Fabrication and Characterization of Hybrid Aluminum Matrix Composites for Structural use in Aerospace Applications

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Certificate

This is to certify that work in this thesis has been carried out by **Abdul Wasay Ghouri, Usman Suleman** and **Ghazi Shoaib Ahmed** and completed under my supervision at school of chemical and materials engineering, National University of Sciences and Technology, H-12, Islamabad, Pakistan.

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Dedication

We dedicate this thesis to our parents and our supervisor Dr. Malik Adeel Umer whose constant guidance and inspiration paved our path.

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Abstract

The development of high specific strength materials with superior mechanical properties have always been of keen interest for the aerospace industry. Modern technological developments have pushed existing engineering materials used in these industries to their limits and the demand for new materials continues to grow to at an ever-increasing rate.

Of the new materials being considered, sintered metal matrix composites are particularly promising because of their superior properties and our ability to tailor these properties for particular applications.

Traditionally, conventional Aluminum alloys have been the structural material to choice for the aerospace industry. This is attributed to their high specific strength, cost effectiveness and partly due to their ability to be easily recycled.

Hence because of the inherent properties suited to the applications, our research will focus on the development of sintered hybrid Aluminum metal matrix composites with optimum properties for structural use in the aerospace industry.

Introduction

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For this purpose we would be using pure Aluminum as the matrix material while pristine CNTs, functionalized CNTs and SiC particles are to be used as reinforcements. The sintering method used will be conventional pressing followed by pressure-less sintering.

Our strategy for fabricating superior Aluminum metal matrix composites will focus initially on optimizing the sintering conditions. It would be most desirable to have maximum densification with a refined grain size that would result in increased strength according to the Hall-Petch effect.

For fabrication, three distinctive routes are employed however, the general process is to blend the matrix material with the reinforcement, uniaxially cold press it in a die, degas and sinter it in an inert environment using a tube furnace.The routes which differ in the blending step : the first one being conventional ball milling for dispersing the reinforcing material with the matrix, the second method would make use of dispersion in Dimethylformamide (DMF) and the third method would make use of dispersion in Dimethylformamide (DMF) followed by high energy impact milling.

The effect of five different weight percentages of reinforcements using both of the mixing techniques will be explored. The selected weight percentages are 0% 0.1%, 0.2% 0.4% and 1%.

The selected composites to be fabricated are Aluminum/Pristine CNT composites, Aluminum/functionalized CNT composites, and Aluminum-/functionalized CNT/SiC composites.

The properties that need to be characterized for verification, comparison and to draw conclusions suited to the application of the composites are to include densification testing, SEM imaging for verification and evaluation , hardness testing, tensile testing using ultra sub size specimens.

In essence, our project aims to achieve the following objectives:

- Fabrication and characterization of Aluminum, CNT, CNT_f, SiC_p based composites.
- Optimization of processing and sintering conditions to refine grain size.
- Investigation of the effects of different fabrication routes.
- Investigation of the effects of functionalization of reinforcement.

Literature Review

Key requirements:
There are four key requirements that we need to keep in mind when choosing our material. We need our material to have all four properties as these will enable it to be utilized in the aerospace industry. They are all listed below.

High specific strength

Specific strength is any material's strength divided by that material's density. It can also be called the strength to weight ratio, and it signifies how much the strength of any material is. Our aerospace metal matrix composite requires a high specific strength as it needs to withstand the strains and pressures caused by being used at high altitudes at high speeds.

High specific stiffness

Specific stiffness is any material's elastic modulus divided by that material's density. It is also commonly referred to as the stiffness to weight ratio. Aluminium used in aerospace applications is very carefully selected, with a large emphasis on it needing to have a high specific stiffness. We require materials which are stiff and have a high percentage elongation on breakpoint, this is why Aluminium is chosen.

High damage tolerance

We need the material with which we construct our aerospace technology to have a high damage tolerance. The boy of the aircrafts will be subjected to environmental conditions, as well as friction and external factors, which is why it is imperative that its damage tolerance is high, so that it can withstand all of this and perform.

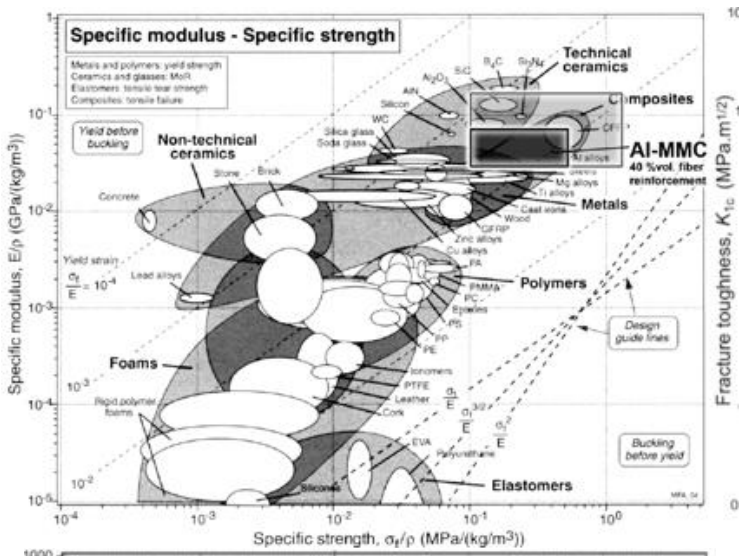
Low coefficient of thermal expansion

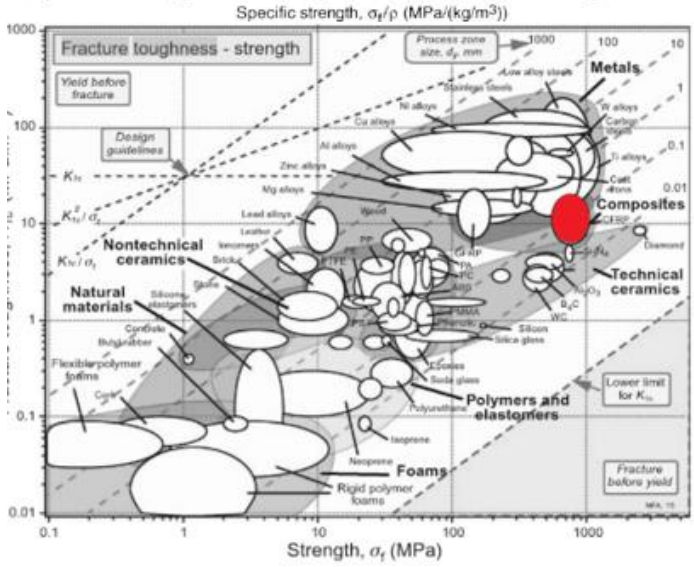
We need for the aerospace materials to have a low coefficient of thermal expansion. A low coefficient of thermal expansion (CTE) means that the material will not readily have a size change when it his heated.

Material selection

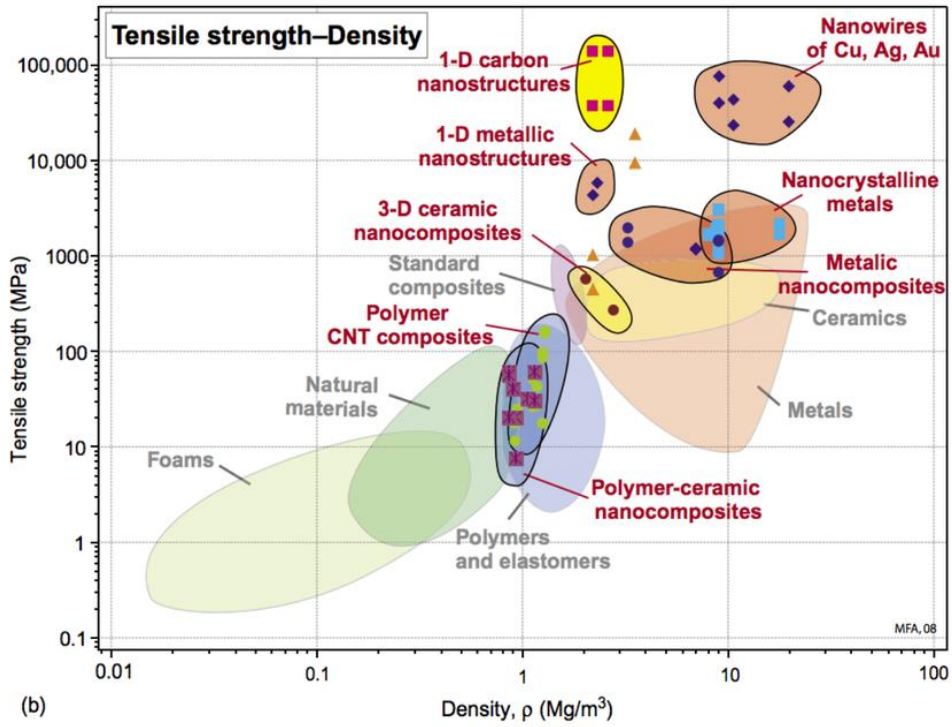
For our material selection phase, we referred to three Ashby diagrams to illustrate which material we should use in our research project. We used to Ashby diagrams, one between specific modulus and specific strength, and the second one between fracture toughness and strength.

In both diagrams below we observed that Aluminium metal matrix composites are found on the top right side of our Ashby plots. This was favourable as it showed that they possessed the properties that we required such as a high specific strength, a high specific modulus, and good fracture toughness.





CNTs are also shown in the Ashby diagram below as one of the strongest nanostructures in existence with moderately low density.



Powder Metallurgy

Basic Concept, Definition, and History

Powder metallurgy has a different concept than traditional metal working processes. Any material whose largest dimension is lesser than 1 mm is considered to be a powder. Normally these are metals, but can occasionally be combined with other phases such as polymers or ceramics. Particles exhibit properties in between solids and liquids, flowing under gravity to fill cavities for example, hence behaving like liquids. They can compress, but this is irreversible, such as plastic deformation in metals. Powder metallurgy is the study of metal powder processing, such as the characterization, fabrication, and conversion of metal powders into useful engineering components. Processing of powders involves applying basic work, heat, and deforming them into the desired shape and structure.

There are three main steps in powder metallurgy. The first part is known as powder technology which concerns itself with the nature of powders, with focus on the handling, classification, characterization and fabrication. Packaging, transportation, safety, and sampling are of secondary concern. Examining powder shape and size are a common and necessary practice in powder metallurgy. Powder consolidation is done traditionally by sintering and compaction. This is the second stage. Densification and formation of powders are the primary concerns at this stage. The final stage involves having a focus on the properties of the formed powder metal. Here we concern ourselves with the ductility, density, strength, conductivity etc. of the formed powders.

