ALUMINUM SILICON CARBIDE COMPOSITE PROCESSED BY FIELD ASSISTED SINTERING TECHNIQUE: MICROSTRUCTURE AND MECHANICAL PROPERTIES

A Thesis in
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by
Sven Alagic

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The thesis of Sven Alagic was reviewed and approved* by the following:

Jogender Singh
Professor of Materials Science and Engineering
Thesis Co-Adviser

Anil K. Kulkarni
Professor of Mechanical Engineering
Thesis Co-Adviser

Douglas E. Wolfe
Professor of Materials Science and Engineering
Department Head, Advanced Coatings at the Applied Research Laboratory

Suzanne Mohney
Professor of Materials Science and Engineering and Electrical Engineering
Chair, Intercollegiate Graduate Degree Program in Materials Science and Engineering

*Signatures are on file in the Graduate School.
ABSTRACT

Aluminum is widely used in aerospace structural components because of its low cost, light weight, and high strength properties. Aluminum silicon carbide composites are an opportunity to reduce weight of these structural components without sacrificing strength. Aluminum and silicon carbide have similar densities (2.6 and 3.21 g/cm$^3$ respectively), but silicon carbide has much higher hardness and strength.

In this thesis, improved hardness, elastic modulus, yield strength, and ultimate tensile strength were achieved in an aluminum alloy based composite system by the addition of silicon carbide particles. Bulk baseline samples as well as samples containing various amounts of SiC were manufactured using Field Assisted Sintering Technology (FAST).

Hardness was found to improve from 130 HV in ECKA Al-Alloy to 207 HV in Al Alloy with 40% SiC composite (60% improvement). This improvement was accompanied with an improvement in elastic modulus from 81 GPa in ECKA Aluminum Alloy to 126 GPa in Aluminum Alloy with 40% SiC (40% improvement). Yield strength improved from 211 to 417 MPa (97% improvement) and ultimate tensile strength improved from 308 to 472 MPa (50% improvement) for these materials.

The microstructure of the samples revealed a homogeneous dispersion of silicon carbide particles throughout the aluminum matrix. The improvement in specific strength was 50% over the baseline aluminum alloy. The samples have been used as extrusion feedstock as a proof of concept for eventual structural applications.
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List of Abbreviations

MMC – Metal Matrix Composite
TiB – Titanium Boride
TiC – Titanium Carbide
ZnO – Zinc Oxide
Al₂O₃ – Alumina
SiC – Silicon Carbide
B₄C – Boron Carbide
CNT – Carbon Nanotube
PS – Pressureless Sintering
HP – Hot Press
HIP – Hot Isostatic Press
FAST – Field Assisted Sintering Technology
SPS – Spark Plasma Sintering
VHN – Vickers Hardness Number
HV – Hardness Vickers
EM – Elastic Modulus
XRD – X-ray Diffraction
OM – Optical Microscopy
SEM – Scanning Electron Microscopy
EDS – Energy Dispersive X-ray Spectroscopy
FIB – Focused Ion Beam
TEM – Transmission Electron Microscopy
HRTEM – High Resolution TEM
EDM – Electric Discharge Machining
RM – Rule of Mixtures
IRM – Inverse Rule of Mixtures
CTE – Coefficient of Thermal Expansion
UTS – Ultimate Tensile Strength
Chapter 1: INTRODUCTION

Metal Matrix Composites (MMC) are material systems which have a metal or metal alloy as the baseline matrix material with an added second material known as the composite material. An illustration of a typical MMC system is shown in Figure 1.1.

![Figure 1.1. Illustration of typical composite material system. The matrix material could be an aluminum alloy and the composite material could be a carbon fiber dispersion to improve tensile properties.](image)

MMC materials include metal-ceramic systems as well as metal-metal composites. This study investigates the aluminum-silicon carbide metal-ceramic system in effort to improve specific strength (strength normalized to weight). This is particularly interesting for applications related to the aerospace and transportation industries. Any reduction in mass while retaining strength of a material used in the body of a vehicle would directly implicate financial savings on fuel as well as potential for higher payload. In the past mechanical property improvements via compositing have been explored using a variety of material systems including magnesium, titanium, and aluminum based metal matrix composites, all of which are interesting in the transportation industry.
1.1: MAGNESIUM BASED METAL MATRIX COMPOSITES

Magnesium based metal matrix composites are commonly used for transportation applications due to their high specific strength and wear resistance [2]. Previous studies have composited magnesium with a variety of carbides, including boron carbide, silicon carbide, and titanium carbide [3]–[7]. Improvements were seen in hardness and wear resistance which are important considerations for structural applications in the transportation industry.

Casting is a very common approach for synthesis of metal matrix composites due to its simplicity. This synthesis method is explained in detail in Section 2.1. Singh et al. [4] have made recent advances in magnesium based metal matrix composites. In this study, the group used commercially pure magnesium metal and stir cast it with a range of wt. % boron carbide particles (3,6,9,12 wt. %). The boron carbide particles were 63 microns in size, with the intended purpose of refining the grain structure and impeding dislocation movement. The casting process was conducted at 700°C, producing a series of 5 samples. The series of samples was tested for Vickers Hardness, showing a significant increase in hardness with increase in boron carbide addition. The results showed 32 HV for pure magnesium, improving to 45 HV for 6 wt. % B4C addition, and nearly 75 HV for a 12 wt. % addition of boron carbide, more than doubling the performance of the baseline magnesium metal. The tests were done with 500g load for 5 measurements where only the average value was reported. This improvement is attributed to the restriction of magnesium matrix deformation due to the boron carbide particle presence hindering dislocation movement. In addition to studying the hardness, the group looked at the wear resistance properties of the reinforced magnesium matrix, and showed that similarly to the hardness, wear resistance almost doubled with the addition of 12 wt. % boron carbide – decreasing material loss from 0.04g under 1 kg load to only 0.02g.
A study conducted by the Poddar group [6] has synthesized a magnesium composite with silicon carbide particle additions. This group used pure magnesium as well as a magnesium alloy in their investigation. The magnesium materials were melted down and two difference silicon carbide powders were added (15 μm and 150 μm) at the 15 vol% proportion. The results showed final densities to be less than theoretical, as well as very large grain sizes larger than 76 μm. Nevertheless, there was also an observed improvement in hardness, doubling from 45 HV to 90 HV with the larger silicon carbide particle size addition. The material with the smaller silicon carbide particle size showed a still higher hardness of 100 HV, but also had a lot of agglomeration in the microstructure. Interestingly, in both cases, the ultimate tensile strength was reduced with the introduction of silicon carbide particles to the system (from 212 MPa to about 200 MPa). The improvement in the hardness is attributed to the particles of silicon carbide constraining dislocation motion and matrix deformation. The low tensile strength is attributed to either separation at the particle matrix interface, or breaking of the carbide particles.

Similarly, Wang et al. [7] stir casted magnesium with 0-20 vol%, 10 μm silicon carbide particles. The major difference in this study was that the melt was treated with an ultrasonic probe before being stirred at 700 °C. The group observed that particles distributed along grain boundaries of the aluminum matrix, and they state that the ultrasonic treatment improved the dispersion of particles by reducing the overall stir time. It was also observed that at the 15% volume fraction of silicon carbide, grains were refined and the ultimate tensile strength was at a maximum (200 MPa) compared to the baseline at about 125 MPa. At the 20% particle volume fraction, there was significant aggregation observed, which acts as a stress concentrator and is attributed to the reason why the ultimate tensile strength dropped off at that point.
M. Yang et al. [5] ran similar experiments where magnesium matrix material was compositied with small amounts of titanium carbide (TiC) particles. This group used ZA84 magnesium alloy as their baseline matrix material, which is simply magnesium with about 8% zinc, 4% aluminum, and 0.25% manganese. The composite material was created by the stir casting method, where 0.5 w t% TiC was added to the alloy melt at 750°C and mechanically stirred. There is no mention of the TiC particle size. After casting, the group conducted a tensile test of these materials to compare performance and room and high temperature (150°C). In both cases, the sample with TiC addition out-performed the baseline alloy. At room temperature ultimate tensile strength and yield strength improved by 18% and 15% respectively. At 150°C the improvement was of lower magnitude, coming out to about 7% and 6% respectively. In both cases this improvement is impressive for such a small addition of titanium carbide, and is attributed to the microstructure refinement of the baseline material.

Powder metallurgy can also be used to produce magnesium based MMCs. Several studies have been done to explore the improvements to the final microstructure and mechanical properties [3], [8]–[10]. An experiment by the Jiang group [8] fits well with the aforementioned studies as it is similarly a magnesium based material which is being reinforced by the addition of boron carbide particles, however it was produced by powder metallurgy. The magnesium particle size was up to 106 μm and the boron carbide particle size was 6 μm. The magnesium matrix material (commercially pure) was compositied with 10, 15, and 20 vol.% boron carbide particles by hot pressing. This was conducted at 620°C and 5MPa for 10 minutes. These samples were then tested for hardness and wear resistance. Hardness improved significantly, by about 400% over the baseline magnesium in the sample with 20% boron carbide addition. Similarly, the wear rate of the sample with 20% boron carbide addition was about four times less than the wear rate.
of the pure magnesium material. The group observed a network of chains of boron carbide within the magnesium matrix, along with the presence of microporosity in all samples. This was attributed to the large size difference in starting powders. The group concluded that the boron carbide contributed to the wear resistance of the material by increasing the matrix resistance to deformation.

