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Binder Jet Additive Manufacturing of Stainless Steel-Hydroxyapatite Bio-composite

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By

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Abstract

Title: Binder Jet Additive Manufacturing of Stainless Steel-Hydroxyapatite Bio-composite

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This study was conducted to create a bio-composite with a porous structure using the binder-jet printing additive manufacturing process. The samples were printed by changing the parameters such as layer thickness, roller speed, sintering time and sintering temperature of the X1-Lab binder jet additive manufacturing printer. The samples were printed 80% stainless steel 316 mixed with 20% Calcium Phosphate Tribasic by volume. Eight experiments were attempted to print according to the fractional factorial design of experiment. The effects of changing parameters on the mechanical properties of the new bio-composite was tested using ASTM E-09 compressive strength standards. The compression testing was conducted by using MTS 810 material testing system following a modified version of the ASTM E9 standards for testing metallic material in room temperature. The samples were scanned using an electron microscope to understand the porous structure of the bio-composite.

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Research Description and background

Identification of the research need

The current United State implantable medical device industry is estimated to be worth around $35 billion. Currently about 500,000 knees and 300,000 hip implant surgeries are performed in United State and about 1 million cardiac stents are used each year. United States demand for implantable medical devices will increase 8.3 % annually till 2020. Hence the implantable market is estimated to be about $100 billion in 2020. Improvements will be driven by the development of next generation devices based on new technologies and improved materials. Spinal implants, cardiac stents and orthopedic implants (knee, hip etc.) will be among the fastest growing product categories (Davis, 2003).

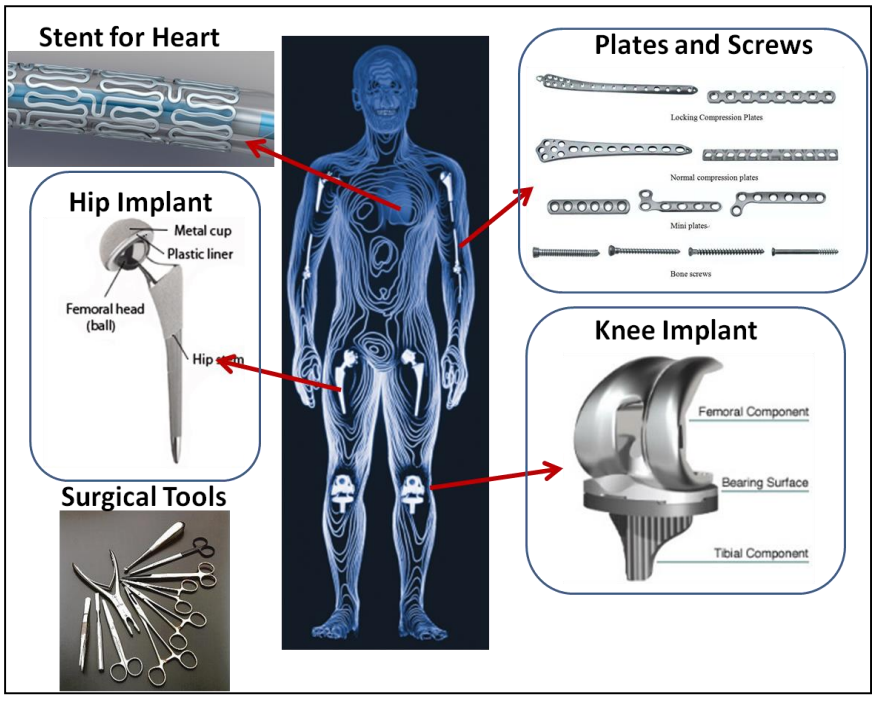


Figure 1 - Medical implants in human body and types of surgical tools

One of the major factors limiting the use of implants is the failure of these implants prematurely. It is estimated that about 50% of the knee implants fail during the first 5-8 years of surgery and about

40% of hip implants need to be replaced within 10 years of surgery (M. Balazic, 2007 December) (Geetha Manivasagam, 2010) (Teoh, 2000) .The environment in which these implants operate is very severe. The material is in constant interaction with cells and undergoes constant cyclic loading (Fig. 2a). The main causes of the failure include fracture, corrosion, tissue reaction, wear etc. (Fig. 2b). This failure causes not only financial loss but also the loss of quality of life for the patient and mental distress for the family. As the population above the age of 50 years continues to increase, it

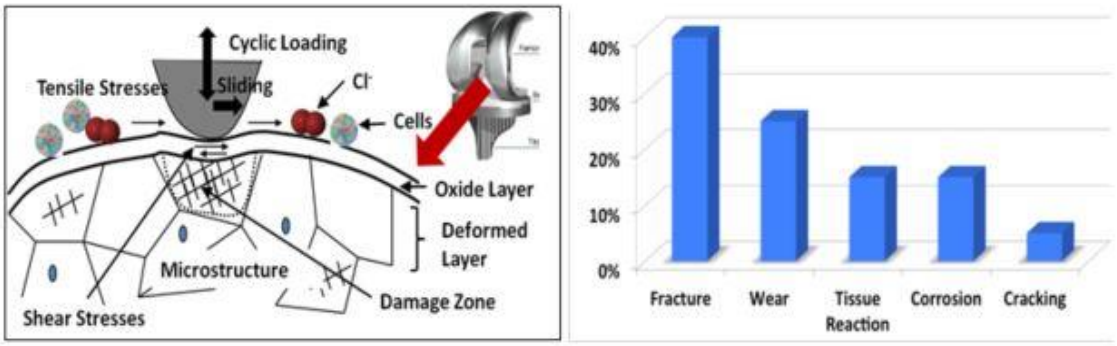


Figure 2 - (a) Complex interactions undergone at surface of implant (b) Main causes of failure of implants (Ratner, Hoffman, Schoen, & Lemons, 2004)

is necessary to find ways to design new materials that would help in increasing the life of medical implants to at least 20 years.

Background of the research

The most common biomaterials for implants and tools are Metals and Alloys (Stainless steel, Ti Alloys, Gold Alloys etc.), Ceramics (Alumina, Zirconia etc.), Polymers and Composites. These metals are most widely used because they have the best load bearing capacity and can be formed easily into the various shapes required. Among the metals, one of the most important classes of materials that have gained a lot of popularity is Stainless Steel (SS) (Walter, 2006) (Gary Winters, 2003) (Ke Yang, 2010). The other materials that are also used for these applications are Co-Cr and Titanium alloys. The first generation of implants focused just on the replacement of bone. However

more research is conducted to create implants that have properties so that tissue can grow around and in to the structure of the implant (osseointegration). These implants behave as scaffolds and are believed to provide better support for the replacement.

A key component in bone implant is the scaffold that serves as a template for cell ingrowth and interactions as well as the formation of extracellular bone matrix to provide structural support to the newly formed tissue. The requirements that allow bone ingrowth are a porosity of 30–70 vol% and a pore diameter between 300 and 800 μm. Scaffolds for bone regeneration should also meet certain criteria regarding mechanical properties, which should be similar to those of the bone to be replaced (0.5–15MPa for cancellous bone) (Rainer Detsch, 2011) (Hing, 2004) (Carter, 1977).

Table 1 (Ratner, Hoffman, Schoen, & Lemons, 2004) (Bartolo P. K., 2012) presents some mechanical properties of several metallic biomaterials. In general, these materials have high tensile and fatigue strength compared with ceramic and polymers. However, the elastic moduli are much higher than that of natural bone, which can cause ‘‘stress shielding,’’ a phenomenon characterized by bone resorption in the vicinity of implants.

	E modulus [GPa]	Yield strength [MPa]	Tensile strength [MPa]
Stainless steel	190	221–1213	586–1351
Co–Cr alloys	210–253	448–1606	655–1896
Titanium	110	485	760
Ti–6Al–4V	116	896–1034	965–1103
Cortical bone	15–30	30–70	70–150

Figure 3 - Mechanical properties of metals compared to bone (Ratner, Hoffman, Schoen, & Lemons, 2004)

Most of the implants are currently manufactured with Titanium alloys because their elastic modulus is better than SS and closer to that of bone (still much higher than needed). However, SS is preferred over these materials, because it has all the desired properties – toughness, ductility, wear, corrosion and fatigue resistance etc. Moreover, SS is easier to be formed into different complicate shapes compared to other alternatives. Various studies have suggested that SS has better fatigue resistance

and toughness than Ti alloys which were commonly used for knee and hip implants. All these benefits led to the development of newer and newer grades of SS. Although SS is available in many different forms – martensitic, ferritic, austenitic, duplex or precipitation hardened, it is the austenitic grade that is used for medical applications. Austenitic SS is relatively inexpensive, can be formed in an easy manner, and its mechanical properties can be controlled over a wide range for desired strength and ductility. However, 316L suffers from drawbacks in case of sustained and long-term use like cytotoxicity, release of metal ions, corrosion, and wear. Also, the elastic modulus needs to be more in the range of bone (15-30 GPa).

Ceramics are inorganic materials with high compressive strength and biological inertness (Dorozhkin, 2010) (Lee KY, 2006). The most commonly used bioceramics are metallic oxides (e.g., Al2O3, MgO), calcium phosphate (e.g., hydroxyapatite (HA), tricalcium phosphate (TCP), and octacalcium phosphate (OCP)), and glass ceramics (e.g. Bioglass, Ceravital) [Binyamin, 2006] [Hench, 1993]. Metallic oxides are considered to be nearly bioinert in biological environments, while calcium phosphate and glass ceramics can bond to bone when implanted. Bioceramics have been successfully used for hard tissue replacement due to their good biocompatibility and bioactivity. Their biocompatibility is a direct result of its chemical compositions, which contain ions commonly found in the physiological environment, such as Ca2+, K+, Mg2+, Na+ (Wang M. , 2003).