Decisions made when selecting the type of powder influence how easy or difficult it is to sinter and compact. In the same way, the consolidation sequence applied to any powder will affect the specific property goals and final compact properties of the powder.

The modern era of P/M Porous can be traced to when Coolidge developed a durable lamp filament from Tungsten for Edison. Porous bronze bearings, cemented carbide were developed by P/M in the 1930's. Refractory metals, ferrous alloys, and tungsten alloys were created in the 1940's through this method. Currently the focus is on using them for their homogeneity, productivity, and better properties. Thermal expansion Aluminium matrix composites is one such example.

Reasons for Powder Metallurgy

Powder metallurgy is useful for three reasons, its cost effectiveness, the unique products that can be formed, and captive applications. In terms of the economics of P/M i.e. its cost effectiveness, the precision of the products formed, with their accurate dimensions and low tolerances are very important. Pre-alloyed powders can be sintered below their melting temperature, eliminating casting defects.

Unique properties and microstructures can be obtained through P/M, such as cermets, oxide dispersion strengthened alloys, cemented carbides, and porous metals. Captive applications mean the materials which are very difficult to process through other methods than powder metallurgy. Examples are refractory metals, melting whom is impractical. Sometimes it's also desirable to process metals at

lower temperatures to avoid microstructural damage at higher temperatures, which is why processing metal powders is preferable. Processing in P/M techniques is done entirely in solid state, which is another benefit.

Aluminum powder metallurgy

One of the main driving forces for using conventional powder metallurgy in materials is to reduce fabrication costs through near net-shape processing. Aluminum powder metallurgy (P/M) is driven by different forces as alternative low cost fabrication techniques are available and cause P/M Being more expensive [1]. With aluminum other attributes are being used such as the properties achieved through adding dispersoids to form composites. The table below shows the comparative study of various powdered alloys in terms of UTS, YS and elasticity.

Table 9 Properties of Extruded P/M-RS Al Alloys at 315°C

Alloy	UTS (MPa)	YS (MPa)	El. (%)
Al-Fe5-Cu2-Ti2-Ce1-Zr1	357	281	3.2
Al-Fe4-Cu2-Ti1-W1-Ce1-Zr1	356	287	3.7
Al-8 Fe-7 Ce	270	225	7
Al-8 Fe-2 Mo	235	210	10
Al-12 Fe-1.2 V-2.2 Sn	310	300	7
Al-4.5 Cr-1.5 Zr-1.2 Mn	235	215	—
7075-T6 Wrought	70	55	60
2024-T81 Wrought	140	115	20

Due to its various uses other than powder metallurgy, aluminum powder is readily available. Conventional press and sinter processes can be used to make parts with all advantages and disadvantages of powder metallurgy. Using elemental powders leads to relatively lower costs of material and fabrication. However, properties are lower than for wrought or even for cast aluminum alloys. Advanced aluminum allows us to incorporate advantages of rapid solidification or mechanical alloying in increasing the alloying elements that can be used to form new aluminum alloys. These alloys extend usability of aluminum alloys, offering lighter weight alternatives to existing materials in high strength, wear resistance and high temperature applications. For example, high temperature aluminum alloys replaced titanium alloys in moderate temperature applications in aerospace. Although being

difficult to fabricate compared to conventional alloys, aluminum alloys can give better properties.

Particulate metal matrix composites based on aluminum are also a significant application for aluminum. P/M Aluminum produces near net shape composites with increased uniformity in the reinforcement distribution and finer microstructure size than for other fabrication techniques, leading to great improvements in properties.

Aluminum P/M traditionally is a small part of the whole P/M market and of the total aluminum market. However, recently aerospace and automotive markets have given aluminum P/M a large boost and Aluminum P/M is expected to see a large growth in the next decade. Research initiatives focused on improving aluminum P/M alloys, reducing cost and complexity of fabrication have sprung up.

Handling & Safety

The safety and handling procedures applied are an important consideration when working with aluminum powder metallurgy. Aluminum powders must be used cautiously due to their volatility, an example of their uses illustrates this as they are used as rocket fuel boosters in aluminum powered spaces shuttles.

The section below details the safety procedures for handling aluminum powder, it however is a primer and not very extensive. Aluminum powder suppliers are a great source for information for particular applications, and should be consulted before being involved in Aluminum powder metallurgy.
Although aluminum powders can be dangerous, aluminum powder manufacturers assume greater risk as more explosions have been reported there than at places where the powder has been used [2]. Anyone wishing to use aluminum powders should be cautious when using them, but shouldn’t be afraid of becoming involved with them.
Any powder which can react with oxygen can ignite, if they are fine enough and disperse into a dust cloud, are explosion can occur. The sensitivity of ignition is

determined by different factors such as the ease of any material to react with oxygen. Aluminum is highly reactive, hence its powder is considered very dangerous. The minimum ignition energy (MIE) indicates the sensitivity of any powder to ignite. Lower values of MIE indicate a higher likelihood to ignite. MIE values below 25 mJ indicate high degrees of sensitivity and can be ignited by electrostatic charge [3].

Elemental Aluminum is below this level, while pre-alloyed aluminum powder is above it, indicating a lower sensitivity to explosion of aluminum alloy powders. The minimum explosive concentration determines the quantity of powder needing to be in a dust cloud to instigate an explosion upon ignition. The maximum rate of pressure rise is used when designing explosion venting. Venting prevents the buildup of pressure to explosion levels. The minimum ignition temperature is the temperature at which metal dust that's lying in a heap can ignite. Properly handled metal powders however, should never be allowed to accumulate.

A minimum value of oxygen must be present to cause an explosion. This amount changes with the atmosphere. In nitrogen at least 9% Oxygen must be present [4], in helium it's 10%, while in Carbon Dioxide only 3% is needed. The powder needs to be suspended, i.e. be in the form of a dust cloud for there to be an explosion. Dust clouds can form easily when small, light-weight powders are used.
The finer the powder particle size, the more likely is the chance of dust clouds forming as it will stay suspended for longer. Finer particles also need lesser energy to ignite, and also explode more powerfully. Aluminum powders of 450 microns diameters pose no hazard, while powders greater than 75 microns are difficult to ignite [2,4]. Powders below 10 microns are very sensitive and great care must be taken in handling them.
Aluminum powder should not come in prolonged contact with water as this causes a reaction producing Hydrogen gas, thus it should be stored and handled with this in mind. This increases the hazard of using aluminum powder. Other practices which should be adopted are similar to any flammable material. Store in appropriate containers and keep away from oxidizers and combustible materials.

There are two areas specific to powder metallurgy operations that need to be considered when discussing the safe handling of aluminum powders [4]. Metal

powders are typically transferred during processing from one container to the other, which may cause dust clouds. Care should be taken to ensure the powder transfers are slow and deliberate. Non sparking elements, and containers attached to the ground and themselves, should be used.
During mixing of aluminum powder in the mixer, dust clouds can also be created. It is advisable that inert atmospheres be used for this [4]. A mixer that reduces that reduces the creation of frictional heat is recommended.

Metal powders, especially aluminum powders must be dealt with using good housekeeping practices. Specific methods for cleaning metal powders should be researched and adopted. Guidelines for handling aluminum powders are available from Aluminum Association [35].

Press & Sinter

Low cost P/M components are routinely produced with press and sinter processing. The low cost is offset with lower densities and properties. Near net shapes can be easily fabricated with the design limitations of the press and sinter process. These limitations include a simple shape in the direction of pressing, while other dimensions can have complex shapes. For general information about limitations in the design of press and sintered parts, refer to the Powder Metallurgy Design Manual, published by MPIF [2]. Tolerances that can be achieved in press and sintering of aluminum are quite good. As-sintered dimensional tolerance is 0.051 mm, while the as-sized tolerance is 0.013 mm [2].
As in most P/M materials the higher the compaction pressure and green density, the higher the final density. Aluminum alloys can be cold compacted to higher green densities than the commonly used ferrous powders. The compaction pressures used are also considerably lower than those used for ferrous P/M, as illustrated in fig.2 [5]. Compaction presses used for aluminum can be much smaller while still achieving excellent green densities. Table 4 shows a comparison of several commercial aluminum P/M alloys and water atomized iron powder. The higher achievable density is clearly shown. Greater pressing pressure leads to greater green density which means the compacted material have nearly zero

porosity. Shown in the figure below

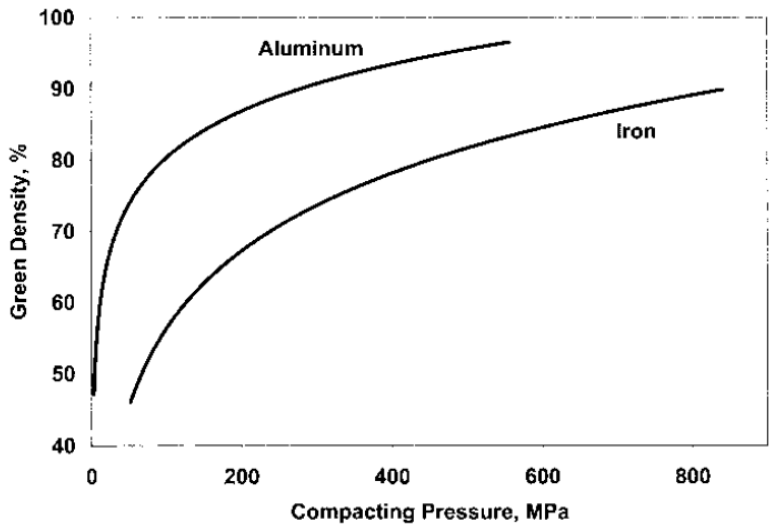


Figure 2 The effect of compacting pressure on the green density of aluminum and iron powders. Note the much lower pressures needed to compact aluminum compared to iron.

One study has shown that aluminum alloy powders can be cold compacted to full density at sufficiently high pressures [3]. Unalloyed atomized aluminum powders (<20mm) were consolidated to 100% density at pressure of 1 GPa. Atomized alloy powders containing Fe and Ni were consolidated to full density at a pressure of 3 GPa. Strength values were not reported. Aluminum alloys are sintered at least 90% of their melting temperature. Many times, a transient liquid phase is involved when sintering elemental blends. A sintering cycle contains three stages, lubrication burn off stage, high temperature sintering stage and a furnace cool-down stage. Good properties require the proper selection of dew point, atmosphere, and temperature.

