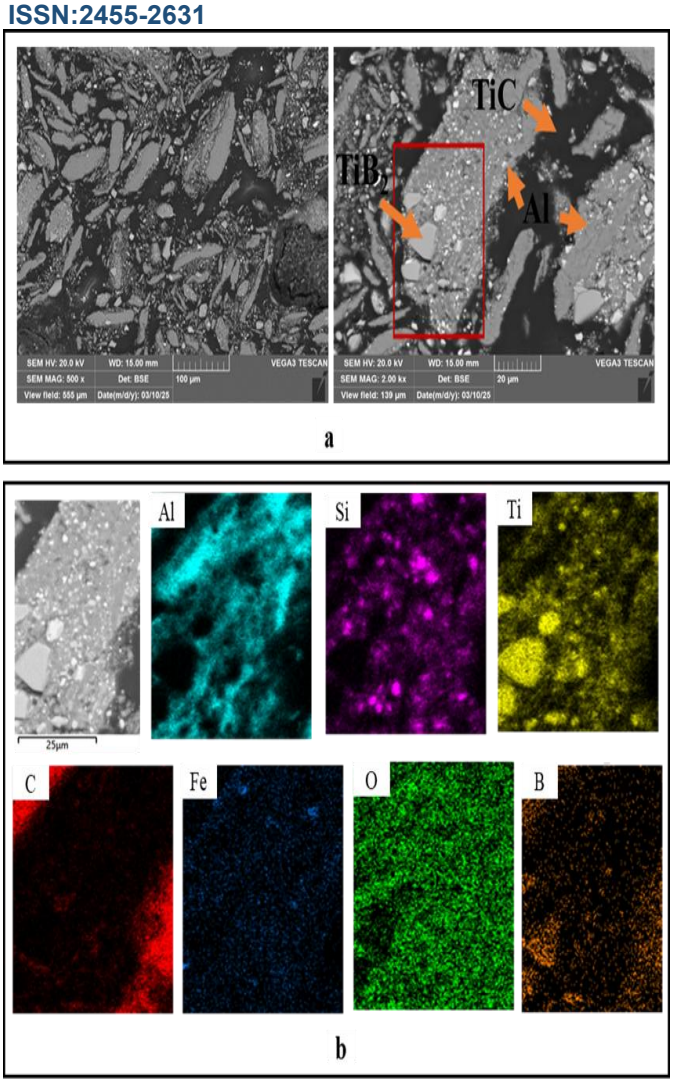


Figure 27 - (a) SEM images and b) EDS maps of ball milled powder at 30 minutes Al-SiC-TiC-TiB_2 sample.

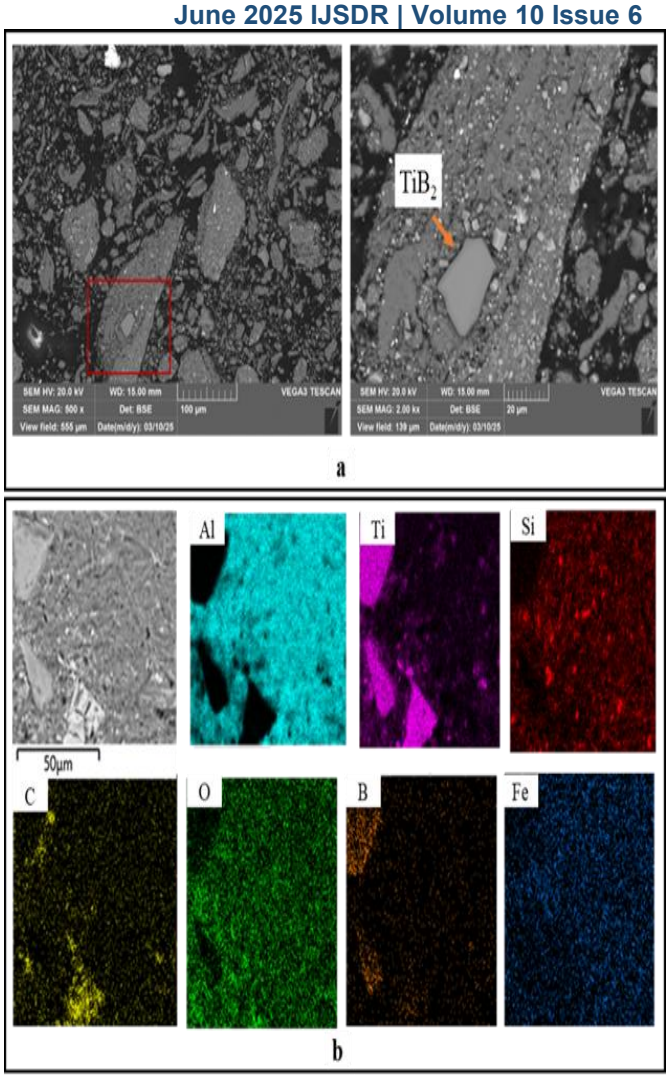


Figure 28 - (a) SEM images and b) EDS maps of ball milled powder at 60 minutes Al-SiC-TiC-TiB2 sample.