The Selvam group [10] and Tun group [11] used zinc oxide (ZnO) as a compositing material for their magnesium composites. The 60-300 μm magnesium particles and the 50-200 nm zinc oxide particles materials were ball milled together and pressed at 510 MPa. They were then sintered at 640 °C for 14 minutes using a microwave sintering technique before being extruded. The groups did a variety of mechanical tests to evaluate the mechanical properties, showing an improvement of hardness from 45 HV to 66 HV in Mg with 1.5 vol% ZnO, as well as in improvement in ultimate tensile strength from 179 MPa to 229 MPa. These improvements were attributed to a couple dislocation mechanisms, those being grain refinement, Orowan bypassing, and coefficient of thermal expansion mismatch effects. Investigation into tensile and compressive responses of Mg–ZnO composites. Investigation into tensile and compressive responses of Mg–ZnO composites. Investigation into tensile and compressive responses of Mg–ZnO composites. Investigation into tensile and compressive responses of Mg–ZnO composites. Investigation into tensile and compressive responses of Mg–ZnO composites.

A more unique study done by the Dudina group [9] has shown that metallic glasses can similarly be used to activate these strengthening mechanisms and improve the overall material properties. This group used magnesium alloy AZ91 as the matrix material and a zirconium based metallic glass at 15 vol% addition. The material was compacted to 50 MPa and sintered at 713 °C. Although the particle sized are not mentioned, the range of sizes seems to be very large as seen in electron microscope images, up to even 100 μm in size. The group showed the
improvement of hardness of the material by 100%, from 68 HV to 123 HV over baseline magnesium alloy.

1.2: TITANIUM BASED METAL MATRIX COMPOSITES

Titanium based metal matrix composites are commonly used in aerospace applications as well as other transportation industries for their similarly desirable strength and weight properties [12]. The literature shows that titanium has been composited thoroughly with materials such as titanium boride and carbon nanotubes [13], [14]. Most studies resulted in the improved toughness and wear properties of the titanium based composite materials. This has been done by mostly powder based processing methods, a couple of which are highlighted as follows.

A study by the Kondoh group [15] added various proportions (0 to 0.35%) carbon nanotubes (CNT) to pure titanium matrix material. The titanium powder was atomized with a size of about 30 μm. The carbon nanotubes were about 20 nm in diameter and 0.5-1 μm long. The materials were blended by coating the titanium particles in a surfactant solution containing the carbon nanotubes, and then drying. From this point the powder was sintered by Field Assisted Sintering Technology (FAST). This was done in a two step process; the first step was 600°C at 20 MPa for 60 minutes and then at 800°C at 41.6 MPa for 30 minutes. Immediately after sintering the samples were hot extruded at 400°C. Upon examining the microstructure, the group found that grains were refined by the addition of CNTs, as compared to the baseline pure titanium. They also found that even though many CNTs were still in the final product, the temperature during sintering was high enough that TiC particles were created at some locations. Tensile tests conducted on the samples showed that a 0.35% addition of CNTs improved the tensile strength by 28%, the yield strength by 47% and the hardness by 9%. This is attributed to
the grain refinement of the titanium grains, and the dispersion strengthening effect of the CNTs and TiC particles.

A study by the Zhang group [16] composited titanium with titanium boride (TiB) by an in situ process, where volume fractions from 0 to 100% TiB in steps of 20% were explored. The group used 32 μm Ti-6Al-4V powder and 4 μm TiB₂ which were mixed by ball milling the materials for 6 h. The materials were sintered by hot press at 30 MPa with a range of temperatures from 1300 to 1500 °C. The samples showed an increase in hardness from 3.8 GPa to 15.3 GPa at 80% TiB, and similarly an improvement in elastic modulus from 103 to 306 GPa. These studies of compositing structural materials to improve the mechanical properties set the stage for applying these concepts to other less explored material systems such as aluminum.

1.3: ALUMINUM BASED METAL MATRIX COMPOSITES

Limited work has been done in the area of aluminum composites with ceramic materials. In the past, aluminum has been composited with silicon carbide, boron carbide, and carbon nanotubes [17]–[22]. Exploration into the strengthening mechanisms of the aluminum matrix with composite material addition has also been done. The most relevant compositing system to this thesis is the aluminum – silicon carbide system.

Casting is a common method for composite production as seen in the magnesium studies mentioned previously. Investigations have been conducted in effort to understand the mechanical properties of aluminum and silicon carbide composites by various casting methods. The Inegbenebor group [17] used stir casting to add 3 μm and 29 μm silicon carbide particles to commercially pure aluminum matrix material. These particles were added in proportions of 2.5, 5, 7.5, and 10 wt. % to an aluminum melt. The aluminum was heated to 750 °C, causing it to
melt. SiC particles were preheated to 1100 °C for 3 hours before being added to the aluminum and stirred for 5 minutes. The samples produced were tested for various mechanical properties including hardness, elastic modulus, and yield strength. The addition of 10 wt. % of 29 μm silicon carbide particles showed a minor improvement in hardness, of about 10% compared to the pure aluminum baseline. However, when the same weight fraction of the 3 μm silicon carbide particles was tested, there was a jump of almost 40% improvement in hardness over baseline. This improvement in general is attributed to the SiC particles impeding dislocation motion, due to the formation of a dislocation loop around the particles. The reason why the finer particles made a larger improvement is theorized to be due to the better dispersion of the particles as observed under SEM. Similarly, elastic modulus was also improved by the addition of SiC particles to the aluminum matrix, showing again the trend that the smaller, better dispersed particles made a more positive impact than the larger SiC particles. In this case, the 7.5% addition of 3 μm silicon carbide resulted in the 375% improvement in elastic modulus over the baseline aluminum.

Ozben et al [18] created an aluminum composite material with silicon carbide particles by squeeze casting. The aluminum matrix material was actually a metal matrix composite in itself, known as AlSi7Mg2. Silicon carbide particles of size range 30-60 μm were added in proportions of 5, 10, and 15 wt. %. The final material was produced by a squeeze casting process of undisclosed parameters, and subjected to a number of tests including hardness, toughness, and tensile strength. Observations showed an increasing trend in overall hardness with increasing silicon carbide addition, with a 10% improvement in hardness of the 15 wt. % composite material over the baseline. In the tensile tests, there was an 8% improvement in tensile strength at 10% SiC addition. The 15% SiC sample was observed to have agglomerations and broken
particles which caused it to fail at a lower load. The final test was an impact toughness measurement. In this test the samples all showed a reduced toughness over the baseline, due to the brittle nature of the added SiC material. The reduction in toughness was also attributed to the formation of porosity and the group speculated that it could be improved if a better bond was formed between the silicon carbide particles and the aluminum matrix material.

Powder metallurgy has proven to be a promising method to obtain high density composite materials. Aluminum and boron carbide composites were produced by Topcu et al. [19] via powder metallurgy to create homogeneously dispersed metal matrix composites. Aluminum powder was atomized at a size of 10 μm, and boron carbide was fabricated externally at a 10 μm specification. A series of samples were mixed by attrition milling at a 5-20 wt. % range. These samples were pressed to 250 MPa by cold isostatic press and then sintered at 600, 625, and 650°C. SEM showed a uniform dispersion of particles. These experiments showed expected results of increasing trend in hardness with increasing weight percent boron carbide addition. The highest hardness was achieved at the 20% boron carbide addition, with a hardness of 82 HV compared to the baseline of 36 HV, an over 100% improvement. This improvement in hardness is claimed to be due to the dispersion strengthening effect. A second set of tests was done to examine the impact resistance of the composite materials. It was shown that at the 20% boron carbide addition, impact resistance had been reduced to 15% of the baseline value.

L. Gomez et al [20] compared properties of aluminum alloy 6061 based composite materials with boron carbide and silicon carbide additions, as well as the differences when heat treatments are brought into the equation. In this study the aluminum powders were blended by ball milling with boron carbide powder and silicon carbide powders up to a ratio 10%. Boron carbide particles were irregularly shaped, faceted, and had a mean size of about 12 μm. Silicon
carbide particles, also irregularly shaped and faceted, had a mean size of 2 μm. The aluminum powders had a much more rounded morphology and seems to have a size range from 10 to 50 μm. The powders were cold pressed to 450 MPa and hot extruded at 530 °C. It is notable that using this processing method the group was not able to achieve 100% theoretical density when silicon carbide or boron carbide exceeded 7.5 vol. %. The samples were hardness tested after extrusion and a positive trend was observed with increasing additions of both silicon carbide and boron carbide, showing a 40% increase in hardness over the baseline aluminum alloy. Tensile tests showed similarly an improvement in specific strength of about 15%, and a decrease in ductility to about 80% of the baseline value. In most of these cases, there was minimal difference between the performances of the silicon carbide samples compared to the boron carbide samples, except in specific strength where boron carbide had a minor advantage due to its low density. Another element to this study was the contribution of different heat treatments to the performance of the composited material. In either case (T1 vs T6) the trends for the improvement of properties was very similar within each heat treatment.

Another interesting study conducted by Najim et al. [23] looked into the effects of composited particle size in aluminum matrix composites. This group added nano (20 nm) and micro (30 μm) alumina (Al₂O₃) powder in different proportions (0, 5, 10, and 15 wt. %) to commercially pure aluminum powder (45 μm). These were mixed in a roller mixer for six hours, and then cold pressed at 500 MPa. The compacts were then sintered at 500 °C for 2 hours. These samples were then tested for compressive properties as well as hardness. Compressive strength of the material was highest in all case for the nano alumina powder in one case exceeding the baseline by 300%. Overall, the best compressive performance was observed at the 10% alumina addition. The lower performance of the 15% alumina samples is attributed to the effects of
agglomeration seen in the microstructure. Hardness testing of the composites showed that in all cases the nano alumina samples out-performed the micro alumina samples. In addition, the highest hardness was seen at the 15% nano alumina addition, showing a 400% improvement over baseline pure aluminum, and a 100% improvement over its 15% micro alumina counterpart. These improvements are explained by the group as effects of the contribution of defects, in conjunction with interfacial area between the nano alumina particles with the aluminum matrix.