Furnaces for aluminum sintering include both batch and continuous conveyor types. Batch furnaces have lower investment, moderate atmospheric requirements and greater control than continuous furnaces, which have an advantage of higher production rates. A humpback furnace can lead to lower atmosphere usage. A vacuum furnace, a special type of batch furnace can also be used to lower atmosphere usage and achieve higher destinations after sintering. If a lubricant is

used, then it must be removed prior to vacuum sintering. Parts are cooled before the vacuum is released and the furnace is opened.

The choice of atmosphere has a significant effect on final properties and dimensional accuracy [44]. Aluminum can be sintered in Nitrogen, dissociated Ammonia, inert gas or in vacuum. Hydrogen has been used but isn't recommended due to lower properties in the sintered part. Humidity should be during sintering, a dew point of 40 C or lower is recommended.
Nitrogen is a particularly good atmosphere for aluminum due to being cheaper and readily available with excellent sintered properties.

Nitrogen atmospheres aid in achieving highest sintered strengths of several commercial aluminum alloys. For example, 601AB, starting with a 95% green density, has a 12% increase in yield in Nitrogen atmosphere compared with dissociated Ammonia as well as ductility increase. Also, the sintering of a 2014(MD-24) PM composite reinforced with various hard phases showed at least a 50% decrease in ultimate tensile strength in Nitrogen as compared to sintering in argon and vacuum [5]. However, Nitrogen used had a dew point of greater than -20 C, which may account for some of the variance.

Dissociated ammonia is an available atmosphere and can be used with aluminum. Properties of parts sintered in this are usually lower than those in Nitrogen. The lower properties are associated with Hydrogen in the dissociated ammonia. Heat treatment in ammonia of 2024 sheet causes a 29% reduction in strength and an 82% reduction in elongation [6].

P/M factors such as green density, sintering temperature, choice of sintering atmosphere and dew point contribute to effecting dimensional changes. The change in dimensional changes can be positive or negative depending on a combination of the green density and the atmosphere.

Using vacuum and varying green density, both 201AB and 601AB can either shrink, remain unchanged or swell. The right choice of atmosphere and green

density leads to good dimensional control. If maximum properties are needed, sintering in nitrogen gives little or no shrinkage in both alloys. The decrease in density that can occur during sintering has been linked with entrapped gases from the atomization process [7]. Swelling can increase with higher compaction pressures and also higher sintering temperatures.

Al MMCs

Powder metallurgy is an excellent fabrication method for aluminum matrix composites.
It offers a method of adding large percentages of hard reinforcing phases
in a uniform distribution, and producing a near net shape. This results in the potential
for significant property improvements. One major drawback to the use of
P/M to produce aluminum MMCs is a potentially higher cost over other methods [8].
Specific advantages of P/M in the fabrication of aluminum MMCs include a
much higher volume percent of reinforcing phase possible when compared to casting
or spray forming techniques [8]. The ability to produce a more uniform dispersion
of the hard particles leads to both higher fracture toughness values and higher
ductilities. In a 15% SiC reinforced 6061 composite, a 50% improvement in KIC
occurred through producing a more uniform dispersion. A 20% SiC reinforced
CP Al alloy also had a 50% improvement in the percent elongation with a more
uniform distribution.
One of the first commercial aluminum matrix composites is called sintered
aluminum powders (SAP) [88]. SAP uses aluminum powder which contains a large
quantity of oxygen. The oxygen forms Al2O3, which acts as a disperoid, providing
significant elevated temperature strength. Parts from SAP are thermally stable at
temperatures up to 500 C, and exhibit good corrosion resistance, thermal and electrical
conductivity, and erosion resistance [7]. Semi finished parts in the form of sheet,
tube, forgings, etc., are available commercially [9].
Aluminum can also be dispersion strengthened by reaction milling with carbon

black. An Al4C disperoid is formed which gives excellent high temperature stability.
This can be heated to 260 C for 100 h without significant loss of strength [9].
Nitrogen can also be incorporated into the structure of aluminum by reaction milling. Cryomilling involves using liquid nitrogen in the milling chamber while mechanically alloying an aluminum powder. AlN particles are formed, which improve the properties of the resulting compacts. The refined microstructure of the MA powders can be retained after sintering through cryomilling. The AlN particles formed appear to stabilize the grain structure during the elevated temperature exposure during sintering. A 5083 alloy treated by cryomilling was sintered to 99.6% density, while retaining most of its refined grain size during either extrusion or hot isostatic pressing [11]. A 30% increase in both the yield strength and ultimate tensile strength were observed when compared to wrought 5083-H343. Surprisingly, there was no decrease in ductility accompanying this increased strength. The grain size of the resulting compact was approximately 30 nm.
A P/M 6061/20% SiC particulate composite showed a 47% increase in stiffness and a 35% increase in tensile strength over wrought AA 6061 [9]. Ductility and fracture toughness are often lower in the composite materials, when compared to the monolithic wrought alloy. One study of layered particulate composites showed that fracture toughness values equivalent to wrought can be achieved [10]. SiC and aluminum 6061 were codeposited by spray atomization into layers of higher and lower SiC volume percent. A layer spacing of 700 mm produced the highest ductility and ultimate tensile strength. The fracture toughness was similar to the wrought alloys toughness. The codeposited layers were effectively graded, unlike similar composites produced by P/M, diffusion bonding and coextrusion. Also, the strength and modulus increase with the amount of reinforcing phase can also be seen. Ductility decreases with the increasing strength.
The effect of volume fraction on the stiffness and thermal coefficient of

expansion (CTE) can be observed for a 6061 alloy reinforced with SiC.
The CTE decrease with increasing volume fraction of SiC. This effect is being used
to develop aluminum alloys with controlled CTE for use in the electronics industry.
Substrates with CTE that closely match silicon reduce the problems of thermal
fatigue in integrated circuit applications.
The route used to fabricate a P/M aluminum MMC is critical to the cost of the
final product [9]. Typical processing sequences would include atomization of the
aluminum alloy, followed by blending of the reinforcement particles with the matrix
powder, then cold pressing or cold isostatic pressing, followed by canning, degassing,
and then a forming technique which produces a high degree of deformation such as
extrusion, forging, or hot pressing. The blending of the aluminum alloy powder
and the reinforcing phase is critical to producing a uniform distribution in the final
part [9]. Alternate routes that have less steps and less cost have been proposed.
Each of these routes typically trades off properties for reduced expense.
Compressibility of aluminum MMCs decreases with increasing particle volume
[11]. As particle volume increases, the green density will typically decrease. A limit
may be reached where additional particle volume may prevent sufficient
densification to get an additional increase in properties.

CNT MMCs

Recently the existence of CNTs, or carbon nanotubes has created an interesting new area of study. As CNTs exhibit incredible physical properties they can bridge the drawback brought on by Ceramic reinforcements with regards to their transfer properties. CNTs however can only give their predicted physical properties if they are in an ideal structural shape, i.e. the CNTs being isolated, the CNTs having no endohedral or exohedral contaminants, and the CNTs having no structural defects on their lattices.

CNTs require us to use various synthesizing methods, their synthesis often leads to contaminants such as catalytic particles etc. being produced. CNTs are most commonly synthesized by performing Chemical Vapour Deposition (CVD). CVD gives a good morphology of the CNTs created and also has the most potential to be scaled for meeting industrial scale synthesis requirements. [24, 25].
MWCNTs are where the current research in CNT synthesis is geared towards. [26–31]. Composite manufacturing uses self-produced CNTs as well. Catalytic Chemical Vapour Deposition (CCVD) is what is utilized for synthesizing the CNTs that are then used for reinforcement in Composites. [26, 27, 30, 52–59].
MWCNTs normally have a diameter ranging from 10 to 80 nm, a length from 0.5 µm to 50 µm, and a purity normally around 95% [26–59].

Blending Methods

Ball Milling

It is done by using attrition or planetary ball mills. Metal powders and reinforcement phase are added together into mixing jars along with some hard balls and are rotated with a particular speed of rotation (Figure 1 a).
The balls fall on top of the powder material while the rotational movement is undergoing, which causes the integration of the CNTs into the matrix powder. It also causes particle welding, as well as a reduction in the size of the particles.
Varying rotational speeds, ball sizes and materials, gas atmospheres, powder to ball ratios and mixing times can be used as mixing parameters. Process control agents are added to the ball milling process so that cold welding can be avoided. However on some occasions such as mechanical alloying, it is preferable to use cold welding. Ball milling gives a homogenous distribution of the reinforcement phase inside of the metal matrix composites (Figure 1 d).
The main issue or shortcoming of low and high energy ball milling is the increase in the defect density of the CNTs when the mixing process occurs. This is as very large contact pressures are applied, pressures as high as 30 GPa [97]. Low energy ball milling cannot entirely end this problem, but can help manage and mitigate it.

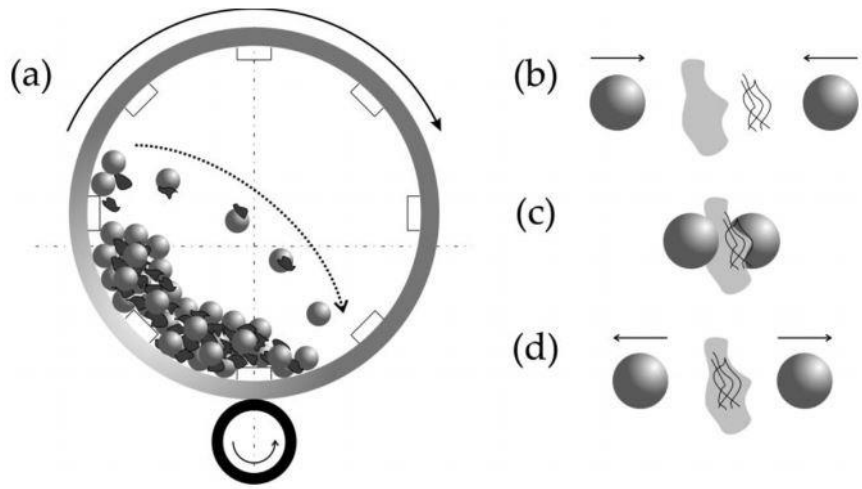


Figure 1. Schematic draft of the ball milling process (a). The used balls are hitting the CNTs and the matrix powder material (b), welding and integrating the two components (c) and bounce of the powder particle to restart this process again at another spot (d).