The main calcium phosphate materials used for medical applications are indicated in Table 2. Synthetic hydroxyapatite (HA, Ca10(PO4)6(OH)2) is a bioactive material, with chemical characteristics similar to hard tissues such as bone and teeth, that promotes hard tissue ingrowth and osseointegration when implanted into the human body (Wang M. , 2003). The porous structure of this material can be tailored to suit the interfacial surfaces of the implant. As a bulk material,

HA lacks sufficient tensile strength and is too brittle to be used in most load bearing applications (Denissen HW, 1980). In such cases, HA is coated onto a metal core or incorporated into polymers as composites. HA is frequently used as a bioactive coating on hip prostheses (Denissen HW, 1980).The ceramic coating on the titanium implants improves the surface bioactivity but often fails as a result of poor ceramic/metal interface bonding. An alternative is the production of composite materials containing titanium and bioceramic as a reinforced phase (Bronzino, 2000).

Several in vitro and vivo works have shown that calcium phosphates support the adhesion, differentiation and proliferation of osseogenesis-related cells (e.g., osteoblasts, mesenchymal stem cells), besides inducing gene expression in bone cells (Shu R, 2003) (Wang J. , 2009) (Bartolo P. K., 2012).

Ca/p molar ratio	Compound	Formula
1.33	Octacalcium phosphate (OCP)	$Ca_8(HPO_4)_2(PO_4)_45H_2O$
1.5	α-Tricalcium phosphate (α-TCP)	α-$Ca_3(PO_4)_2$
1.5	β-Tricalcium phosphate (β-TCP)	β-$Ca_3(PO_4)_2$
1.67	Hydroxyapatite (HA)	$Ca_{10}(PO_4)_6(OH)_2$
2.0	Tetracalcium phosphate	$Ca_4(PO_4)_2O$

Figure 4 - Main Calcium Phosphate compounds (Bartolo P. K., 2012)

The manufacturing processes for implants currently involve in casting, forging and machining. This limits the use of mainly homogenous materials and metals. To incorporate porosity or calcium phosphate into the implant, newer manufacturing processes need to be explored. Also, the current manufacturing process creates implants that behave like “one size fits all”. This leads to problems in the long run. Hence there is a need to develop processes that can create in-situ porosity and also incorporate both hydroxyapatite and metals in the same implant.

In the recent years additive manufacturing processes (SLS, SLM, EBM) have been explored for development of metallic implants. The research is mainly focused on Titanium and Co-Cr alloys and optimization of properties in these materials. Multi material systems have been tried in the past

(Partee B, 2006), but with limited success. None of the research, however has focused on porous materials with both metals and HA in a heterogeneous manner. In some cases, Titanium implants have been created with SLS and later on coated with HA via sol gel or electrodeposition methods. This does not result in correct properties as the ceramic-metal interface is weak and degrades over time.

Research hypothesis

This study was conducted to develop a new bio-composite using binder jet additive manufacturing process with a structure similar to the bone. The structure of the bio-composite should be porous and should be capable for load bearing as a medical implant. The developed bio-composite should be tested for required mechanical properties.

Chapter 1

Introduction

In the present day medical implants play a huge role in the field of advanced medicine. Dental implants, hip replacement, knee replacement components, plates and screws can be named as some of the applications of biomaterial. There are many biomaterials used in a variety of medical applications.

Biomaterials

Biomaterial can be defined as a natural or synthetic material which is applicable to be combined or to be introduced in to a live tissue. Biomaterials can be used in a form of implant or as a medical device. (Merriam-Webster Medical Definition of BIOMATERIAL, 2015)There are many types of biomaterials that are currently available for human use. Metallic, ceramic, polymeric and composites are used to manufacture implants currently in the medical field (Park & Lakes , Biomaterials An Introduction, 2007)

Metallic Martials

Metals are the most common medical implant material found today. High strength, ductility, wear resistance, higher electrical and thermal conductivity are well known properties of metals. Fe, Cr, Ti and, Co are some of the metallic elements which used in manufacturing medical implants. (Park & Lakes , Biomaterials An Introduction, 2007, p. 100)

Stainless steel (SS)

The corrosion resistance and strength properties make stainless steel a better candidate than Vanadium steel for bio implants. Stainless steel 302 was the first type of stainless steel to be used in manufacturing medical implants which replaced Vanadium. Later stainless steel 316 which contained Molybdenum was used to replace the stainless steel 302 for medical implants. (Park & Lakes , Biomaterials An Introduction, 2007, pp. 100-103)

Application, properties and composition of stainless steel

The stainless steel 316 and 316L are the current stainless steel types that are been used to produce medical implants. They are also known as austenitic stainless steel. Stainless steel usually contains around 12% of Chromium which makes it corrosion resistant. Nickel and Molybdenum content in

Stainless steel 316 improves its corrosion resistance properties (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 4-5).

Figure 5- Stainless Steel Bone Screws (Autocam Medical - Bone Screws, 2015)

Figure 6 - Stainless Steel Bone Plates (Autocam Medical - Bone Plates , 2015)

Commercial grade stainless steel materials were commonly been used to manufacture surgical instruments. The austenitic steel were the common candidate for medical implants. Stainless steel is a nonmagnetic material (AK Steel , 2007). The stainless steel 316L contains less amount of Carbon comparing to the Stainless steel 316. Stainless steel is hardened by cold working. American Society of Testing Martials (ASTM) recommends SS type 316L than 316 for medical implant manufacturing. (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 4-5). The SS316L can be heat treated to obtain wide variety of mechanical properties. Even though SS316L has non corrosive properties, under high stress and low Oxygen level environments it tends to corrode. Because of this reason stainless steel is generally used in short term implants such as fracture plates, screws, and hip and knee components. (Park & Lakes , Biomaterials An Introduction, 2007, pp. 100-104)

Titanium (Ti)

Titanium is extracted from Rutile (TiO_2). Due to titanium's ductility it cannot be found in pure form in the nature. However, Rutile can be found as mineral deposits in the nature. (Bartolo &

Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 2-3). Titanium was used as an implant material since early 1930s.

Application, properties and composition of Titanium

Titanium has a higher strength to weight ratio compared to SS316. Comparing density of SS316 (7.99g/cm^3) (AK Steel , 2007) to the density of Titanium (4.5g/cm^3) (RMI Titanium Company , 2000) indicates a higher strength to weight ratio in Titanium (AK steel data sheet/ Titanium Ally guide RMI). Four grades of unalloyed titanium were recommended for implant applications. Also Ti6A14V Titanium alloy were commonly used in the medical implant manufacturing industry (Park & Lakes , Biomaterials An Introduction, 2007, p. 108). Titanium and its alloys can be evaluated as an oxide-passivated material. It has noncorrosive quality due to the thin oxide layer which forms on the surface. Compared to SS316L, Titanium indicates similar corrosion resistant properties. Titanium shows high reactivity with the cell culture in implants than SS316L (Brunette, Tengvall, Textor, & Thomsen, 2001).

Titanium is used to manufacture major components in hip replacements. Titanium is used to manufacture pins, screws and spinal fusion devices for fracture repair. Most of the dental implants were also made using titanium alloys. (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 3)

Figure 5 Titanium Knee Joint Implant (MOTOR SICH JSC, 2015)

Figure 6 - Titanium Hip Replacement Implant (Ory,

Cobalt (Co)

Cobalt based materials were Cobalt-Chromium alloys. ASTM standards defines four Cobalt based alloys (CoCrMo, CocrWNi, CoNiCrMo and CoNiCrMoWFe) which are used for surgical implant manufacturing. CoCrMo is commonly used to cast products while the other three types are used for wrought implants (Park & Lakes , Biomaterials An Introduction, 2007, p. 103).

Application, properties and composition of Cobalt

When casting Cobalt based alloys, Molybdenum is added to produce a strong grain structure. Cold working cobalt based alloys are difficult to make but it increases the strength properties in implants. The wear properties were similar in both wrought and cast Cobalt implant products. Wrought Cobalt based alloy materials indicates superior fatigue and ultimate tensile strength. This makes cobalt based implants suitable for long term based implant applications. (Park & Lakes , Biomaterials An Introduction, 2007, pp. 104-107)

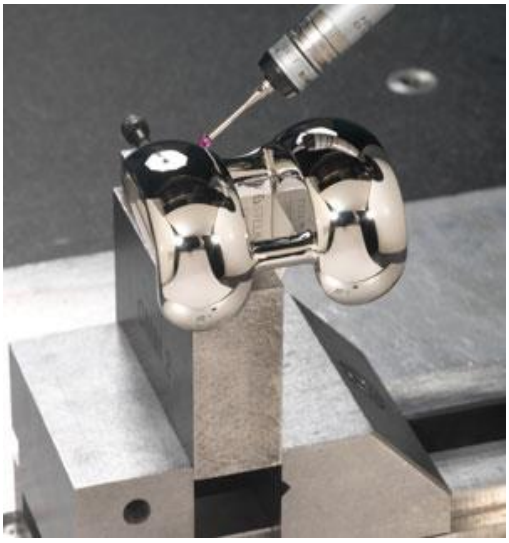


Figure 8 - Cobalt Chromium-Molybdenum Knee Implant (Tooling & Production , 2009)

Figure 7 - Cobalt Hip Implant (Loughney, 2014)

Gold

Gold is primarily used for dental implant applications. The durability and corrosion resistant are the prime qualities of gold. The dental fillings were done by casting and melting gold. Copper

Figure 9 - Cast Gold Dental Crowns (Glidewell Laboratories, 2015)

alloyed with Gold add strengths to the material. Gold has higher conductivity and being used in wires in heart pacemakers. (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 9)

Platinum

Platinum is known due to its corrosion resistance. Due to bio-compatibility of platinum, it's used to produce miniature coils to treat cardio vascular therapy. Also the electrical conductivity of

Figure 10 - Platinum Eyelid Implants (MedDev Corporation, n.d.)

platinum makes it suitable for manufacturing pacemakers (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 9)

Ceramic Martials

Ceramic materials are formed by ionic bonds. Typically ceramic materials are inorganic. (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 10). Ceramic material includes silicates, hydrides, carbides, sulfides and metallic oxides (Al_2O_3, MgO).