Microstructures and elemental distribution of powder during ball milling process at 60 minutes

Figure 28 (a) illustrates, with an increase in the milling time of the powder mixture to 60 minutes, significant adhesion of particles to each other occurs with the formation of granules. Large TiB2 particles are crushed and their size is reduced compared to the milling time previously described. Fragments formed because of the destruction of titanium diboride and particles were redistributed during subsequent deformation. In addition, an increase in the processing time leads to welding of small particles with larger particles as shown in figure 28 (b), the maps show that titanium and boron are uniformly distributed in the bulk of the granules.

Microstructures and elemental distribution ball milled sample

Microstructures and elemental distribution ball milled sample and melted at energy input of 250 V

Figure 29 illustrates the micro- and EDS maps of an Al-SiC-TiC-TiB2 composite that was ball-milled and melted at 250 V. As clearly shown in figure 29 (a), the distribution of reinforcement particles was coarse, poor, or uneven, indicating considerable porosity and weak interfacial bonding. Even small amounts of energy in this regime could trigger the initial formation of phases, however these were formed on a small scale. That means Ti, Al and C may migrate a bit and react inside the material and

parts of the sample are less affected by the laser. EDS maps of the elemental distribution on the sample of this track are shown in figure 29 (b). As examined before, the map highlights the distribution of Ti and C in a limited manner. A small amount of the MAX phase was formed, since the Ti-Al-C overlap was minimal.

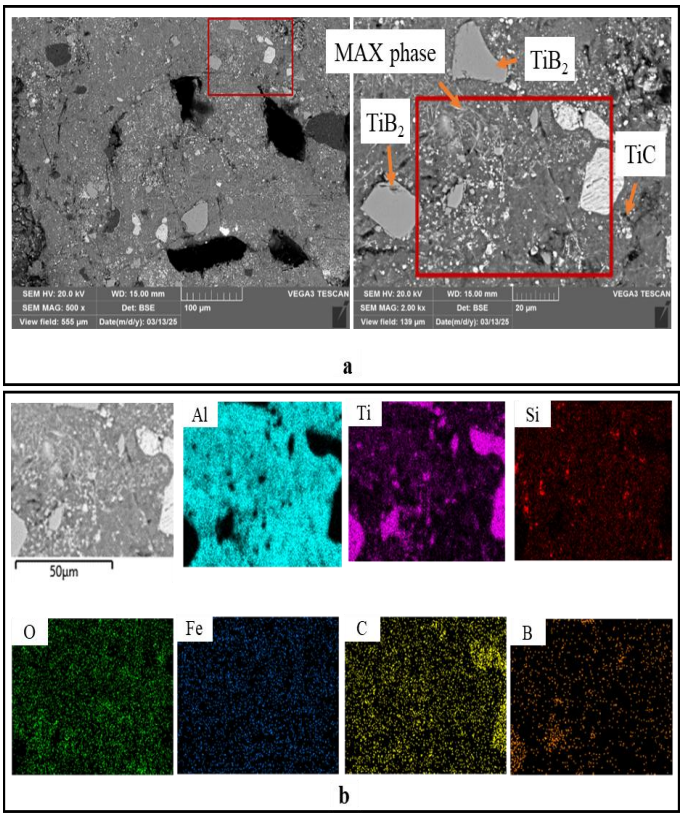


Figure 29 - (a) shows an overview of the microstructure and (b) EDS maps of a ball milled Al-SiC-TiC-TiB2compositeand melted at 250 V

Microstructures and elemental distribution of ball milled sample and melted at energy input of 300 V

Figure 30 shows the microstructure of an Al-SiC-TiC-TiB2 alloy that was processed of ball milling and melted at 300 V. As clearly shown in figure 30 (a), there was a better distribution of the reinforcement particles across the aluminum matrix and a more refined and homogenous microstructure was formed due to better preparation method and melting from high energy. Increasing the energy input here helped melting and mixing which spread the particles more evenly and made the bond between all the particles stronger. Also, a higher level of input energy helps particles interact more fully which boosts the amount of layered or needle-like structures that indicate MAX phase development. Because of the structure formed here, known as the phase and the excellent bonding found, the materials show much better hardness, wear resistance, ability to be machined and heat stability, as stated in literature reviews. As shown in figure 30 (b), more uniform and fine-grained distribution elements are shown by EDS elemental maps. There was a strong match between Ti and Al–C which explains the observation of needle-like structures in SEM. Increased energy input resulted in the most interfacial reactions, suggesting that this helped improve both the movement of molecules and their binding force.