Work has been done by Chen et al [21] in effort to consolidate aluminum 6061 material (particle size of 13 μm) with boron carbide powder with an average size of 18 μm. This group added boron carbide via shaker mixer and consolidated the composite by field assisted sintering technology. This resulted in high densities of consolidation to be achieved as described by Chen to be higher than densities achievable by counterpart processing techniques like conventional sintering and hot isostatic press (HIP). The samples prepared included a range from 0-50% boron carbide addition in increments of 10%. Sintering was conducted in 2 stages, the first being heating to 480 °C and 32 MPa for 2 min. The second stage was holding at 550 °C and 40 MPa for 3 min. These samples showed an increasing trend in hardness, yield strength, and ultimate tensile strength with increasing boron carbide addition. Hardness increased by 300% (52 HV – 220HV) and ultimate tensile strength increased by about 100% at 30% B4C addition (120-240 MPa). Above 30% addition, detrimental effects were seen. These improvements were attributed to a couple of causes. The hardness was said to have been improved by the good interface between the particles and matrix material, as well as the creation of dislocations around boron carbide particles. The homogeneous dispersion of boron carbide particles is also said to have an impact on the improved hardness, and it is said that the agglomeration of these particles is what reduced performance at compositions above 30%. The improvement in tensile properties is
attributed to multiple factors. The primary contributor is said to be due to grain refinement by the short processing cycle of FAST as compared to HP or HIP. It is also said that contributions come from load transfer due to the interface between boron carbide and the aluminum matrix. It is also hypothesized that orowan strengthening and dislocation strengthening contributed to the improvement of the tensile properties.

Carbon nanotubes have been added to aluminum matrix in similar ways to those discussed above and have had interesting impacts on the composite system. Esawi et al [22] has conducted a study in which small (2-3 wt%) additions of carbon nanotubes were composited with an aluminum matrix material. The CNTs were 3-4 μm long with a 140 nm diameter. The powder was processed via ball milling and compacted at 475 MPa. The compact was extruded at 500 °C and machined. The group did a hardness test on the samples comparing to a baseline of unmilled aluminum. The milled aluminum as well as the aluminum with CNTs showed a 100% improvement in hardness over the unmilled sample, and this is attributed to the grain refinement during milling. However, in this study, there was no observed contribution to hardness by the CNTs. Additionally, tensile tests were conducted on these samples, which showed that the addition of CNTs improved the tensile strength by about 21%. This is attributed to the load transfer mechanism. Additional interesting observations included that ball milling was an effective method for CNT dispersion and that the CNTs acted as nucleation sites for void formation during tensile testing.
1.4: STRENGTHENING MECHANISMS

The strength of the aluminum can be improved by taking advantage of strengthening mechanisms, as discussed by H. Chen and B. Chen with regard to studies on aluminum composited with boron carbide and carbon nanotubes [21], [24]. The following points are a summary of key factors contributing to strengthening of aluminum composites.

1) Grain refinement: The presence of a composite material in the aluminum matrix can act as a site for grain boundary pinning. The Hall-Petch relationship shows that this refinement of grains increases strength of the overall composite.

\[ \sigma_y = \sigma_i + \frac{k}{\sqrt{D}} \]

Where \( \sigma_y \) is yield strength, \( \sigma_i \) is strength, \( D \) is grain size, and \( k \) is a material specific constant. The relationship suggests that grain size is inversely related to material strength.

2) Dislocation pinning: The presence of carbide nanoparticles in the aluminum matrix reinforces the material by Orowan looping, where carbide particles act as pinning points for dislocations.

3) Load transfer: The homogeneous dispersion of carbide particles within the matrix strengthens the aluminum if quality interfaces are formed between the particles and the matrix. This effect transfers the load from the aluminum matrix to the composite material, improving its performance in tensile and compressive conditions.

4) Solid solution strengthening: Diffusion of carbon into the aluminum matrix forming \( \text{Al}_4\text{C}_3 \). This is unlikely to occur at low sintering temperatures below 660 °C, so in an
aluminum composite system it is unlikely to be observed. However in the case that
the aluminum matrix material is an alloy which is typically heat treated (e.g. T6 –
solutionize, quench, and age), the formation of Guinier Preston zones (precipitates
which are coherent with the lattice) can act to improve mechanical properties by solid
solution strengthening.

In general, these mechanisms will be the primary contributors to strength improvements in
aluminum composite systems, and are considered throughout this study.

1.5: SUMMARY

In summary, metal matrix composites have been synthesized by various processing
methods including casting and powder metallurgy. The more promising composites were
produced via sintering due to the short processing cycle and energy savings, as well as finer
grain structure and more uniform dispersion of compositing material. The FAST processing
method shortens processing cycles even more than conventional sintering methods.

Although aluminum has been explored thoroughly as a MMC candidate material, there has
been limited work done on MMCs where the matrix material is an aluminum alloy as opposed to
pure aluminum, and even fewer studies have explored structural alloys such as Al-6061 or 2124
as a matrix material [20], [21]. In this thesis, the aluminum alloy 6061 and 2124 is explored with
SiC as the compositing material.

The homogenous addition of silicon carbide particles to the aluminum matrix has not been
successfully achieved in proportions greater than 30%. Also, limited work has been done in the
size range of 0.7 - 3 μm, where work that has been done shows promise in terms of mechanical
improvements [17]. In this thesis, SiC additions up to 40% were explored.
The work in this thesis strives to fill a gap in available structural materials by exploring FAST as a method to fabricate stronger composites than previously thought possible. The powder blending technology combined with FAST sintering allows for the fabrication of well dispersed aluminum alloy with silicon carbide composite materials, with high density and with superior mechanical properties, up to the 40% fraction by mass.

1.6: HYPOTHESIS

Attrition milled aluminum alloy powder with embedded silicon carbide particles in the 0.7 to 3 μm size range will be consolidated via field assisted sintering technology (FAST). This will produce a high volume fraction (20% - 40%) evenly dispersed Al-SiC composite microstructure. The composite system will take advantage of various strengthening mechanisms including grain refinement, dislocation pinning, and load transfer. This will create a composite material which has improved hardness and specific strength over conventional structural alloys.
Chapter 2: MATERIAL PROCESSING TECHNIQUES

Casting and sintering are the two main methods to process aluminum alloys. During casting, a material is brought above its melting temperature and shaped into an end product. Alternately, during sintering, consolidation of powder by solid state diffusion is a technique governed by diffusion, commonly referred to as sintering. Sintering in its essence refers to the densification of a powder material in a time and energy efficient manner. Sintering has been around in many forms for many years and noteworthy iterations of the process are described below. Alternate processing techniques have been discussed including casting of materials, which is a slow process requiring very high temperatures to actually melt the material being processed. Each process comes with its challenges and has the capability to generate parts with different mechanical and microstructural characteristics. This chapter includes a summary of the melting process as well as variations of the sintering process to assess their values and applications.

2.1: CASTING

Casting is one of the oldest methods of processing metals, dating back thousands of years [25]. The casting process is conducted with either a permanent mold or an expendable mold (generally made of sand) [26]. This batch process is capable of producing a near-net shape part, requiring surface machining of the part surface, the sprue, and interconnecting channels within the mold. A schematic of a typical sand-casting mold is illustrated in Figure 2.1.
The metal in a casting process is brought to its melting point and liquified. Upon liquification the metal is poured into a mold. Normally this mold is oversized to account for shrinkage of the material due to thermal expansion when it is in molten state. There is a long cooling time, due to the insulating nature of the surrounding mold. This contributes to large grains in the end product. In addition, segregation of secondary phases is a commonly reported undesirable side effect. Compositional segregation and inhomogeneous cooling of the part will affect microstructure and can result in a part that does not meet performance standards. The characteristic properties of a cast part include high density (100% of theoretical), coarse grain structure (depending on cooling times), and rough surface finish (depending on mold material) [27].

Issues with part chemistry and surface roughness are generally resolved with a post-process heat treatment and with machining [27]. In a cast aluminum alloy, a common issue is inhomogeneity in the grain structure and composition of the part. This arises due to the rapid cooling of the part surface in contact with the mold and slower cooling near the center of the part. The result is that alloying elements will preferentially migrate towards the center, making
the part weaker towards the outer, fast cooling regions. For this reason, it is typical to follow aluminum casting with a homogenization process, which is conducted near the melting temperature, to allow for the redistribution of these precipitates and create more uniform mechanical properties throughout the part.

Between the creation of the mold, the heating, the cooling, and the post process heat treatment and machining, casting is a very time-consuming process. This makes sintering an attractive alternative processing method.

2.2: PRESSURELESS SINTERING (PS)

Pressureless sintering is a consolidation method which relies solely on the application of heat to transform a green (non-sintered powder compact) part into a dense part. This process is different from casting in that temperatures never reach the melting point of the material. Sintering is typically conducted at 75% of the casting (melting) temperature of a material [28], meaning the energy cost is lower than that of casting. PS is a two-step process where first the green part needs to be made, and then it is sintered.

