Fabrication and characterization of aluminum-carbon nanotubes (Al-CNT) functionally graded cylinders

Sherry Samy Morad

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Fabrication and Characterization of Aluminum-Carbon Nanotubes (Al-CNT) Functionally Graded Cylinders

BY

Sherry Samy Morad

A thesis submitted in partial fulfillment of the requirements for the degree of

Master of Science in Mechanical Engineering

Under the supervision of:

Dr. Amal Esawi

Professor, Department of Mechanical Engineering

The American University in Cairo

The American University in Cairo

December, 2016
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Has been approved by

Dr. Amal Esawi
Thesis Committee Chair / Advisor
Professor, Department of Mechanical Engineering, The American University in Cairo.

Dr. Mohamed Serry
Thesis Committee Reader / Examiner
Professor, Department of Mechanical Engineering, The American University in Cairo.

Dr. Randa Abdel Kereem
Thesis Committee Reader / Examiner
Professor of Metallurgy- Faculty of Engineering- Cairo University
ABSTRACT

Attention to carbon nanotubes (CNT) reinforced aluminum matrix composites has been growing considerably for the past decade owing to the expected improvement in specific properties that this nano-composite could offer. Functionally graded material (FGM), is also a rapidly developing field of materials science offering the possibility to manufacture components with desired properties at selective locations to serve different applications. Nano FGMs are the newest generation of this class of materials that is currently being theoretically researched intensely with more requirement for experimental research to support and verify the theoretical and numerical models.

In the current research, an experimental study was conducted to develop a route to fabricate Al-CNT functionally graded cylinders varying materials along radial direction. Larger content of CNT reinforcement was selectively used at the outer layers to provide the highest strength, hardness and wear resistance at the surface subjected to higher stresses and more severe environments. Pure Al was chosen as the core material since it is subjected to lower stresses and to provide overall ductility to the material. The gradient selected is also cost effective as the expensive CNT is placed only in the layers that require most strengthening while maintaining a soft tough core leading to a balanced set of properties. The produced specimens having increased strength and hardness at the surface in addition to overall combination of high strength, ductility and light weight, together with the cylindrical geometry of the produced FGMs could be used in various applications especially mobility related applications such as drive shafts for aerospace and automotive industries. Testing and characterization of the produced samples were carried out to evaluate the performance of the cylindrical FGMs and the interfacial bonding between the different layers.

Two sets of functionally graded specimens were produced; first is “FG2%” having the 2%CNT-Al at the surface then followed by layers of 1%CNT-Al, milled Al and reaching pure Al at the core. The second is “FG5%” having the 5%CNT-Al at the surface then followed by layers of 2%CNT-Al, milled Al and reaching pure Al at the core. Two specifically designed molds were manufactured to produce the functionally graded cylinders; mold A and mold B with enhancements implemented in mold B to produce well bonded cylindrical FGMs. Al-CNT composite powders with 1, 2, 5wt.% CNT fraction as well as milled Al were manufactured deploying planetary high energy
ball milling technique (HEBM) of powders of CNT and Al. These were loaded in the designated layers of the manufactured molds. Powder metallurgy process was followed; starting with cold compaction and followed by sintering, then hot extrusion to produce compacts of Al-CNT FGMs of high density. Mechanical testing and characterization were carried out on the produced specimens and these included; mechanical compression, tension, optical microscopy, scanning electron microscopy as well as nanoindentation.

It was concluded that the developed route and novel manufactured molds were successful to produce well bonded functionally graded cylindrical structures of Al-CNT. Adding CNT to Al reaching 2%wt. CNT at the outer layer with varying composition to reach pure Al at the core resulted in very unique balanced properties. The samples showed high strength while retaining a higher percentage of the material ductility compared to pure Al and homogeneous composite competitor having the same overall CNT content. The compression strength and hardness values also showed significant enhancement. Increase in content of CNT up to 5%wt. showed a remarkable increase in compression strength and hardness with some enhancement in modulus of elasticity. However, its full potential of strength and ductility could not be confirmed in this study due to fracture of the specimens under tension outside the gauge length. This could be due to increased sample notch sensitivity or increased internal stresses between layers as CNT percentage increases.
ACKNOWLEDGMENTS

My deep gratitude goes out to every single person who has contributed to this work in every possible way. First of all, I am sincerely thankful to my very supportive wise advisor and professor, Dr. Amal Esawi for all her valuable contributions to this piece of work. Her scientific input, personal encouragement, and continuous true support during different times were the reasons behind completing this study.

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I also sincerely acknowledge the valuable contributions from Research and Development Engineer Ehab Salama, as a member of our small research group for all his efforts, great team spirit and passion to assist in this research study.

I would like to extend my thanks to Testing Lab Engineer Zakarya Taha, Workshop Engineer Khaled Iraqi, and all responsible for providing adequate facilities at the testing lab and workshop for their help during experimental and testing stages of this study.

Last and certainly not least, I would like to thank Yousef Jameel Science and Technology Research Center team for facilitating testing and characterization requirements for this research. I am greatly thankful to Engineer Ahmed Nour, Engineer Ahmed El Beltagy, Mrs. Nelly Kamel and Eng. Ahmed Ghazaly for all their assistance during the testing phase of my study.
DEDICATION

I sincerely dedicate this work to my beloved family and true friends; Mum and Dad my lifetime inspiration and extraordinary true role models in every possible way, my husband and backbone, my sister and wise advisor, my mother in law and supporter and my two gorgeous daughters Joy and Angela who are my whole life....
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<td>Al</td>
<td>Aluminum</td>
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<tr>
<td>BPR</td>
<td>Ball to Powder Weight ratio</td>
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<td>CC</td>
<td>Centrifugal Casting</td>
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<td>CNT</td>
<td>Carbon Nanotubes</td>
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<td>CVD</td>
<td>Chemical Vapor Deposition</td>
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<td>EBSD</td>
<td>Electron Backscattered Diffraction</td>
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<td>FGL</td>
<td>Functionally Graded Laminates</td>
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<td>MA</td>
<td>Mechanical Alloying</td>
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<td>MM</td>
<td>Mechanical Milling</td>
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<td>Metal Matrix Composites</td>
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<td>MMNC</td>
<td>Metal Matrix Nano Composites</td>
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<td>MWCNT</td>
<td>Multi-walled carbon nanotubes</td>
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<td>OM</td>
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<td>PCA</td>
<td>Process Control Agent</td>
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<td>Raman</td>
<td>Raman spectroscopy</td>
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<td>SEM</td>
<td>Scanning electron microscopy</td>
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<td>SFF</td>
<td>Solid Free Form</td>
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<td>SPS</td>
<td>Spark Plasma Sintering</td>
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<td>TEM</td>
<td>Transmission electron microscopy</td>
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CHAPTER ONE

INTRODUCTION

Many engineering applications nowadays require unusual combinations of properties to match the rapid advances in various fields and environments hence the need for novel materials instead of the pure or traditional ones. Composites are advanced materials, where two or more materials in solid state are combined, offering properties different from original materials [1]. Metal matrix composites (MMCs) are created by dispersing particles, fibers or whiskers as reinforcements in a metal matrix to obtain a new homogenous material with enhanced properties that match the needs of the target application. The advantage of MMCs compared with alloys is their ability to be tailored to specific design needs by altering the reinforcement shape, size, content, and distribution [2-4].

Recent advances in ultra-small engineering sciences and severe competition for producing more effective products during the last two decades has motivated scientists to work on new composite materials with nano scale fillers. One type of nano fillers is carbon nanotubes (CNT). The outstanding characteristics of carbon nanotubes, positioned them as superior reinforcement for composite materials owing to their high aspect ratio, low density together with high specific strength and stiffness [5]. Aluminum is also an interesting material as it is light in weight, easy to form, with high electric and thermal conductivity as well as high corrosion resistance. Hence, the attention to CNT-Aluminum composites was increasing significantly over the past decade owing to the improved properties that this nano-composite could offer [6].

Recent studies on Al-CNT composites have reported two main problems, one is the agglomeration of CNTs and difficulty of uniform dispersion in the Al matrix and the second is the diminished ductility of the composite accompanied with enhancements in mechanical properties with respect to specific strength [3, 7]. Diminished ductility could be attributed to mechanical milling which strain hardens the matrix, the addition of reinforcements which are known to reduce ductility or a combination of both. Extensive focus was made on coming up with new techniques to achieve better bonding and disperse CNTs more homogeneously in the matrix. Powder metallurgy as a fabrication method using High Energy Ball Milling (HEBM) proved being a very good technique to
evenly disperse CNTs in Al matrix. This is because in PM during fracturing and cold welding, the particles are deformed and the particle distribution is continuously being improved during deformation. HEBM evenly embeds reinforcements into aluminum particles to produce Al-CNT composites of high compaction density. However, milling conditions should be carefully controlled and percentages of CNT reinforcement well optimized to prevent excessive matrix strain-hardening and deteriorated properties. [8, 9]

A number of studies were carried out in an attempt to improve the ductility of Al-CNT composites using bi/multi modal grain sizes so that high strength would be attained from the finer grains while plasticity is retained through the coarser grains [3, 10]. Results on dual matrix showed that the composite could maintain some ductility provided that the suitable milling conditions and mixing ratios are used [10].

Further attempt to improve ductility was to use the Functionally Graded Materials (FGMs) concept in Al-CNT composites to attain the desired combination of high strength while maintaining adequate ductility [11]. FGMs are heterogeneous composites, comprising of different materials, with varying compositions [2]. FGMs were initially utilized in Japan during a space plane project in the year 1984 and since then their characterization, superior properties and production methods have been critical issues in this rapidly developing field of materials science [9, 12]. A detailed review of literature on FGMs and nano reinforced FGMs is provided in chapter two.

For FGMs, the ability to improve certain properties in specific locations of the material hence obtaining more specific functionalities across the end product is the most interesting attribute to researchers and eventually to industries. The added value of introducing FGMs appears in materials operating under combined loads experiencing mechanical stresses and extreme conditions as of large temperature gradients. Several countries nowadays have research groups working on FGMs as they are expecting an increase in the wide range of applications of FGMs in different areas as aerospace, automotive, defense, energy and medicine [13]. However, majority of work on FGMs used analytical and numerical methods and while improved new approaches are developed yet barely sufficient experimental work is done to verify the models[14].

Components rotating around their longitudinal axis find their way in many applications as power transmission using shafts, helicopter drive and automotive as
well as heavy machines as turbines, motors and flywheels [15, 16]. In order for these components to withstand high and at many times combined loads; novel structures such as FGMs were developed to reduce the deficiencies faced using homogenous composites in components of cylindrical geometries [17].

This research is an extension of previous studies done by the research group on Al-CNT composites with the following objectives:

- Investigating the possibility of reaching a compromise between strength and ductility using light weight Al metal and CNT as the reinforcement to produce co-axial functionally graded cylinders that could be potentially used in engineering applications such as drive shafts.

- Producing nano reinforced FGM samples with cylindrical geometry as opposed to the commonly fabricated layered FGM specimen films and laminates.

- Evaluating the performance of Al-CNT functionally graded cylinders with different combinations of Al-CNT composites, milled Al and pure Al by characterization techniques and testing of fabricated FGM samples.

- Characterizing the overall mechanical behavior of the FGM samples, as opposed to the majority of experimental researches in literature that measured tension and hardness properties of the individual layers.

The thesis is organized as follows; Chapter two "Literature Review" presents the most important and relevant literature on FGMs and nano FGMs in terms of properties, applications, and processing techniques, with a specific focus on relevant experimental studies done by other researchers in the field of Al-CNT FGMs or closely related fields. Chapter Three "Materials and Experimental Procedure" provides details of methodology, testing and characterization followed. Chapter Four "Results and Discussions" shows results of this study for the different compositions of the co-axial cylindrical Al-CNT samples. Mechanical properties and results of used characterization techniques involved are presented. Chapter Five "Conclusions" captures the main outcomes of the current research on Al-CNT coaxial cylindrical FGMs and this is then followed by future work recommendations in the same area.
CHAPTER TWO

LITERATURE REVIEW

2.1. Composites

Pure metals are not very often used due to the requirement of different and sometimes opposing properties of certain engineering applications. Combination of materials in the molten state to produce a characteristic unlike that of starting materials is termed “conventional alloying”, whereas, composite materials are obtained by combining materials in solid state [18]. Different composites are obtained depending on the material of the matrix (metal, ceramic, polymer, etc.) and the reinforcement.[4]

A metal matrix composite (MMC) is created by dispersing particles, fibers or whiskers, in a metal to form a multi-constituents material. The advantage of MMCs compared with alloys is their ability to be tailored to specific design needs by altering the reinforcement shape, size, content, and distribution. [2-4]. Li et al. explains that for MMCs there are two strengthening mechanisms: direct and indirect. The direct strengthening occurs by transferring the load from the matrix onto the reinforcement; however changes caused in the matrix microstructure due to addition of reinforcement or due to the deformation mode result in indirect strengthening [3].

Recently, studies on MMCs extended to include metal matrix nano composites (MMNCs). The size of nanoscale reinforcement particles affects properties of MMCs. Nanoscale fillers give improved properties, compared with the traditional bulk fillers owing to their high surface areas [9]. The main target of researchers working on MMNCs is to overcome some of the shortcomings of MMCs such as difficulty in dispersing nano fillers in addition to poor ductility, machinability, and fracture toughness [2]. Amongst different types of MMCs, Al-based MMCs are especially interesting as they are light in weight, easily formed, having high electric and thermal conductivity as well as high corrosion resistance. Such properties, in addition to current interest in nanoparticles have encouraged research of nano reinforced Al based matrices MMCs [6].

Carbon nanotubes (CNTs) are very interesting nano materials owing to their exceptional properties in mechanical, electrical and thermal applications [5, 19]. Carbon nanotubes were discovered by Iijima and are formed with a high length to diameter ratio
in a hexagonal tubular network of carbon atoms [20]. Mechanical properties of CNTs excel properties of many materials with reported values of high modulus values reaching 1 TPa and tensile strength reaching 30 GPa. These properties together with low density of CNTs make them very suitable in aerospace and sports industries [11, 12].

Carbon nanotubes exist in single-walled (SWCNT) or multi-walled (MWCNT) structures, to date MWCNTs are much cheaper than SWCNTs and hence used in many experimental studies for cost-effectiveness [19]. Esawi and Farag, studied the feasibility of using CNTs to reinforce polymer matrix composites both economically and technically [21]. The study showed that in order for CNT composites to become more commercial, longer lengths and bigger quantities should be produced as well as improved techniques for evenly distributing them in the matrix.[21]

A main difficulty to CNTs reinforcement of MMCs has been poor dispersion of CNTs in the metallic matrix due to agglomeration resulting in low interfacial bonding of CNT reinforcements and the matrices [12]. Dedicated studies were conducted to find workable techniques to disperse the carbon evenly in the matrix including sonication, ball milling or combination of both techniques.[7]

Ball milling as a technique to homogeneously disperse CNTs in aluminum powder has been researched in many studies. Esawi and Morsi, studied in details the effect of ball milling on Al-2wt% CNT composite powders morphology. The results showed that carbon nanotubes were evenly distributed within the Al matrix, however milling times and speeds used caused significant strain hardening of the powders causing further processing problems by conventional powder metallurgy techniques [8]. In a continued study by the same authors, improvement in mechanical properties occurred as CNT percentage increased until reaching a 5 wt.% when the mechanical properties were recorded to be unexpectedly below the predicted figures. This was attributed to CNTs large aspect ratio having a tendency to agglomerate at large volume fractions [7]. Sridhar et al. also report that techniques of cold compaction followed by sintering and extrusion were successful at producing near net shape components of aluminum matrix composites reinforced with 0.5, 1.0, and 2.0 % milled MWCNTs. No agglomeration of the reinforcement using CNT content reaching 2wt% volume fraction were reported [19].
Despite all these researches, limited achievement was attained, this is due to the challenge of the significantly diminished ductility accompanying the high strength of Al-CNT composites [3, 7]. Some ways are being investigated by the research group in an attempt to enhance the ductility without significantly compromising strength for example introducing bi/multi grain sizes [10] so that nanoscale grains provide the required strength whereas the coarse grains provides formability [3]. For bi/multi modal structures and composites, it is important to know the operating strengthening mechanisms in the Al-CNT composites such as, Orowan’s secondary phase dispersions and Taylor’s dislocation strengthening mechanisms [3, 6].