Figure 11 -Ceramic Dental Implants (Zsystems, 2013)

Properties of Ceramic Materials

The compressive strengths of ceramic materials are generally higher. The ceramic materials demonstrates low tensile strengths. (Park & Lakes, Biomaterials An Introduction , 2007, pp. 139-124), (Bronzino, 2000). Measurement of hardness is standardized against ceramic materials. According to the Moh's hardness index Diamond is the hardest martial which indicates a hardness index of 10. The lowest hardness index was showed by talc ($Mg_3Si_3O_{10}COH$) with a hardness index of 1 (Bronzino, 2000). The ceramic materials has low electrical conductivity. Ceramic materials indicates high melting temperatures and low heat conductivity due to its ionic bonds. The ceramic materials have higher shear strengths than metals. Ceramic material are brittle. Ceramic implants were produced under a high temperature heat treatment method called firing.

Aluminum Oxides (Alumina) (Al_2O_3)

The single crystal form of Alumina is commonly used to manufacture medical implants. The implants are manufactured by feeding fine alumina powder to a crystal surface which is then extracted from the surface with an electrically created arc or oxy-hydrogen flame. ASTM standards require 99.5% pure alumina combined with less than 0.1% SiO_2 for medical implants (Park & Lakes, Biomaterials An Introduction , 2007, pp. 142-143). Hardness and the low frictional wear are some of the advantages of using alumina as an implant material. (Park & Lakes, Biomaterials An Introduction , 2007, p. 143)

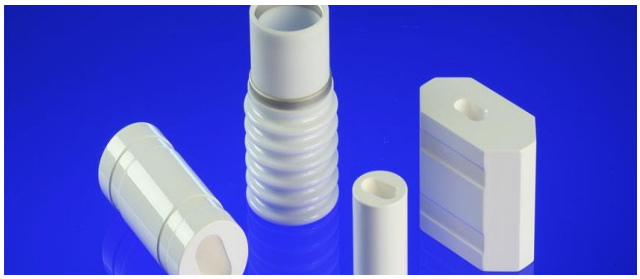


Figure 12 - Alumina Ceramic (Morgan Advanced Materials and its affiliates, 2015)

Zirconium Oxides (Zirconia) (ZrO_2)

This material is also known as fake diamonds. Zirconia is commonly used to produce medical implants especially in the dental industry. The Zirconia is prepared for manufacturing implants by phase transformation and controlling grain structures. This provides strength to the materials.

Figure 13 - Zirconium Dental Implants (Inclusive Dental Solutions, Glidewell Laboratories, 2015)

Calcium Oxide (CaO) and Yttrium (Y_2O_3) have been used as stabilizers in the strengthening process. (Park & Lakes, Biomaterials An Introduction , 2007, pp. 143-150)

The Yttrium stabilized Zirconia has been used for manufacturing femoral head of complete hip replacement implants and dental implants. Fine grain structure makes it a good bio compatible material. (Park & Lakes, Biomaterials An Introduction , 2007, p. 151)

Calcium Phosphate - (Hydroxyapatite) (HA)

This material has mainly been used to manufacture artificial bones. This ceramic material is used to build bone implants and also used as a solid or porous coating on other types of implants. Calcium phosphate can be crystalized in to different salts such as mono calcium phosphate, di calcium phosphate, tri calcium phosphate, tetra calcium phosphate, hydroxyapatite, and β-whitlockite. This is done by varying the Calcium and Phosphorus ratios. Hydroxyapatite can be named as the most important material due to its existence in the human bones and teeth. (Park & Lakes, Biomaterials An Introduction , 2007, pp. 152-153)

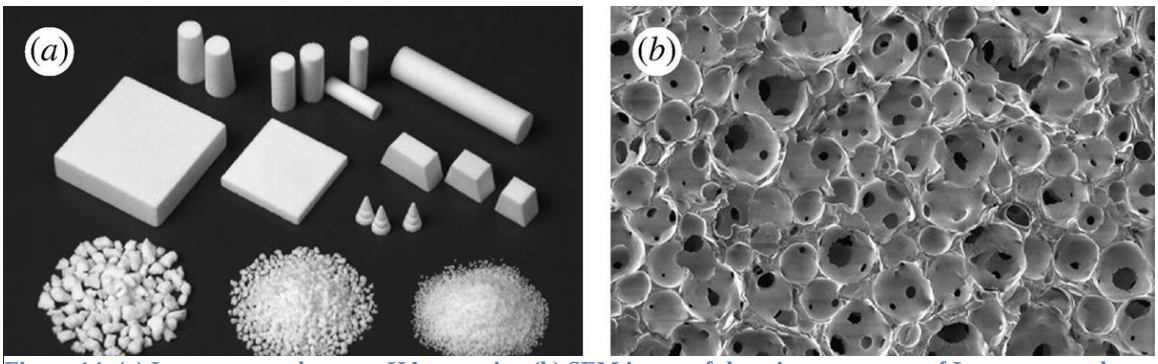


Figure 14- (a) Interconnected porous HA ceramics, (b) SEM image of the microstructures of Interconnected porous HA (Yoshikawa, Tamai , Murase, & Myoui, 2009)

Depending on the manufacturing process, Hydroxyapatite displays a variety of mechanical properties. Hydroxyapatite has an outstanding biocompatibility due to this reason it forms a direct chemical bond with hard tissues. Many procedures are used in production of Hydroxyapatite.

Precipitation, filtration and dehydration methods are used to form a Hydroxyapatite powder which is commonly used in manufacturing Hydroxyapatite based implants. (Park & Lakes, Biomaterials An Introduction , 2007, pp. 153-154)

Hydroxyapatite is commonly used to creating scaffolds in tissue engineering. In high load bearing implant applications, hydroxyapatite used as a coating for metallic core material plasma spray technology. Maxillofacial and orbitals can be named as commonly used applications of hydroxyapatite. (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 10)

Glass Ceramics

Glass ceramics are also called as polycrystalline ceramics. These type of ceramic are produced by controlled crystallization which were developed around 1960s. Bioglass® and Ceravital® are two of the major glass ceramics used in manufacturing implants (Biomaterial an Introduction). Injection molding methods are used to manufacture glass ceramic implants.

Glass ceramics indicates soft characteristics and can be easily machined in to any shape using diamond tools (Biomaterials and tissue Engineering).Also this material is brittle and it has a low bending strength. Low bending strength make glass ceramics not suitable for load bearing applications.

Polymeric Material

Polymers are the largest class of biomaterial. Natural polymer materials and synthetic polymer materials are used in medical applications (Ratner, Hoffman, Schoen, & Lemons, 2004). Synthetic

Figure 15 - Polymeric Spinal Implant - Zeniva® polyetheretherketone (PEEK) resin from Solvay Advanced Polymers, LLC. (SOLVAY, 2011)

polymers are made by linking molecules together using primary covalent bonds. Primary molar-bonds in polymers contains Carbon, Nitrogen and Silicone. The bonding is completed by using chemical processes such as condensation and addition (Park & Lakes, Biomaterials An Introduction, 2007, pp. 174-175)

Condensation Polymers

This method is also known as the step reaction. In this method, small molecules will be condensed out. Polyester, Polyurethane and Polyamide are some of the condensation polymers. In the downside, this process does not form longer polymer chains. (Park & Lakes, Biomaterials An Introduction , 2007, p. 175)

Addition polymers

Free radical polymerization is another name for addition polymers. This process re-organize the bonds in monomers. Ethylene, Propylene and Acrylonitrile are some of the monomers which are suitable for this process. (Park & Lakes, Biomaterials An Introduction , 2007, p. 176)

Mechanical properties of polymers

Polymers can be amorphous or semi-crystalline. The amorphous polymers exhibit a lower stiffness and it can handle higher stress before failure. Semi-Crystalline polymers have a lower extensibility. This material can handle repeated cycles of stress and strain (Ratner, Hoffman, Schoen, & Lemons, 2004). Amorphous polymers change properties according to the temperature.

Polymers have a wide range of medical applications. These polymers can be fabricated easily in to many forms. Fibers, films, textiles and rods can be named as few forms that the polymers have been fabricated into.