In pressureless sintering, no pressure is applied to the parts during the sintering step. The green part, however, is made by applying pressure to a powder, often with the assistance of organic materials to help hold the desired shape [29]. The addition of organics complicates the chemistry of the process because chemical compatibility between the powder and the surroundings needs to be considered to prevent any undesired chemical reactions. When the powder compact is made without the assistant of organics, this is referred to a “dry” process. Titanium and other metals are a good candidate material for a dry process because of their ductility and malleability [30].
An advantage to pressureless sintering is that multiple parts can be produced at a time, the only limitation being the size of the furnace. Two important characteristics of a material which has been sintered without the application of pressure are relatively low density and long sintering times [31]. Diffusion is being driven only by the heating process, and pressure is necessary for the elimination of porosity.

2.3: HOT PRESS (HP)

Hot pressing is a process by which densification is achieved by the concurrent application of heat and axial pressure to a powder. A schematic of a hot press system is shown in Figure 2.2. The combination of heat and pressure leads to the formation of necks between particles followed by densification of the material. Densification in hot pressing occurs by the diffusion of atoms between two adjacent particles forming a neck. The addition of uniaxial pressure to the system aids in elimination of porosity in the sintered part (Figure 2.3) [32].
Figure 2.2. Schematic diagram of HP processing setup.

Figure 2.3. (a) Packed powder. (b) Necking due to heat application. (c) Applied pressure eliminates porosity.
HP sintering temperatures are similarly approximately 75% of the melting point of a material. Heat is applied by indirect resistance heating, where a heating element outside of the die mold will heat the mold and the powder. Due to the indirect heating method, heating rates for hot press are relatively slow heating rates of up to 25 °C/min [33]. Applied pressure will encourage the elimination of porosity as illustrated in Figure 2.3. Due to the constraints of heating rate and pressure application, it is challenging to sinter samples at high production rates. A key characteristic of a hot pressed sample is its high density, and relatively fine grain microstructure as compared to casting [32]. A material that would be a good candidate for hot pressing would be one that has a low sintering temperature (due to low heating rates).

2.4: HOT ISOSTATIC PRESS (HIP)

Hot isostatic pressing, is a process that differs from hot pressing by the introduction of a pressure vessel. This pressure vessel is used to help achieve full densification by applying a more uniform pressure in all directions across the surface of the sample. Similar to hot pressing, this system relies on the combination of heat and pressure to achieve full density in a material system. It is no longer limited by a single axis of pressure application, rather it has the freedom to apply pressure from all directions. This allows for more complex geometries of parts to be produced which would fail to densify under uniaxial pressure [32]. A schematic of a hot isostatic press is shown in Figure 2.4. A sample material is placed into a pressure vessel which is the final desired shape. This can is placed into a pressure vessel which is subsequently filled with an inert gas. Typical pressures for a HIP process are in excess of 100 MPa. High temperature is achieved by a similar furnace heating process such as indirect resistive heating. HIP is capable of producing very high density parts as compared to other processing methods. Due to the high processing time and energy cost, it is used for materials where primary concern is the shape and
density. For instance, HIP is commonly used as a post-process technique for additively manufactured metal components [34], [35].

A key characteristic of a HIP part is that it will have superior microstructural uniformity as well as mechanical performance due to uniformly applied pressure. HIP is summarized as a two step process. First is the fabrication of a green or pre-compacted component, followed by the HIP process. These extra steps add to the manufacturing cost and also contribute to long processing cycles. The entire system is much bigger and more complex that its uniaxial counterpart HP [32].

Figure 2.4. Schematic of HIP process setup.
2.5: FIELD ASSISTED SINTERING TECHNIQUE (FAST)

Field Assisted Sintering is a relatively new sintering process. It is known by different names, one of them being Spark Plasma Sintering (SPS). Nomenclature of SPS is associated with localized plasma generated during the sintering of metal materials. There is no evidence for localized plasma formation during ceramic material sintering [36]. Therefore, a more appropriate, generic name, FAST, is chosen that is applied to metal, ceramic, and composite materials. The combination of heat, pressure, and high density current allows for rapid densification of powder [37], illustrated in Figure 2.5.

![Schematic diagram of the Field Assisted Sintering Technique (FAST) system.](image)

**Figure 2.5.** Schematic diagram of the Field Assisted Sintering Technique (FAST) system.
FAST is similar to hot pressing, that a uniaxial pressure is applied to the sample for compaction. A typical FAST setup has a die loaded with powder to be sintered. The die will have a graphite foil lining to protect the die from reacting with the powder during sintering. The loaded die is then placed into the FAST system, where the mold is insulated with graphite blankets to minimize heat loss. Sintering is done either in vacuum or back filled with argon or nitrogen gas. There are four major sintering process valuables – pressure, temperature coupled with high current density, heating rate and time. A typical sintering curve can be seen in the sections to follow. (3.2.1)

High density current plays a significant role in the sintering of metal alloys via FAST. Part of the current applied to the system will flow through the die (radiation) and part will flow through the powder (volumetric heating). This comes from resistive heating at the interface area between particle contacts. Combined radiation heating and volumetric heating allows for rapid heating rates that can range up to 300 °C/min [38] – depending mold material and size. The high heating rate is favorable in minimizing grain growth in a sintered material. Another important contribution of current is the electromigration effect [39], which is basically the current itself contributing to the transport of atoms between adjacent particles. This enhances the diffusion process.

The Peltier effect is an electric effect which will also transfer heat between particles. This effect can contribute up to 10% of the total heating to the system. The Peltier effect takes place primarily at the interface between the die punch and the powder, and is related to the applied current direction. In the case of FAST, it is expected to be observed during the sintering of semiconducting powders such as silicon carbide, whereas in metals it is very minimal and can be ignored [40], [41]. This effect is illustrated in Figure 2.6, where the thermal gradient can be
observed to be migrating heat downward, implying that in this case holes are the majority charge carrier. This will have effects on the resulting material properties, as well as influencing pyrometer readings during sintering.

The Peltier effect along with radiative and resistive heating are the primary contributors to the rapid heating and reduced activation energy observed in FAST sintering. The key characteristics of a sample produced by the FAST process are a fine, uniform grain microstructure, high density, and superior mechanical properties. FAST is a very short processing cycle as compared to casting, PS, HP, and HIP.

**Figure 2.6.** Schematic diagram of the Peltier Effect. Heat is being redistributed down through the sample. This will affect material properties and densification.
Chapter 3: EXPERIMENTAL PROCEDURE

3.1: DEVELOPMENT OF POWDER

Al-SiC powder was provided by Materion Performance Alloys in Ohio. Two similar powders were provided, one with a silicon carbide particle size of 0.7 microns, and one with silicon carbide particle size of 3 microns. Different volume fractions of SiC particles were blended with aluminum alloy powder by attrition milling. Due to the repeated impact of high hardness silicon carbide powder particles with the relatively low hardness and high ductility aluminum particles, powders were able to mechanically bond during the milling process. Overall powder composition is given in Table 3.1, followed by alloy composition in Table 3.2. The blended AMC powders were provided by Materion and therefore the information about the original aluminum particle size is not available. The post-milling particle sizes listed below were obtained from measurements using the Malvern Mastersizer 3000 (Laser Diffraction). This piece of equipment has a resolution of 10 nm to 3500 μm, so it is sufficient to capture the full distribution of particle sizes in this study.

Table 3.1. Aluminum Samples Sintered by FAST Process

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Aluminum</th>
<th>ECKA* Alloy</th>
<th>AMC620XF</th>
<th>AMC225XE</th>
<th>AMC640XA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>100% Al-104</td>
<td>Proprietary</td>
<td>6061B</td>
<td>2124A</td>
<td>6061B</td>
</tr>
<tr>
<td>SiC Addition</td>
<td>0%</td>
<td>0%</td>
<td>20%</td>
<td>25%</td>
<td>40%</td>
</tr>
<tr>
<td>SiC Particle Size</td>
<td>N/A</td>
<td>N/A</td>
<td>0.7 μm</td>
<td>3 μm</td>
<td>3 μm</td>
</tr>
<tr>
<td>Al-SiC Particle Size</td>
<td>( \bar{x} ): 71.8 μm</td>
<td>( \bar{x} ): 43.2 μm</td>
<td>( \bar{x} ): 272 μm</td>
<td>( \bar{x} ): 347 μm</td>
<td>( \bar{x} ): 370 μm</td>
</tr>
<tr>
<td>( D_{50} )</td>
<td>69 μm</td>
<td>35.4 μm</td>
<td>163 μm</td>
<td>283 μm</td>
<td>238 μm</td>
</tr>
</tbody>
</table>

* ECKA is the trade name of the company. The powder composition is similar to 6061B (Table 3.2)
Composite powders were placed in a graphite mold and transferred to the FAST system to be sintered at various pressures (35-55 MPa) temperatures (450-550 °C), and times (5-20 minutes) under vacuum. Two sets of samples were sintered using two different FAST units. The first set was the sintering of 40 mm diameter by 5 mm thickness samples on a 25 metric ton FAST unit. Optimal sintering conditions were determined for samples of such compositions as listed in Table 3.1. In both cases samples were afterwards sandblasted to remove any graphite residue and density was evaluated using Archimedes Principle. These samples were cut and polished for microstructural characterization, which included SEM, EDS, XRD, TEM, and hardness measurements. The second set of samples sintered was on a 250 metric ton FAST system. These samples were discs of dimension 120 mm diameter by 25 mm thickness. The