A recent study was conducted on dual matrix of aluminum-CNT reinforced composites with percentages of 1%, and 2.5 wt.% CNT produced from single matrix of Al-2 and 5 wt.% CNT composite and pure Al using high energy ball milling and powder metallurgy. This study showed that dual matrix structure was able to retain strength and ductility provided that the right milling conditions and mixing ratios are used. [10]

Emergence of functionally graded materials (FGMs) which could be considered as heterogeneous composites in which properties vary with position is another strategy that was developed to enhance composites’ performance such as reducing delamination (separation of fibers from the matrix) as well as providing unique combination of properties [18]. FGMs concept was recently researched in few studies for Al-CNT composites in order to attain this desired combination of high strength while maintaining adequate ductility. Detailed review of literature on FGMs and nano FGMs is provided in the coming sections.
2.2. Functionally Graded Material

2.2.1. Definition

Functionally graded materials (FGMs), including their properties and production methods are a rapidly developing field of materials science [22]. Many definitions of FGMs are used in literature, in an attempt to describe it as close and precise as possible the concept of FGMs [1, 18, 22-26]. Functionally Graded Materials are defined as “inhomogeneous materials, consisting of two (or more) different materials, engineered to have a continuously varying spatial composition profile” [23], “a homogeneous material in which the physical, chemical and mechanical properties change continuously from point to point” [24], “composite materials with controlled composition or structure” [25, 27], and “the selective and reproducible adjustment of the microstructure of a material to the setting of macroscopic component properties (eg. hardness, strength)”[26].

The application of these definitions prompts to the production of parts being specifically controlled in areas requiring certain properties such as enhanced strength, toughness as well as corrosion and wear resistance [22].

2.2.2. History and Background

In 1972, graded materials were introduced in theoretical papers by Bever and Duwez [28] and also by Shen and Bever [29], but had little effect, likely because of absence of fabrication techniques for FGMs around then [1, 30]. The idea of FGM was practically used 15 years later in Japan in a space plane venture. FGMs were used as a thermal barrier to tolerate surface temperature of 2000 K and a temperature gradient of 1000 K across a 10 mm section [22, 26]. Currently, FGMs have triggered world-wide research interest and studies are being conducted on metals, organic and ceramics composites to provide better-quality components with superior combinations of properties. [22]

The concept of FGMs is present in nature in the form of the human bone and teeth. Human bones are combination of a ductile protein polymer known as collagen and brittle calcium phosphate known as ceramic hydroxyapatite. The human bone has high strength at the surface which gradually lowers toward the inside by altering the porosity
Hence the idea of FGMs is taken from nature to answer some problems similar to man-made neural network that matches human brain [1].

2.2.3. Classifications and Types

Literature uses different categorizations to classify FGMs into different types according to gradient type, thickness and graded structure. Since FGMs are materials with gradual variation of properties with location, therefore they are referred to as gradient materials [24]. Gradient effect could result from different micro-structures and chemical composition and it could be along the whole component or at certain regions depending on application and requirements [30]. Depending on the type of gradient as shown in Figure 1, FGMs could be grouped into: % content gradient (a), shape gradient (b), orientation gradient (c) and size (of material) gradient (d) [24, 26].

![Figure 1 FGMs according to Types of Gradient [24]](image)

Ruys et al. [31], divides FGMs according to their thickness into: films, interface FGMs and bulks. Functionally graded films are graded coatings providing a solution to thermo mechanical mismatch that occurs at the film-substrate regions. Interface functionally graded films are used as a bonding region between two unlike materials. Functionally graded bulks have large graded cross-sections [27].

Mahamood, R.M., et al., broadly divide FGMs to two main categories: thin and bulk FGMs. Thin sections are processed by Physical Vapor Deposition or Chemical Vapor Deposition methods (PVD/CVD) in addition to self-propagating high temperature Synthesis (SHS) and plasma spraying. Centrifugal casting, powder metallurgy, and solid freeform could be used to produce bulk FGMs [18].
Kieback et al. and Udupa et al. classify FGMs into continuous structured FGMs presented in Figure 2 (a) and step FGMs presented in Figure 2 (b) [23, 30]. The variation in structure is shown with varying location in continuous FGMs, whereas for stepwise FGMs multilayers are formed with interface existing between layers in a stepwise fashion.

![Figure 2 Schematic Diagram of Classification of FGM by Gradation Structure][23]

### 2.2.4. Advantages

The main breakthrough and advantage that is of interest to researchers in FMGs is the possibility to manufacture components with desired local properties [27, 32]. In certain applications, for example drive shafts and gears, having different properties in the cross-section of the component depending on the position is very desirable. That is having for example increased wear resistance and hardness on the outside and a higher ductility at the inside [32]. According to Udupa et.al, the main idea behind having FGM is to attain opposing properties in one material, such as having a material that possesses at high temperatures a high hardness level and at low temperatures adequate “structural toughness” [23], enabling the bulk to have the best of both materials. Producing such structures extends the industrial applications of the considered materials.[22]

Also, sudden jumps of properties, as in hard coatings, are unwanted as they could be the reason for stress concentrations at regions of interface, accompanied by failure of the coating [33]. According to Carvalho, O. et al., the key benefits of FGMs are: thermal stress reduction; especially at critical locations, diminishing the sudden transitions of stresses at the interfacial regions and increase of the interfacial bond strength [32]. This is also supported by Bohadir, S. et al. stating that the introduction of
FGMs helps to eliminate the sharp interfaces present in composites at which failure could start [1] as the gradient produces smooth variation between different materials.

It is fair to say that introducing FGMs helps to solve many problems appearing at the material interface and also enhances bonding [24]. Moreover, adjusting the properties of only a part of the material, and not the whole bulk is cost efficient as the reinforcement material may not be wasted on unwanted locations of the bulk, making the process economical, especially on the industrial scale. Also development of such properties of FGMs can significantly reduce the requirement for preventive maintenance, increase safety, and reduce downtime leading to significant reduction of the operating cost [2].

2.2.5. Applications

There are numerous applications for FGMs and there exists a potential for them to increase if costs of processing and powder decrease by continual enhancements of processes[18]. FGMs have unique mechanical, thermal, optical, biomedical and electronic advantages [34], hence they find their applications in mobility engineering as aerospace, automobile and marine as well as defense, medicine, sport, energy, sensors and optoelectronics, etc [1, 23, 26].

FGM bulks offer wide prospective for components serving in severe operating conditions; for example, heat exchanger tubes, heat-engine components, wear-resistant drilling linings and rocket heat buffers [1, 35].

There are some commercial products being produced with graded materials but those commercially produced are mostly functionally graded laminates not bulks. Mitsubishi Materials developed their Indexable Inserts (Figure 3) made of a carbide substrate having a newly developed graded structure on the surface and CVD coated layers with a triple-structure. As reported, using FGM structure at the surface, improved the plastic deformation and damage resistance of the inserts [36].
A recent optimization study was also conducted for AlSi–CNT functionally graded composites processed by powder metallurgy (PM) for potential use in engine piston rings [32]. The problem with the traditional rings is the different coefficients of thermal expansion at high temperature occurring at the region between the two unlike materials; the coating and the metal, leading to delamination of the coating. Benefit of introducing FGMs is that it creates a transitional region of CNT reinforcement along the thickness of the piston ring.

Udupa et al. in his review paper on FGMs presents a list of some of the most common FGMs that could be used in different applications as per the specific functional requirements of these components replacing traditional composites and pure metal [37]. Table 1 extracted from Udupa’s review paper; shows many FGM applications in different fields. We have highlighted the Al based FGMs being of interest to the focus of our current research. Different reinforcements are used with the Al or Al alloy base metal, depending on the required application; however it is shown that Al-C or Al-CNT FGMs are mainly used for the unique combination of light weight, hardness, strength and ductility required for the applications highlighted below.
Table 1 List of Functionally Graded Composite Material and the Applications[37]

<table>
<thead>
<tr>
<th>SLNo</th>
<th>FGCM type</th>
<th>Requirements</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SiC-SiC</td>
<td>Corrosion resistance and hardness</td>
<td>Combustion chambers</td>
</tr>
<tr>
<td>2</td>
<td>Al-SiC</td>
<td>Hardness and toughness</td>
<td>Combustion chambers</td>
</tr>
<tr>
<td>3</td>
<td>SiCw/Al-alloy</td>
<td>Thermal resistance and chemical resistance</td>
<td>CNG storage cylinders, Diesel Engine pistons</td>
</tr>
<tr>
<td>4</td>
<td>(E-glass/epoxy)</td>
<td>Hardness and damping properties</td>
<td>Brake rotors, Leaf springs</td>
</tr>
<tr>
<td>5</td>
<td>Al-C</td>
<td>Drive shafts, Hubble Space telescope metering truss assembly, Turbine rotor, Turbine wheels</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>SiCp/Al-alloy</td>
<td>High melting point, low plasticity and high hardness</td>
<td>Motorcycle drive sprocket, Pulleys, Torque converter reactor, Shock absorber</td>
</tr>
<tr>
<td>7</td>
<td>Carbon and glass fibers</td>
<td></td>
<td>Propulsion shaft</td>
</tr>
<tr>
<td>8</td>
<td>Glass/Epoxy</td>
<td>Cylindrical pressure hull, Sonar domes</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>TiAl-SiC fibers</td>
<td>Composite piping system, Scuba diving cylinders</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Be-Al</td>
<td>Floats, Boat hull, Wind tunnel blades, Spacecraft truss structure, Reflectors, Solar panels, Camera housing</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Al,O2/Al-alloy</td>
<td>Good thermal and corrosive resistance</td>
<td>Rocket nozzle, Wings, Rotary launchers, Engine casing</td>
</tr>
<tr>
<td>12</td>
<td>Carbon/Bismakridide</td>
<td>Drive shaft, Propeller blades, Landing gear doors, Thrust reverser, Heat exchanger panels, Engine parts</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>Carbon/Epoxy</td>
<td>Lightweight and good damping properties</td>
<td>Helicopter components viz. Rotor drive shaft, Mast mount, Main rotor blades</td>
</tr>
<tr>
<td>14</td>
<td>SiCw/6061</td>
<td>Hard and toughness</td>
<td>Racing bicycle frame, Racing vehicle frame</td>
</tr>
<tr>
<td>15</td>
<td>(Al-alloy/CNT), Light weight and high stiffness</td>
<td>Artificial ligaments, MRI scanner cryogenic tubes, Wheelchairs, Hip joint implants, Eyeglass frames, camera lenses, Musical instruments</td>
<td></td>
</tr>
</tbody>
</table>

2.2.5.1 Application of FGMs for Drive Shafts

Referring to Table 1, drive shafts are among the various applications of FGMs. Drive shafts deployed in mobility applications as in automotives (Figure 4), aircraft and aerospace, are an interesting area to our research focus where the combination of light weight, high strength and toughness is of utmost importance to energy efficiency, higher accelerations and reliability of transport applications [38-40].

![Figure 4 Schematic arrangement of Underbody of an Automobile [38]](image)

Replacing the conventional metals with new high tech composites and nanocomposites as the constitutive material of a drive shaft, provides lighter and longer
drive shafts for a special critical speed [41]. Shojaeefard et al. in their optimization study of CNT nanocomposite drive shafts, proposed a new method to model the properties of a functionally graded nanocomposite using ANSYS and ABAQUS software products. The numerical and finite element analysis showed that designing a drive shaft with a particular distribution of CNT FGM replacing homogenous composite or aluminum one, will ultimately remarkably enhance mechanical properties of the shaft [41].

Combined loads are exerted on the drive shafts as axial and torsional loads hence its design is of great importance. Bankar et al. [39] conducted a study to use a composite drive shaft instead of a connected two piece metallic drive shaft. One piece shafts eliminate the assembly connecting the two metal shafts hence reducing overall weight, vibrations and cost [39, 40]. As concluded from their study, solid or hollow cylinders are the two choices for drive shaft design. Solid shafts provide maximum torque but as their weight increase the frequency decreases. In addition, the shaft’s outer surface is subjected to maximum stresses while the inner material layer was subjected to lower magnitudes of stress, so the inner materials add to the total weight of the shaft while not being properly used for efficient distribution of stresses. FGM cylindrical components could be a solution to challenges of drive shaft designs.

2.2.6. Processing Techniques

Sobczak J. et al. broadly divides the production methods of FGMs into two groups. In the first group components of the desired bulk undergo some preliminary preparations and are arranged in a graded manner then processed by a suitable technique as sintering or melt infiltration to finally reach the graded product. In the second group, FGM structure is formed by application of external forces such as gravity, centrifugal or magnetic on the separate components in certain liquid composite slurry [22].

The most commonly reported fabrication methods according to O. Carvalho et al. for bulk FGMs are: thermal/chemical processes, centrifugal casting and powder metallurgy [27]. As for Gupta, K. M. he categorizes the processing techniques into: thermal spraying, powder metallurgy and coating processes, [24], where choosing the suitable method depends on the materials used, geometry of end product and transition required. For powder metallurgy, FGM is fabricated first by mixing different materials in powder form with the calculated required percentages and loading the mixed powders
in consecutive layers. On the other hand, thermal spraying combines high strength phases with metals of low melting temperatures, whereas melt processing produces FGMs by centrifuging of high melting temperature materials with metals of low melting temperatures. As for coating, a layer of a hard material is force applied on the surface to the interior while varying the binder content [24].

Kieback et al. presents a comprehensive detailed review on the different processing techniques for FGMs and the involved parameters and conditions for fabrication together with an in depth review of modeling of FGM processing [30]. In the review, they divide the manufacturing process of FGMs to mainly include: gradation of structure and consolidation of component. First step is gradation which is forming the inhomogeneous structure and consolidation is transforming it to a bulk material[30]. Consolidation takes place by pressure and sintering where high attention is given to selection of process conditions so that the gradient is not damaged or changed in any manner.

According to Rasheedat M. et al. processing of FGMs depends on whether the end product is a thin FGM or a bulk FGM [18]. Surface coating, laminates or thin FGMs are produced by Plasma Spraying, PVD, CVD or Self-propagating High temperature SHS [42]. These techniques apply thin graded coatings on the surface with superior properties, however they have disadvantages of being slow as well as emitting poisonous byproducts [1, 18]. On the other hand, bulk FGMs could be produced by Powder Metallurgy (PM), centrifugal casting method or Solid freeform (SFF) [36].

Powder metallurgy is a widely used processes to manufacture well compacted FGM bulks and fundamentally follows procedures used for ceramics manufacturing where the operating parameters include particle size distribution, mixing techniques, pressing, and finally sintering [43]. PM technique or as sometimes referred to as Mechanical Alloying (MA) was developed in early 1960s [9] and is also used now to produce functionally graded material. PM process goes through three main processes: powder mixing as per the functional requirement, powders loading and sintering [16]. PM produces stepwise functionally graded products. Centrifugal method could be chosen if more continuous gradient is required.

In the centrifugal method, force of gravity to spin the mold and differences in material densities are used to produce FGMs [17]. Despite the fact that centrifugal
casting produces continuous grading, yet tubular or cylindrical geometries could only be produced. Also centrifugal casting limits the type of gradient produced [18] since gradation is a byproduct of natural process of centrifugal force and difference in densities. A very recent research was conducted by Radhika et al. using centrifugal method to develop FG aluminum composites and hollow cylindrical components were produced [44]. Reinforcements resided at the surface of the functionally graded composites owing to their higher densities in comparison to the matrix material, but not in an accurately controlled manner regarding their percentage and distribution.