Composite Materials

Composites contain two or more different basic materials or phases on a micro or macroscopic scale. Properties of the composite materials depend on the structure. Some properties of the material depend on the shape of the heterogeneities based on the volume fraction gained by the material and the margin among the elements (Park & Lakes, Biomaterials An Introduction, 2007, pp. 209-210). Two phase composite materials can be classified using the microstructure. The enclosures with in the matrix could be fiber particles or platelets. If either enclosures or the matrix contained liquid or air, the material could be a cellular solid (Park & Lakes, Biomaterials An Introduction, 2007, pp. 219-222). The strength of the fiber composite materials depends on the brittleness or ductility of the enclosures and the matrix of the material. Composite materials could fail due the fiber brakeage, matrix failure, deboning fiber from the matrix and buckling. Composite materials are currently used

Figure 16 - Composite Restoration Dental Implant (Harmony Dental Lab, 2013)

as dental fillings and to reinforce the some bone cements and some porous orthopedic implants. (Park & Lakes, Biomaterials An Introduction, 2007, p. 213)

Bone Implants

Natural bones can be named as unique tissue which has the capability of healing and remolding. Bones provide structural support to the human body. Load bearing, acting as a mineral fountain to the muscle tissues, and protecting internal organs are some of the important duties of the bone tissue. Due to injuries and defects bone tissue may sometime need repair. Bone grafts procedures are required to repair defective bone tissue. Grafts can be taken from the patient's own bones, or from another human individual. Research experiments developed synthetic substitutes to replace these bone-graft procedures. This help to eliminate complicated surgeries and stop the spread of infectious diseases (Bartolo & Bidanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 79-80).

Because of the complications of treatment, researchers developed methods of bone reconstruction by using metal, ceramic, polymers, and composite materials. Advance methods are required to produce these complex implant structures. Generic range of implant designs can be found in the production market. The correct size is selected by the surgeon by pre operation scans (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 79-80). The Implant parts are manufactured in many different methods such as casting, bar stock milling, compression molding, and sintering. These manufacturing methods have some disadvantages. Incapability of rapid design changes, high material wastage and geometrical restrictions are some of constrains that the implant manufactures face (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 80).

The modern implants are modeled by using the following methods: Reverse engineering (RE), scanned base imaging such as magnetic resonance imaging (MRI), Computerized tomography (CT), X ray and laser scanning. 3D models of the human organs were created using these methods. Then Rapid prototyping(RP) and solid free form fabrication manufacturing methods has been used to create physical models for surgical training purposes, develop customized implants, scaffolds, and pre surgical planning. These advanced manufacturing methods have revalorized the medical industry and it has been providing higher quality of life (Bartolo & Bindanda, 2008, p. 80).

Modeling and design biomedical implants

Geometrical data gathered from scanning and reverse engineering have been used as the drivers for the medical rapid prototyping, computer aided design (CAD) and computer aided manufacturing (CAM). In the biomedical application, modeling and design can be defined in four major steps; data acquisition, data processing, design phase, and development of the medical application. (Bartolo & Bindanda, 2008, pp. 84-85)

CAD models provide many advantages for biomedical implant development. Defining external shapes, providing ability to introduce internal structural characteristics, test simulation such as finite element analysis (FEA) to analyze load bearing capacities can be named as some of the valuable features of CAD (Bartolo & Bindanda, 2008, p. 85).

Implant Manufacturing

In biomedical applications bone implants could be categorized as highly important. Preassembled implants with correct geometrical requirements absorb the complexity from surgical procedures.

Layer manufacturing can be named as the key for the rapid manufacturing (RM) of directly useable implants. RM is a

Different method than RP. RP produce the tools and parts for prototyping while RM produced the implants for direct applications. RM helps to produce customized products in mass scale and layer manufacturing provides the ability develop complex shapes. In the modern day there are many layer manufacturing methods has been used to develop bone medical implants (Bartolo & Bindanda, 2008, pp. 85-87).

Chapter 2

Additive Manufacturing

Basic principle of additive manufacturing can be defined as producing a model using a 3D computer-aided design without a pre-planned process. Complex objects can be directly manufactured by additive manufacturing (AM) using CAD models (Gibson, Rosen, & Stucker, 2010, p. 1). In AM the parts are produced by adding materials in layers. Every layer is a small cross section of the CAD model which acts as the source. AM is the key feature for the product development. The AM developed models were used by manufacturers to test the functional ability of the product. The necessary improvements are made after these tests. AM is not only used for developing models it's also being used to develop end products in areas such as custom-made bio medical implants. (Gibson, Rosen, & Stucker, 2010, pp. 1-3)

Basic Steps of Additive manufacturing

Due to the layer based manufacturing, sometimes additive manufactured parts require post-conditioning. As a result of these additional steps, AM can be divided into many categories. From the CAD model to the actual part, AM technology is separated to eight different steps (Gibson, Rosen, & Stucker, 2010, p. 3)

General Steps for additive manufacturing

Computer Aided Design

AM starts with designing a model with any professional CAD software. The output model has to be a 3D or surface representation of the actual part. Scanning and reverse engineering equipment could be also be used to generate this model. (Gibson, Rosen, & Stucker, 2010, p. 4)

Conversion for AM accepted file type

STL is the standard file type for the AM machines. Once a CAD model is created it should be saved in STL format through the CAD software. STL file translates the surface in the CAD model to a mesh of triangles. The number of the triangles controls the precision of the rounded surfaces. The CAD software allows the user to control the number and the size of those triangles (Stratasys Ltd., 2015).

Transfer STL files to AM machine

The STL file should be transferred to the machine. The file should be verified for its right size and build orientation. Also if there are multiple parts they should be properly placed so they wouldn't overlap with other parts.

AM machine setup

Machine setup is a crucial step in the process. The parameters should be properly set up to achieve the tolerances of the manufactured part. Layer thickness, orientation, energy provided, timing and roller speeds can be named as some of these parameters.

Build phase

This is step is more of automated process done by the machine. Partial monitoring will be required to ensure there are no errors and the machine would not run out of material.

Removal

In this phase the machine is done producing the part and the user has to take it out. The user should follow the safety protocols and proper shut down procedures. This will ensure the safety of the user and the machine.

Post Processing of AM parts

Once the parts are produced they might need some additional treatment such as curing, sintering and cleaning. At these stages parts may be weak and they should be handled with care.

Application

Some parts may be ready to use by this time. Some parts may require additional processing. Also priming, painting, and polishing should be performed depending on the application requirements. If there are multiple parts being produced they might need additional assembly work done before using. Handling with care and good engineering practices may help to produce high quality parts using AM methods.

Categories of Additive Manufacturing

Additive manufacturing spreads out in to many divisions which are defined by the method and materials used. These categories are as shown below.

Powder bed Process

This method fuses small layers of powdered martial using laser or electron beams. The CAD data will provide the geometry of the manufacturing part. (Additive Manufacturing(AMCRC), 2012)

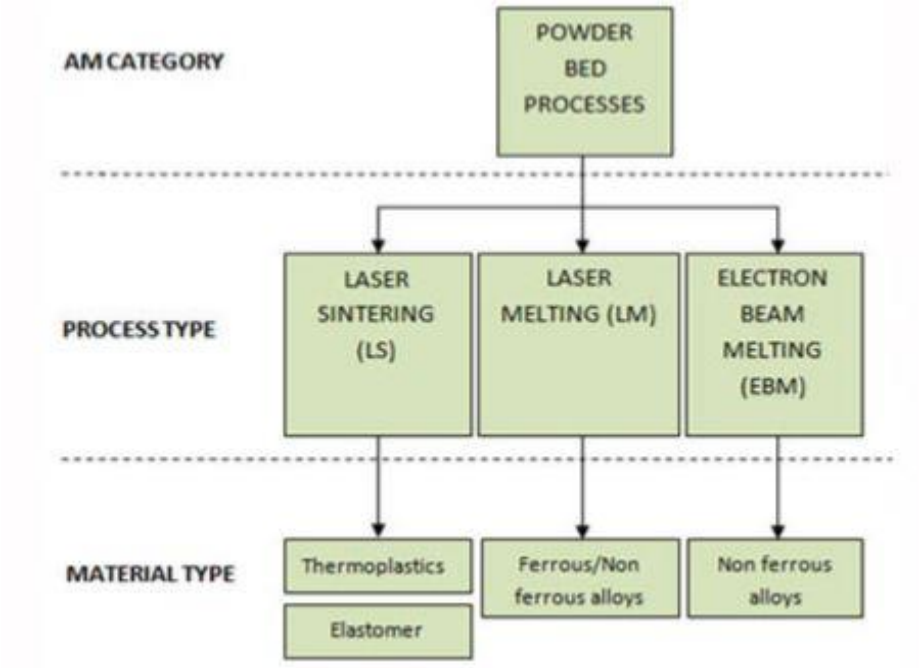


Figure 17 - Flow Chart for Power Bed process including the process and material type (Additive Manufacturing(AMCRC), 2012, p. 1)

The figure 19 flow chart indicates the process and the material types in the powder bed AM category.