<table>
<thead>
<tr>
<th>Element</th>
<th>Al - 104</th>
<th>6061B</th>
<th>2124A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>&gt; 99.0</td>
<td>96</td>
<td>93</td>
</tr>
<tr>
<td>Magnesium</td>
<td>&lt; 0.01</td>
<td>1</td>
<td>1.5</td>
</tr>
<tr>
<td>Silicon</td>
<td>&lt; 0.01</td>
<td>0.5</td>
<td>0.2</td>
</tr>
<tr>
<td>Chromium</td>
<td>&lt; 0.01</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>Copper</td>
<td>&lt; 0.01</td>
<td>0.3</td>
<td>4.5</td>
</tr>
<tr>
<td>Iron</td>
<td>&lt; 0.01</td>
<td>0.7</td>
<td>0.3</td>
</tr>
<tr>
<td>Zinc</td>
<td>&lt; 0.01</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Manganese</td>
<td>&lt; 0.01</td>
<td>0.15</td>
<td>0.6</td>
</tr>
<tr>
<td>Titanium</td>
<td>&lt; 0.01</td>
<td>0.15</td>
<td>0.15</td>
</tr>
</tbody>
</table>

3.2: FAST PROCESS

Table 3.2. Percent Composition of Aluminum Alloys

<table>
<thead>
<tr>
<th>Element</th>
<th>Al - 104</th>
<th>6061B</th>
<th>2124A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>&gt; 99.0</td>
<td>96</td>
<td>93</td>
</tr>
<tr>
<td>Magnesium</td>
<td>&lt; 0.01</td>
<td>1</td>
<td>1.5</td>
</tr>
<tr>
<td>Silicon</td>
<td>&lt; 0.01</td>
<td>0.5</td>
<td>0.2</td>
</tr>
<tr>
<td>Chromium</td>
<td>&lt; 0.01</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>Copper</td>
<td>&lt; 0.01</td>
<td>0.3</td>
<td>4.5</td>
</tr>
<tr>
<td>Iron</td>
<td>&lt; 0.01</td>
<td>0.7</td>
<td>0.3</td>
</tr>
<tr>
<td>Zinc</td>
<td>&lt; 0.01</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Manganese</td>
<td>&lt; 0.01</td>
<td>0.15</td>
<td>0.6</td>
</tr>
<tr>
<td>Titanium</td>
<td>&lt; 0.01</td>
<td>0.15</td>
<td>0.15</td>
</tr>
</tbody>
</table>
larger samples were cut by wire EDM into tensile bars with surface finish Ra 23 for further mechanical testing. A schematic of the tensile bars is shown in Figure 3.1.

![Figure 3.1. Schematic diagram of tensile bars cut by wire EDM from large aluminum discs.](image)

### 3.2.1: Sintering Plots

The aluminum and silicon carbide powder mixtures were all sintered using FAST, with sintering parameters shown in Figures 3.2 to 3.6. Sintering among all samples follows a general trend of an initial increase in pressure to 35 MPa, where it is held until temperature reaches 425°C. At this point temperature and pressure are simultaneously ramped up to the sintering point (45 MPa and 550°C). Once there, the sample is held at the sintering point for 25 minutes until full density is achieved as evidenced by no change in the relative ram position on the sintering plots. The relative ram position gives information about the densification of the powder, where piston travel is directly related to powder compaction. It can be seen that at the 25 minute mark in all cases (Figures 3.2 – 3.6) the piston travel has levelled out, which signals that maximum compaction density has been achieved for the given temperature and pressure setting.

Densification speeds differ between the pure aluminum and samples and the aluminum with SiC additions, as seen in Figures 3.2 and 3.5, where relative ram position levels out much
faster during the sintering of pure aluminum powder. This indicates that pure aluminum is densifying much faster than the alloy material, which makes sense due to the lower sintering temperature of aluminum. The material is densifying before the secondary increase in temperature and pressure takes place. The densification of the three aluminum alloy with silicon carbide samples (Figures 3.4-3.6) are very similar in behavior, where densification begins at the initial temperature and pressure hold step (at 425°C), and but does not reach 100% completion until the temperature and pressure ramp up to 550°C and 45 MPa.
Figure 3.2. 100% aluminum sintering plot.

Figure 3.3. 100% aluminum alloy sintering plot.
Figure 3.4. Aluminum alloy with 20% SiC addition sintering plot.

Figure 3.5. Aluminum alloy with 25% SiC addition sintering plot.
In order to evaluate microstructural characteristics, polished 40 mm diameter samples were etched using Keller’s reagent to reveal microstructure for optical and SEM evaluation. Table 3.3 shows the recipe for Keller’s reagent.

**Table 3.3.** Keller’s Reagent Recipe

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Volume (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>190</td>
</tr>
<tr>
<td>Nitric acid</td>
<td>5</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>3</td>
</tr>
<tr>
<td>Hydrofluoric acid</td>
<td>2</td>
</tr>
</tbody>
</table>

**Figure 3.6.** Aluminum alloy with 40% SiC addition sintering plot.

### 3.3: ETCHING

In order to evaluate microstructural characteristics, polished 40 mm diameter samples were etched using Keller’s reagent to reveal microstructure for optical and SEM evaluation. Table 3.3 shows the recipe for Keller’s reagent.
Etching is a method of revealing grain structure on a polished material’s surface. Commonly this is done by selective chemical reaction at the grain boundaries of a material so that the grains themselves will be visible when observed through a microscope. There are two methods which are possible for etching samples. One method of etching the samples is chemically treating the surface with an acid using a swab. Another method is to immerse the sample in an acid bath. In this study the surface of the aluminum samples were immersed in Keller’s reagent for 0-30 seconds to reveal microstructural characteristics of the sample, before observation with SEM and optical microscopes.

3.4: CHARACTERIZATION

Sintered samples were characterized by various techniques including optical, scanning electron microscopy, x-ray diffraction, transmission electron microscopy, and mechanical evaluation by hardness and tensile testing.

3.4.1: Vickers Hardness

Samples of 40 mm diameter were cut, mounted, and polished to a 0.05 micron finish. Polished samples were tested for hardness using a Leco LM248AT indenter with a standard diamond tip for Vickers measurements at 500 g load as per ASTM standard E92-17.

Vickers hardness operates on a relative scale ranging from 0 to 10,000 VHN (Vickers Hardness Number). The maximum rating of 10,000 is assigned to diamond, which is fittingly the material used as the tip of the indentation device. The shape of the diamond tip is resembling that of a pyramid. The instrument works on the basis that an indentation is format on the polished surface at a given load. The surface area of the indent is directly proportional to the length of the diagonals of the indent formed on the surface of the sample. This measurement is then assigned a
VHN. Typically a series of five random indents is taken across the surface of the sample, and the average VHN is the reported value. Calculation of the VHN is illustrated below.

\[
Vickers \text{ Hardness (VHN)} = \frac{\text{applied force}}{\text{surface area of indent}}
\]

The surface area of the indent can be calculated by multiplying the diagonal of the indent by a constant (0.5393). This constant is derived from the fact that the standardized angle of the pyramid face is 136° [42]. Where \(d\) is the length of the diagonal and \(F\) is the force applied.

\[
surface \text{ area of pyramid} = d^2 \cdot \frac{1}{2 \cdot \sin\left(\frac{\text{face angle}}{2}\right)} = d^2 \cdot \frac{1}{2 \cdot \sin\left(\frac{136}{2}\right)} = d^2 \cdot 0.5393
\]

\[
VHN = \frac{F_{(kgf)}}{d_{(mm)} \cdot 0.5393}
\]

**Figure 3.7.** Schematic of a Vickers indentation.

The multiple indents on each sample were measured using an optical microscope. The plotted data is reported in the results and discussion (Section 4.1.2). The hardness in this study was conducted at 500 g load, with a 10 second dwell time and measured with a 25x lens objective. Five indentations were averaged per sample.
3.4.2: Elastic Modulus via Sound Velocity

Measurements of sound velocity were conducted using Mistras Acousto-Ultrasonic Technology instrument. Two transducers each for longitudinal and shear wave measurement via ultrasonic wave reflections were implemented in this process. A couplant was applied to each side of the sample and waves were sent through using the pulse-echo technology. For longitudinal waves the couplant of choice was glycerin, and for transverse waves the couplant is a honey based substance which has high a transmission coefficient. A setup of the experiment is shown in Figure 3.8.

![Figure 3.8. Schematic of sound velocity measurement setup.](image)

The velocity of sound through a material can be used to estimate a material’s elastic modulus. The principle of this approach depends upon the bonding between atoms in a material that will affect the speed at which a wave propagates. A material with a higher stiffness will allow acoustic waves to propagate through the sample at a higher velocity than a material with a lower stiffness. The velocity of the wave is determined by dividing the thickness of the sample by the time that it takes for the sound to transmit.

\[
Velocity = \frac{Distance}{Time}
\]
\[ V_T = \text{Transverse wave speed} \]
\[ V_L = \text{Longitudinal wave speed} \]

\[
\text{Poisson's Ratio } (\nu) = \frac{1 - 2(V_T/V_L)^2}{2 - 2(V_T/V_L)^2}
\]

\[ \rho = \text{sample density} \]

\[
\text{Elastic Modulus} = \frac{V_L^2 \rho (1 + \nu)(1 - 2\nu)}{1 - \nu}
\]

The elastic modulus is calculated as seen in equation 3 [43]. These values are discussed in the results and discussion, Section 4.1.3.