Researchers are also developing models and prototypes for producing FGMs using additive manufacturing and rapid manufacturing processes utilizing computer controlled, less-tools manufacturing methods. Knoppers, et al. explains that producing complex graded material distributions in complicated geometry parts can only be accurate and applied in wider scale if driven by computerized optimizations [36]. Rapid manufacturing for FGMs enables the creation of products with changing material properties in any direction not only perpendicular to the surface of the part as is the case for most manufacturing methods. In their research, Knoppers and his team were able to find a solution to define graded material property distributions in a CAD like environment to enable sending graded material information to rapid manufacturing machines for graded materials production based on the assignment of certain properties to specific areas within the enclosed volume of a solid.

Solid Freeform (SFF) Fabrication Method is regarded as the most commonly recognized additive manufacturing process that can be adopted for FGM production [18, 27, 30]. SFF offers advantages of high production speed, with optimum material use as well as possibility of having complex shapes, however poor surface finish is obtained and hence it is important to undergo secondary finishing [18]. SFF broadly goes through five main steps [18, 45]; CAD data models of the required part is generated and then converted to a Standard Triangulation language (STL) file. STL file is then sliced into two dimensional profiles to enable building of the component layer by layer [46] until reaching the final steps of finishing. SFF technologies include laser SFF processes used in production of FGMs as referenced in literature including laser cladding, selective laser sintering, 3D printing and selective laser melting [18, 47, 48].
According to Kieback et al. deciding on the most suitable processing method depends on the component’s geometry, the used materials and reinforcements in addition to the type of the gradient to be formed.

Table 2 provides a list of processing methods for FGMs, parameters of choice and limitations in each process [23, 30, 49].

Table 2 Processing techniques for FGMs [23, 30, 49]

<table>
<thead>
<tr>
<th>Process</th>
<th>Variability of transition function</th>
<th>Layer thickness(^a)</th>
<th>Versatility in phase content</th>
<th>Type of FGM</th>
<th>Versatility in component geometry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder stacking</td>
<td>Very good</td>
<td>M, I</td>
<td>Very good</td>
<td>Bulk</td>
<td>Moderate</td>
</tr>
<tr>
<td>Sheet lamination</td>
<td>Very good</td>
<td>T, M</td>
<td>Very good</td>
<td>Bulk</td>
<td>Moderate</td>
</tr>
<tr>
<td>Wet powder spraying</td>
<td>Very good</td>
<td>UT, T(^b)</td>
<td>Very good</td>
<td>Bulk</td>
<td>Moderate</td>
</tr>
<tr>
<td>Slurry dipping</td>
<td>Very good</td>
<td>M, L</td>
<td>Very good</td>
<td>Bulk</td>
<td>Very good</td>
</tr>
<tr>
<td>Jet solidification</td>
<td>Good</td>
<td>C</td>
<td>Very good</td>
<td>Bulk(^c)</td>
<td>Good</td>
</tr>
<tr>
<td>Sedimentation/centrifuging</td>
<td>Good</td>
<td>C</td>
<td>Very good</td>
<td>Bulk(^c)</td>
<td>Good</td>
</tr>
<tr>
<td>Filtration/dip casting</td>
<td>Very good</td>
<td>M</td>
<td>Very good</td>
<td>Bulk(^c)</td>
<td>Very good</td>
</tr>
<tr>
<td>Laser cladding</td>
<td>Very good</td>
<td>T</td>
<td>Very good</td>
<td>Coating</td>
<td>Good</td>
</tr>
<tr>
<td>Thermal spraying</td>
<td>Very good</td>
<td>T</td>
<td>Very good</td>
<td>Coating bulk</td>
<td>Good</td>
</tr>
<tr>
<td>Diffusion</td>
<td>Moderate</td>
<td>C</td>
<td>Very good</td>
<td>Joint, coating</td>
<td>Good</td>
</tr>
<tr>
<td>Directed solidification</td>
<td>Moderate</td>
<td>C</td>
<td>Good</td>
<td>Bulk</td>
<td>Good</td>
</tr>
<tr>
<td>Electrochemical gradation</td>
<td>Moderate</td>
<td>C</td>
<td>Good</td>
<td>Bulk</td>
<td>Good</td>
</tr>
<tr>
<td>Foaming of polymers</td>
<td>Moderate</td>
<td>C</td>
<td>Good</td>
<td>Bulk</td>
<td>Good</td>
</tr>
<tr>
<td>PVD, CVD</td>
<td>Very good</td>
<td>M, L, C</td>
<td>Very good</td>
<td>Coating</td>
<td>Moderate</td>
</tr>
<tr>
<td>GMFC process</td>
<td>Very good</td>
<td>M, L, C</td>
<td>Moderate</td>
<td>Bulk</td>
<td>Good</td>
</tr>
</tbody>
</table>

\(^a\)T: large (>1 mm); M: medium (100–1000 μm); UT: very thin (<10 μm); C: continuous.
\(^b\)Depending on available powder size.
\(^c\)Maximum thickness is limited.

2.2.7. Modeling and Simulation

Studies on behavior of FGMs are many and literature provides many examples due to the extended applications of this material. Numerical approaches were established to model FGM designs and others to simulate their mechanical behavior to predict their responses [27].

A review on modeling and theoretical analysis of FGMs was documented in 2007 by Birman and Byrd [49] presenting different characteristics of FGMs including, stress distribution, homogenization, fracture, static and dynamic analysis in addition to heat transfer. Cracks created in FGMs due to different phases of the material are a critical issue. Kieback el al. [30] discusses modeling used for prediction of cracks; suggesting that cracking at the interface between the different layers of FGMs could potentially occur during processing. This is because different regions sinter at different rates thus creating incompatibilities between the different layers. A summary regarding fracture analysis of functionally graded materials was carried out by Shanmugavel et al. [18, 50] and a study on crack propagation in FGMs was also conducted by Bahr et al. explaining the damage resulting from shrinkage due to sintering and also due to thermal
cycling causing high-temperature creep, that increase the rate of energy released and end up into delamination cracks [51].

A recent crack propagation analysis was conducted for Aluminum matrix-SiC reinforced functionally graded cylinder obtained by centrifugal casting [14]. Tensile strength was not obtained on the full bulk, however the specimen was vertically sliced into four sections and tension testing was done on each individual section as shown in Figure 5. Results of analysis showed that cracks started later in sections with higher content of SiC compared to aluminum sections, fatigue life also improved due to increase in SiC for specimens notched at the center.

Figure 5 Tensile Test Sample by Vertical Slicing[14]

Other reviews on functionally graded cylinders and shafts are also available in the literature since as previously mentioned; cylindrical structures (cylindrical shafts, cylindrical shells and cylindrical panels) consisting of FGMs make their way to mobility and military applications [15]. A very interesting and comprehensive review paper was presented in early 2016 by Hong-Liang et al. covering the majority of studies conducted on FGM cylinders during the period of 2005-2015, with a focus on coupled mechanics as thermo-elastic and fluid-solid couplings, etc.[15]. Rooney and Ferrari also studied bending, flexure and tension behavior of functionally graded cylinders [52]. This study concludes that for tension applications, any grading will improve the performance, however if the application requires bending or flexure, then if the phase having highest Young’s modulus is far from the bending axis, this will provide less deformation for a specific bending moment.
Additional studies for anisotropic cylinders under mechanical and thermal stresses [53], dynamic response of cracks in FGMs under impact load and thermo-mechanical loading of FG hollow cylinders have been presented. Boukhalfa et al. presents a finite element study of a spinning shaft made of functionally graded material to predict dynamic behaviors as natural and whirling frequencies, and the spinning FGM shaft critical speeds [54].

The list of conducted studies and models is long and more in research yet to be completed to further enhance properties, performance and economics of processing methods of FGMs [18].

2.2.8. Challenges for FGMs

Despite of all advantages, progress and very promising applications, there are still some challenges that will affect how FGMs will evolve. For example how the processing techniques will be fit for mass production, reliability of the produced FGMs and quality control. Although simulation could ease some of these challenges [30], more experimental researches should be conducted to support and validate the results of numerical modeling in addition to generating a solid database of gradient material (Process parameters, material preparation and evaluation) [23, 55]

Cost-effectiveness of production processes is the serious challenge. Techniques used to produce graded components, increase the part’s cost [24] with significant portion spent on powder processing and fabrication [18, 21]. However it could be argued that FGMs would actually decrease required maintenance downtime for many operations due to enhanced properties hence reducing production costs [2]. Also expensive reinforcements are placed where they are most needed so in general the material is cost-effective

In addition, thin FGM films are widely investigated and are more readily becoming used by industry, however many severe-environment applications require bulk FGMs; yet such bulks have not yet found their way commercially. Most of the proposed fabrication methods are still high cost, labor-intensive, specialized laboratory techniques and not commercially competent [35].
2.3. Nano Reinforced Functionally Graded Materials

As part of the current researches in the area of functionally graded materials (FGMs), studies of nano reinforced FGMs have also been emerging with efforts mainly concentrating on the theoretical models and numerical solutions of the many factors affecting the performances of Nano FGMs [15, 20, 56, 57]. Limited researches have been conducted to date on the production of nano reinforced FGMs. This could be attributed to inadequately developed processing technologies required to produce graded nano reinforced composites as compared to homogeneous composites, in addition, to dispersion and interface compatibility issues [33].

CNT reinforced functionally graded composites possess a great potential of being the next generation material as it holds many uninvestigated properties that could solve many problems in various technological areas [58]. Attention in research was given to fabrication and testing of homogenous composites having CNT reinforcement within polymer, ceramic and metal matrices, whereas fabrication and testing of heterogeneous composites of CNT reinforced FGMs is still in the very early stages of investigation [59]. CNT reinforced functionally graded materials were first studied by Estili [60] and Shen [61] to enhance mechanical properties of the components. In a recent paper, Liew et al. presented a literature review of various studies on functionally graded CNT reinforced composites (FG-CNTRC) in terms of mechanical properties in addition to static and dynamic analysis, free vibration as well as buckling and non-linear analyses [11].

Most investigations of functionally graded CNT-reinforced composites are still based on numerical calculations or models. Vibration analysis of CNT-reinforced functionally graded materials was studied by Lei and Yas et al. for cylindrical panels [62, 63] and for sandwich beams by Kamarian et al. [64]. Factors affecting performance such as waviness of CNTs and their aspect ratio were analyzed by Moradi et al. on the stress and displacement distributions of FG nano composites of cylindrical geometry having wavy carbon nanotubes as the reinforcement [56]. They reported that CNT waviness plays a critical role for distributions and values of stress and displacement of the CNT reinforced cylinders. Thermal post buckling behavior of FGM CNT-reinforced circular shells under steady increase in temperature was studied by Shen and results reported show that the functionally graded reinforced composites affected positively the
results by increasing the thermal post buckling strength of the shells as well as their buckling temperature [57].

Literature presented some limited experimental case studies of nano FGMs using different matrices, reinforcements, as well as different manufacturing techniques. Nano reinforced polymer FGM study is presented by Yi Wang et al. who used centrifugal method to fabricate nano TiO2-epoxy FGM [33]. A coupling agent was used to avoid agglomeration of TiO2 during curing. The study confirmed that wear resistant materials could be produced in a graded manner of varying hardness and modulus along the FGM layers, hence reducing the cost by eliminating reinforcement consumption in unnecessary areas.

Nano reinforced ceramic FGMs fabrication studies are also presented in literature. X.Tian et al. fabricated an FGM ceramic tool of Si3N4/(W, Ti)C/Co nano-composite using hot pressing [65]. Results show that the FGM structure improved the properties particularly fracture toughness and flexural strength while maintaining the ceramic advantage of increased hardness. Some interesting conclusions show that the formation of the elongated grains introduced toughness mechanism similar to whiskers. Also, thermal mismatch between Si3N4 and nano (W,Ti)C reinforcement, is suggested to have induced some dislocations in the intragranular structure of the Si3N4 grains, these could actually prevent cracks propagation and ultimately delay fracture. The “pin effect” of the nanoparticle along the boundary also strengthened the grain boundaries.

Another study of fabrication of nano reinforced ceramics was presented by S. Markovic et al. [66]. Nanostructured calcium phosphate FGMs were produced by pressing of stoichiometric (SHAp) and calcium deficient (CDHAp) hydroxyapatite powders in layers stacked samples as shown in Figure 6. Spatial change of porosity, phase compositions, hardness and young’s modulus promotes this fabricated bioceramic FGM to provide an appropriate model for functionalities of artificial bone material.

![Figure 6 Schematic of Uniaxial Layered FGMs](image)

With regards to Al-CNT FGMs, the current experimental data is still very much limited [37, 59]. In addition, the available experimental studies focused on linear
variations of materials in layered structured specimens [23, 37, 59, 67], with none to the best of the author’s knowledge performed on cylindrical FGM specimens with coaxial variation of materials or compositions. Also very few studies investigated the mechanical performance as in tension and compression behaviors with most of the studies investigating hardness and microstructure.

Kwon et al. report that they successfully fabricated Al-CNT reinforced FGM using powder metallurgy in layered structure, in which the Al-CNT powders were prepared using the planetary ball mill followed by hot pressing in a layered structure using different weight percentages of CNT: 0, 5, 10 and 15% [59]. Hardness tests and SEM characterization were used to evaluate the samples produced in the study. SEM characterization was performed to the as received and composite powders as well as the Al-CNT FGM specimens as shown in Figure 7.

![Figure 7](image)

Figure 7 (a) Digital image of CNT-Al FGM, SEM images of (b) 10 & 15% CNT-Al FGM layer, (c) pure Al, (d) 5% CNT-Al (e) 10%CNT-Al and (f)15% CNT-Al [59].

Results show that CNTs were well dispersed and that a fully dense Al-CNT FGM bulk was successfully fabricated by powder metallurgy process including ball milling and hot pressing. With increase in CNT content, composite powders were finer in terms of mean particle size. Vickers hardness increased with increase in CNT content reaching a maximum enhancement of seven times compared to pure Al as shown in
Table 3. In addition, the author suggests that hardness records in FGM layers were enhanced due to reinforcement by CNT, homogeneous dispersion, pin effect as well as internal stress created at the interfacial regions. 

*Table 3 Density and Hardness of CNT-based Al FGM [59]*

<table>
<thead>
<tr>
<th>CNT (%)</th>
<th>Theoretical Density</th>
<th>Measured Density ±2%</th>
<th>Vickers Hardness ±5 Powder</th>
<th>Vickers Hardness ±5 FGM</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.69</td>
<td>2.69</td>
<td>34.0</td>
<td>33.2</td>
</tr>
<tr>
<td>5</td>
<td>2.65</td>
<td>2.65</td>
<td>120.1</td>
<td>164.5</td>
</tr>
<tr>
<td>10</td>
<td>2.61</td>
<td>2.61</td>
<td>188.6</td>
<td>217.0</td>
</tr>
<tr>
<td>15</td>
<td>2.56</td>
<td>2.56</td>
<td>192.2</td>
<td>258.1</td>
</tr>
</tbody>
</table>

Udupa et al. produced Al-CNT functionally graded laminates (FGL) [23, 37]. The author suggests that the mismatch of the gradients in a specific direction that could lead to damage in case of conventional FGM, can be avoided by FGL using very thin layers of laminates with different CNT content varying from one layer to the other as shown in Figure 8. Pressure is applied to bond the laminates of 0.5wt% -0.1wt% CNT to reach pure Al at the other side of the structure [37].