Molecular level Mixing

CNTs have to be functionalized if we are to have this method be successful, for example by using an acid bath (Figure 2 a). Metal salts are added and reduced by the use of reducing agents so that CNT metal oxide suspension can be established. The CNTs behave as nucleation points from which metal oxide formation can occur. This is illustrated in Figure 2 b.

After we wash off whatever chemcials remain, we calcinate the powder and then reduce it, an example is the reduction under a hydrogen atmosphere so that we can have the CNT/metal powder (Figure 2 c and d). As the CNTs are coated by the MMC, we have very homogeneous distribution within the metal matrix, which is a positive (Figure 2 d). homogeneity can be reduced if the reinforcement phase has a limitation of only being able to be placed near grain boundaries, this is seen in many other mixing methods, which is why molecular level mixing is employed.

The major problem with using molecular level mixing however is the functionalization that is needed, which involves the breaking up of covalent carbon bonds so that functional groups may be attached to the CNT surfaces, which would greatly reduce their unique properties [27, 29, 31, 32, 14, 44, 45, 70–74].

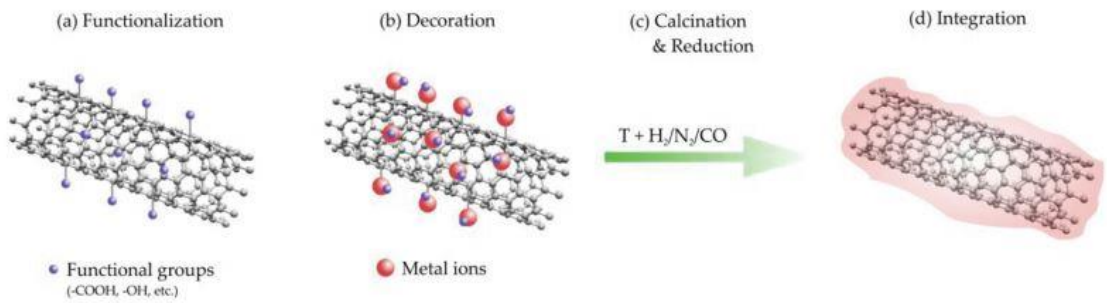


Figure 2. Schematic draft of the molecular-level mixing method. CNTs are functionalized by functional groups, which will be covalently bond onto the CNTs surface (a). After this, metal ions can electrostatically interact with the functional groups, thus coating the CNT surface with metal ions (b). These ions are transformed to a pure metal layer by calcination and reduction under temperature and H_2, N_2 or CO atmosphere (c). Finally, the CNTs are fully decorated or even integrated in the metal matrix material (d).

Collodial Mixing

In this we disperse the CNTs with the help of magnetic stirrers in solvents, homogenizers, or ultrasonic baths. Many solvents exist which can help in stable and fine dispersal of the CNTs, for example ethylene glycol or DMF. The surface of the used CNTs, as well as the solvents that are used both determine the stability and the dispersion grade of the CNT suspensions. Some research works avoid functionalizing the CNTs and use them as pure/pristine so that their original properties can be used. This however decreases the dispersion quality. Other research works involve the functionalizing of CNTs so that electrochemically stable dispersions can occur, however this is at the cost of the physical properties of CNTs.

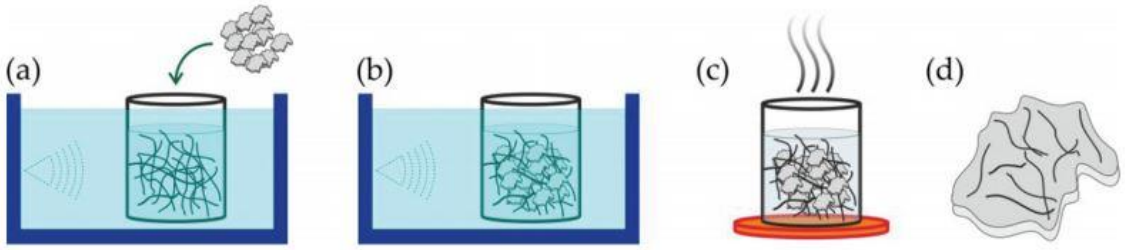


Figure 3. Schematic draft of the colloidal mixing method. First, the CNTs are dispersed in a liquid solvent using ultrasonic agitation, after which the matrix material powder is added (a). The two components are mixed again by ultrasonic agitation (c), and the solvent is evaporated (c) to finally obtain the mixed CNT/metal matrix powder (d).

One point is debated with regards to the effect of ultrasonic agitation when it

comes to the defect density of the CNTs. Studies both claim a decrease as well as an increase in the defect density with regards to the time spent in an ultrasonic bath. The decrease observed in defect density can be caused by misinterpreting the Raman spectra of disentangled CNTs

Once the CNTs are dispersed the metal powder is mixed and dispersed by using the CNTs through ultrasonic agitation, or by using homogenizers, or through stirring (Figure 3 b). in the final step the solvent is evaporated so that we can obtain a dry powder that is mixed (Figure 3 c and d). The capability of up scaling and having the same results regardless through this method is a large advantage of the process [26, 36, 42, 46–51, 61–69].

Cold Pressed Sintering (CPS)

This is the easiest and most uncomplicated process through which we can densify our blended powder. We can use isotactic or uniaxial presses to pre compact our powder into the shape that we require (Figure 4).

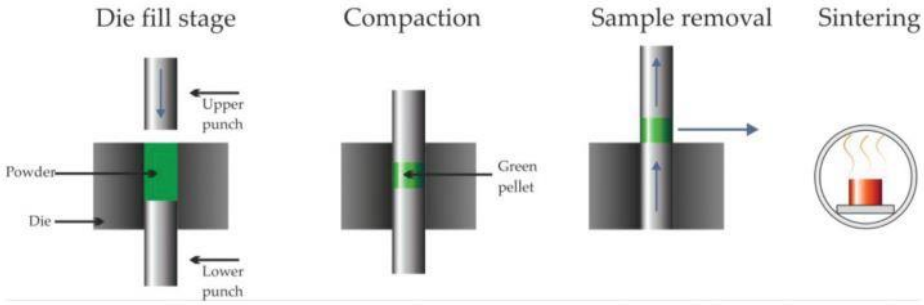


Figure 4. Schematic draft of pressureless sintering or cold pressed sintering. A die is filled with the powder material, which is then pressed uniaxial to a green pellet. The sample is then removed and sintered without pressure in a furnace under vacuum or inert gas atmosphere.

Once this is completed, the sample is then sintered, but without applying any more pressure. This is done under either a vacuum or inert gas atmosphere as this will consolidate the sample even more (Figure 4). One heating-cooling cycle may take a fair amount of time but it gives us an advantage of being able to sinter many samples which have dissimilar shapes simultaneously, this makes sintering time efficient. Densification however is done based upon the grain boundary and lattice diffusion [81], and we cannot close larger porosities without using pressure, hence we have a poorer final density of our composite than what is seen with other processes [28,34,37,40,47,48,54,55,67,73,83,94,98].

The Al/CNT System

A detailed overview of the research papers in the Al/CNT system can be found in the table below:

Reference	Blending method	Sintering method	CNT content Value wt%	Relative density Value %
Kuzumaki et al. (1998) [60]	**Stirring**: Stirring in ethanol at 300 rpm for 0.5h, drying in vacuum furnace.	**HUP and hot extrusion**: Packed in an Al case and preheated for 1.5 h at 873 K in vacuum (0.53 Pa) and then compressed with 100 MPa in steel dies for 60 min. The heating and loading rates were 29.1 K/min and 10 MPa/ min, respectively. Then, the composites are extruded at 773 K (extrusion ratio = 25 : 1) at a speed of 10 mm/min	5 vol% 10 vol%	94% 96.2%
Xu et al. (1999) [59]	**Hand grinding**: For > 30 min.	**HUP**: At 793 K under a pressure of 25 MPa for more than 30 min.	1 wt% 4 wt% 10 wt%	XXX
Tokunaga et al. (2008) [61]	**Colloidal mixing process**: Mixing by sonication for 5min and then evaporation of the solvent	**HPT**: At room temperature with an applied pressure of 2.5GPa. The rotation is initiated 5s after the load application and terminated after 30 turns.	5 wt%	98.4%
Kwon et al. (2009) [38]	**NSD**: Consisting of commercial gas atomized Al powder, CNTs and natural rubber. The precursor is heat treated at 500 °C for 2 h in an argon atmosphere (1 l/min) to evaporate the natural rubber.	**SPS and hot extrusion**: Sintering at 600 °C, holding time of 20 min, heating rate of 40 °C/min and pressure of 50 MPa. The sintered compact was extruded in a 60° conical die at 400 °C with a pressure of 500 kN. The extrusion velocity and the extrusion ratio were fixed at 2mm/min and 20, respectively.	5 vol%	Sintered: 96.1% Hot extruded: 98%
Esawi et al. (2009) [78]	**Ball milling**: At 200 rpm for 3h and 6h using 75 stainless steel milling balls (10mm diameter); giving a ball-to-powder weight ratio of 10:1. The jars are filled with argon and 2ml of methanol is added.	**Hot extrusion**: Compacted at 475MPa. Hot extrusion of the compact is conducted at 500°C using an extrusion ratio of 4:1.	2 wt%	XXX
Kwon et	**NSD:** Consisting of	**SPS and hot extrusion**: Sintered	1 vol%	Up to