3.4.3: X-Ray Diffraction

X-Ray Diffraction (XRD) scans were done to identify the phases present in the samples after sintering. XRD patterns were obtained from all 40 mm samples using the Empyrean system by Malvern Panalytical. The scan was conducted with a copper target, 20-90 degrees with a 15 minute duration. This system is used to confirm present phases in a material. X-ray diffraction operates on the principle of Bragg’s Law.

\[ n \lambda = 2 d \sin(\theta) \]

Where \( d \) is the lattice spacing, \( \lambda \) is the wavelength, \( \theta \) is the angle of the incident beam, and \( n \) is an integer. When the lattice spacing of a material is related to the wavelength of the x-ray beam by an integer, \( n \), there is an observation of constructive interference. The pattern of constructive interference at given angles \( \theta \) is characteristic of specific material phases. Using these peaks as guidance, the phases present in a fabricated material can be identified.
### 3.4.4: Scanning Electron Microscopy

Scanning electron microscopy (SEM) was a useful tool in this study to look at the dispersion of silicon carbide particles within the aluminum matrix, as well as the aluminum grain structures. This was done using the FEI Nova NanoSEM 630, with a FEG emitter. SEM was conducted on the surfaces of the polished samples after etching. Additional SEM images were taken of fracture surfaces of the tensile tested samples as well as the cross section of the samples.

SEM operates on the principle of obtaining contrast by atomic number of particular elements. In the SEM a focused electron beam is scanned across the surface of a sample, where the difference in energy of scattered or secondary electrons allows for contrast to be obtained at any given point. Secondary electrons arise from closer to the surface of the sample and so they are primarily used in collecting information about the sample’s surface features. Backscatter electrons come from deeper within the sample and are more useful for discerning the chemical composition of a sample.

### 3.4.5: Energy Dispersive X-ray Spectroscopy

Energy dispersive x-ray spectroscopy (EDS) is a characterization method which is used to identify elements present in a particular region of a sample. It is often built in as an additional feature to an SEM where elemental mapping of the surface can be done. EDS detects the aforementioned backscatter electrons. It operates on the principle that an incident electron’s interaction with a particular element’s outer shell electrons will cause it to scatter. This will leave the backscattered electron with a specific energy upon returning to the detector. These backscattered electrons are collected and mapped across the surface of a sample so that a map of elemental composition is constructed.
EDS maps were obtained for confirmation of silicon carbide dispersion throughout the sample after sintering, as well as for identifying elements at the points of failure on the fracture surface of the tensile samples.

3.4.6: Transmission Electron Microscopy

Transmission electron microscopy (TEM) is a technique used to examine internal microstructure of a material at much higher magnification. The machine used was the FEI Titan G2. High resolution TEM was employed to examine the interface and bonding between silicon carbide particles with the aluminum sample.

For high resolution TEM (HRTEM), samples were prepared by manual polishing followed by ion milling by focused ion beam (FIB). High Resolution TEM imaging was conducted at 200kV on 6061B aluminum sample with 40% silicon carbide.

3.5: MECHANICAL TESTING

Cylindrical tensile specimens were made and cut via wire electric discharge machining (EDM) from a large sintered disc. These samples were then mechanically tested at Case Western Reserve University. Samples were clamped into the fixture as shown in the Figure 3.10. Samples were machined to approximately 60.45 mm in length and 5.05 mm in diameter with a surface finish 23 Ra using diamond impregnated cutting tool as shown in schematic in Figure 3.1. Figure 3.9 is an image of a tensile sample before testing. Test conditions are listed in Table 3.4. The setup of the tensile test is pictured in Figures 3.10 (a) and (b).
Table 3.4. Mechanical testing conditions.

<table>
<thead>
<tr>
<th>Test Input</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strain Rate</td>
<td>1.3716 mm/min</td>
</tr>
<tr>
<td>Starting Load</td>
<td>150.000 N</td>
</tr>
<tr>
<td>Data Acquisition Rate</td>
<td>5.0 Hz</td>
</tr>
<tr>
<td>Break Threshold</td>
<td>222.411 N</td>
</tr>
<tr>
<td>Break Sensitivity</td>
<td>90%</td>
</tr>
</tbody>
</table>
Figure 3.10. Mechanical testing setup at Case Western Reserve University. (a) Whole machine setup. (b) Close up of tensile bar and grips.
Chapter 4: RESULTS AND DISCUSSION

This chapter summarizes the results of the destructive and nondestructive evaluation of mechanical and microstructural properties of sintered aluminum samples.

4.1: PHYSICAL PROPERTY NON DESTRUCTIVE EVALUATION

Non destructive evaluations included optical microscopy (OM), SEM, EDS, TEM, XRD, sound velocity measurements, and hardness.

4.1.1: Powder SEM and EDS

First, the aluminum and silicon carbide powder mixtures were characterized using SEM and EDS. SEM images of powder particles for aluminum samples with silicon carbide addition can be seen in Figures 4.1 to 4.5, along with corresponding EDS maps.

Particle size and shape were observed using SEM. Powder size ranges were compared with reported values as seen in Table 3.1. SEM images revealed that silicon particle sizes corresponded well with the reported values. Aluminum particles for pure powder appear to be irregularly shaped, and about 50 μm in size. ECKA alloy powder particles are spherical and range in size from 10 to 50 μm. The attrition milled powders from Materion had a much broader size range due to the agglomerations that formed during milling. The aluminum particles with embedded silicon carbide size range was measured to be very large, from 50 to 500 μm for all three composite powders. Additionally, the irregular shape of the particles shows that the aluminum particles have been fractured during the attrition milling process, which confirms that the particles were mechanically bonded with silicon carbide by attrition milling. The SEM
images give a clear indication of embedded silicon carbide particles within the aluminum matrix material.

Figure 4.1. SEM of aluminum powder with EDS map. The EDS map indicates that the entirety of the particle is pure aluminum metal.
Figure 4.2. SEM of ECKA Al alloy powder with EDS map. The EDS map confirms presence of majority aluminum as well as several alloying elements.
**Figure 4.3.** SEM of Al alloy + 20% SiC powder with EDS map. SiC particles are embedded in the aluminum agglomerations.
Figure 4.4. SEM of Al alloy + 25% SiC powder with EDS map. Again, SiC can be seen embedded in the aluminum agglomerations.
4.1.2: Vickers Hardness

The Vickers Hardness Numbers (VHN) of various sintered samples are shown in Figure 4.6. The Figure shows that VHN increases as a function of silicon carbide addition to the aluminum matrix. Samples with 40% silicon carbide addition are observed to have over 500%
improvement in hardness as compared to pure aluminum. This translates to a 58% improvement over the ECKA aluminum alloy.

This observation is expected because of the previously mentioned strengthening mechanisms involved with the compositing of aluminum with silicon carbide. Primarily, with grain refinement (Hall-Petch equation) which predicts that there should be an increase in hardness. This refinement of grain size comes from the silicon carbide particles acting as grain boundary inhibitors. In addition, the inclusion of silicon carbide in the matrix inhibits the flow of aluminum during compression or indentation, improving the overall hardness measurement.

![Aluminum - SiC Hardness Vickers (500g load)](image)

**Figure 4.6.** Vickers hardness indentation values.

There will be additional contribution to overall hardness increase from the Coefficient of Thermal Expansion (CTE) mismatch effect, which causes strains in aluminum matrix material surrounding silicon carbide particles. There will also be improvement in hardness by the presence of silicon carbide and the load transfer mechanism, which suggests that the silicon
carbide in the matrix is improving the overall material’s strength by absorbing load that is imposed on the material.

The rule of mixtures is a useful calculation which can be used to predict a composite system’s performance. In the case of a metal matrix composite system, work has been done by the Kim group [44] in effort to understand the behavior of composite systems. This group has shown that in a constant stress condition, the hardness of a composite will behave more similarly to the inverse rule of mixtures. However, in a constant strain condition, the material will perform more similarly to the rule of mixtures.

- Rule of mixtures (RM):

\[ H_{\text{composite}} = f_{\text{Al}} \times H_{\text{Al}} + f_{\text{SiC}} \times H_{\text{SiC}} \]

- Inverse rule of mixtures (IRM):

\[ H_{\text{composite}} = \left( \frac{f_{\text{Al}}}{H_{\text{Al}}} + \frac{f_{\text{SiC}}}{H_{\text{SiC}}} \right)^{-1} \]

Where \( f \) is the volume fraction and \( H \) is the hardness. Comparing the Al-SiC system to the two different rules of mixtures reveals that overall behavior is more similar to the constant stress condition (IRM), which would be expected in a composite where one material is very soft compared to the other. Figure 4.7 shows the comparison with the RM and IRM, where RM is overestimating the actual hardness values and IRM is very closely predicting.
Figure 4.7. Hardness of composite materials compared to expected values to rule of mixtures and inverse rule of mixtures, showing trend much closer to IRM.

Due to hardness of silicon carbide (2628 HV,[45]), the RM predicts a high hardness, especially at the 40% silicon carbide addition. The reason why this prediction is not experimentally observed is because hardness indentations were conducted at 500g load, which is high enough that most of the aluminum is flowing around the silicon carbide particles. This negates the SiC contribution to the overall hardness, and the constant strain condition is not met. This experimental setup gives a better understanding of overall bulk properties, which is preferred for the purpose of this study.

Performing the same measurements with a lower load shows a trend closer to the RM, as would be expected due to the silicon carbides ability to absorb more of the load, and reduced deformation of aluminum. It can be stated that the behavior of a composite system will shift between the RM and IRM as a function of both the volume fraction of particle addition as well as
the load used in measuring the hardness. The hardness was measured for each sample at varying loads and plotted in Figure 4.8.

![Hardness Trend with Varying Load](image)

**Figure 4.8.** Hardness trend at varying loads shows that lower loads tend to reveal more of SiC contribution.

The difference in hardness at varying loads is most clear in the 40% silicon carbide addition with 10g load. The standard deviation is very large at this point because the hardness is much more sensitive to the microstructure beneath the surface of the sample. If there is a high density of silicon carbide particles in a location where the indent has been made, the hardness will be much higher than if it is mostly aluminum beneath the surface. The behavior in this case is as expected, where at a much lower load the hardness trend shifts towards the RM predition, approaching a constant strain condition.
4.1.3: Elastic Modulus via Sound Velocity

Elastic modulus of samples was estimated using the sound velocity technique, with results shown in Figure 4.9.