![Figure 8 Schematic diagram of FGCM component][37]

PM technique was used for this research, where CNT reinforced Al based powder were prepared using a mixing process involving intensive sonication followed by planetary ball milling for 15 hours. The layered structure was manufactured successfully using cold compaction followed by sintering. Microscopic and SEM characterization, hardness tests, density measurements and XRD, were used to evaluate performance of the Al-CNT FGL. SEM of milled powders showed good CNT distribution within matrix (Al); however the time of milling to above 15hrs seems to have damaged the CNT. The microstructure results showed decrease in the grain size as
CNT % increases as shown in Figure 9 from the value of 127 µm to 53.4 µm. Decrease of density of the nanocomposites resulted from increase in CNT content. An increase in hardness from 31 HV on one side to 68 HV from top to bottom layer of CNT–Al FGL was also reported, hence an improvement in hardness by 129% along the linear direction compared to pure Al.

![Figure 9 FGCM of Al with CNT reinforcement microstructure with different layers.][37]

Kwon et al. also presented another study for Al matrix FGM, reinforced by dual nano particulate of CNT and nanoSiC and manufactured using HEBM and spark plasma sintering [67]. Nano-SiC (nSiC) particle was added as a mixer during milling process to improve CNT dispersion in Al powders and to increase strength due to presence of fine particle [68]. Figure 10 shows a digital image of the dual-nanoparticulate reinforced FGM various layer compositions of pure Al, Al-10%CNT, Al-10%CNT-30%SiC, Al-30%CNT-10%SiC.

![Figure 10. Functionally graded dual-nano reinforced aluminum.][67]

Samples were analyzed based on microstructure and micro hardness properties. Results show that layers were tightly bonded with no serious pores or micro cracks as shown in Figure 11. Some nano Al₄C₃ particles were observed in the CNT layer and embedded across the Al matrix, but it could have further enhanced strength. The
Vickers hardness showed different values depending on composition with a maximum hardness value four times greater than pure Al when a high amount of nSiC was added to the sample. This study suggests that the FG multilayer concept is successful and not only applicable on dual nano particulate-reinforced Al matrix nanocomposites but could also be deployed in polymer and ceramic systems.

A recent investigation was conducted by O. Carvalho et al. on AlSi-CNT to come out with an approach to produce FGMs by powder metallurgy (PM) that can fulfill the requirements of engine compression piston rings [32]. The optimum CNT content to be added in each layer of the FGMs for enhanced fatigue and mechanical behavior was studied. Mechanical properties (tensile, fatigue and wear) of FGMs with CNT content (0 wt.% up to 6 wt.%) were compared to homogeneous AlSi–CNT composites. The 2 wt.% CNT-AlSi was selected for the intended FGM creating a gradient of 2%CNT decreasing to 0%CNT providing the optimum combination of mechanical as well as wear properties. The results of AlSi–CNT FGM as shown in Figure 12 show improvement in properties when compared to the base alloy whereas properties of FGM fall in between those of base alloy and 2 wt.% CNT homogenous composite. As reported by the author, a greater CNT content would have enhanced wear properties however decreasing mechanical properties. Finally, it was proposed that the 2% CNTs of the FGM developed be positioned at the surface of the piston ring since these are the locations experiencing most of the mechanical loads and wear.

**Figure 11** FE-SEM micrograph of the interfaces of functionally graded multilayers with various compositions.[67]
2.4. Summary of Literature Review

1. According to literature, FGMs have many superior advantages promising a great potential for applications in diverse areas especially applications requiring special combinations of properties or severe conditions.

2. FGMs’ experimental and theoretical investigations are very critical and require more in depth attention to discover unrevealed yet possible properties for practical applications at reasonable costs.

3. Specifically in the field of nano FGMs, although many researches using modeling have been carried out, little experimental work has been conducted to produce and comprehensively test nano FGMs. Furthermore, concerning the particular field of our research of Al-CNT FGMs, the current experimental data is still very much limited.

4. The available experimental studies of Al-CNT FGMs also focused on linear variations of materials in layered structured specimens with none to the best of the author’s knowledge performed on cylindrical FGM specimens with coaxial variation of materials or compositions.

5. Also very few experimental studies in the field of nano FGMs and in particular for Al-CNT FGMs investigated the mechanical performance as in tension and compression behaviors with most of the available studies investigating hardness using Vickers hardness and microstructure. In addition, the majority of experimental researches measured tension and hardness properties of the individual layers not of the FGM specimen as one component.
CHAPTER THREE

MATERIALS AND EXPERIMENTAL PROCEDURE

3.1. Materials

Pure Aluminum powder that is gas atomized was used in this research as well as Elicarb® CNTs, to produce composite powders.

Al powder with purity >99.7 % and a density of nearly 2700 kg/m³ was provided by Aluminum Powder Company Limited ALPOCO©. An average particle size of 75μm was reported by the supplier and observed by SEM images. Particle size and morphologies of as received Al powder are shown in SEM images (Figure 13).

![Figure 13 SEM of the as received aluminum powder (a, b).](image)

Carbon nanotubes supplied by THOMAS SWAN© as dry powder agglomerates are shown in Figure 14. CVD process was used to produce CNTs providing purities between 70-90%.

![Figure 14 SEM at Low Magnification showing CNT Agglomeration](image)
Nanotubes diameters reported by the supplier are of an average of 10-12 nm with length up to tens of microns. Range of stated relative density is 1.7-1.9 g/cm³. Some minor Iron and Ash impurities exist. Images in Figure 15 (a, b) show SEM of the as received CNTs indicating high degree of entanglements and some structural imperfections.

![Figure 15 SEM images of CNTs](image1.png)

**Figure 15 SEM images of CNTs a) Moderate Entanglement b) Some Structural Imperfections**

TEM images in Figure 16 showing coaxial CNT illustrating diameters and multi walls of individual CNTs.

![Figure 16 High resolution TEM images of CNTs](image2.png)

**Figure 16 High resolution TEM images of CNTs (a,b)**

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3.2. Experimental Procedure

3.2.1. Overview

This research is an extension of earlier studies conducted by the research group in the field of Al-CNT nano composites with an objective to reach a compromise between strength and ductility using light weight Al metal and CNT as the reinforcement [7, 10, 21, 69, 70]. However, in this study, focus is mainly on fabrication, characterization and testing of FGMs of cylindrical co-axial geometry with different combinations of Al-CNT nano composites, milled and pure Al. Efforts in this research have not been to re-investigate parameters for Al-CNT composites production but rather directed to a novel experimental technique to produce FGM specimens with cylindrical geometry as opposed to the commonly fabricated layered FGM specimens. In this study, powder metallurgy route is used. The same process parameters and conditions that proved in previous studies to be efficient in producing fine Al-CNT powders with the CNT being well dispersed and with minimal damage to the CNTs were used in this study.

Planetary ball mill of 400 rpm speed and ball to powder ratio (BPR) of 5:1 were used [10]. 1 wt. %, 2 wt. %, and 5 wt. % CNTs – Al composites in addition to milled Al were produced. Pure Al samples were also milled using the same milling parameters. Characterization and testing methods used include SEM, optical microscopy, EBSD, density calculation and mechanical testing of powders, FGM specimens and fracture surface of FGMs. Mechanical testing included compression, tension and hardness tests.

The below experimental work is carried out for two combinations of co-axially graded cylinders; FG2% with maximum CNT content of 2% wt. at the outer layer and with the four layers arranged as follows (2% wt CNT-Al, 1% CNT-Al, Milled Al, Pure Al) and FG5% with maximum CNT content of 5% wt. at the outer layer and with the four layers arranged as follows (5% CNT-Al, 2%-CNT-Al, Milled Al, Pure Al). Fabrication of three similar samples was carried out to take the average results.

General experimental procedure included conventional powder metallurgy routes as shown in Figure 17; powder mixing using High Energy Planetary Ball Milling (HEBM), powder loading, powder compaction, sintering, hot extrusion, machining and finally testing and characterization.
3.2.2. Sample Description

The FGM samples prepared for this project are cylindrical specimens with FGM layers stacked in a coaxial manner as shown in the schematic representation of the cross-sectional view of the sample in Figure 18; where notations of 1, 2, 3 and 4 refer to the four layers of the FGM specimens containing the different materials. As previously mentioned two combinations of FGMs were chosen to be fabricated; having the larger content of CNT reinforcement at the outer layer and gradually decreasing the CNT percentage to reach pure Al at the core. Pure Al was chosen as the core material to provide overall ductility to the material. Milled Al was also used to act as an intermediate region between the very ductile pure Al and the consecutive less ductile layers which contain CNTs. This gradient was selected to serve as a hard coating for applications that require more hardness at the surface while having a compromise of high strength and ductility; however the combination of layers can be adjusted depending on the functional requirement of the intended application. Other researchers have used the simple layered sandwich-like approach to FGMs, however the co-axial design implemented in the current study necessitated the design and fabrication of a special mold machined with separate slots for each layer to be compacted and formed producing the coaxial FGM Al-CNT specimen.
3.2.3. Powder Dispersion

The first step in this process is powder weighing followed by mechanical milling using the high energy planetary ball mill to synthesize different concentrations of Al-CNT composites. Reference to literature and to previous researches [7, 9, 10, 59] was made to use and control the milling process parameters including Process Control Agent (PCA) amount, speed, time and BPR. In addition, handling of powders was done in a very careful manner during the process.

3.2.3.1 Powder Weighing

The as received elemental powders of Al and CNTs were accurately weighed and loaded into stainless steel jars having a capacity of 250 ml as shown in Figure 19. PCA was added in varying amounts to ensure that both processes of cold welding and re-fracturing will occur while also ensuring that powder does not stick to the jars’ walls or balls. The PCA amount decreased with increase in CNT content as CNT alone acts as a lubricant that doesn’t readily allow cold welding process during milling. The powders and PCA were loaded according to the final required composite composition of each layer as shown in Table 4 below.
Table 4 Powder Composition

<table>
<thead>
<tr>
<th>Concentration of CNT by Weight</th>
<th>CNT (grams)</th>
<th>Al (grams)</th>
<th>PCA (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5%</td>
<td>1.48</td>
<td>28.06</td>
<td>30</td>
</tr>
<tr>
<td>2%</td>
<td>0.59</td>
<td>28.95</td>
<td>170</td>
</tr>
<tr>
<td>1%</td>
<td>0.30</td>
<td>29.24</td>
<td>180</td>
</tr>
<tr>
<td>0% (Milled Al)</td>
<td>-</td>
<td>29.54</td>
<td>270</td>
</tr>
</tbody>
</table>

Figure 19  a) Materials and handling tools used, b) Milling jar with milling balls

Powder handling performed under Argon gas in a LABCONCO® glove box as shown in Figure 20, hence providing a clean safe environment for operation. Argon, an inert gas, prevents Al powders from quickly reacting with the air and forming an oxide layer. It also prevents the powders from bursting into flames when they are removed after milling, the latter being a high energy process that shall be described in the coming sections. Attention was given to the cleanliness and dryness of the glove box chamber, milling jars and the handling tools.

Figure 20  LABCONCO® Glove Box
3.2.3.2 High Energy Ball Milling

High energy ball milling (HEBM) which is often referred to as mechanical milling is a solid state powder processing method in which two processes are happening simultaneously; cold welding and fracturing. Retsch® PM400 MA-type high energy planetary ball mill, was used as shown in Figure 21. According to literature and previous studies, HEBM proved effective in dispersing the CNTs homogeneously within the Al matrix; this will also be verified by the characterization of the milled powders presented in the Chapter Four. The planetary ball mill consists of a main sun gear that rotates in one direction, while the containers placed in the docks, acting as the planets, rotate along the sun gear and at the same time in an opposite direction along their own axes (Figure 22).
A stainless steel, special alloy milling jar is used to contain the powders. There are 4 milling jars, where 42 balls having identical diameters of 10 mm, and an overall mass of 147.69 grams are placed in each jar. After being carefully weighed as stated in the previous step, the Al and CNT powders are loaded in the stainless steel jars together with the milling balls, then the jars are sealed and fixed into position with a clamp as shown in Figure 23.

Ball milling is a high energy process, so to avoid having the powders bursting into flames or overheating of the milling jars, the machine is automatically set to process for 10 min, then to remain inactive for twice the period, i.e. 20 min. So, for a total one hour of milling, the machine will work 3 hours; 1 hour milling and 2 hours break. For this study, the ball mill operated at a speed of 400 rpm for 1 effective hour with a BPR of 5:1 at room temperature and high purity ethanol as PCA. These operating parameters were sustained during all the experiments. After the process of milling, the steel containers are taken again to the glove box. Powders are removed from the stainless-steel containers and stored separately, inside air-tight glass jars.

Prior to each milling run the milling jars and balls are cleaned and dried. Premilling cleaning and drying of milling jars and balls are carried out to remove any residual particles that could have remained from a previous run. The residuals are removed using a concentrated solution of sodium hydroxide and inserting the jars in the ball mill for 10 minutes at 300 rpm speed. Milling jars and balls are then removed from the ball mill and washed using soap and water, and then all components dried.

Three homogenous Al-CNT composite powders were produced in addition to milled Al. These concentrations were 1wt.% CNT-Al, 2wt.%CNT-Al, and 5wt.%CNT-Al. Powders from each concentration were produced using the same set of parameters during milling to avoid any variables in fabrication of several replicates of the sample.
3.2.4. Production of Functionally Graded Co-axial Cylinders

In this study, one of the main challenges was to manufacture the cylindrical co-axial graded structure. As mentioned in the Literature Review, many theoretical researches were conducted on nano FGMs, some experimental efforts have been carried out on nano FGMs or more specifically on CNT-Al FGMs using powder metallurgy but in layered graded structure and up to the time of this research, none have used PM to fabricate Al-CNT multi layered coaxial functionally graded cylinders.

3.2.4.1. Powder Loading

Two attempts were made to manufacture a mold/die that would allow powder loading in co-axial graded manner, taking into consideration the importance of dimensional accuracy, powder compactness in each layer and proper bonding at the interfaces.

Mold A: Tool Steel Coaxial-Multi Layered Mold/Die

The die is manufactured of tool steel Bohler-K245 as it is easily machinable to allow production of all the required components with the given dimensions and tolerances and can withstand high pressures and temperatures during compaction and sintering. The die tooling drawing (Figure 24) provides details of all parts, components, and assemblies machined to produce the FGM cylindrical coaxial specimens.

![Figure 24 FGM Mold A Tooling Drawings]
Mold A consists of 16 parts, including the base block, cores, punchers, gaskets, washers and stopper. All these parts fit together to create the 5 assemblies shown in Figure 24. The mold assemblies are designed to allow powder compaction of the 4 consecutive layers of the FGM specimen, producing a green compact of FGM of diameter 32mm and height 30mm.

This mold was used to produce FG2% composition. The components of Assembly1- Figure 25 (outer diameter 32mm, inner diameter 24mm) were arranged to stack the outer layer of the 2%wt CNT-Al composite milled powder. A Ø24mm core (part 6) is fixed in the middle to the lower end block and gasket1 (parts 2,15) to allow loading of the outer layer in the clearance between the core (part 6) and the walls of the base block (part1). The powder with specific calculated mass is carefully added in the clearance using a funnel to guide the powder into the designated space. Punch1 (part12) is placed covering the core and the powder layer to keep the powder in place. Slight axial force using the press is applied on Assembly1 for a few minutes to keep the layer in place. A section view for Assembly1 is shown in Figure 25 for enhanced visualization.

![Figure 25 Section View of Assembly 1 of FGM Mold A](image-url)
Punch1 and the Ø24mm core (parts 6,12) are ejected leaving the outer powder layer in place. We placed washer 32-24 (part 9) on top of the first layer to enable loading the following layer of powder. The above procedure is repeated for each layer replacing the cores, gaskets, washers and punchers to suit the dimensions of the powder layer being stacked until the final core of pure Al is reached; as shown in Assemblies (2-4). The die as in Assembly 5 is now ready for the compaction process. Section views for Assembly 2,3 are shown in Figure 26, Figure 27.