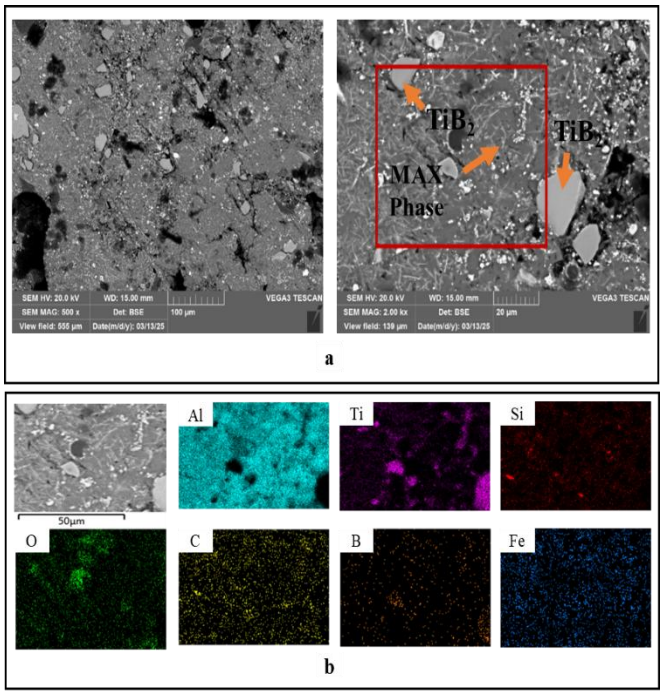


Figure 30 - (a) shows an overview of the microstructure and (b) EDS maps of a ball milled Al-SiC-TiC-TiB2 alloy for 60 min and melted at 300 V

Microstructures and elemental distribution of ball milled sample and melted at energy input of 300 V after preheating at 400 °C

Figure 31 shows an overview of the microstructure and EDS maps of a ball milled Al-SiC-TiC-TiB2 alloy for 60 min and melted at 300v after undergo preheating at 400 °C. As shown in figure 31 (a), the SEM microstructure of this regime shows a more diffused and thermally relaxed microstructure owing to preheating. In contrast to the sample melted at 300 V without preheating, the MAX phase development was noticeably reduced, even though the particle

distribution seemed to be rather uniform. This was probably because preheating reduces the thermal gradient and slows the solidification rate, which restricts the rapid solid-state processes normally required for MAX phase development. Moreover, because of the slower cooling rate, stress relaxation was encouraged, which can negatively affect the creation of the MAX phase. As illustrated in figure 31 (b) in this regime, the distribution of the elements is better than 300 V without preheating, because preheating decreases the thermal shock, which encourages slower and more even solidification that helps the elements to have enough time to be evenly distributed across the matrix.