![Elastic Modulus by Sound Velocity](image)

**Figure 4.9.** Elastic modulus of samples showing improvement with added silicon carbide.

The measurements reveal very similar elastic modulus between ECKA Alloy, AMC620XF (Al + 20% SiC) and AMC225XE (Al + 25% SiC). There is a 38% improvement in elastic modulus in AMC640XA (Al + 40% SiC) over pure aluminum material. This result makes sense as the silicon carbide addition to the matrix is increasing the material’s overall stiffness. (The elastic modulus of silicon carbide is around 434 GPa [45].) However, comparing this to the rule of mixtures, it is revealed that the values are lower than what would be expected as seen in Figure 4.10.
It should be considered that the rule of mixtures is derived from the assumption that there is equal strain on the lattice as well as the silicon carbide particles in the composite system, similarly to the situation with the hardness measurements. In this case, it is expected that the aluminum should deform much more than the silicon carbide material, due to the large difference in elastic modulus. It may be more accurate to assume that there is an equal stress being experienced in the composite system, rather than strain. Deriving this yields the inverse rule of mixtures.

**Figure 4.10.** Comparison of elastic modulus measure by sound velocity with predicted values by rule of mixtures.
mixtures, where the $f$ is the fraction of the respective material and $E$ is the corresponding elastic modulus.

$$E_{\text{composite}} = \left( \frac{f_{\text{Al}}}{E_{\text{Al}}} + \frac{f_{\text{SiC}}}{E_{\text{SiC}}} \right)^{-1}$$

Plotting this with the data shows a much more accurate prediction of the composite material performance as seen in Figure 4.11.

![Elastic Modulus by Sound Velocity compared to Rule of Mixtures](image)

**Figure 4.11.** The plot of inverse rule of mixtures compared to the experimental results for elastic modulus by sound velocity. This shows a much stronger correlation between predicted and experimental values.

This relationship makes more sense and supports that this behavior is dominated by the load transfer mechanism allowing for near equal stress distribution between the materials.
4.1.4: Sintered Samples: Microstructure by SEM and EDS

Figures 4.12 and 4.13 are SEM images of the pure aluminum and baseline aluminum alloy samples, where large grain structures can be seen (on the order of 20 μm). SEM images of the sintered composite samples, as shown in Figures 4.14 to 4.16, are clear indicators of uniform dispersion of the SiC particles throughout the Al matrix. It is difficult to see the grain structure of the composite materials, because the high volume fraction of SiC has caused the grains to become very small. Additionally, there is evidence of porosity which can be accounted for as pullout of silicon carbide from the sample surface during polishing process. This is due to the difference in hardness between the silicon carbide particles and the aluminum matrix, allowing for aluminum to polish away more quickly causing silicon carbide particles to pull out of the material.
Figure 4.12. SEM image of aluminum sample etched microstructure.

Aluminum 100%
Figure 4.13. SEM image of ECKA aluminum alloy sample etched microstructure.
Figure 4.14. Al alloy + 20% SiC sintered image. SiC particles evenly distributed throughout microstructure.
Figure 4.15. Al alloy + 25% SiC sintered image. SiC particles are still well distributed but there is a large amount of Al₂Cu also formed and distributed throughout. This is supported by XRD scans in Section 4.1.5.
Image analysis has been conducted on the three composite samples using MATLAB R2018a to evaluate whether the surface area fraction of silicon carbide is consistent with the volumetric additions. Due to the low contrast between aluminum and silicon carbide under the SEM, it is difficult to completely eliminate noise within the SiC particles and the aluminum.

Figure 4.16. Al alloy + 40% SiC sintered image. Even at this high volume fraction, SiC is well dispersed throughout the microstructure.
matrix. Performing analyses at four random locations throughout the microstructure, the results turned out to be reasonably near the correct fractions as illustrated in Figures 4.17 to 4.19.

**Figure 4.17.** Al alloy + 20% SiC image analysis. This sample has some noise due to the smaller silicon carbide particle size as compared to the 25 and 40% samples.
**Figure 4.18.** Al alloy + 25% SiC image analysis. This sample has fewest contrast issues, although the primary concern was that some of the Al$_2$Cu phase could be mistaken for SiC particles. This was avoided as best as possible for the analysis.
Figure 4.19. Al alloy + 40% SiC image analysis. Due to the large size of many of the silicon carbide particles in this sample, some are falsely attributed as matrix material. This is reflected in the slightly low calculated fraction in three cases.
4.1.5: X-Ray Diffraction

XRD scans show that among the silicon carbide and aluminum peaks, there are a few extra phases that are present in the aluminum alloys materials. XRD scans of the aluminum and silicon carbide sintered samples shows the increased percentage of silicon carbide addition as observed by increased intensity of silicon carbide peaks across scans included.

![Aluminum with Silicon Carbide XRD peaks](image)

**Figure 4.20.** XRD scan comparison of aluminum samples with SiC additive. ECKA Alloy and Al+25% SiC sample both have extra phase formation.
4.1.6: Transmission Electron Microscopy

TEM was employed to examine the interface between the SiC and aluminum matrix. Due to a difference in milling rate during FIB, it was difficult to obtain a high resolution image of both the SiC and the aluminum regions at the same time. This is because aluminum is damaged more than silicon carbide during ion milling due to lower bond strength as indicated by low hardness. Images of the aluminum and silicon carbide interface are shown in Figure 4.21. There is evidence of possible twinning structure in the silicon carbide region.

![TEM Low Mag and HRTEM](image)

**Figure 4.21.** TEM of SiC particle and aluminum interface after sintering.

At the interface between the silicon carbide and aluminum, there is a very thin amorphous region that is rich in copper, magnesium, and oxygen as evidenced by elemental mapping in Figure 4.22. The thickness of this region is approximately 1-2 nanometers. The amorphous region can be seen by the lack of planar periodicity in the TEM image. It is hypothesized that this amorphous region arises due to the migration of alloying elements with the current during sintering. High temperature, current, and pressure allow alloying elements to...
diffuse from the outer edge of aluminum matrix toward the silicon carbide particles, where it will settle on the boundary and form a high entropy amorphous region. It is interesting to note that although there is a mismatch in coefficient of thermal expansion (CTE) between these two materials, no delamination has been observed between the matrix and the particles.

Figure 4.22. EDX Scan of Al - SiC interface at amorphous interface region.
4.2: PHYSICAL PROPERTY DESTRUCTIVE EVALUATION

The mechanical testing of these samples generated data about the materials tensile behavior, which was used to extract information such as the materials’ elastic modulus, yield strength, ultimate tensile strength, and fracture behavior.

4.2.1: Tensile Test

Digital images of tensile samples after mechanical testing was performed are shown in Figure 4.23. An unbroken sample is included in the images to show elongation effects of the silicon carbide addition. Pure aluminum exhibited elongation about 30% during tensile testing, whereas aluminum alloy and silicon carbide samples had very little elongation (<1%).

![Digital image of tensile samples after testing.](image)

**Figure 4.23.** Digital image of tensile samples after testing. (a) Pure aluminum sample with approximately 30% elongation. (b) Aluminum alloy sample with very little elongation (<1%). (c,d,e) Aluminum with 20%, 25%, 40% silicon carbide addition, showing very little elongation.

Elastic modulus values derived from sound velocity measurements were compared to the modulus obtained from tensile testing. This was done to evaluate the accuracy of the estimate from sound velocity. Both approaches exhibited similar results as can be seen in Figure 4.24.
Elastic modulus comparison of mechanical test versus values derived from sound velocity method.

Elastic modulus increased with addition of silicon carbide particles to the aluminum matrix.

Using the mechanical testing data, 55% improvement was observed in elastic modulus over baseline Al alloy. Variation in calculation arises from selection of the elastic region within the stress-strain plots. As would be expected from a material system with improved stiffness, ductility is minimal among all the samples except for the pure aluminum, as can be seen in Figure 4.23. This can also be observed in the fracture surface images included in Section 4.2.2, where the ductility is so minimal that it is difficult to measure or distinguish amongst the alloy and composite samples. Information about the material yield strength and ultimate tensile strength can also be extracted from a tensile test. An example for Al + 40% SiC is shown in Figure 4.25. Additional plots for other samples are included in the Appendix.
Figure 4.25. Stress strain curve generated by plotting raw data from tensile test. Key properties highlighted.

The plot in Figure 4.25 is constructed by calculating stress and strain raw data generated during tensile testing. Ultimate tensile strength (UTS) is the maximum stress that a material can withstand before. In the case of brittle failure – the UTS is the applied force at failure divided by the cross sectional area of the samples. The elastic modulus of the samples is extracted by calculating the slope in the linear region of the stress strain curve. This may vary slightly from person to person, depending on what is considered the linear region. For this reason the calculations were repeated by Penn State as well as Case Western to assure consistency. The yield strength of the material was calculated by drawing a line at a 0.2% offset of the strain
parallel to the elastic modulus line. The point of intersection between the offset line and the original stress-strain curve is the yield stress of the material.

Yield stress calculations resulted in minimal variation between calculations. It was observed that yield strength increased with increased percentage of added silicon carbide. There is about a 29% improvement over the baseline alloy with a 20% silicon carbide addition. The improvement increases to 97% with a 40% silicon carbide addition.