Prior to powder loading all components of the die shown in Figure 28 were cleaned very well using Isopropyl Alcohol (IPA) and dried completely. Following that, all parts had to be thoroughly lubricated by high temperature graphite grease to allow easy ejection of the parts and avoid any damages to the previously formed layers.
Figure 29, shows digital images of some of the steps (steps 1-4) performed during powder loading of the FGM layers in Mold A.

As observed the process involved multiple steps starting from assembly of the die parts, powder loading to ejection of parts to allow for loading the following layers. Although coaxial cylindrical FG2% was successfully produced using mold A, however the problems faced during manufacturing and testing due to presence of the lubricant between the FGM layers and the multiple components of the die, necessitated the design of a new die/mold and hence mold B was developed.
Mold B: 3D Printed Coaxial-Multi Layered Mold

Mold B is an FGM slicer which fits in Aluminum can (Figure 30) manufactured from Aluminum alloy (Al 6061) hollow bars.

![3D Printed Slicer Mold in Aluminum Can](image)

The design of the slicer is shown in the engineering drawing in Figure 31. The slicer was 3D printed on a polymeric material. It is a cylinder of length 80 mm and an outer diameter of 32 mm. The circular cross section is internally sliced into 3 different layers of Ø24mm, Ø16mm and Ø8mm with wedges of 0.5 mm thickness separating them and extending along the vertical axis of the cylinder. Each layer is also divided into 3 slots to provide rigidity to the mold structure and aid in powder loading (Figure 32).

![Engineering Drawing of FGM Slicer Mold](image)
The Aluminum can is a simple cylindrical shape can of 32mm diameter, 62mm height and 0.5mm thickness (Figure 33), with a movable top lid to allow closing the can after loading the powders inside the can.

![Figure 32 3D printed FGM Slicer Mold](image)

![Figure 33 Engineering Drawing for Aluminum Can for Powder Stacking](image)

Each FGM sample is prepared in a new Al can (Figure 34 a, b) and the powders together with the Al can go through the process of consolidation. The very thin Al can is later removed from the extrudate by machining.

![Figure 34 Aluminum Can and Lid; a) Opened prior to powder stacking & b) Closed after powder stacking](image)
Powder loading for the layers of the FGM sample became much easier, cleaner and more efficient using Mold B. Only two parts are used to stack the powders in each layer (slicer and Al can) using a small funnel and a pipette to guide the powder flow into the slots (Figure 35). No lubricant is used between the layers and hence no contamination to the FGM powders. After each composition of the powder is introduced in the designated layer and slots, the slicer is slowly removed leaving the layers of powder in the Al can which is then covered by the upper lid in preparation for the consolidation process. FG2% and FG5% specimens were produced using Mold B.

Figure 35 Powder Stacking in Mold B and Can
3.2.4.2 Powder Mass Calculation

The diameters of the four layers of the co-axial graded cylindrical specimen are shown in Figure 36. Each layer is 4mm in thickness and the below equations are used to calculate the mass of the powder to be loaded in the mold for each layer. Table 5 shows the mass values for each layer which varies according to the volume of each layer and density of the specific material loaded into it.

**Mass Calculation**

\[ m = \rho \times V \quad \text{eq. 1} \]

**Volume Calculation**

\[ V = A \times L \quad \text{eq. 2} \]

**Area Calculation**

\[ A = \frac{\pi (d_o^2 - d_i^2)}{4} \quad \text{eq. 3} \]

(where; \(d_o = \text{outer diameter},\)
\(d_i = \text{inner diameter}\))

*Figure 36 Schematic Drawing of an FGM Specimen*

<table>
<thead>
<tr>
<th>Table 5 FGM Powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>FG2%</td>
</tr>
<tr>
<td>---</td>
</tr>
<tr>
<td><strong>Composition</strong></td>
</tr>
<tr>
<td>Layer 1 (\varnothing=8\text{mm})</td>
</tr>
<tr>
<td>Layer 2 (\varnothing=16\text{mm})</td>
</tr>
<tr>
<td>Layer 3 (\varnothing=24\text{mm})</td>
</tr>
<tr>
<td>Layer 4 (\varnothing=32\text{mm})</td>
</tr>
</tbody>
</table>
3.2.4.3. Powder Compaction

The compaction process was carried out using 100 ton capacity FOSTER® hydraulic press (Figure 37). Parameters used were a pressure of 525MPa for one hour at room temperature (cold compaction). The output is called a green compact. For samples produced by mold A, the main block of the die with the powder loaded in it is directly placed on the press for compaction. For samples produced by mold B, the Al can with the co-axial stacked powder from the previous step is placed inside a hardened tool steel alloy BÖHLER® W302 die 32 mm diameter opening. Figure 38 shows the sample to be compacted which is properly sealed, the die used for compaction with the Ø32 opening, the Ø16mm, the punch and stopper.

Pressure exerted on the die was passed on to the powder to ensure elimination of internal spaces and possible air gaps present. A cylindrical green compact is produced after compaction and is further subjected to sintering and hot extrusion.
3.2.4.4. Sintering

Sintering is the process of powder densification under heat without reaching the melting temperature of the metal. It is an essential step in the PM process where heat reduces the porosity and enhances bonding. The green compact contained in the die is sintered at a temperature of 500°C, for 1 hour using an electric heater. The extrusion die is fixed in the green compact die prior to sintering in preparation for hot extrusion directly after sintering is completed.

3.2.4.5. Hot Compaction

Hot Compaction was used for consolidation of only 2 specimens of FG5% (samples #S22, #S23) in an attempt to enhance bonding between the layers especially for FGMs with high concentrations of CNTs. This route was used to investigate possible improvements in mechanical properties.

In this study, Genesis-WABASH compression molding press machine (Figure 39) was used to perform hot compaction at a temperature of 350°F (176°C) and at a 25 tones force for 1 hour. This testing machine is digitally controlled and can operate either automatically or manually according to the given sets of commands and parameters. Specially machined steel rectangular plates and a circular guide were fixed to uniformly apply pressure and heat during the hot compaction test.
3.2.4.6. Hot Extrusion

Extrusion is carried out to produce constant cross-sections or reduce dimensions of a part under compressive forces as shown in the schematic drawing (Figure 40) [71]. In our research, hot extrusion is also carried out to enhance orientation of carbon nanotubes along direction of extrusion, improve densification of material, in addition to increased work hardening in the extruded FGM. Hot extrusion directly followed the sintering process to utilize the heat used for sintering and avoid another heating cycle. Extrusion was carried out at a 500°C temperature with a reduction ratio of 4:1 in area. The diameter of the part hence decreases from 32 mm to 16 mm.

Following extrusion, the extruded part still attached to the die is left to cool for about 8-10 hours. During the cooling process a thermal insulation is added to avoid quenching of the sample and the die. After cooling, the part is removed from the die and the product is the FGM extrudate with diameter of 16 mm and length of 90 mm as shown in Figure 41.

![Figure 40 Extrusion Schematic [71]](image)

![Figure 41 FGM Extrudate](image)

3.2.5. Annealing

Annealing is a heat treatment process where material is heated to more than its recrystallization temperature, left for some time at an appropriate temperature followed by cooling [72]. The high temperatures at which annealing occurs, serve to accelerate the process of internal stresses relief in the material hence making it more workable and ductile. In our research, annealing was done for only one specimen of FG5% (sample
S24) at a temperature of 500°C for 1 hour, then the specimen was left to cool slowly till the following day. Annealing was used with the higher concentration of CNT in an attempt to release internal stress and obtain better mechanical properties and eliminate the problem of notch sensitivity that faced us during tensile testing of FG5% sample that failed outside the gauge length.

3.2.6. Machining

Machining was carried out on the cylindrical extrudates according to ASTM standards to produce compression test specimens. ASTM standards E9-09 for metallic materials compression testing performed at room temperatures were followed in terms of dimensions and surface finish. However for tension tests, samples were not machined according to ASTM E8-08 for tension testing of metallic materials since this would have removed the outer layer of the Al-CNT reinforced FGM affecting the mechanical properties. However, the standard was followed for testing procedure.

For tension testing, round specimens were machined from the extrudate with 15.7mm in diameter and 40 mm in gauge length (Figure 42). The specimens were machined using a manual center lathe including rough, fine, thread turning and finally finishing. Machined specimens (Figure 43) were labeled and placed in sealed bags until tested. Fabrication of three similar samples was carried out to take the average results.

![Figure 42 Standard Dimensions of Tension Test Specimens](image)

For compression testing, specimens with length to diameter ratio (L/D) of 1.5 were produced as per the compression testing ASTM E9-09 standard. Round specimens
of 15.5 mm diameter and 23.25 mm length were machined from the extrudates (Figure 44). Machining process also included rough, fine and thread turning followed by finishing. Three samples from each composition were machined, labeled and sealed until tested.

![Machined Specimen for Compression Testing](image)

*Figure 44 Machined Specimen for Compression Testing*

For nano indentation, the extrudates were machined to produce cylinders of 16 mm diameter and 15 mm height. Samples were then mounted in a polymeric mold to ensure that the sample would fit easily inside the sample tray of the nano-indentation device. The sample surface is machined using the lathe machine to ensure that the FGM and the sleeve are on the same surface level. The sample surface is then grinded and polished until it reaches a mirror like surface (Figure 45).

![Nano Indentation FGM Sample](image)

*Figure 45 Nano Indentation FGM Sample; (a) Before Preparation, (b) After Preparation*

### 3.3. Testing and Characterization

In this study, tension, compression and hardness testing of the different samples were carried out (Table 6). The microstructural characterization techniques carried out included Optical Microscopy (OM), Scanning Electron Microscope (SEM) as well as Electron Backscatter Diffraction (EBSD). Density measurement was also carried out for the samples of FG2% and FG5%.
Table 6 Mechanical Tests Conducted in the Current Study

<table>
<thead>
<tr>
<th></th>
<th>FG2% Old Mold (with Lubricant)</th>
<th>FG2% New Mold (No Lubricant)</th>
<th>FG5% New Mold (No Lubricant)</th>
<th>FG5% New Mold Hot Compacted</th>
<th>FG5% New Mold Annealed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension</td>
<td>--</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Compression</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Nano Indentation</td>
<td>--</td>
<td>✓</td>
<td>✓</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

3.3.1. Tension Testing

Universal testing machine INSTRON® 3382 (Figure 46) was used to carry out the tension tests at room temperature. The 100KN capacity machine was operated with an extension rate of 0.5 mm/min using the V-Shaped jaws designated for the tension tested specimens. Aluminum foil was wrapped around the specimen shoulders to diminish stresses at shoulder. ASTM E8-08 standard procedure was followed for the test procedure although the tested specimens were not as standard in order to accommodate for the nature and structure of the FGM specimens. Stress-strain curves were computer generated based on the given specimen dimensions and input operating parameters. The ultimate strengths and strains at failure were recorded. Pen marks technique was used to measure extension at break.

*Figure 46 INSTRON® 3382 Universal Testing Machine*
3.3.2. Compression Testing

Compression tests were also carried out by the universal testing machine INSTRON® 3382. At room temperature, the 100 KN capacity machine operated with an extension rate of 0.5 mm/min using the plates designated for the compression tested specimens. Lubricant was applied to the upper and lower plates before the test as shown in Figure 47. Similar to tension test, stress-strain curves were computer generated based on the given specimen dimensions and input operating parameters. The test was stopped when 50% compressive strain was reached or the maximum loading of 95KN whichever happens first.

![Compression Testing](image)

Figure 47 Compression Testing

3.3.3. Nano-Indentation

Nano-indentation test is used to determine the hardness and the modulus of elasticity of the sample. It employs load-displacement measurement technique in which the indenter displacement is measured as the load is exerted as shown in schematic drawing in Figure 48 (a). The extent of penetration into the material is recorded as the load is applied. Figure 48 (b) illustrates a typical load-displacement curve of a nano-indentation test with the vertical axis showing the applied load and the horizontal one showing the displacement [73].
In this study, an MTS® nanoindentor XP device was used to conduct the nanoindentation tests (Figure 49). The tests were conducted using the 3-sided Berkovich diamond indenter and 0.05 nm/sec drift correction. The Basic Hardness and Modulus method was used by setting a maximum force of 500mN with a defined matrix of 176 columns x 3 rows of indentation 100 µm interspaced (Figure 50). For all indentations, force versus displacement records were obtained, nano indentation hardness and modulus recorded. Samples of FG2% and FG5% were tested. The defined matrix covered the radius of the cylindrical specimen in a way to ensure readings were taken at each of the four layers of the graded specimens as well as at the three interfaces between each layer.
3.3.4. Optical Microscopy (OM)

Microstructure examination was carried out using LEICA® optical microscope (OM) shown in Figure 51. The OM is connected to a computer that saves the images captured by the digital camera attached to it. The samples intended for examination are prepared in accordance to standards of preparation of metallographic specimens-ASTM E3-01. Sample preparation process included grinding, polishing and chemical etching. Abrasive grinding papers from rough to fine was used for grinding. Following the grinding process, polishing is carried out using Aluminum oxide solution until the specimens reach a mirror like surface. Etching of the polished specimens is the last step of sample preparation where the polished sample is placed in a 95 ml of distilled water solution containing 2 grams of sodium hydroxide (NaOH) and 4 grams of sodium carbonate (Na₂CO₃) for 1 minute. The etched specimen is then removed, washed using distilled water then left to dry.
3.3.5. Scanning Electron Microscopy (SEM)

For the Scanning Electron Microscope (SEM) to operate, a focused beam of electrons interacts with the atoms at the surface of the sample producing images with high magnifications and providing different information about the topography and composition of the specimen [74]. Secondary electrons (SE) and back-scattered electrons (BSE) are types of SEM signals. For SEM, specimens or at least the surface of the specimens must be electrically conductive to interact with the electric beam. Metal based specimens require little preparation except for cleaning and mounting.

In our research a LEO® Supra55 field emission SEM shown in Figure 52 (a) with a resolution up to 10 nm was used. The specimens were placed inside the SEM specimen chamber and fixed with a carbon tape to conduct the electrons more efficiently. The images were shown on the computer attached to the SEM as shown in Figure 52 (b). Different images were obtained using different magnifications and using the two different detectors Inlens and SE to obtain the required information and complete characterization of the specimens.

SEM was used in this study to examine the characteristics, surface topography and microstructure of the as received elementary powders of pure Al and MWCNT as well as the milled powders of different Al-CNT composites. SEM was also used to examine fracture surface of fractured FGM bulks under tension. The different layers were examined as well as the bonding at the interfaces which was of special interest in this study as we are examining multi layered graded heterogeneous composites.

Figure 52 Scanning Electron Microscope (SEM); a) SEM Chamber, b) Images on attached Computer
3.3.6. Electron Backscatter Diffraction (EBSD)

Electron Backscatter Diffraction is a microstructure characterization technique of crystalline materials [75]. It shows size of grains, grain boundaries, as well as the sample orientation and texture. The data was collected on FEI Nova 600 Nanolab FIB/FEG-SEM, operating at 10 kV and at 23 nA. Hikari XP (EDAX) camera at 6 x 6 binning at a collection speed of 150fps was used to collect the data which was then analysed using OIM version 7.1 (EDAX).

3.3.7. Densitometer

Density of the produced bulks was measured by Mettler Toledo XS205 digital densitometer shown in Figure 53. The operation of the densitometer is based on the Archimedes buoyancy principle with the auxiliary liquid being distilled water. The density of the FGMs produced is compared with theoretical densities to indicate level of samples compaction. The weight of the sample is first recorded in air by the upper scale of densitometer then the sample is inserted in the distilled water where the density is calculated and recorded.