al. (2010) [99]	commercial gas atomized Al powder, MWCNTs and natural rubber. The precursor was heat treated at 500 °C for 2 h in an argon atmosphere (1 l/min) to evaporate the natural rubber.	at 480, 500, 560, and 600°C with a heating rate and holding time of 40 °C/min and 20 min, respectively. A pressure of 50 MPa is used. SPSed compacts were extruded in a 60° conical die at 400 °C with a 500 kN press. The extrusion velocity and extrusion ratio were fixed at 2mm/min and 20, respectively.		96.8%
Choi et al. (2011) [79]	**Ball milling**: A2024 chips are ball-milled at 500 rpm under argon atmosphere for up to 48 h. The ball-to-chip weight ratio was 15:1. A control agent of 1.0 wt% stearic acid is added. The composite powder is then produced by ball-milling 18-h A2024 powder and CNTs.	**Hot rolling**: Heated to a temperature of 450 °C and then hot rolled with every 12% reduction per a pass; the initial thickness was 20mm, and the final thickness was 1 mm.	1 vol% 2 vol% 3 vol%	XXX
Choi et al. (2011) [80]	**Ball milling**: Aluminium powder was solely ball-milled for 18h or 12h and then mixed with CNTs by ball-milling for 6h. Or Aluminium powder is directly mixed with CNTs by ball milling for 6h	**Hot rolling**: Heated to a temperature of 450°C, 480°C or 530°C and then hot rolled with every 12% reduction per a pass; the initial thickness was 20mm and the final thickness was 1 mm.	1.5 vol% 3 vol% 4.5 vol% 6 vol% 7.5 vol% 9 vol%	96.3%-99.9% depending on parame ters.
Kwon et al. (2011) [39]	**Ball milling:** For 3h under an argon atmosphere; 360 rpm, Ø 10mm ball, 10:1 ball to powder weight ratio, and 20 wt% heptane was used as the process control agent.	**HUP:** Consolidating at 500 °C for 5min under a uniaxial pressure of 57 MPa.	5 vol% 10 vol% 15 vol%	100%
Jiang et al. (2012) [40]	**Ball milling**: Flake powder is obtained by ball milling of nearspherical powder with an initial ball-to-powder weight ratio of 20:1 and 423 rpm for several hours in flowing Ar atmosphere with water cooling. The as-prepared Al nanoflakes are first surface modified by PVA with 1700–1800 repeat units and then mixed with a CNT suspension, which	**CPS and Hot extrusion**: Compacted under 500 MPa pressure, and then sintered in flowing Ar atmosphere at 550 °C for 2h. Then heated to 440 °C with a heating rate of 10 °C/min within a vacuum furnace installed with the extruder. And extruded with an extrusion ratio of 20:1 at a ram speed of 0.5 mm/min.	0.5 vol% 2 vol%	After CPS: 80-85% After hot extrusion: > 99.5%

	is then heated in flowing Ar atmosphere at 500 °C for 2 h to remove the PVA. 1kg blend in 8 hours.			
Nam et al. (2012) [70]	**Molecular-level mixing and ball milling**: Poly-vinyl alcohol aqueous solution and CNTs are mixed by tumbler ball milling for 48 h, followed by drying in vacuum at 100 °C. The PVA-coated CNTs, Cu(CH3COO)•2 H2O and 2 M NaOH aqueous solution are dispersed in water and heated to 80 °C to form a CNT/CuO composite. This is followed by vacuum filtering and reduction at 300 °C under a hydrogen atmosphere. CNT/Cu composite powders and Al powders are then mixed using a planetary mill for 3 h with rotation speed of 200 rpm; the ball to powder ratio was 10:1. The composition of the Al–Cu matrix is Al-4wt%.	**SPS and cold rolling**: Sintered at 500 °C for 5 min in a vacuum of 10-3 torr under a pressure of 50 MPa . The CNT/Al–Cu composites are solution heat treated at 550 °C for 12 h and quenched in a water bath at room temperature. They were then cold rolled to achieve 5% plastic deformation. The composites are finally aged at 130 °C for 0 to 24 h.	2 vol% 4 vol%	XXX
Kwon et al. (2013) [41]	**NSD**: The precursor was heattreated at 500°C for 2 h in an argon atmosphere (1 l/min) in order to evaporate the natural rubber.	**SPS**: Sintering at 600 °C, holding time 20 min, heating rate 40 °C/min, and pressure 50 MPa. Then heattreated in an alumina pan at 670 °C and at 800 °C for 1 h in an argon atmosphere (1 l/min) inside a tube furnace.	5 vol%	96%
Liu et al. (2013) [76]	**Friction stir processing (FSP):** Plates of aluminium alloy are used as the substrate materials. Holes with a depth of 3.5 mm and a diameter of 0 mm, 2 mm, 4 mm, 6 mm, 8 mm and 10 mm respectively were drilled in the aluminium plates, which are then filled with various quantities of CNTs.	**Friction stir processing:** A tilt angle of 2° is applied on the fixed pin tool during FSP. The pitch distance was 0.3 mm. The forward velocity of the rotating pin is kept as a constant of about 30 mm/min, and a rotational speed of 950 rpm is used. Five passes on the same position were conducted on each plate.	1.6 vol%	XXX
Asgharz	**Ball milling**	**HPT**: Pre-compacted under	3 vol%	98.5% -

adeh et al. (2014) [81]		pressure of ~1 GPa. Then, the green compacts are compressed and deformed under the applied pressure of 6 GPa at room temperature. The rotation of the lower anvil was started after 30 seconds of load application with a rotational speed of 1 rpm and was terminated after various numbers of revolutions up to 15.		99.5%
Isaza Merino et al. (2016) [77]	**Layer stacking**: CNTs are added to a solution composed of 4 wt.%, PVA dissolved in distilled water. This mixture is magnetically stirred at 1000 rpm during 1 h, followed by a sonication at energies between 40 and 60 kJ and a power of 100W. Finally, this mixture is cured at room temperature in a Petri dish for 8 days. The composite sheets are cut into small sections and stretched at a rate of 2mm/min in a tensile machine at 80 °C. Then, two composite sheets of polymer/CNTs are alternately stacked with three aluminium sheets.	**HIP**: In argon atmosphere. The pressure and temperature are raised gradually during 1.5 h to 40MPa and 650 °C for 30 min. This allows for the evaporation of PVA and aluminium diffusion between the sheets to finally produce the consolidation of the composite	0.5 wt% 2 wt%	XXX
Carvalho et al. (2016) [82]	**Ball milling**: Al-Si 88-12 wt% and CNTs are mixed using 12 steel milling balls with 10-mm diameter, presenting a ball-to-powder weight ratio of 10:1. The jar was placed in a rotation device, and the mixing was made with a constant rotation speed of 40 rpm, during 6 days (low-energy ball milling).	**HUP**: Uniaxial load pressure of 35MPa and a temperature of 550°C during 10min.	2 wt% 4 wt% 6 wt%	XXX
Chen et al. (2016) [43]	**Ball milling**: Al powder is milled with 2 wt.% stearic acid. The powder is sealed in a ZrO2 jar together with 600 g ZrO2 media balls (10mm in diameter) in an argon gas atmosphere. The	**SPS and hot extrusion**: Sintering at 800 K, 850 K and 900 K. The heating rate is 20 K/min and the holding time at target temperatures is 60min. A pressure of 30MPa is applied on the sample under a vacuum of ~5	1 wt%	>99%

	rotation speed is 200 for 240 min. Then, Al-flake powder	Pa. Before		

Distribution and interaction with the matrix material

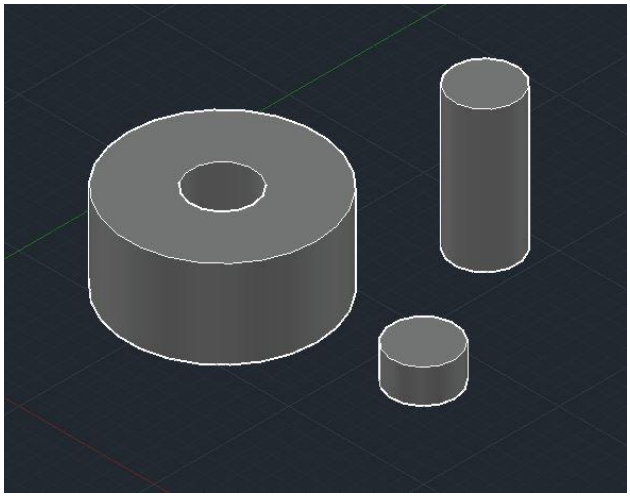
We can see a certain amount of agglomeration of CNTs in all of our analysed samples. The amount of agglomeration corresponds to the dispersion method applied. Ball milling contributes to a large degree of clustering, while functionalized and non-functionalised CNTs, both would produce this result. However a homogeneous dispersion coupled with a very fine dispersion of the clusters can be seen regardless of the agglomeration. Cluster formation does not greatly affect the composite densification. We need to establish an ideal compromise between the lowest possible CNT structure damage and optimal initial disentanglement of agglomerates.

We can observe the amount of agglomerates in our composite through two methods, we can evaluate the fracture surfaces of our composites, or we can evaluate the polished surfaces through electron microscopy. SEM imaging however enables us to have improved views of the size of the agglomerates, as well as their distribution. We can observe this by segmenting the C-containing phases.
If we observe the CNT degradation after densification and during blending, we see dissimilar results through Raman spectroscopy, even if the same manufacturing and fabrication methods are used. CNT degradation is a big cause for concern whenever metals that are strong carbide builders are being researched and used [115]. Aluminum it is commonly known forms a stable carbide (Al_4C_3) in a widespread range of compositions [116].

Experimental Procedures

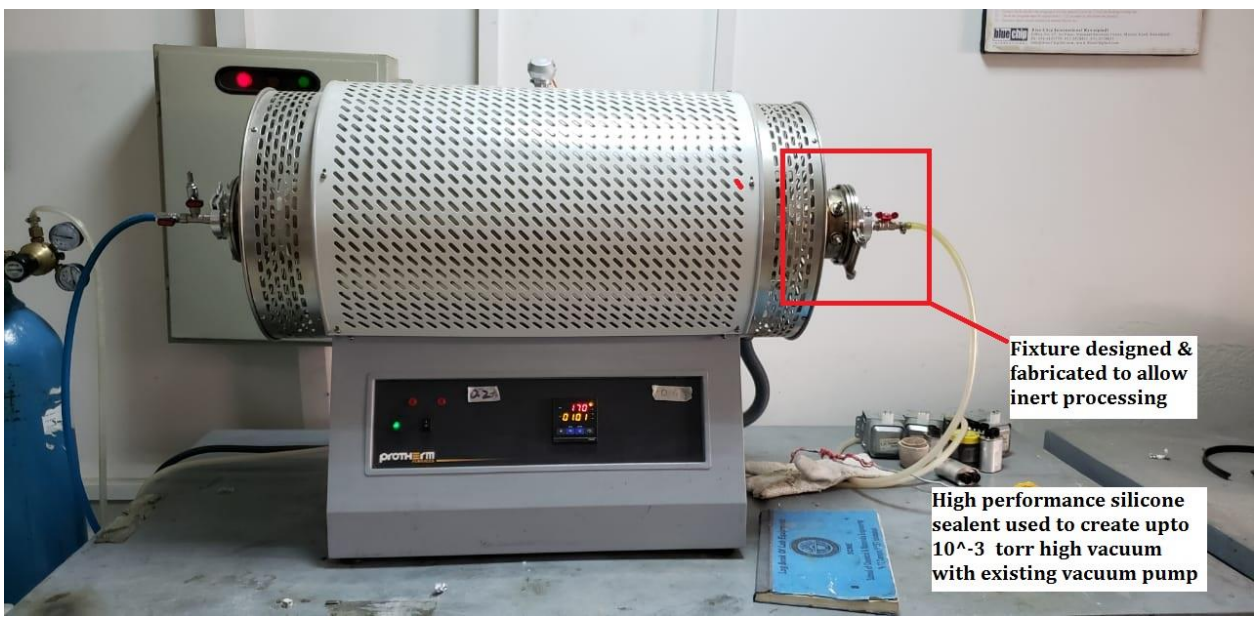
Design and fabrication of the die to be used for cold pressing.