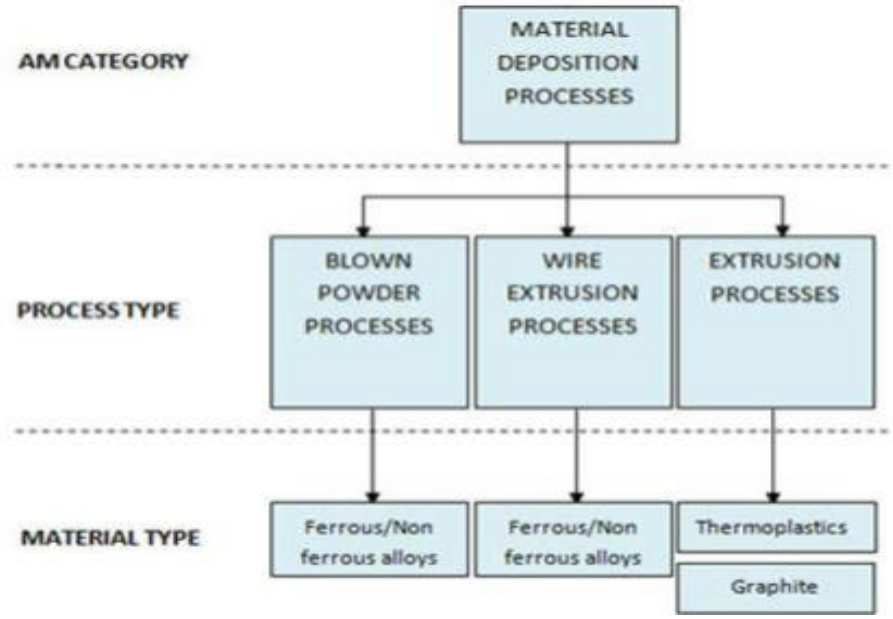


Figure 18 - Flow Chart for material deposition process including the process and material type (Additive Manufacturing(AMCRC), 2012, p. 2)

Material Deposition Process

Materials will be pre heated through an extrusion nozzle which works through a predefined trail. Material layers will build on top of each other to build the 3D object. Figure 20 below shows how the processes and some of the materials which are used in this methods (Additive Manufacturing(AMCRC), 2012).

3dPrinting Process

The 3D printing method lays down a thin layer of heated material to the platform. The print head or the print bed continually move in a sequence according to the CAD model. This method lays thin material layers on top of each other to build the part. Also some of the 3D printing machines print binder layers on to the powder bed. Figure 19 indicates some of the material that is used in 3D printing process.

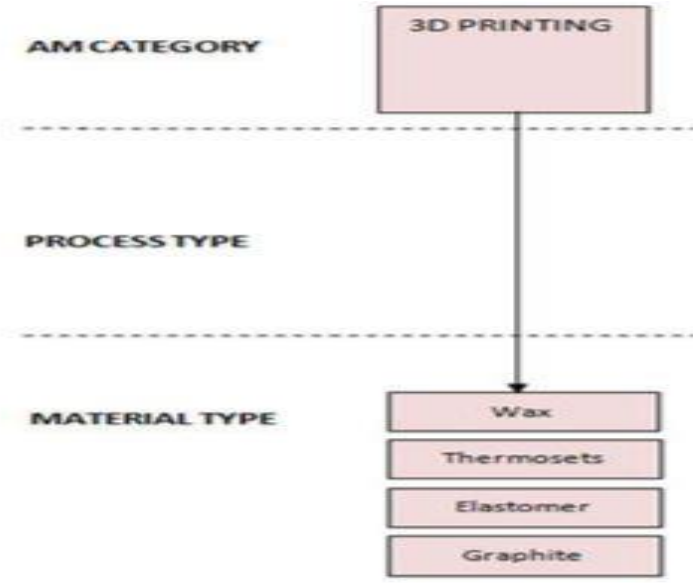


Figure 19 - Flow Chart for 3D printing process including the process and some material types (Additive Manufacturing(AMCRC), 2012, p. 3)

Liquid Polymer Process

This process hardens thin layer of ultraviolet thermoset liquid. This method uses a solid state crystal laser to build the part layer by layer according to the CAD model. The ultraviolet radiation from

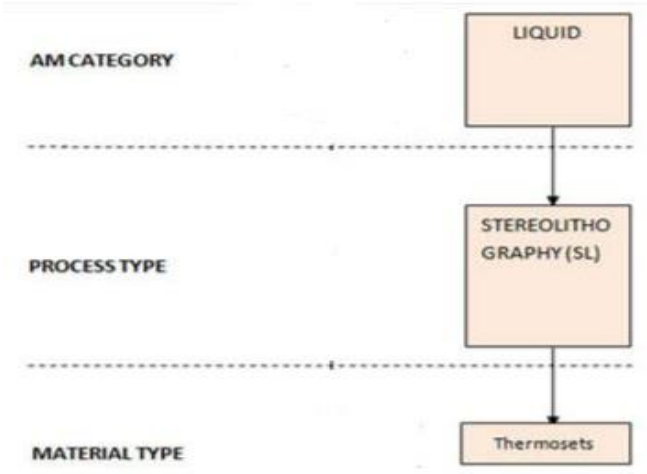


Figure 20 - Flow Chart for liquid polymer process including the process and material type (Additive Manufacturing(AMCRC), 2012)

laser cures the liquid polymer. The above figure 20 shows the process type and the materials that are used in liquid polymer process. This process has the unique ability to build high resolution parts.

Further information about AM processes

Additive manufacturing categories that were discussed earlier have many different processes. Those methods are used in modern day additive manufacturing. This section discusses the additive manufacturing processes and some of the materials which are commonly used with these processes.

Laser based methods – Laser engineered Net shaping (LENS®)

Laser engineered net shaping (LENS) is a CAD based system. In this method a high powered laser is focused on to a metal which creates a molten pool. Then the powder is injected in to the molten pool to increase the volume. The CAD data moves the laser overlapping each line. This process continues till the metal object is formed.

Titanium, stainless steel and cobalt based products are manufactured using this process (Bartolo & Bindanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 85-87).

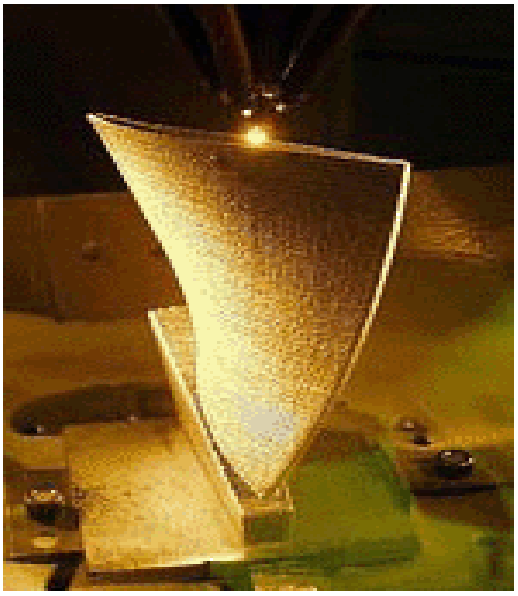


Figure 21 - Building a Processing Blade (Sandia National Laboratories , 1997-2014)

LENS® technology was developed by Sandia National Laboratories. This process reduces time and cost to build metallic parts. Also the LENS® process is capable of manufacturing fully dense shapes. It has the ability to fabricate parts with high tolerance levels. A wide range of materials can be used to manufacture parts using this technology. The LENS® process has been improved for three and four axis systems which deliver the ability to construct complex shapes with a better finish (Sandia National Laboratories , 1997-2014). Detailed explanation in figure 22 provides an Idea of LENS® manufacturing technology.

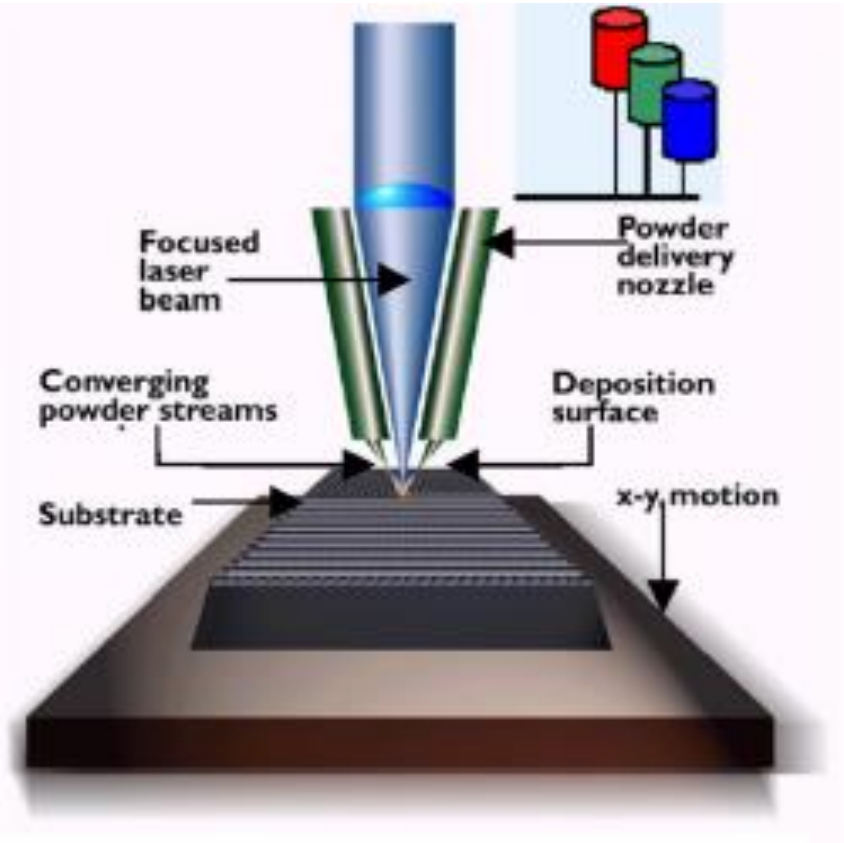


Figure 22 - Layout of LENS® System (Mudge & Wald, 2007 January)