![Densitometer](image-url)

*Figure 53 Densitometer*
CHAPTER FOUR

RESULTS AND DISCUSSION

In this chapter, results related to the produced functionally graded co-axial cylinders are presented and discussed. Testing and characterization of the elementary powders and the FGMs produced by the different molds are presented. The results are presented as shown below:

1. Powder results using SEM showing; particle morphology, CNT dispersion, milling effect and grain size analysis.
2. Density of FGM samples.
3. FG2% produced by mold A (FG2%A) with lubricant applied between the consecutive layers, including compression test results in terms of compressive strength at a specified constant strain and microstructure investigation by SEM. EBSD analysis was also conducted for FG2%A.
4. FG2% produced by the modified 3D printed mold B (FG2%B) with no lubricant at the interface. These included microstructure investigation by optical microscopy, compression tests results in terms of compressive strength, at a specified constant strain, tension tests recording ultimate strengths and strains at failure, nanoindentation results recording hardness and modulus in addition to fractographic investigations by SEM.
5. Effect of increasing the weight percentage of CNT in the FGM layers on the mechanical properties was investigated through results of FG5% produced by mold B (FG5%B). These also included microstructure investigation by optical microscopy, compression tests results in terms of compressive strength at a specified constant strain, tension tests, nanoindentation results of hardness and modulus in addition to SEM investigations.
6. Comparison of the obtained results to pure Al samples and homogeneous composites of equivalent CNT concentration processed by the same technique and parameters as the FGM specimens.
4.1. Powder Characterization

4.1.1. SEM of Elementary Powders

SEM micrographs of as received aluminum, milled aluminum and composite milled powders of 1wt.%CNT-Al, 2wt.%CNT-Al and 5wt.%CNT-Al are collectively shown in Figure 54(a-j). The SEM images are displayed for each composition in both low and high magnifications for ease of comparison.

<table>
<thead>
<tr>
<th></th>
<th>Low Magnification</th>
<th>High Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Al</td>
<td><img src="a" alt="Image" /></td>
<td><img src="b" alt="Image" /></td>
</tr>
<tr>
<td>Milled Al</td>
<td><img src="c" alt="Image" /></td>
<td><img src="d" alt="Image" /></td>
</tr>
<tr>
<td>1% CNT-Al</td>
<td><img src="e" alt="Image" /></td>
<td><img src="f" alt="Image" /></td>
</tr>
</tbody>
</table>
Grains and grain boundaries are clearly observed for pure Al under high magnification as shown in Figure 54(b). The images show that particles of Al powder coarsen as they undergo milling. A clear distinction between pure unmilled and milled Al powder appears as shown in (Figure 54 (a-d)), where during HEBM, particle sizes increase due to cold welding and agglomeration that occurs during the process. Particle sizes are generally uniform in unmilled Al powder as shown in Figure 54 (a), compared to that of milled Al that shows a mix of different particle sizes.

Further coarsening occurs to Al-CNT powders during milling and upon increasing the CNT content as shown in Figure 54 (e-j). SEM analysis showed a mixture of larger agglomerates of bonded particles in addition to a number of smaller not yet agglomerated particles. The agglomerates of milled Al-CNT composites are observed to have irregular morphologies compared to that of milled aluminum. It is also noted that as CNT content increases, surface roughness also increases as shown in Figure 54 (e,g,i) compared to pure and milled aluminum in Figure 54 (a,c).

Unexpectedly, it is observed that as we increased the concentration of reinforcement to 5wt.% CNT in the Al matrix, the particle sizes did not increase further.
and were observed with sizes close to those of 2\%CNT-Al as shown in Figure 54 (g,i). The increase in agglomeration trend that was seen with addition of 1\% and 2\%CNT concentration and milling would have suggested a further increase in particle sizes for 5wt.\% CNT-Al composition. However, this unchanged particle size with the 5\%CNT content could be attributed to CNT characteristics in acting as a lubricant [76]. Therefore with increased CNT percentages, lubrication effect counteracts the cold welding of particles during milling and hence doesn’t further increase particle size of the composite powder.

SEM examination of milled composite powders verified efficiency of HEBM to produce homogenous composite powders. The milling conditions (time, speed, BPR, PCA amounts) used in this research also proved effective in dispersing the CNTs as CNTs were seen well embedded with no clusters in the Al particles as shown in Figure 54 (f,h,j).

Figure 55 presents an SEM image of well embedded CNTs in a 5\% Al-CNT composite powder. The CNT tips are appearing in 5\%CNT-Al milled powders, indicating the good dispersion of CNTs by ball milling. CNT tip appearing well embedded in 5\%CNT-Al powders.

Figure 55 CNT tip appearing well embedded in 5\%CNT-Al powders
4.2. Density

The theoretical density of the different FGM samples were calculated by the rule of mixtures and volume fraction equation and tabulated in Table 7 together with the measured densities using the densitometer.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Theoretical Density (g/cm³)</th>
<th>Measured Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FG2% B</td>
<td>2.695</td>
<td>2.825</td>
</tr>
<tr>
<td>FG5% B</td>
<td>2.674</td>
<td>2.794</td>
</tr>
<tr>
<td>FG5% B – Hot Compacted</td>
<td>2.674</td>
<td>2.760</td>
</tr>
<tr>
<td>FG5% B - Annealed</td>
<td>2.674</td>
<td>2.736</td>
</tr>
</tbody>
</table>

Whereas; $\rho_{Al} = 2.7\text{ g/cm}^3$, $\rho_{CNT} = 1.8\text{ g/cm}^3$

**Rule of Mixtures**

$$\rho_C = \rho_{Al} \cdot \frac{VF_{Al}}{100} + \rho_{CNT} \cdot \frac{VF_{CNT}}{100}$$  \hspace{1cm} eq. 4

**Volume Fraction**

$$VF_{CNT\%} = \left( \frac{1}{1 + \left( \frac{m_{\text{wt.\% Al}} \cdot \rho_{CNT}}{m_{\text{wt.\% CNT}} \cdot \rho_{Al}} \right)} \right) \cdot 100$$  \hspace{1cm} eq. 5

The measured density of the produced FGMs shows that the compaction process has been successful in producing well compacted powders and bulks with no pores entrapped.

The increase in densification could be attributed to presence of small amount of Alumina [77] formed during the process that increases the actual densities of the FGMs versus theoretical densities.
4.3. FG2% Produced by Mold A (FG2%A)

FG2% cylindrical samples were produced using mold A explained in details in chapter three and denoted by FG2%A in this thesis for ease of reference. Many trials were carried out to produce a well compacted FGM sample using this mold. Initial attempts (Figure 56) were not successful until finally compact FGMs were produced (Figure 57). Figure 56 (a, b) shows a cracked sample inside the mold and after removal. This specimen failed at the initial production stages during powder loading and removal of die cores to allow for loading of consecutive layers. After several trials of adjusting the amount of lubricant between the layers and die cores, applied pressure after loading of each layer and with increased experience in using mold A, FG2%A samples were produced. However, in order to confirm success of this mold as a recommended method for manufacturing co-axial cylindrical FGMs, further tests were carried out including compression test and SEM of microstructure.

![Figure 56 Failed FG2%A Fabrication Attempts a) Sample inside mold, b) Damaged Sample after removal from mold](image1)

![Figure 57 FG2% produced by Mold A](image2)
4.3.1. Compression Test

Stress-Strain curves based on machine measurements, for pure Al, homogenous 1% CNT-Al composite [10] and FG2% A are shown in Figure 58. The 1% CNT-Al sample with homogeneously dispersed CNTs was selected for comparison with the FGM sample because both have approximately the same CNT content. The compression test was set-up to stop at a strain of 50% if the specimen has not fractured prior to reaching this strain value to avoid damaging the testing machine components. All the tested samples represented in the below plot did not fracture at the end of the test. Both the 1% CNT-Al homogenous composite and FGM samples exhibited a significant increase in compressive strength compared to pure unmilled Al. This can be attributed to both milling effect and strengthening effect of CNT. However, the homogeneous 1% CNT-Al composite showed higher compressive strength compared to that of FG2% A. This could be attributed to the different behavior of the layers of FGM due to the different compositions or due to presence of lubrication between the layers that decreases the extent of compactness.

![Stress-Strain Behaviour of FG2% A](image)

_Figure 58 Stress strain plot comparing compressive behaviour of FG2% A to pure Al and 1% CNT-Al._

ASTM E9-89a standard for metallic materials compression testing conducted at room temperature, section 10.1.10, states that compressive strength is reported for materials exhibiting brittle failure at the fracture point. However, “a compressive strength at a specified total strain may be reported for ductile materials. If so report the strain at which the compressive stress was determined.” [78]. Thus as the specimens
tested did not fracture under the compression test, the measured mechanical properties in terms of compressive strengths at 5% strain are graphically presented in Figure 59. Reported records represent averages of three sample replicates for each identified composition. The error bars are shown representing 90% confidence of values of mean compressive strength. At 5% strain, pure Al reported a mean compressive strength of 69.26 MPa and FG2%A reported a value of 186.7MPa exceeding by 169.5% that of pure Al. 1%CNT-Al homogeneous composite reported a mean compressive strength of 232.43 MPa exceeding by 24.45% that of FG2%A.

![Figure 59 A bar chart comparing compressive stress at 5% strain of FG2%A to pure Al and 1%CNT-Al.](image)

Although some FG2%A specimens sustained compressive stress up to end of test without fracture, however several specimens suffered either from fracture (Figure 60-a) or complete delamination of layers (Figure 60-b) in addition to micro cracks appearing in the layers at the interfaces (Figure 60-c). These behaviors appear to have occurred due to presence of lubricant that is applied during the fabrication process between the cores of the die and the layers of the FGM powders to assist in parts ejection.
4.3.2 SEM of FG2%A

An FG2%A specimen which has undergone a compression test is shown in Figure 61 (a). SEM characterization of microstructure of section A-A of the compressed specimen (Figure 61-b) was performed after the section has been prepared and polished.

On a microscopic scale, the SEM image in Figure 62(a), shows the milled Al and pure Al layers of the FG2%A specimen and the interface between the two layers. A major crack appears along the circumferential interface of the two layers with a rough surface that is believed to contain impurities of residuals of the lubricant. Figure 62(b) is a magnification of the framed section in Figure 62(a), showing a clearer capture of the
surface with the major cracks, with an obvious separation of layers at the interface, which is also suggested to be due to the presence of lubricant.

Figure 62 SEM of Compressed FG2% A Specimen a) Low Magnification, b) High Magnification
4.3.3. EBSD

EBSD characterization was carried out for FG2%A sample. The EBSD images are shown in Figure 63 for pure Al (1), milled Al (2), and composite milled powders of 1wt. %CNT-Al (3) and 2wt.%CNT-Al (4). Grains color coding and the corresponding planes and crystallographic information are not the focus of this study, however the point of interest is the effect of milling and addition of CNT on the grain size of the produced FGM.

The images show that grain size decreases by milling and further decreases by adding CNT in 1% and 2% weight content. Percentage of reductions of average grain size and maximum grain size are demonstrated and compared for each composition with pure Al as the baseline in Table 8. A significant reduction in grain size occurred due to milling, and further yet smaller incremental reductions in grain size by addition of CNT. This grain size reduction is expected to increase strength by increasing the barriers to movement of dislocations hence, increased work hardening and increased strength.

It is worth noting that EBSD shows decrease in grain size by milling and addition of CNT; whereas SEM images indicate an increase in particle size by milling and addition of CNT due to agglomeration effect caused by HEBM.

Figure 63 EBSD Images of Grain Size Distribution for each layer FG2%A
As shown in Table 8 the difference in average grain size between 1\%CNT-Al and 2\%CNT-Al layers is small, thus we decided to study effect of addition of higher percentage of CNT. 5\%CNT-Al was chosen to be used in the composition of the other FGM samples that were produced and investigated in this study (FG5\%).

**Table 8 EBSD Grain Size Comparison**

<table>
<thead>
<tr>
<th>Layers</th>
<th>Average Grain size Diameter (microns)</th>
<th>Max Grain size Diameter (microns)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Al</td>
<td>8.23626</td>
<td>20.6054</td>
<td>Baseline</td>
</tr>
<tr>
<td>Milled Al</td>
<td>1.24955</td>
<td>6.60872</td>
<td>84% reduction in avg. grain size</td>
</tr>
<tr>
<td>1% CNT-Al</td>
<td>0.954659</td>
<td>3.41884</td>
<td>88.4% reduction in avg. grain size</td>
</tr>
<tr>
<td>2% CNT-Al</td>
<td>0.737577</td>
<td>2.35984</td>
<td>91% reduction in avg. grain size</td>
</tr>
</tbody>
</table>

**4.3.4. Summary of Results for FG2\%A**

FG2\% co-axial cylindrical bulk was produced using mold A detailed in chapter three. Compression test results and SEM micrographs show the enhancement of strength due to milling and addition of CNT, however the lubricant seems to affect the compactness of the FGM layers causing delamination and entrapment of impurities at the interfaces that is in turn not resulting in further enhancement of mechanical properties. Hence it is concluded that this mold is not efficient in production of intact layered cylindrical FGMs and an improvement to the design was implemented.
4.4. FG2% Produced by Mold B (FG2%B)

As detailed in chapter three, mold B was developed and manufactured using a 3D printing machine in accordance with the specified design and dimensions. FG2% was re-produced using the newly developed mold that primarily eliminates the need of adding lubricant during the process of powder loading in addition to other advantages related to reduced number of parts of the mold, reduced time of the process and operating in clean environment to avoid contamination of the powders during processing. FG2% produced by mold B is denoted by FG2%B in this thesis for ease of reference. Several testing and characterization techniques were carried out to evaluate the effectiveness of this design to produce coaxial cylindrical FGMs.

4.4.1. Optical Microscopy (OM)

Microscopic imaging of the surface of FG2%B is shown in Figure 64 (a-c). The focus is on the interface between each two layers of FG2%B specimen. Interfaces are clearly observed and distinguished by OM for the three interfaces of 2%CNT-Al and 1%CNT-Al layers (Figure 64-a), 1%CNT-Al and milled Al layers (Figure 64-b) and finally milled Al and pure Al layers (Figure 64-c).

At this magnification, all four individual layers appear to be well compacted with different microstructures observed identifying the different compositions of each layer. Moreover, the images reveal the intact bonding between the three interfaces of the different layers. No cracks appear or any sort of delamination. The defined interface in (a) versus the more gradual interface in (b,c) could be due to different levels of powder compactness during processing or loading.

![Optical Microscopic Images of FG2%B](image-url)
4.4.2. Compression Test

FG2%B samples showed bonded layers along the radial direction after compression, with slight barreling similar to that of ductile material under compression. The specimens did not fracture under compression until they reached the maximum machine set-up strain of 50%. No delamination of layers was observed nor cracks initiated at the interfaces as shown in Figure 65. Elimination of the lubricant by using the new mold is believed to have enhanced this bonding to enable the different layers to behave as one bulk.

Stress-Strain curves based on machine measurements, for pure Al, homogenous 1%CNT-Al composite [10] and FG2%B are shown in Figure 66. FG2%B is also compared to the homogenous 1%CNT-Al composite of the same overall CNT content. The tested specimens did not fracture at the end of the test as shown in the plot below. FG2%B exhibited a significant increase in compressive strength compared to pure unmilled Al. It also recorded a higher compressive strength compared to homogeneous 1%CNT-Al composite unlike that recorded for FG2%A.
Figure 66 Stress-strain plot comparing compressive behavior of FG2%B to pure Al and 1%CNT-Al.

The measured mechanical properties in terms of compressive strengths at 5% strain [78] are graphically presented in Figure 67. Reported records represent averages of three sample replicates for each identified composition. The error bars are shown representing 90% confidence of values of mean compressive strength at 5% strain. At 5% strain, pure Al reported a mean compressive strength of 69.26 MPa and FG2%B reported a value of 242.26 MPa exceeding by 250% that of pure Al and exceeding by 4% that of homogeneous 1%CNT-Al which reported a mean compressive strength of 232.43 MPa.

Figure 67 A bar chart comparing the compressive stress at 5% strain of FG2%B, pure Al and 1%CNT-Al.
In order to validate the advantage of using Mold B, stress-strain curves based on machine measurements, of FG2%B compared to FG2%A are presented in Figure 68. FG2%B exhibited an increase in compressive strength compared to FG2%A. This increase could be attributed to the elimination of the lubricant during production as all the other process factors remained unchanged.