A die was designed using AutoCAD to produce cylindrical green samples of 25.4 mm diameter. This provides a sample size adequate for testing and the fabrication of a USS sample for tensile testing.
Once the die was finalized, the die was manufactured on a lathe and subsequently heat treated using D-2 tool steel. This grade of alloy was chosen as it provides an effective combination of toughness, hardness, tool performance, corrosion resistance and price.



Design and fabrication of air tight fixtures for the tube furnace

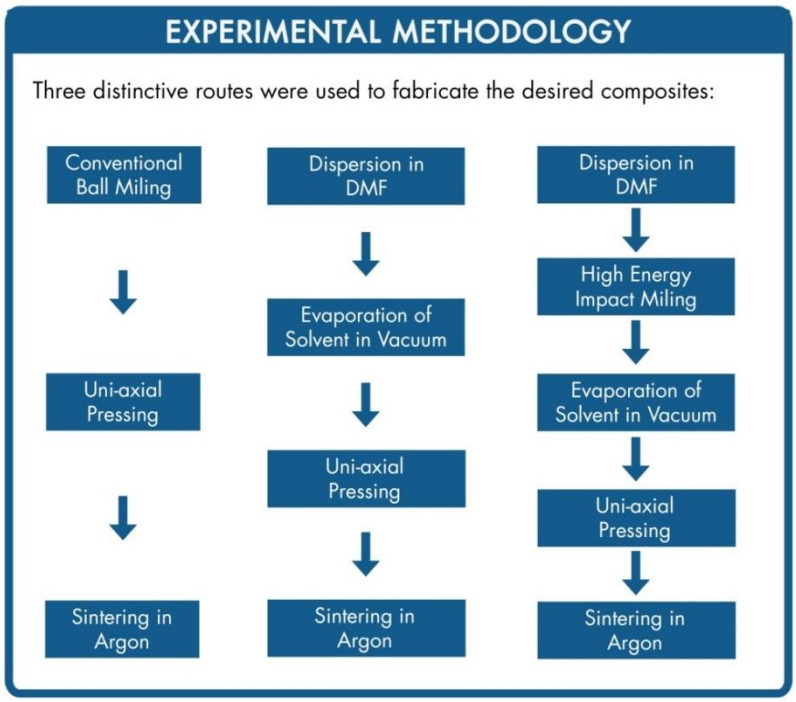
Missing fixtures were designed with valves that would allow the control of gases in and out of the tube furnace that were used to carry out the sintering operations throughout the study. Parts were manufactured using 316 SS on a lathe and then assembled onto the furnace. A high performance silicone sealant was also used to create a vacuum up to 10^{-3} Torr with the existing vacuum pump. This allowed for an inert processing environment, free of oxygen, crucial to prevent unwanted oxidation or other undesirable reactions keeping in mind the reactivity of aluminum powders.



Process Overview

Although three distinctive routes were used to fabricate composites of the desired composition, the general process employed was to blend the matrix material with the reinforcement, uniaxially cold press it in a die, degas and sinter it in an inert environment using a tube furnace. The routes differed in the blending step; this is

illustrated below:



Each route will be explained in detail in section 3.6

Experimental setup:

Throughout the fabrication process, exposure of Aluminum powders to air was avoided where possible due to reasons discussed earlier. Therefore, the use of a vacuum glove box in conjunction with Nitrogen gas was employed whenever possible. This allowed processing in an inert environment and prevented oxidation and other undesirable reactions.

Cold pressing was carried out with a die and hydraulic press.

To sinter the green composites, the tube furnace was connected to a vacuum pump and an argon gas cylinder using pipes and the fixtures made earlier. An Erlenmeyer flask filled with viscous synthetic oil was also used to bubble the outlet argon gas. This was to prevent any backflow of air.

The sintering conditions were first optimized as a function of sintering time and temperature. For this purpose five different conditions were tested out and the optimal conditions to be used to fabricate the composites were thus chosen.

Weighing of precursors:

Both Aluminum powder and the reinforcements were weighed on a high precision analytical balance to produce the desired the weight fractions in a vacuum glove box. Nitrogen gas connected to the vacuum glove box was used to provide an invert processing environment.

Blending

Blending was carried out using three distinctive routes explained below.

Conventional ball milling

To carry out conventional ball milling, CNTs and Aluminum powder was weighed and placed in polypropylene bottles along with alumina balls purged with nitrogen gas. This was important to prevent any undesirable reactions that Aluminum powder may undergo. It would be worth mentioning here that Alumina balls were used instead of steel to minimize contamination. The balls were used in 10:1 ratio with the precursors. Using this ratio is standard practice.

The polypropylene bottles were then placed in a ball mill for 10 hours at 200 RPM to ensure through mixing. This method was used solely to fabricate Pristine CNT/Al composites only.

Dispersion in DMF

CNT_f were dispersed uniformly in DMF solvent using ultrasonic agitation. DMF is chosen as it is one of the best solvents that form stable suspensions with CNTs. Once fully dispersed, Aluminum powder was mixed in and the resulting suspension was transferred to a rotary evaporator. The solvent was thus extracted under vacuum while continuously being stirred. When all of the solvent had been extracted the resulting mixture was allowed to cool under vacuum to prevent oxidation. The powdered mixture is then cold pressed, degassed and sintered in an inert argon atmosphere. This method was used solely to fabricate CNT_f/Al composites only.

Dispersion in DMF followed by High energy impact milling

For the technique involving high energy impact milling, the precursors were weighed and slurry was formed using DMF. The mixtures were then placed in steel containers designed specifically for this purpose. The purpose of forming a slurry is to avoid cold welding of Aluminum to the walls of the container caused by high energy impact milling. Molybdenum tool steel balls were used instead of Alumina for this operation as Alumina balls lack the toughness required. The container was purged with Nitrogen gas and the precursors were milled for 4 hours at 400 RPM to ensure thorough mixing. This method was used to fabricate CNT_f/Al, pristine CNT/Al and CNT/SiC_p/Al composites.

Pressing

The mixtures produced in the preceding steps were then placed in the die and pressed using a hydraulic press until compaction was achieved. A holding time of 5 minutes using 100 MPa pressure was used to ensure even compaction 100 MPa. The green sample was then removed from the die and placed in an inert environment to prevent oxidation.

Degassing & Sintering

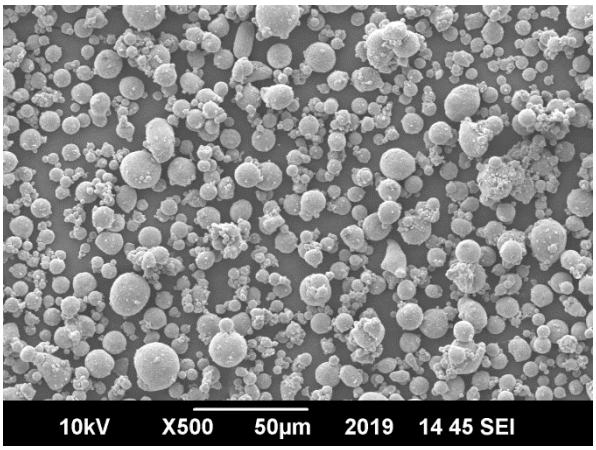
Degassing and sintering were integrated into the furnace heating program. First the green sample was placed in the tube furnace with the vacuum pump on and heated to 100 C in vacuum. This step is important for the desorption of any moisture from the green sample which could otherwise form undesirable side product. After degassing, the green sample was allowed to continue to heat up to 600 C in inert Argon atmosphere. The green sample was allowed to soak in this temperature for a total of 135 minutes and then allowed to furnace cool with the gas trapped inside the tube. The sample is then removed from the furnace

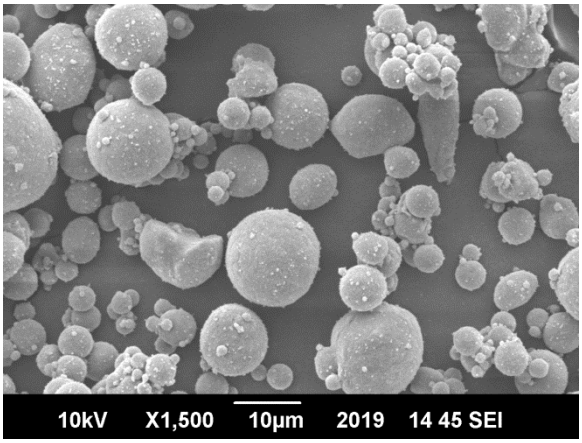
.

Results & Discussion

SEM micrographs of the Aluminum powder

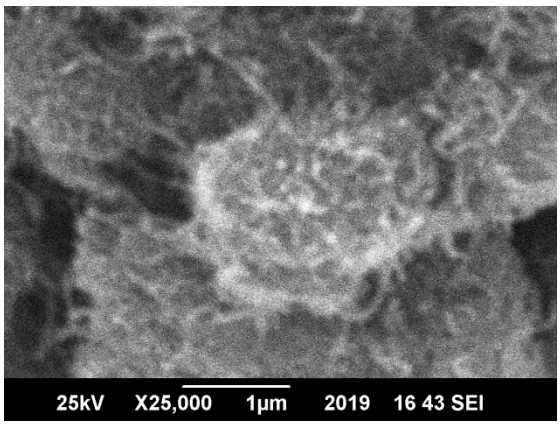
SEM micrographs of the Aluminum powder used throughout are shown below. This is done for verification of the powder size which is indeed <160µm in size and roughly spherical in morphology.