Laser based methods – Selective laser sintering (SLS) / Selective Laser Melting (SLM)

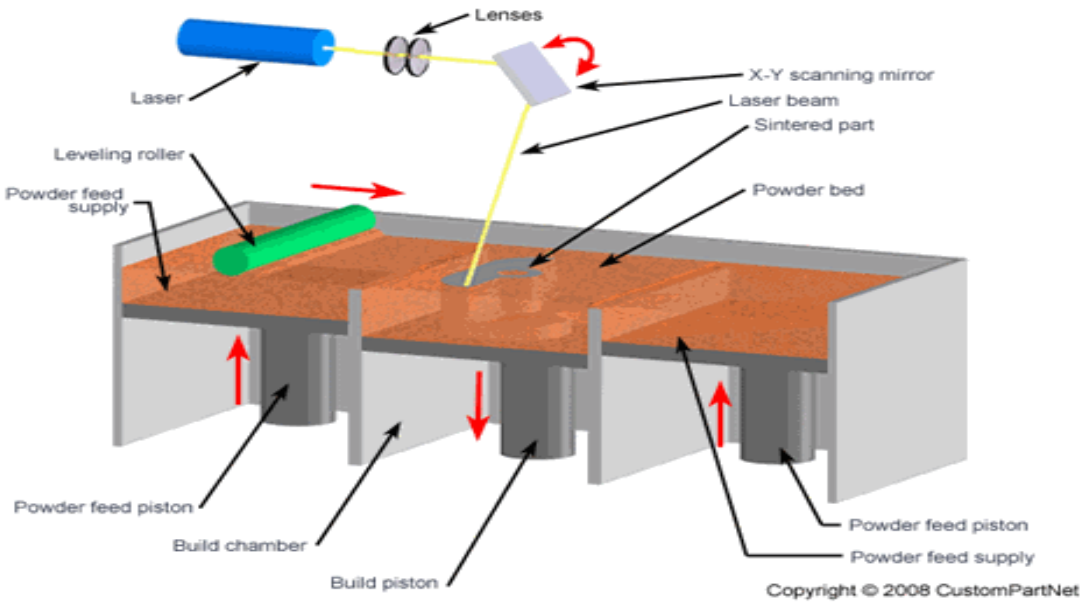


Figure 23 - SLS Process Diagram (CustomPart.Net, 2008)

The selective laser sintering (SLS) process operates by selectively sintering powdered material heated by a laser. The process forms the objects fusing 2D layers. Many materials have been experientially printed in this method. Nylon, polymer, and ceramic materials are printed using this method. The parts were created by fully melting powdered materials. As the figure 23 indicates, raw powder is pushed through the feed bed. The leveling roller pushes the power on the cured part. The high power laser reflects through a mirror and moves in x and y planes. The laser path is defined by the CAD model. In this process, layer by layer get sintered after the roller pushes the material on top of each layer.

Selective laser melting (SLM) process is primarily used with powdered metallic materials. SLM process works as same as the SLS method. It uses a very precise laser beam for melting the powdered metallic material, which allows this process to manufacture complex parts. The finished

parts need post conditioning before being used. Stainless steel and titanium alloys are some of the powdered materials used with this method

Electron based method – Electron Beam Melting (EBM®)

This technology was developed by a company called Arcam AB®. This AM process uses a high power electron beam which generates energy needed for melting the material (Arcam AB, n.d.). The parts are built by melting layer by layer. The EBM® process provides many advantages.

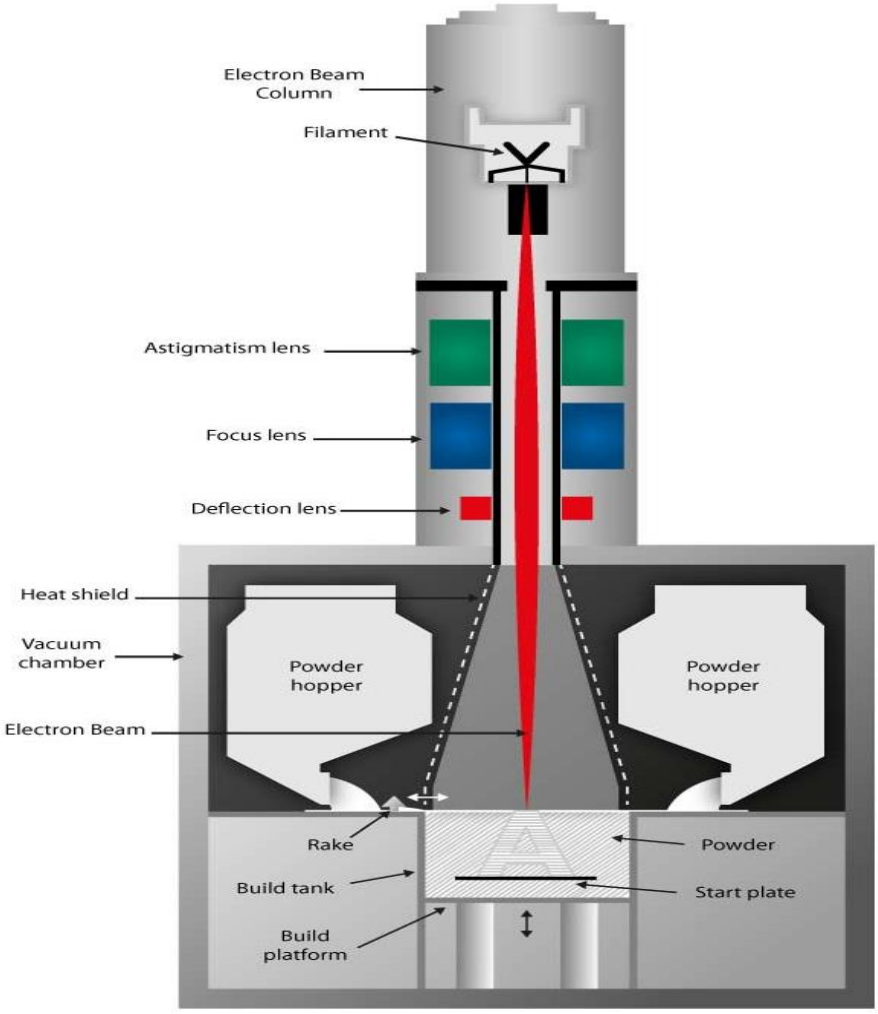


Figure 24 - EBM Process (Arcam AB, n.d.)

Small spot size and high power beam adds efficiency and high scanning speeds to this method. (Bartolo & Bidanda, 2008, p. 92) The EBM® process takes place in vacuumed environment with a high temperature, which makes the end parts of better finished quality comparing to cast and wrought material (Arcam AB, n.d.)

Arcam has developed EBM® processing further by introducing controlled vacuum technology, warm processing and Arcam MultiBeam™ technology.

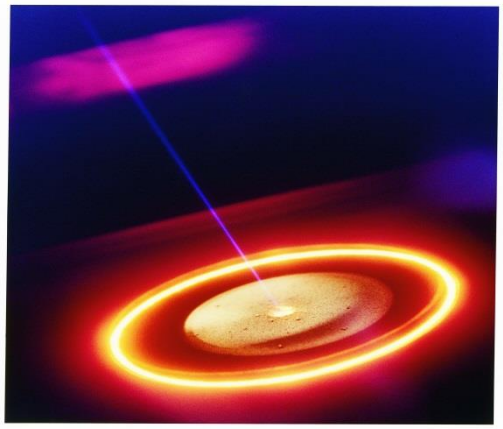


Figure 25 - Electron Beam Melting Pool (Arcam AB, n.d.)

Controlled Vacuum technology

This technology maintains $1*10^{-5}$ mbar base pressure throughout the build process. Then the process Introduces Helium gas at $2*10^{-3}$ mbar when it actually melts the material. This controlled environment provides high quality finished products (Arcam AB, n.d.).

Warm Process

This technology heats the powder bed to the optimal ambient temperature specific for the material. The process provides the ability to process parts without residue. This allows the micro structure of the end product to have no martensitic structures (Arcam AB, n.d.)

Acrcam MultiBeam™ Process

This technology provides the ability to create multiple melted pools maintained at the same time. This approach reduces the build time, Improves surface finish and builds precise parts.

Extrusion based methods – Fused Deposition Molding (FDM)

Fuse deposition molding (FDM) melts and extrudes the materials layer by layer. The build materials stay as a filament stock. The filament is then continuously melted and extruded through a nozzle in layers (Bartolo & Bidanda, Bio-Materials and Prototyping Applications in Medicine, 2008, pp. 93-94). This process can be named as the most common extrusion based manufacturing process. This method was developed by Stratasys.