![Yield Strength](image)

**Figure 4.26.** Yield Stress data compared with percentage improvement compared to aluminum and aluminum alloy.

The elongation of the materials was difficult to measure due to the brittle failure mode of the materials. The only accurate measurements could be made on the 100% Aluminum samples, which was about 40% elongation. The remainder of the samples all had elongations of less than 1%, based on a rough measurement of the final length. Among the composite and alloy samples a difference in elongation percentage is indistinguishable.
Ultimate tensile strength shows similar trend of increasing strength with increasing silicon carbide content. A 53% increase in UTS is observed over baseline alloy in the 40% silicon carbide sample, whereas over 600% improvement over pure aluminum.

![Ultimate Tensile Strength](image)

**Figure 4.27.** Ultimate Tensile Strength (UTS) of each sample.

### 4.2.2: SEM of Fractured Surface

Figures 4.28 to 4.32 are images of a top view of the fractured tensile bars. For the pure aluminum sample, Figure 4.28, there is evidence of large amounts of dimpling in the surface, as would be expected for very ductile failure. The fracture surface for the ECKA aluminum alloy, Figure 4.29, shows less dimpling, reflecting the more brittle nature of the failure. The fracture surfaces shown in Figures 4.30 to 4.32 shows that there is evidence of fracture origin in regions rich in silicon carbide. This is also observed to be a low point on the surface of the sample which is likely the point of failure. High carbon concentration in this region supports this claim.
Figure 4.28. 100% aluminum high magnification SEM image. Evidence of dimpling on surface.
Figure 4.29. 100% ECKA alloy high mag SEM images. More evidence of dimpling on surface showing some signs of ductility.
Figure 4.30. Al alloy + 20% SiC high mag SEM images. Evidence of low amounts of dimpling in these surfaces.
Figure 4.31. Al + 25% SiC EDS scan of particle on surface of high mag SEM image. Possible origin of fracture.
The dimpling observed at this surface is an indication that the matrix is still retaining some ductile properties even though samples showed minimal elongation during tensile testing (less than 1%). This observation also supports the idea that origin of fracture is occurring at the interfaces between aluminum and silicon carbide particles, which is further clarified in the side view images seen in Section 4.2.3.

**Figure 4.32.** Al alloy 40% SiC high mag SEM image with EDS scan confirming fracture at high SiC content region.
4.2.3: SEM of Side View

SEM images are taken of the neck region of the sample as shown in Figure 4.33. This was done to assist in identification of the origin of the cracks that led to failure during tensile testing.

**Figure 4.33.** Schematic diagram of location for side view images.

The samples were expected to show evidence of elongated grains near the point of fracture and this can be seen in SEM images in Figures 4.34 to 4.38. Figure 4.34 shows the expected elongation of grains near the point of failure, which occurred along the grain boundaries of the pure aluminum. As for the aluminum alloy, it can be seen in Figure 4.35 that the crack originates at the interface between two phases in the alloy and then propagates along the boundary of the different phases in the microstructure.

In a similar way the crack is being redirected by silicon carbide particles as seen in Figures 4.36 to 4.38. In these images it seems as though the crack is generally originating at or near the boundary between the matrix and a silicon carbide particle. This is expected because these interfaces are where stress is concentrated during tensile load. The charging in these images is due to the bakelite mounting material and is not the tensile sample itself.
Figure 4.34. 100% Al sample shows little to no cracking at interface, but there is evidence of elongation on larger scale. It also appears that the crack is propagating along grain boundaries upon failure.
Figure 4.35. ECKA Al alloy high mag SEM of crack. This crack appears to originate between phases in the alloy and propagated between them as can be seen in the fourth picture.
Figure 4.36. Al + 20% SiC high mag SEM of crack at fracture surface. Charged area is bakelite material in which sample is mounted. The crack in the high mag image seems to originate in the matrix or possibly near a silicon carbide particle beneath the surface. The crack propagates between the matrix and silicon carbide particles.
Figure 4.37. Al + 25% SiC high mag SEM of crack. It is difficult to see the origin of the crack but it appears to be originating at the interface between the matrix and the silicon carbide. The crack similarly propagates between the particles and the matrix.
Figure 4.38. Al + 40% SiC crack high mag SEM. At this location the crack appears to originate more clearly at the interface between silicon carbide particles and the aluminum matrix. Again it travels along these interfaces to the fracture surface.
Chapter 5: CONCLUSIONS

FAST sintering of attrition milled aluminum and silicon carbide particles allowed for the production of composite materials with higher volume fractions of silicon carbide than has been achieved in the past. The key to the homogeneous dispersion of the silicon carbide particles was the attrition milling process, which created an embedded particle structure resistant to agglomeration. The sintered samples showed improved hardness, yield strength, and ultimate tensile strength properties over the baseline materials.

FAST was able to produce fully dense samples with a 25 minute hold time. This resulted in excellent homogeneity of the final sintered part, as seen in the SEM images. A uniform distribution of SiC particles throughout the system was observed from 20 to 40% silicon carbide additions. TEM revealed that excellent interfaces, with good diffusion and no porosity, were formed by the FAST process. FAST has proven to be an effective method to enhance diffusion at the interface of the materials, improve densification, and allow controlled grain growth due to its short processing cycle. This sets the stage for the observed mechanical improvements of the sintered samples.

Addition of silicon carbide to the aluminum matrix had effects at the 25% and 40% composite level. Hardness was improved by 58% (131 HV to 207 HV) over the ECKA aluminum alloy. These improvements are attributed to the grain refinement (due to FAST sintering as well as grain boundary pinning effects), dislocation pinning (due to the presence of composite particles), and load transfer (due to the high quality interface and large fraction of silicon carbide particles in the matrix) mechanisms. Similarly, elastic modulus has been shown to improve by 38% with a 40% silicon carbide addition (97 GPa to 134 GPa). It is also interesting
to note that the elastic modulus by sound velocity was an effective predictor of elastic modulus data produced by tensile testing. The ultimate tensile strength of the materials improved by 53% at 40% silicon carbide addition compared to baseline alloy (308 to 472 GPa). The yield strength also improved, showing an 88% improvement over the baseline alloy (217 to 409 MPa). This is again due to the load transfer mechanism thanks to the strong interface between silicon carbide and the aluminum matrix. However, silicon carbide additions resulted in decreased elongation during tensile testing.

To further realize the benefits of this composite system, it is useful to look at the specific strength of these materials compared to other metals used in structural, aerospace, or automotive applications. Figure 5.1 shows a comparison between aluminum silicon carbide specific strength performance with titanium and steel. There is a 56% improvement in specific strength over the baseline aluminum alloy and there is a 156% improvement over a conventional structural material such as stainless steel. When this is applied to a transportation structural application, strength requirements can be met with less material mass. These weight savings turn into financial savings in terms of either decreased fuel consumption or increased payload.
Figure 5.1. Specific strength comparison between Al-SiC composites and Titanium [46], [47], Steel [46], and Magnesium [46], [48].

It was shown that extrusion of this material is possible. These sintered discs of aluminum have been used as feedstock for extrusion of rods as a proof-of-concept for future structural applications as seen in Figure 5.2.
Further research can be done on the properties of these extruded parts to assess how the silicon carbide particles flow during extrusion and evaluate whether the improvement in mechanical properties is retained. Additionally, it could be worthwhile to explore even higher volume fractions of silicon carbide addition, to allow the system to approach behavior resembling what is predicted by the rule of mixtures.

Figure 5.2. Hot extruded Z-shape bar using Al-SiC composite powder.
APPENDIX: Stress Strain Plots

This is a collection of the stress-strain data used to calculate results from tensile testing.

Figure A.1. 100% Aluminum stress-strain curve for sample 1. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.2. 100% Aluminum stress-strain curve for sample 2. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.3. 100% Aluminum stress-strain curve for sample 3. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.4. 100% Aluminum stress-strain curve for sample 4. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.5. 100% Aluminum stress-strain curve for sample 5. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.6. ECKA Al Alloy stress strain curve for sample 1. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.7. ECKA Al Alloy stress strain curve for sample 2. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.8. ECKA Al Alloy stress strain curve for sample 3. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.9. ECKA Al Alloy stress strain curve for sample 4. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
**Figure A.10.** ECKA Al Alloy stress strain curve for sample 5. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
**Figure A.11.** Al + 20% SiC stress strain curve for sample 1. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.12. Al + 20% SiC stress strain curve for sample 2. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.

\[ y = 80422x - 0.7333 \]
Figure A.13. Al + 20% SiC stress strain curve for sample 3. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.14. Al + 20% SiC stress strain curve for sample 4. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.15. Al + 20% SiC stress strain curve for sample 5. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.

\[ y = 90611x - 0.6729 \]
Figure A.16. Al + 25% SiC stress strain curve for sample 1. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.17. Al + 25% SiC stress strain curve for sample 2. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.18. Al + 25% SiC stress strain curve for sample 3. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.19. Al + 25% SiC stress strain curve for sample 4. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.20. Al + 25% SiC stress strain curve for sample 5. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.21. Al + 40% SiC stress strain curve for sample 1. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
**Figure A.22.** Al + 40% SiC stress strain curve for sample 2. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
Figure A.23. Al + 40% SiC stress strain curve for sample 3. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
**Figure A.24.** Al + 40% SiC stress strain curve for sample 4. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.

\[ y = 125555x + 6.5493 \]
Figure A.25. Al + 40% SiC stress strain curve for sample 5. Gray line is 0.002 mm/mm offset and equation corresponds to the highlighted region.
REFERENCES


B. S. Al-shaer, *Evolution of Metal Casting Technologies A Historical Perspective* .