![Figure 68 Stress strain plot comparing the compressive behaviour of FG2%A and FG2%B.](image)

At 5% strain, FG2%B reported a value of 242.26 MPa exceeding by 30% that of FG2%A that reported a value of 186.76 MPa as shown in Figure 69.

![Figure 69 A bar chart comparing the compressive stress at 5% strain for FG2% by Molds A and B](image)
4.4.3. Tension Test

Tension tests were conducted to further investigate and record the mechanical behavior of FG2%B. Conducting a tension test on an FGM specimen is very challenging, hence has been rarely conducted by other researchers working in the field of FGMs. The challenge faced to conduct the tension test was the machining of the FGM specimen to the standard dimensions of the tensile specimens. This is because machining the cylindrical FGM specimen to create a reduced section would essentially remove part of the outer layer of the FGM. The removal of part of the outer layer which includes the higher content of CNT reinforcement affects the measured mechanical properties and does not reflect the full mechanical properties of the materials.

To carry out the tensile test, non-standard specimens were used. Many trials were attempted, some of which were to test the specimen without creating a reduced section (Figure 70-a) or machine threaded shoulders (Figure 70-b) to the specimen but these trials concentrated the stresses at the shoulders and the specimens broke at the jaw section. Also a thicker Al can for loading the powder was examined to be able to machine a larger thickness of the specimen that would actually be the Al can and hence not waste the FGM layers in machining. This required using a compaction die with different dimensions, which altered the dimensions of the extrudate and the structure of the FGM during extrusion. It also resulted in inaccurate records of additional ductility as part of the can is believed not to have been fully removed during machining prior to the test, so these results were discarded as they were not a precise record of the behavior of the tested FGM bulk.

Figure 70  Failed Specimens under Tension
It was concluded that the above trials were not the solution to the tension testing requirement and that it was necessary to use the original thickness of the Al can together with creating a reduced section with minimum removal of the outer layer material. Again, many trials were carried out until reaching the optimum minimum possible tolerances for machining the specimens to the dimensions detailed in chapter three and these made it possible to test the FG2%B samples under tension.

FG2%B images of tensile tested specimens, showed typical ductile fracture appearance. Crack initiation and fracture occurred in the middle of the gauge length for all three tested samples, nearly at an inclination of 45° as shown in Figure 71. On a macro scale, images of the fracture surface in Figure 72 shows that layers of FG2%B were still well bonded with no evidence of cracking or delamination.

![Figure 71 FG2%B Specimen during Tension Test](image1)

![Figure 72 Side and Top view of FG2%B fractured surface at tension](image2)
Stress-Strain curves of tensile behavior based on machine measurements, of pure Al, homogenous 1%CNT-Al composite [10] and FG2%B are shown in Figure 73. Both 1%CNT-Al homogeneous and functionally graded composites exhibited a significant increase in ultimate tensile strength compared to pure unmilled Al. However there was a loss in ductility with the increase in strength. This loss in ductility occurred by a higher percentage in the 1%CNT-Al homogenous composite compared to FG2%B as observed in the plot below. The behavior of FG2%B provided a unique compromise of increase in strength while showing a higher percentage of ductility and toughness which is one of the main objectives and advantages of FGMs.

![Stress-Strain Behaviour of FG2%B](image)

*Figure 73 Stress strain plot comparing the tensile behavior of FG2%B to pure Al and 1%CNT-Al.*

Measured properties in terms of average ultimate strengths and elongation are shown in Table 9 and presented graphically in Figure 74 and Figure 75. Reported records represent averages of three sample replicates for each identified composition. The error bars are shown representing 90% confidence of mean strengths and strains values at failure.

*Table 9 Ultimate Strength and Elongation*

<table>
<thead>
<tr>
<th>Composition</th>
<th>Ultimate Strength (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Al</td>
<td>101.2 ± 2.1</td>
<td>35.43 ± 2.03</td>
</tr>
<tr>
<td><strong>FG2%B</strong></td>
<td><strong>209.56 ± 0.89</strong></td>
<td><strong>12.69 ± 1.00</strong></td>
</tr>
<tr>
<td>Homogeneous 1% CNT- Al Composite</td>
<td>189.62 ± 14.59</td>
<td>9.05 ± 1.38</td>
</tr>
</tbody>
</table>

71
As observed from the plots, FG2%B reported 107.1% and 10% increase in ultimate tensile strength compared to pure Al and homogeneous composite 1%CNT-Al respectively.

![Ultimate Tensile Strength of FG2%B](image1)

*Figure 74 A bar chart comparing the Ultimate Tensile Strength of FG2%B to pure Al and 1%CNT-Al*

As for ductility, FG2%B reported 40% increase in elongation percentage compared to 1%CNT-Al homogeneous composite, thus it is concluded that FG2%B has a unique mix of high strength and ductility.

![Elongation % of FG2%B](image2)

*Figure 75 A bar chart comparing the Elongation% of FG2% to, pure Al and 1%CNT-Al.*
4.4.4. NanoIndentation Test

Since this study is conducted on a multilayered co-axial material, therefore it is not accurate to provide a single hardness value representing the whole bulk, and this is where the nano indentation hardness tests come to be of a great use. The nanoindentation tests were carried out to investigate behavior of FG2%B in terms of gradual change in hardness and Young’s modulus along the radius of the sample to capture the behavior of the different layers included in the FGM specimen. It is also conducted to investigate the change in behavior at the interfaces between the different layers.

Typical nanoindentation hardness records of FG2%B are graphically demonstrated in Figure 76. The chart shows four sections of increasing hardness values. Each section corresponds to a different layer and composition of the FGM specimen as illustrated on the chart below. It is clear that strong bonding exists at the interfaces between the different layers indicated by the continuous hardness records on the chart. Gradual increase in hardness is observed between the layers except for the sharp increase between the pure and milled Al due to significant increase in hardness as a result of mechanical milling. A gradual yet clear increase in hardness is observed between layers of 1%CNT-Al and 2%CNT-Al. An increase in hardness is also shown as we get closer to the surface with a value of hardness reaching approximately 1.35 GPa.

![Nanoindentation Hardness of FG2%B](image)

*Figure 76 Nanoindentation Hardness of FG2%B*
Figure 76 also shows enhancement in hardness due to milling alone then further enhancement due to reinforcement by CNT as we reach the outer layer of 2% wt. CNT-Al. On average for FG2%B, hardness values range from 0.42 GPa at the core reaching 1.35 GPa at the outer layer of the FG2%B cylinder, hence exceeding that of pure Al at the core by 220%. This controlled variation in hardness is provided in the same cylinder, hence making it of special interest for applications requiring harder outer layers while maintaining a soft tough core.

The modulus of elasticity records of FG2%B are graphically presented in Figure 77. An increasing trend could be observed along the different layers as we move from the core to the surface. Despite that increase in the recorded modulus with milling and higher CNT concentrations is relatively small, yet there is still a range of increase in the modulus from approximately 71 GPa to 80 GPa which is around 12.7% increase. Also the indentation modulus chart confirms the bonding and compatibility of the different compositions of each layer with one another. This is indicated by the smooth, not step-like transitions and absence of blank areas of readings at any of the sections.
4.4.5. SEM Fracture Surface Investigations

Fractography of FG2%B specimens that failed during tension test was characterized by SEM. Figure 78 shows an image capturing the three bonded layers of FG2%B (2%CNT-Al, 1%CNT-Al and Milled Al) and the interface between them. No cracks, delamination or impurities are observed at this section of the specimen. Each layer could be clearly seen, showing distinctive topographies indicative of its composition. Unlike the FG2%A samples, the layers appear to be well bonded with no apparent defects which conform to nanoindentation results previously discussed.

![Figure 78 SEM Image of three FG2%B layers](image_url)

Figure 78 SEM Image of three FG2%B layers

Figure 79 provides a closer capture of the two outer layers, showing slight difference in surface features due to the difference in the CNT content in both. The red curved line is drawn to illustrate the interface between the two layers.

![Figure 79 SEM of the Outer Layers Interface of FG2%B](image_url)

Figure 79 SEM of the Outer Layers Interface of FG2%B
Ductile fracture features were a common factor in all the FG2%B layers but with different extents. The more we added CNT together with the effect of HEBM, the less ductile the fracture surface of the layers appeared and the more rough, flat and distorted the layers were, indicating different failure mechanisms.

Figure 80 (a), shows a cracked section at fracture, which is detected at the interfacial layer between 1%CNT-Al and milled Al. The crack in Figure 80 (a) is magnified showing the tearing location at the interface in Figure 80 (b). This tearing is believed to have occurred due to the mismatch between the ductility of the milled Al and the more brittle CNT reinforced layer. According to literature, Kieback el al. [30] states that cracking at the interface between the different layers is a potential danger during processing of functionally graded materials. That is because different regions sinter at different rates thus creating incompatibilities between the different layers. The author further explains that such differences in the material could be relieved in different forms, either as distortions, creep or could ultimately end into cracks. The different forms of relief depend on material properties and geometry.

![Figure 80 SEM of interface between 1%CNT-Al and Milled Al](image)

Typical fracture surfaces images of pure and milled Al are shown in Figure 81 and Figure 82 at different magnifications. Differences between pure and milled aluminum fracture surfaces are observed. High ductility of Al is indicated by the deep dimples in Figure 81. Material homogeneity over the core of the specimen is also verified through the uniform size and distribution of these dimples.
The fracture surface of milled aluminum Figure 82 is also very typical of its morphology. Although dimples still exist, however they are finer, shallower and less uniform in size compared to those of the unmilled aluminum.

The images also provide a vivid picture of the successful intact bonding between these two layers. And although these layers have different mechanical properties, yet the bond is very strong which in turn enhances the strengthening of the specimen.

Figure 81 Pure Al and Milled Al Interface- Low Magnification

Figure 82 Pure Al and Milled Al Interface- High Magnification
4.4.6 Summary of Results for FG2%B

FG2%B co-axial cylindrical samples were produced using Mold B detailed in chapter three. The OM images demonstrate the success of the powder metallurgy processes used in producing well bonded co-axial FGM cylinders.

Compression test results show enhancement of compressive strength due to milling and addition of CNT. Tensile tests also show very positive results confirming that the FGM design provided a better trade-off between strength and ductility in comparison to homogeneous composite competitor. SEM analysis also confirms the efficiency of bonding across FG2%B layers. In addition, the results of nanoindentation are of great interest as they demonstrated in a single plot the gradual change in properties due to effect of milling and CNT addition providing a harder outer surface material with a softer core. Hence it is concluded that this newly manufactured mold design is very efficient in production of intact layered cylindrical FGMs.
### 4.4.7 Comparison between Mold A and Mold B

Table 10 provides a concise comparison of the two molds manufactured to produce FG2%.

**Table 10 Comparison between Mold A and Mold B**

<table>
<thead>
<tr>
<th>Mold A</th>
<th>Mold B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lubrication applied between each layer of the FGM</td>
<td>Total elimination of lubricant during powder loading</td>
</tr>
<tr>
<td>Many parts, 16 different sized cores and plungers</td>
<td>Only one part</td>
</tr>
<tr>
<td>Difficult to manufacture with precision</td>
<td>3D printed with exact dimensions</td>
</tr>
<tr>
<td>Heavy weight, not easily handled and stored</td>
<td>Very light weight, easily handled and stored</td>
</tr>
<tr>
<td>Powder loading must be done at the press to apply pressure after loading each layer, need workshop tools to eject stuck parts</td>
<td>Powder loading is done at an uncontaminated atmosphere, no need for press or tools; no parts ejection is involved</td>
</tr>
<tr>
<td>Time taken to stack the powder of FGM sample is too long</td>
<td>Reduced time of producing samples to about one third; time efficient</td>
</tr>
<tr>
<td>Produced FGM specimen, showed lower mechanical properties under compression in addition to layers delamination due to presence of lubricant at the interfaces.</td>
<td>Produced samples of FGMs have more intact layers, withstand testing with higher compression results and no delamination of layers</td>
</tr>
<tr>
<td>SEM images reveal cracks, de-bonding and entrapment of impurities at interfaces.</td>
<td>SEM images reveal intact bonding and no evidence of cracks or delamination at the interfaces.</td>
</tr>
</tbody>
</table>
4.5. FG5% Produced by Mold B (FG5%B)

Following the successful results of FG2% cylinders produced by mold B and the enhanced mechanical properties recorded, a higher content of CNT in the FGM was studied. CNT percentage was increased at the two outer layers in an attempt to study the effect of this increased reinforcement on the mechanical properties of the FG5%. FG5% produced by mold B is denoted by FG5%B in this thesis for ease of reference.

4.5.1. Optical Microscopy

Microscopic imaging of the surface of FG5%B bulk is shown in Figure 83 (a-c). Focus is on the area of interface between each two layers of the FG5% specimen. Interfaces are very clearly observed and distinguished by OM for the three interfaces of 5%CNT-Al and 2%CNT-Al layers (Figure 83-a), 2%CNT-Al and milled Al layers (Figure 83-b) and finally milled Al and pure Al layers (Figure 83-c). At this magnification, all four individual layers appear to be well compacted with different microstructures observed corresponding to the different composition of each layer. Moreover, the images reveal the intact bonding and smooth transition between the three interfaces of the different layers. No cracks or any sort of delamination appear.

The microstructure of 5% CNT-Al layer shows particles with darker color due to increased CNT content compared to 2%CNT-Al and milled Al.

![Figure 83 Optical Microscope Images of FG5%B](image-url)
4.5.2. Compression Test

On a macro scale FG5%B shows bonded layers along the radial direction after compression. Although some cracks started to appear at the outer layer closer to the end of the test as shown in Figure 84, yet the specimens did not fracture under compression until they reached the maximum machine set-up strain of 50%.

![Figure 84 FG5%B Specimen after Compression](image)

Stress-Strain curves derived from machine measurements, of pure Al, homogenous 2%wt. CNT-Al composite [10] and FG5%B are shown in Figure 85. The overall content of CNT in the FG5%B specimens was calculated to be approximately 2%wt., hence FG5%B is compared to the homogenous 2%wt.CNT-Al composite of the same overall content of CNT. FG5%B showed a very good behavior under compression as it exhibited a significant increase in compressive strength compared to pure unmilled Al. It also recorded a much higher compressive yield strength compared to homogenous 2%CNT-Al composite. However closer to the end of the test some cracks started to appear at the outer layer 5%CNT-Al, which explains the slight decrease of the FG5%B curve slope shown in Figure 85.

The measured mechanical properties in terms of compressive strengths at 5% strain [78] are graphically presented in Figure 86. Reported records represent averages of three sample replicates for the identified compositions. The error bars shown are representing 90% confidence of values of mean compressive strength. So, at 5% strain, pure Al reported a mean compressive strength of 69.26MPa and FG5%B reported a value of 304.64MPa exceeding by 340% that of pure Al. Moreover, the mean compressive strength at 5% strain of FG5%B exceeded by 28.5% that of homogenous 2%CNT-Al which reported a value of 236.98MPa.
4.5.3. Tension Test

FG5%B tensile specimens were machined to the dimensions detailed in chapter three similar to those of FG2%B specimens. At this relatively high CNT concentration
of FG5%B layers, tensile testing fractured the material in a nearly brittle manner as shown in Figure 87. The fracture occurred outside the gauge length for all the tested specimens. This could be attributed to the increased brittleness of the 5%CNT-Al outer layer that caused the specimens to be very notch sensitive and any defects in machining could have caused stress concentration and hence fracture at the shoulders.

On a macro scale images of the fracture surface of FG5%B images show that cracking and delamination between the milled Al and 2%CNT-Al layers occurred at some sections of the specimen. These will be further investigated by SEM. However the rest of the interfaces between the other layers remained intact.