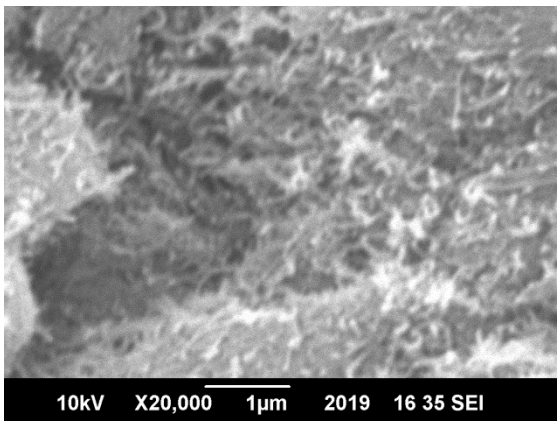




SEM micrographs of the CNTs

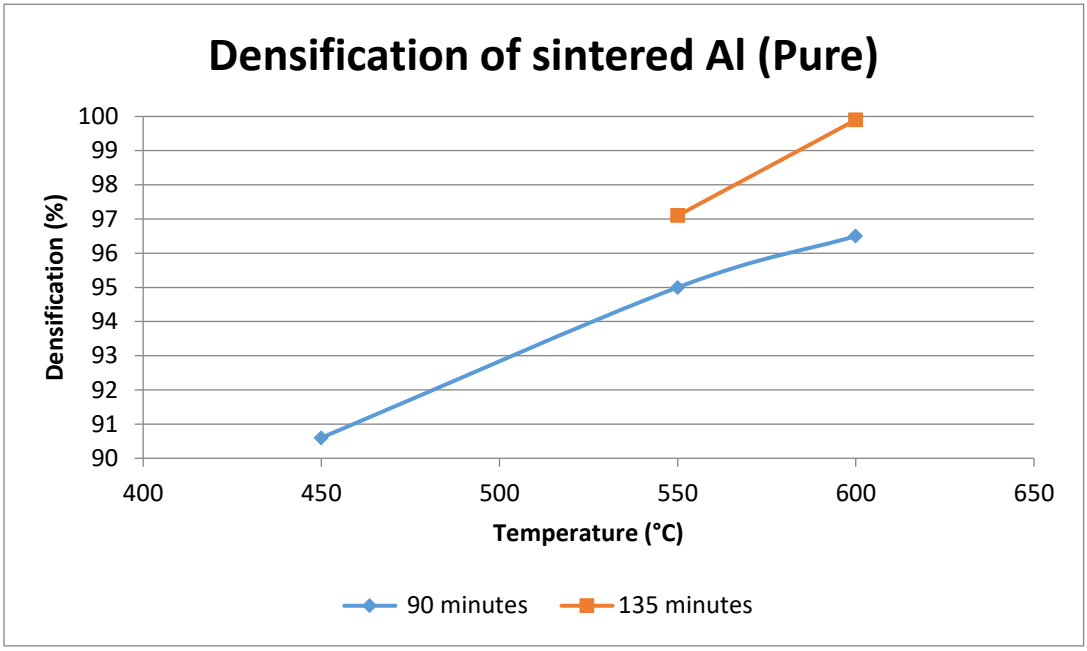
SEM micrographs of the CNTs used throughout are shown below. This is done for visual verification.



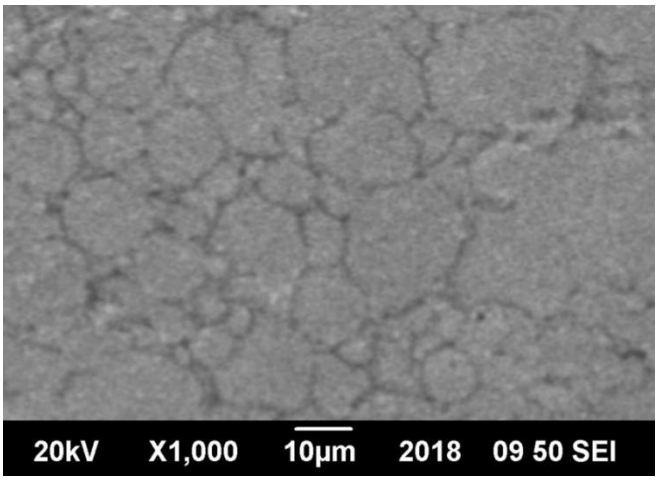


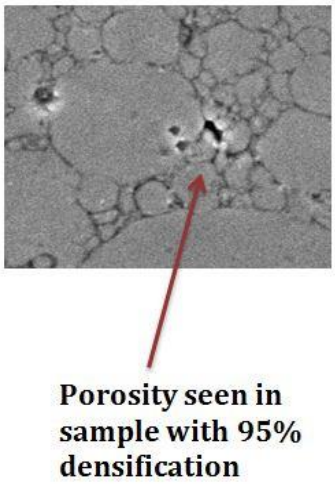
Optimization of sintering conditions

Five different conditions were tested out to find out the optimal sintering conditions. Shown below is how the final densification of the samples varied as a function of sintering time and temperature.

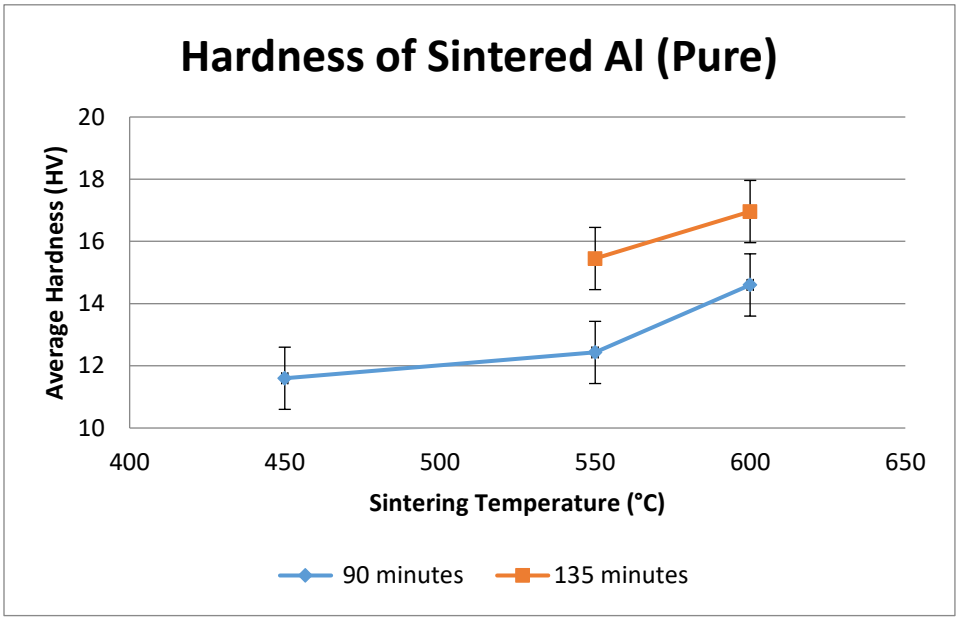


The maximum densification of 99.9% occurred at a sintering temperature of 600 C and a holding time of 135 minutes. Such a high densification value was not expected due the nature of cold uniaxial pressing and the pressure-less sintering method used but this value, did in fact, after verification, turn out to be true after examining the sample under a SEM and comparing it to a sample with porosity:





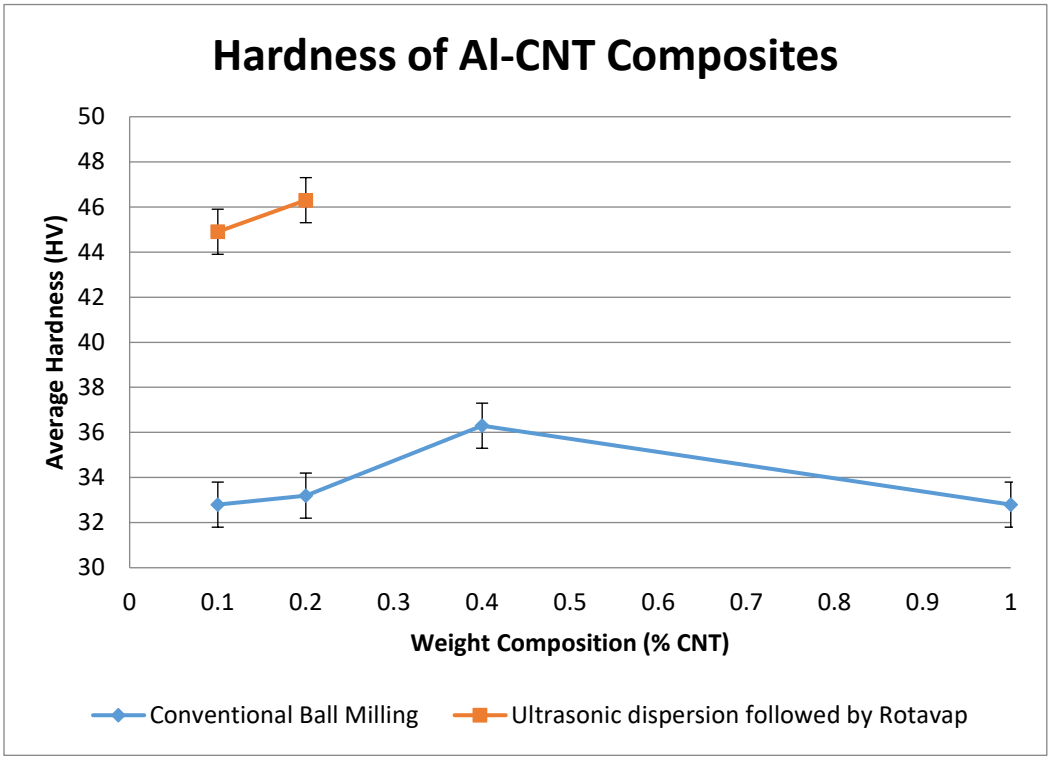
The hardness values were calculated using the micro Vikers method according to ASTM-E384 test procedures. A load of 200g was used with a dwell time of 10 seconds. The hardness values are shown below for pure Aluminum as a function of sintering time and temperature.



The maximum hardness of 16.96 HV occurred at a sintering temperature of 600 C and a holding time of 135 minutes. The difference in grain size was found to have a negligible effect on the hardness. This may be because the effect of pores far outweighed any change in grain size due to the different sintering conditions tested out here.