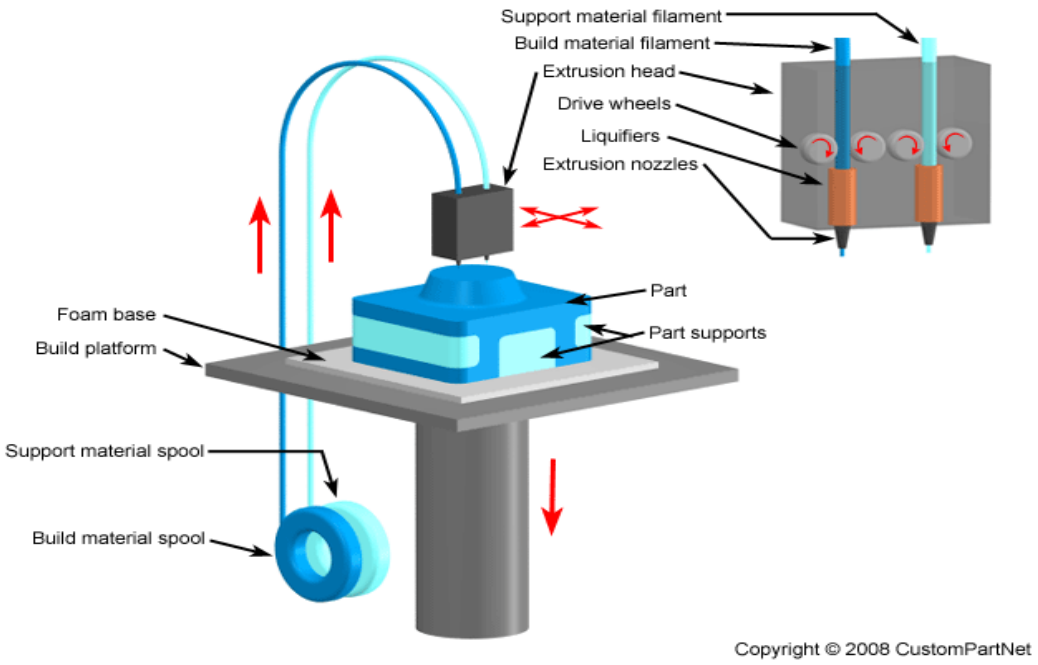


Figure 26 - FDM Process (CustomPart.Net, 2008)

FDM process has a preprocessing production and a post-processing phase in the production cycle. The path to extrude the built material and the support material is calculated through the CAD model.

This task is done during the preprocess stage. The build materials and the support material will then be heated to semi-liquid phase. Then the materials are deposited in fine droplets along the extrusion pathway. During the post-production process the support material then needs to be broken out or dissolved. The final parts should be inspected for flaws before using them as prototypes or actual application. (Stratasys, 2015)

FDM materials requires certain types of material properties. The materials also have to meet specific geometric specifications in order to feed it through the extrusion nozzle. This situation limits the materials that can be used to build products using FDM process. This is the major disadvantage of this process. Resolutions of the final products are relatively low comparing to some of the other AM process methods such as Stereolithography and LENS®. (Bartolo & Bidanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 95).

Printing based methods – Binder Jet process

The printing based method deposits liquid binder in to the powder bed according to a pre-defined path by the CAD model. This method is similar to the traditional paper printing. Binder jet process does not generate heat during the build process. After the printing process is completed the printed parts should be extracted and then cured or sintered (ExOne , n.d.). This process is capable of using a wide variety of material from metals to ceramics. Producing large objects is a major advantage of this process. The binder jet process was initially developed at MIT (Bartolo & Bidanda, Bio-Materials and Prototyping Applications in Medicine, 2008, p. 97).

ExOne™ – X1-Lab™

ExOne™ is one of the leading binder jet 3D printing manufacturers. The experimental parts for this study was printed using X1-Lab™ 3D printer. This section will discuss the machine and the operating process.The X1-Lab™ is specially designed for research and educational wok.

The machine contains two beds. One of the beds is used to build the part and the other is used to supply the powder material. This machine will print each layer by laying binder on the powder of the build bed. Then the leveling roller spreads a new layer of materials by pushing the materials from the supply bed to the print bed (ExOne, 2013). Once a layer gets printed the bed assembly moves under the heater for a few seconds to dry the fresh laid binder. After the layer is dried the print bed travels down while the supply bed moves up and provides a new powder layer. The bed assembly is then moved through the roller to level and spread the powder on the build bed. This process continues until the entire part is completed. The printed layers are supported by the loose powder lying around the printed area on the build bed. After the printing is completed the parts need to be extracted from the build bed. The build plate should be then transferred to the oven for initial curing using an extraction assembly. The build plate under the print bed is attached to the extraction assembly. This complete assembly can be paced in the oven as the figure 33. Depending on the density of the parts, the material for infiltration is added to the sintering bowl. The sintering bowl is then placed on a furnace. Air is evacuated out from the furnace and Argon is supplied to the furnace at atmospheric pressure by a compressed gas tank. The temperature is increased in steps till the maximum sintering temperature is achieved.

After holding the maximum temperature the furnace is brought back to room temperature in steps. The figure 27 flow chart shows the process for this machine. The parts may still need post processing such as surfacing and grinding before use.

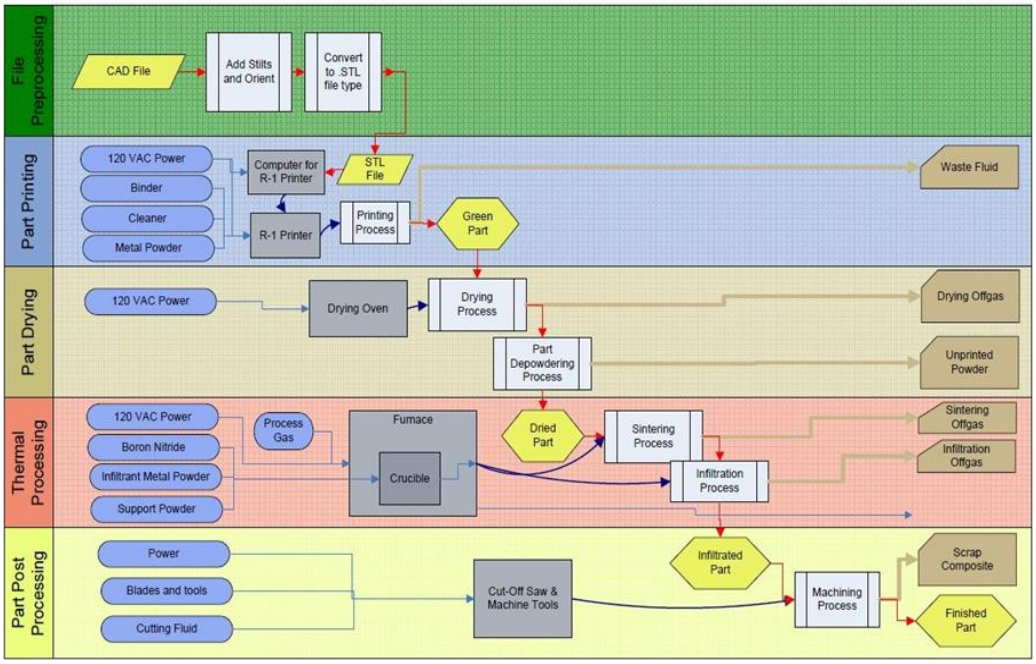


Figure 27 - X1-Lab process flow

Liquid polymer method - Stereolithography (SL)

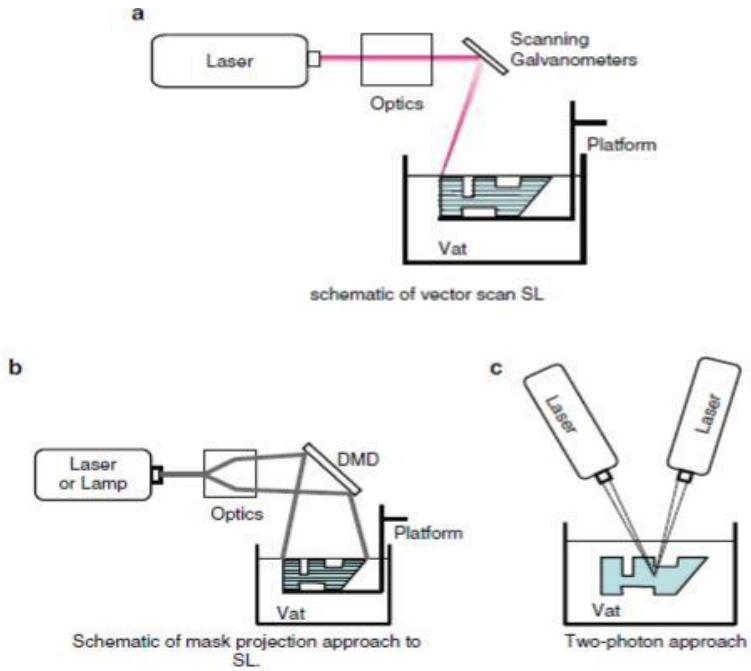


Figure 28 - SL methods a) Vector Scan b) Mask Projection c) Two-Photon (Gibson, Rosen, & Stucker, 2010, p. 62)

This process uses liquid resins or photopolymers which is curable by a radiation method. Various radiation methods are used for SL. Ultraviolet and visible light radiation are the most common radiation methods used in this process. Commercial SL machines commonly use vector scan process. Mark Projection and Two-Photon are some of the other SL process methods. 3D systems are the major SL machine manufacturers in the present. The figure 28 explains the difference between each method (Gibson, Rosen, & Stucker, 2010, pp. 64 - 65).

Chapter 3

Compressive strength

According to the Mariam Webster dictionary the compressive strength can be defined as the maximum compressive stress that a solid material can hold under a gradually applied load without fracture. (merriam-webster, 2015)

Compression testing

Compression is one of the main test methods which can be used to understand the characteristic qualities of material such as metal, rocks, composites, plastic, concrete, and wood. Static compression tests are conducted by applying a compressive load gradually till the material fails or applying a load through a certain time period. The dynamic compression is tested by varying the load between two or more loads.