Stress-Strain curves of tensile behavior based on machine measurements, for pure Al, 2%CNT-Al homogeneous composite [10] and FG5%B are shown in Figure 88. Homogenous and FGM composites exhibited a significant increase in ultimate tensile strength compared to pure Al reaching about 260MPa. However, the accompanied loss in ductility for FG5% could not be confirmed as the samples fractured outside the gauge length. Hot compaction and annealing of FG5%B samples were also investigated as possible solutions for this behavior of FG5%B under tension.
4.5.4. Nano Indentation Test

Typical nanoindentation hardness records of FG5%B are graphically presented in Figure 89. The chart very clearly shows four distinctive sections of different average hardness values. Each section corresponds to a different layer and composition of the FG5%B specimen as illustrated on the chart below. Figure 89 also shows values of hardness ranging from 0.42GPa at the core reaching approximately 1.6GPa at the outer layer of the FG5%B cylinder, hence exceeding that of pure Al at the core by 281%.

It is also evident that good bonding existed at the interfaces between the different layers, since the nanoindentor was able to measure hardness values at all these interfacial areas, hence the existence of bonded material. Smooth transition is also observed between the milled Al and 2%CNT-Al layers. However a sharper transition appears between the pure and milled Al layers due to significant increase in the hardness due to milling, as reported earlier for FG2%B samples. Another stepwise transition occurred between the 2%CNT-Al and 5%CNT-Al layers, and this could be attributed to the exponential CNT hardening effect as we move from 2%CNT directly to 5%CNT reinforcement content. The sharp stepwise increase in hardness between some layers could be the cause of internal stresses developing in the bulk and affecting the mechanical properties during testing especially as noted during tension test.
The modulus of elasticity records of FG5%B are graphically presented in Figure 90. An increasing trend could be observed along the different layers as we move from the core to the surface. Variation in modulus with milling and increased concentration of reinforcement is also relatively small similar to the FG2%B variation, yet there is still a range of increase in modulus from approximately 65GPa to 82GPa in the same specimen. Also the indentation modulus chart confirms the bonding of the different layers with one another.
4.5.5. SEM Fracture Surface Investigations

Fractography of FG5%B specimens that failed during tension test was characterized by SEM and qualitatively analyzed. Layers of 5%CNT-Al and 2%CNT-Al and layers of pure and milled Al are seen in Figure 91 (a,b) respectively with indication to the interfaces between the layers as well. No cracks, delamination or impurities are observed at these sections of the specimen. Each layer could be clearly seen, showing distinctive topographies indicative of its composition. The 5%CNT-Al layer showed a less ductile fracture surface and a rough, flat structure was observed.

![Figure 91 SEM of the FG5%B layers and the interfaces](image)

On the other hand, Figure 92 shows a cracked section at fracture, which is seen at the interfacial region between 2% CNT-Al and milled Al. Cracking also, appeared in the fracture surface of FG2%B between 1%CNT-Al and milled Al layers. This confirms that the mismatch between the milled Al and the more brittle CNT reinforced layer could be a source of internal stresses creating cracks and ultimately fracture. The observation reported earlier by Kieback et al. [30] related to different sintering rates of FGM layers and the associated stresses created is also applicable in this case.
4.5.6. FG5%B - Hot Compacted

Hot compaction was carried out on FG5%B specimen in an attempt to find a solution to eliminate the stress concentration causing fracture under tension at the shoulders. The hot compaction process and parameters are detailed in chapter three.

4.5.6.1 Optical Microscopy

Microscopic imaging of the cross section of FG5%B specimen produced by hot compaction is shown in Figure 93 (a-c). 5%CNT-Al and 2%CNT-Al layers (Figure 93 a), were not clearly distinguishable under the microscope. The other layers were clearly distinguished by OM with different microstructures observed corresponding to the different compositions of each layer. Moreover, at this magnification, the intact bonding and smooth transition between the three interfaces of the different layers was observed with no cracks appearing or any sort of delamination.
4.5.6.2 Tension Test

The hot compacted FG5%B specimen fractured in a nearly brittle manner as shown in Figure 94. The fracture occurred outside the gauge length as well. On a macro scale the fracture surface of FG5%B image shows that cracking and delamination occurred at the same location; that is between the milled Al and 2%CNT-Al layers. However the rest of the interfaces between the other layers remained intact.

![Figure 94 Fractured Hot Compacted FG5%B Tensile Specimen](image)

Stress-Strain curves of tensile behavior based on machine measurements, for pure Al, 2%CNT-Al homogeneous composite [10] and hot compacted FG5%B are shown in Figure 95. Although, FG5%B exhibited a significant increase in ultimate tensile strength compared to pure Al reaching about 245MPa, however, it exhibited slightly lower strength compared to its homogeneous composite competitor. Hot compaction didn’t cause any improvement in terms of tensile properties compared to FG5%B produced by cold compaction.

![Figure 95 Stress strain plot comparing the tensile behaviour of hot compacted FG5%B to pure Al and 2%CNT-Al](image)
4.4.6.3. SEM Fracture Surface Investigations

The hot compacted FG5%B specimen was characterized by SEM and qualitatively analyzed. Figure 96 shows a collective capture of three layers of FG5%B specimen (5%CNT-Al, 2%CNT-Al and Milled Al) and the interface between them. Each layer could be clearly seen, showing distinctive topographies indicative of its composition. Figure 96 (a), shows a section of the specimen where no cracks, delamination or impurities are observed between these three layers. Figure 96 (b) on the other hand, shows another section capturing the same layers but with a major crack between the milled Al and 2%CNT-Al layer.

![Figure 96 SEM Image of three Hot Compacted FG5%B Layers a) Bonded Layers, b) One Cracked Layer](image)

It can be concluded that no more investigations should be done using hot compaction on the FG5%B as it didn’t seem to provide a relief of the internal stresses that were causing the development of cracks and ultimately premature failure of the specimens. Another route was sought, which is annealing after extrusion explained in the coming section.

4.5.7 Annealed FG5%B

Annealing was carried out for FG5%B specimen after extrusion, in order to release the internal stresses that might have been developed during extrusion and sintering. The parameters used for annealing are detailed in chapter three.

4.5.7.1 Tension Test

Tension test was carried out using the same sample dimensions used for all the previous specimens. Fracture occurred inside the gauge length for the annealed specimen, in a mixed brittle/ductile mechanism as shown in Figure 97.
On a macro scale the fracture surface of annealed FG5%B in Figure 98 shows that cracking and delamination occurred again at the same location; between the milled Al and 2%CNT-Al layers. However the rest of the interfaces between the other layers remained intact.

Figure 98 Side and Top view of Annealed FG5%B Fractured Surface at Tension

Stress-Strain curves of tensile behavior based on machine measurements, for pure Al, 2%CNT-Al homogeneous composite [10] and annealed FG5%B are shown in Figure 99. Although, FG5%B exhibited a significant increase in ultimate tensile strength compared to pure Al reaching about 250MPa, however, it still showed lower values compared to its homogeneous competitor for both strength and ductility.
A comparison of stress-strain records of tensile behavior of FG5%B samples produced by different conditions versus pure Al is shown in Figure 100. The representative figure below shows a comparison between FG5%B samples compacted at room temperature, hot compacted and annealed.

The added value provided by annealing for FG5%B is that the specimen fractured inside the gauge length not at the shoulders as in the previous FG5%B specimens. However, since only one specimen was examined, more samples need to be tested in future work to confirm whether this behavior was due to annealing or due to improved experience in machining of the specimens to the desired tolerances.
As expected the strength results of the annealed FG5%B went down due to annealing, however unexpectedly, the ductility was not improved either. During annealing a more equilibrium state is believed to be created hence minimizing the residual stresses and ultimately allowing the tensile fracture to happen in the middle of the specimen. However, since only one sample was tested results are not conclusive and further samples and tests should be conducted.

4.5.8 Comparison of Results for FG2%B vs. FG5%B

Table 11 shows a summary of comparison between behaviours of FG2%B and FG5%B in terms of OM, compression testing, nanoindentation hardness and modulus results.

*Table 11 FG2%B vs. FG5%B*

<table>
<thead>
<tr>
<th></th>
<th>FG2%B</th>
<th>FG5%B</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Optical Microscopy</strong></td>
<td>At magnification used by OM, layers appeared well bonded, no cracks or delamination.</td>
<td>At magnification used by OM, layers appeared well bonded, no cracks or delamination.</td>
</tr>
<tr>
<td><strong>Compression Testing</strong></td>
<td><em>Mean compressive strength increased compared to:</em></td>
<td><em>Mean compressive strength increased compared to:</em></td>
</tr>
<tr>
<td>(at 5% strain)</td>
<td>• Al by 250%</td>
<td>• Al by 340%</td>
</tr>
<tr>
<td></td>
<td>• Homogeneous composite by 4%</td>
<td>• Homogeneous composite by 28%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• FG2%B by 25%</td>
</tr>
<tr>
<td><strong>Nanoindentation</strong></td>
<td>Ranged from 0.42GPa-1.35GPa</td>
<td>Ranged from 0.42GPa-1.6GPa</td>
</tr>
<tr>
<td><strong>Hardness</strong></td>
<td>Surface values higher than core by 221%</td>
<td>Surface values higher than core by 281%</td>
</tr>
<tr>
<td><strong>Nanoindentation</strong></td>
<td>Ranged from 71GPa-80GPa</td>
<td>Ranged from 67GPa-82GPa</td>
</tr>
<tr>
<td><strong>Modulus</strong></td>
<td>Surface values higher than core by 12.7%</td>
<td>Surface values higher than core by 22%</td>
</tr>
</tbody>
</table>
4.5.9 Summary of Results for FG5%B

FG5%B was manufactured to investigate the effect of increasing carbon nanotubes content on FGM behavior and mechanical properties. FG5% co-axial cylindrical samples were produced using mold B detailed in chapter three. The OM images demonstrate the success of the powder metallurgy process used for producing well bonded co-axial FGM cylinders with up to 5wt% CNT.

Compression test results show significant enhancement of compressive strength due to milling and addition of CNT up to 5% at the outer layer compared to pure Al and to the homogeneous competitor. This was further confirmed by the nanoindentation test results providing an increasing trend of hardness and modulus of elasticity properties as we move from the core to the surface of the specimen. So for applications requiring high compressive strengths and increased hardness, FG5%B would be very desirable.

Tensile tests were the real challenge for FG5%B due to fracture occurring outside the gauge length, hence not recording the full potential of the mechanical properties of the material. Hot compaction was conducted and same problems were faced during testing, so it was not further investigated. Annealing was also carried out, and although it resulted in fracture of the specimen inside the gauge length, yet it didn’t provide enhanced ductility in comparison to its competitors. Therefore annealing was not further investigated in our study but could be examined in future studies with other combinations and compositions of FGM layers.

It is also believed that the problem faced with tensile specimens could be related to machining as specimens become more notch sensitive with the increase in brittle phase of 5%CNT-Al. The incompatibilities between layers due to increased CNT content to 5% and the sudden jump from 2% to 5% could have also created internal stresses.

In summary, FG5%B shows significant enhancement of compressive strength and hardness, however tensile properties need further investigation.
CHAPTER FIVE

CONCLUSIONS

1. The larger content of CNT reinforcement was selectively used at the outer layers of the FGM cylinder to provide the highest strength and hardness at the surface subjected to higher stresses and more severe environments. Pure Al was chosen as the core material since it is subjected to lower stresses and to provide overall ductility to the material.

2. Specifically designed and manufactured mold A is not effective in producing Al-CNT intact layered functionally graded co-axial cylinders. The lubricant applied between the graded layers during processing is believed to have affected the compactness of layers causing delamination with no further enhancement of compressive strength compared to the homogeneous composite competitor. An improvement to the design was implemented.

3. Newly designed 3D printed - mold B, is very efficient in the production of intact Al-CNT co-axial cylindrical specimens.

4. Powder metallurgy using HEBM in addition to the novel mold B; are recommended as a technique for fabrication of nano metal matrix functionally graded co-axial cylinders. This technique offers high precision, very good compaction of layers, flexibility in deciding the material or composition of composite to be loaded in each layer according to required functional properties and application in addition to very good surface finish of the end product.

5. FG2%B (2%CNT-Al, 1%CNT-Al, Milled Al, Pure Al using mold B) reported a mean compressive strength exceeding by 250% that of pure Al and exceeding by 4% that of 1%CNT-Al homogeneous composite at 5% strain.

6. FG2%B reported a mean compressive strength exceeding by 30% that of FG2%A (2%CNT-Al, 1%CNT-Al, Milled Al, Pure Al using mold A) at 5% strain, indicating the enhancement due to elimination of lubricant using the new mold.

7. FG2%B reported 107.1% increase in ultimate tensile strength compared to pure Al and 10% increase compared to 1%CNT-Al homogeneous composite.
8. FG2%B reported 40% increase in elongation percentage compared to 1%CNT-Al homogeneous composite.

9. On average for FG2%B, nanohardness values ranged from 0.42GPa at the core reaching 1.35GPa at the outer layer of the FG2%B cylinder, hence exceeding that of pure Al at the core by 221%.

10. On average for FG2%B, modulus of elasticity values ranged from approximately 71GPa at the core reaching 80GPa at the outer layer which is around 12.7% increase.

11. It is concluded that FG2%B has a very unique mix of high strength and ductility in addition to a gradual increase of hardness and modulus of elasticity values from the core to the outer layer containing the maximum CNT content.

12. FG5%B (5%CNT-Al, 2%CNT-Al, Milled Al, Pure Al using mold B) reported a mean compressive strength values exceeding by 340% that of pure Al and by 28.5% that of homogenous 2%CNT-Al at 5% strain.

13. FG5%B tensile tests were not successful due to fracture occurring outside the gauge length, hence not recording the full potential of the mechanical properties of the material.

14. FG5%B produced by hot compaction faced the same problems of fracture outside the gauge length during tensile testing, so it was not further investigated.

15. Tensile testing for annealed FG5%B resulted in fracture of the specimen inside the gauge length, yet it didn’t provide the expected enhanced ductility in comparison to its competitors. Annealing was not further investigated in our study but could be examined in future studies.

16. Problems faced in tensile testing in FG5%B samples could be related to one or more of these reasons; machining, increase in notch sensitivity with the increase in brittle phase of 5%CNT-Al, the incompatibilities created between layers due to increased CNT content and internal stresses due to sudden jump from 2% to 5% CNT content.
17. On average for FG5%B, hardness values ranged from 0.42GPa at the core reaching 1.6GPa at the outer layer of the FG5%B cylinder, hence exceeding that of pure Al at the core by 281%.

18. On average for FG5%B, modulus of elasticity values ranged from approximately 67GPa at the core reaching 82GPa at the outer layer which is around 22% increase.

19. EBSD shows decrease in grain size by milling and addition of CNT; whereas SEM shows increase in particle size by milling and addition of CNT due to agglomeration effect caused by HEBM.

20. Powder metallurgy including compaction and extrusion proved effective in consolidation of Al-CNT composites to above 99% densification.

21. The produced FG2%B cylindrical component could be a solution to the challenges of drive shaft designs discussed in literature review, section 2.2.5.1. Having higher percentage of CNT reinforcement at the surface and graded to reach pure Al at the core will combine the benefits of both the solid and hollow shaft designs as well as allow replacing two-piece drive shafts with one nano FGM piece shaft. This is because FG2%B will provide the required outer strength, overall toughness, and overall weight reduction while maintaining a solid shaft design for achieving maximum torque.
RECOMMENDATION FOR FUTURE WORK

- Further investigation of the interfacial regions between different layers.
- Devise ways to make the milled Al and CNT reinforced transition more gradual.
- Investigate the Al-CNT FG2%B behavior under torsion, fatigue as well as its corrosion resistance and wear behavior.
- Study heat treatment methods to improve ductility and performance with higher CNT percentages.
- Perform a cost-benefit analysis of using FG2%B as an alternate material for drive shafts of mobility applications.
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