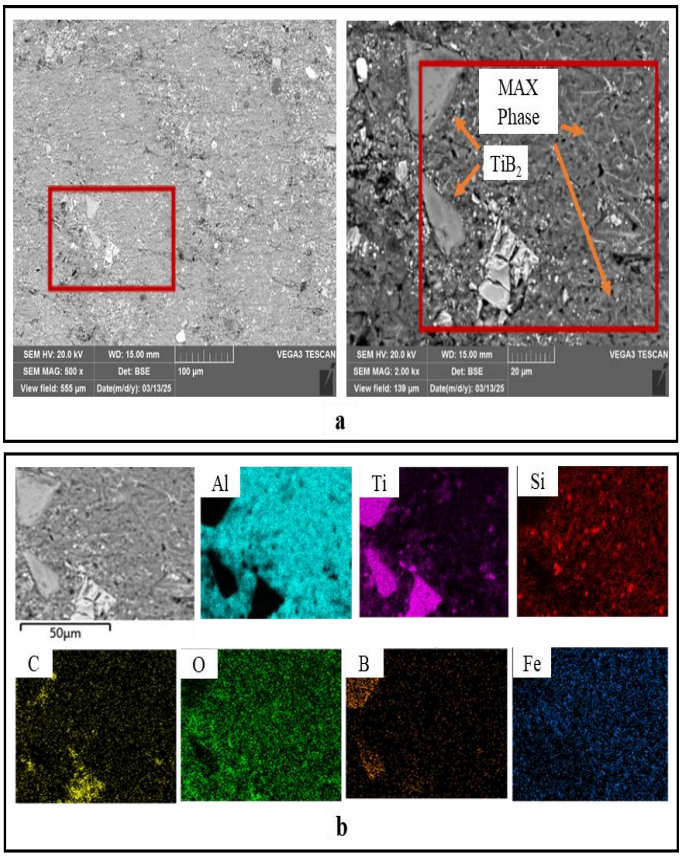
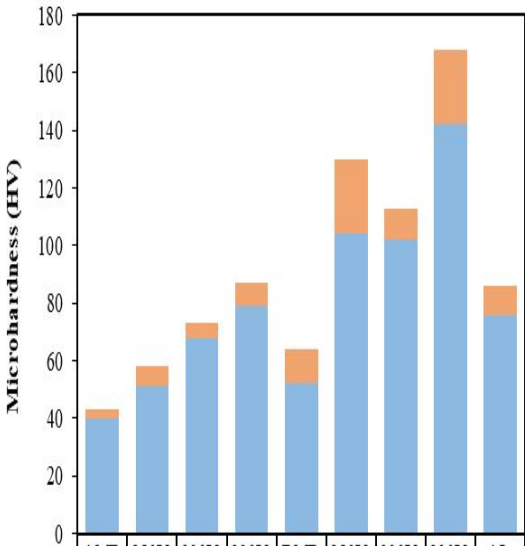


Figure 31 - (a) shows an overview of the microstructure and (b) EDS maps of a ball milled Al-SiC-TiC-TiB_2 composites and melted at 300 V after preheated at 400 °C

Microhardness

Figure 32 shows the microhardness results air of mixed sample before laser melting (AMBL), Air mixed sample (AM) melted at 250 V and 300, Air mixed sample after preheating (AMH) , Ball milled sample before laser melting (BMBL),Ball milled sample (BM) melted at 250 V and 300 V , Ball milled sample after preheating (BMH), pure aluminum sample (Al) and how the samples' microhardness behaved in response to preheating and laser melting at various voltages, as well as how the initial processing technique affected these results.



	AMBL	250V, AM	300V, AMH	300V, AM	BMBL	250V, BM	300V, BMH	300V, BM	AL, 300V
sample standard deviation	3	7	5	8	12	26	11	26	10
Average	40	51	68	79	52	104	102	142	76

Figure 32 - Microhardness results for all tracks

Effect of processing methods on the microhardness

It was clear from microhardness testing that the ball milled sample had a stronger mechanical quality than the air mixed sample before laser melting. The microhardness readings for the air-mixed sample varied from 21 HV to 48 HV and had an average of 40 HV (± 3 HV). On the contrary, the Vickers hardness values for the ball-milled samples were between 35 HV and 72 HV and their average was 52 HV (±12 HV). Because each manufacturing process is unique, this impacts the outcome. Because air mixing was not done well, ceramic particles tended to have uneven amounts and could clump together.

Due to this non-uniformity, sections of the specimens had soft areas with limited reinforcement which was why the microhardness result was not very high. However, ball milling is an approach that provides mechanical alloying which results in an even finer microstructure and the reinforcements are distributed more uniformly throughout the material. By ball milling with increasing of energy, SiC, TiC and TiB_2 particles are well-distributed throughout the aluminum matrix. Extreme plastic deformation and better grain structure help the Hall-Petch mechanism work which makes it harder for dislocations to move in small grains. Ball milling decreases the occurrence of porosity and clusters

in powders which happen because the powders are not held together well enough. When there are many large particles in air mix, they tend to draw the stress in one place which makes cracks appear more quickly and reduces the strength of materials. For example, the ball-milled sample melted at 300V which was an average of 142 HV (±26 HV) showed almost twice as much hardness as its air-mixed counterpart which was 79 HV (±8 HV), highlighting how important uniform particle distribution is for optimizing dispersion strengthening.