Hardness of Al/CNT composites

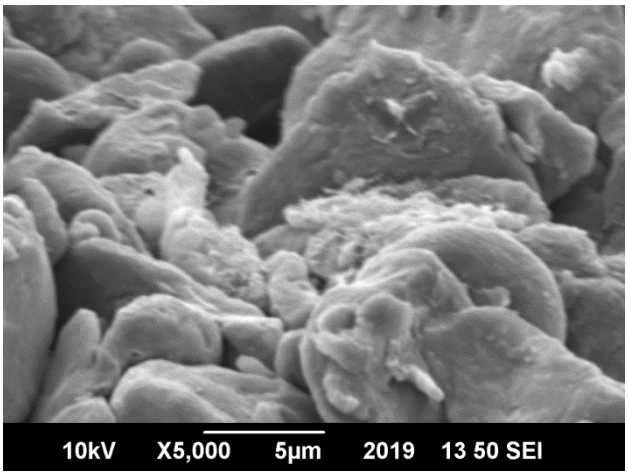
The hardness values were calculated using the micro Vikers method according to ASTM-E384 test procedures. A load of 200g was used with a dwell time of 10 seconds. The hardness values are shown below for two sets of composites, one fabricated by the conventional ball milling route using pristine CNTs and the other by the dispersion in DMF route using functionalized CNTs.

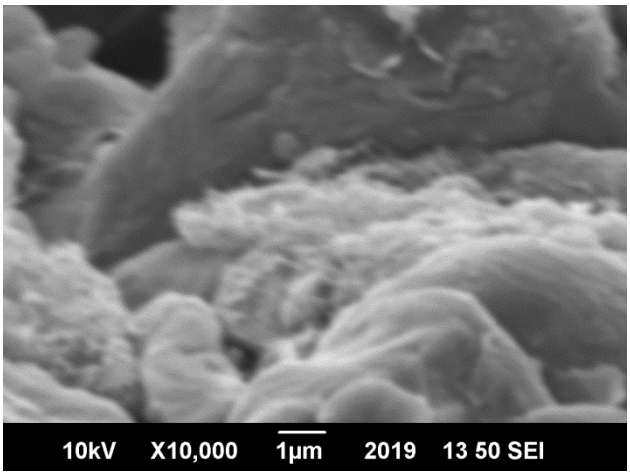


A maximum hardness of 46.3 HV was obtained using the second route with a CNT_f
weight fraction of 0.2%. For the first method it can be observed that hardness for the 1% weight fraction sample is less than that of the 0.4% weight fraction sample. This may be because of increased agglomeration in the 1% weight fraction sample

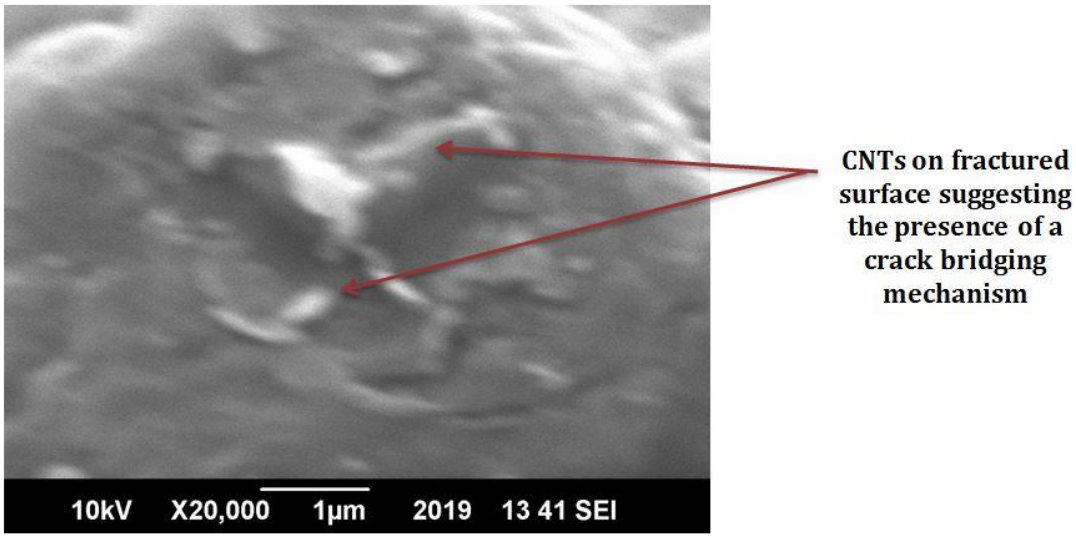
Fracture Surface

SEM micrographs of the fracture surface of a sample, this particular one having 0.4% weight fraction CNTs fabricated using the conventional ball milling route, is shown below:



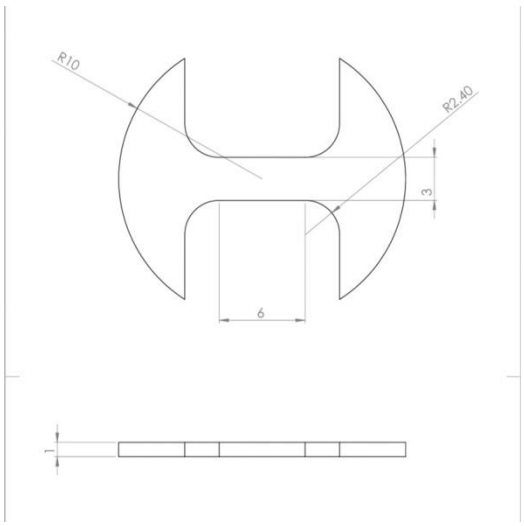


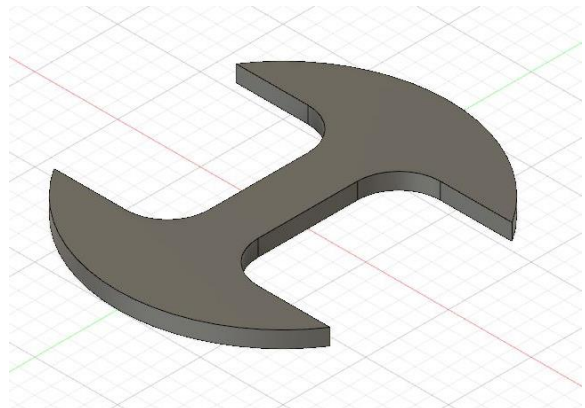
CNTs can been seen protruding out of the fractured surface; this suggests the presence of a crack bridging failure mechanism that can possibly increase the UTS and fracture toughness.



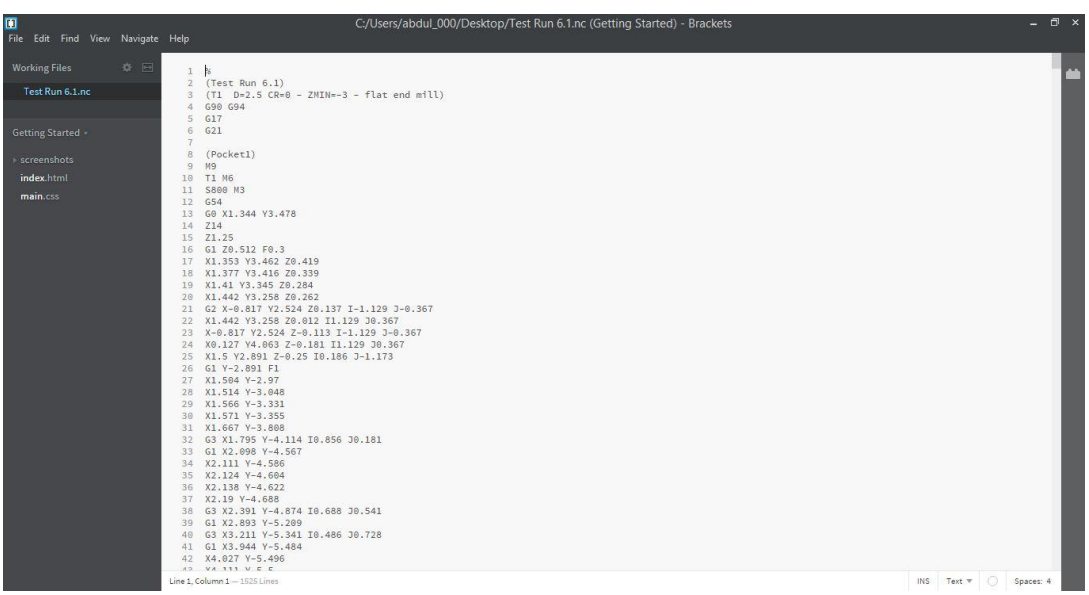
Utra Sub-size (USS) tensile specimen

Since the size of the samples did not allow for a standard ASTM tensile specimen to be made, an ultra-sub size specimen had to be used instead [117]. The dimensions were modified while keeping the critical ratios constant. An orthogonal drawing with units in mm and a CAD model is shown below:





The initial strategy employed to manufacture these USS tensile specimen were to use a 3-axis CNC milling machine to machine the sample. For this purpose a number of GRBL based G & M codes were devised. Part of a sample code by the name of ‘Test Run 6.1’ is shown below:



Although the latter codes worked with somewhat success, this strategy had to be dropped due to the following reasons:

With the existing setup, the optimal machining code had a calculated machining time of over 50 hours; this could not be pursued due to impracticality.

Secondly, as machining does impart residual stresses on the specimen, it was thought that this would affect the stress-strain curve to an extent that the specimen may not represent bulk properties. This was indeed proven to be true with test samples from an Aluminum bar.

Another strategy was chosen that has shown promising results so far, this employed a route that fabricates the USS samples in three steps shown in the diagram below:

A sinker electro discharge machining is first used to reduce the thickness of the composite sample, the shape is then imparted using wire electro discharge machining and finally electropolishing is then used to impart a smooth surface and remove the heat effected zones and residual stresses created due to electro discharge machining.

Future Work

Future work may be focused on:

- Using Aluminum 2024 as matrix material instead.
- Studying the effect of different surfactant treatments of reinforcement on dispersion & mechanical properties.
- Studying the effect of different sintering atmospheres on mechanical properties.
- Exploring new fabrication routes.

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You will find me a dedicated professional and an enthusiastic individual. I have the utmost faith in my capabilities, expertise and the willingness to learn, improve and adapt. I would therefore request you to please give me the opportunity to join KAIST and get enrolled in the Master's degree of "Nuclear and quantum engineering" and work in in your laboratory as one of your maters students.