Laser melting and energy Input Impacts

Laser melting the air-mixed samples using 250 V made slightly harder, ranging from 39 HV to 67 HV and averaging about 51 HV (±7 HV). This shows that the bonding between the particle and the matrix material better and the microstructure was improved because of the laser process. Unlike with the milled samples, laser melting caused major effects in the ball milled samples. Results showed hardness levels from 63 HV to 153 HV and the average was 104 HV (±26 HV). This was a very impressive growth. The significant improvement mainly resulted from laser melting adding heat impact to ball-milled metal which led to stronger metallurgical bonding, smaller grains and more dislocations, all of which make the alloy tougher and harder by impacting the boundaries between grains (Hall-Petch effect). It was more obvious that the effect occurred when the laser's voltage was increased to 300V. Air-mixed samples were in the range of 67 to 97 HV, averaging 79 HV (8 HV). But the ball-milled samples had a clear improvement, with the hardness averaging around 142 HV (±26 HV) and reaching up to 197 HV. Applying higher voltage allowed more energy to, accelerate matrix and reinforcing particle fusion which improved wetting at the interface and better immersed the particles in the matrix.

Due to the energy introduced, there were better diffusion and lower air porosity. Still, the influence is held back by the varied way particles are spread. The mixture of the sample is improved by lasers, taking advantage of the original work hardening. Because laser treatment happens instantly, it leads to microstructural refinement which results in greater mechanical strength.

Preheating (400°C) effect before laser melting at 300 V

Prior to melting at 300V, the samples were baked at 400°C and this brought a significant difference in their hardness. At those conditions, the hardness values for air-mixed samples varied from 50 to 80 HV with an adjusted average of 68 HV (±5 HV) which was not significantly more than at the same voltage without preheating. Additionally, the average hardness of ball-milled samples decreased, with values ranging from 84 to 125 HV and an average of roughly 102 HV (±11 HV). Preheating plays a part in lowering the temperature gradient during laser processing, which explains the hardness drop even with comparable or greater energy input due to slower cooling rate which leads to grain coarsening and reduced dislocation density.

In addition, 300 V and preheating regime is higher than 250v regime in air mixed and vice versa in ball milling for the following reasons:

a. Since the air-mixed samples are in poor beginning condition, they benefit from the extra thermal energy and extended exposure at 300V + preheating. When compared to the lower-energy 250V regime which was an average of 51 HV (±7 HV), the preheating regime (300v heated to 400°C), results in higher hardness which was 68 HV (±5 HV) in average by stabilizing the melt pool, enhancing bonding, and correcting defects.

b. On the other hand, when the ball-milled samples were exposed to the extended heat of preheating, they lose some of their beneficial microstructural characteristics (such as fine grains and strain hardening), even if they were already optimized in terms of reinforcement and grain structure. This leads to a minor decrease in hardness in contrast to the 250V condition which was decreased from 104 HV (±26 HV) to

102 HV (±11 HV), where the microstructural advantages of ball milling are better preserved by quick melting and solidification.

CONCLUSION

The main results of this work could be summarized as follows:

1. Contrary to air mixing, ball milling made the microstructure much more uniform. With this approach, the composite was packed more closely, Al-SiC-TiC-TiB2 particles were evenly distributed and the grain size decreased which are important for strong aluminum composites.

2. Increasing the energy input to 300V made matrix-reinforcement better, enhanced melting and increased the diffusion of particles in ball-milled samples. In addition, structure gets refined and interfacial reactions begin largely depends on processing method.

3. Ball-milled samples at 300V showed the most evidence of layered and needle-like structures suggestive of MAX phase development. The absence of these structures in air-mixed samples demonstrated that phase formation requires homogeneous dispersion.

4. In air-mixed samples, preheating before laser melting improved particle visibility, slightly reduced oxide formation, and lessened temperature gradients. Preheating, on the other hand, decreased stress fields, slowed solidification, slightly softened the microstructure and, because of slower cooling rates, somewhat prevented the formation of the MAX phase in ball-milled samples.

5. It was consistently noticed that ball-milling produced samples with higher microhardness compared to those air-mixed samples. Mechanical alloying improved hardness by enhancing dislocation blocking (Hall-Petch effect) and making the material more homogeneous.

6. Raising the laser voltage to (300V) greatly improved the hardness in both samples. Using 300V for a ball-milled sample produced the best ranking, a high microhardness value (197 HV) which stresses that high energy is needed and better processing to ensure good fusion and mechanical strengthening.

7. Preheating to 400°C enhanced microhardness in air-mixed samples by lowering defects and stabilizing the melt pool, whereas ball-milled samples had somewhat less hardness because of slower cooling-induced dislocation density reduction, grain coarsening and softening microstructure due to preheating.

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