Brittle materials generally express grate compressive strengths. When they fail the materials tend to fracture. Materials such as concrete and cast iron indicate this type of failure. Ductile materials such as mild steel deforms elastically under a load. Ductile materials also generally indicate very good compressive strengths.

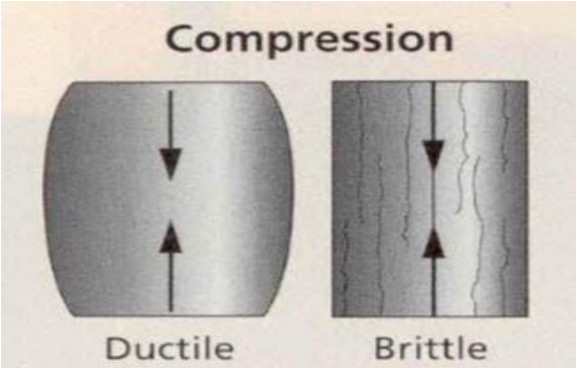


Figure 29 - How Ductile and Brittle material act under Compressive loads (Klopman, 1998)

Material Test System 810 (MTS)

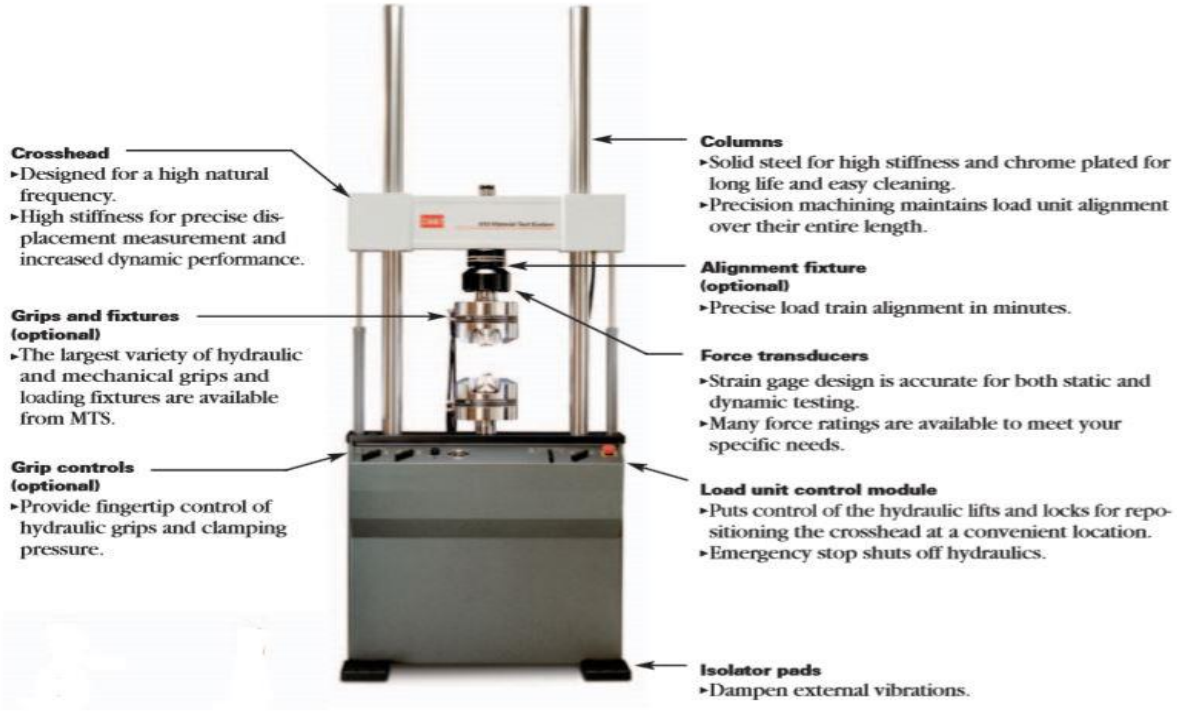


Figure 30 - MTS 810 in its standard step with the tensile testing jaws (MTS Systems Corporation, 2006)

Material testing of this study is conducted using MTS 810 in its compression test set up. MTS 810 is capable of conducting a variety of tests on multiple materials.

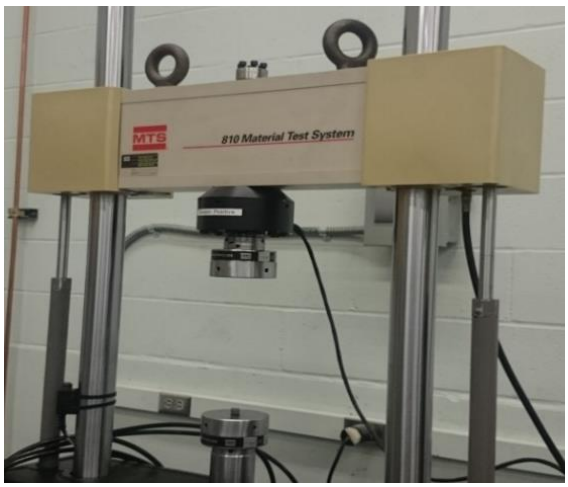


Figure 31 - MTS 810 with compression test setup

The machine has the ability to conduct both high and low force application testing, as well as both static and dynamic testing. The computerized control ability and with the accessories available to this system it could be easily setup for different material testing needs. MTS 810 has force range from 25KN (5.5kip) to 500KN (110kip).Because of this wide range MTS 810 is capable of conducting tests on wide range of materials. The design of this machine allows the user to test specimens in different sizes. The movements of the machine and the jaws are driven with a hydraulic power unit attached to the system. For the purpose of this study the MTS 810 machine is used with compression accessories. The Calibration data is provided for the machine in the appendix 1. Figure 31 shows the compression jaws used for this study. (MTS Systems Corporation, 2006)

ASTM standards (E-9)

ASTM provides guidelines to conduct compression testing. The ASTM standard E9-09 provides the guidelines for standard test method of compression testing for metallic materials at room temperature. This standard cover guide lines, apparatus, specimens and procedure for axle-load compression testing at the room temperature. If there is any testing beyond that temperature range it should follow E209 guidelines.

Test summery

The test specimen should be exposed to increasing axial compressive loads. Stress and strain monitoring should be done continually or in steps. The data gathered from these tests may include, stress strain curves, yield strength and compressive strength. If the material does not fail by fracture, compressive strength is determined by the total strain and the geometry of the sample.

Apparatus

Test machines used for compression testing should confirm their calibration. The bearing heads should be parallel at all times with 0.0002 in. /in. Bearing blocks should be parallel to the surface of the test sample with in 0.0002 in. /in. The blocks should be made out of hard material and shall be faced. (ASTM International, 2015)

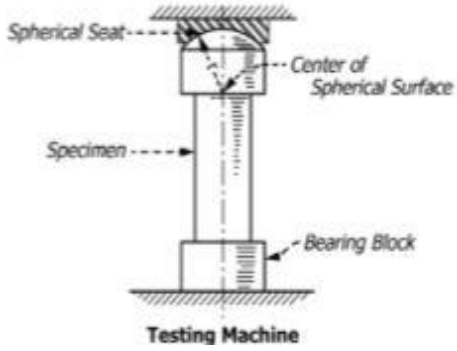


Figure 32 - Sample centered on the test heads (ASTM International, 2015)

The sample should be carefully centered in the test machine heads. The purpose of the sample aligning in the center of the compression heads is to uniformly apply the axle load to the test sample.

Test samples

Samples are recommended to be prepared in the form of solid cylinders. There are three types of cylindrical samples recommended for testing metallic materials depending on their length. Long, short and medium length samples can be used for the test. This study uses samples in the short range. ASTM standards recommend that the length to diameter (L/D) ratio of the sample be around 1.5 to 2.0. With the shorter samples this ratio is important to obtain good results for high strength materials. For the purpose of this study the samples have been post processed to fit this L/D ratio (ASTM International, 2015).

The lateral surfaces of the gauge length of the samples should not differ in diameter or width by more than 1% or 0.0002 in. The centerline of the all lateral surfaces should coaxial within 0.01in. Surface finish should be 1.6µm or better. The end of the sample should be flat and parallel within 0.0005 in. /in. and perpendicular to the lateral surface within a 3 in. arc. Machining or grinding

methods should be used to achieve these specimen requirements. The gauge length of the sample should not be closer to the ends of the sample. The gauge length should have an equal or greater length of the sample radius from the boundaries of sample (ASTM International, 2015).

The specimens that are greater than 0.10 need to be measured to the nearest 0.001in. The test bed should be cleaned with acetone. Lubrication of the bearing surfaces is recommended. For the purpose of this study lubrication was not done due the porous quality of the samples. The reason for this was lubrication might enter in to the material and it might affect the of the sample properties. The testing speed is recommended to be around 0.005 in / in. min (ASTM International, 2015).

The number of sample should be determined according to the test purpose and needs. Protective gear should be used when conducting tests to ensure the safety of the operator.

Loading

The machines that do not use the strain-spacing attachments or auto feedback controls should maintain a constant head speed from the beginning to the end of loading. The average strain can be calculated by the time interval and the load strain recorded (ASTM International, 2015).

Test Run

When the samples are installed and aligned properly, run the test until complete as follows. For ductile materials run the test till the yield strength or the yield point is reached, even though in some occasions strength and strain of the material is greater than the yield strength. In this situation the test for understanding yield strength and compression are the same. Material that does not show sharp neck on the stress and strain diagram will need a yield point initially estimated. Then the test should be run passing that point to make sure the yield strength can be determined after finishing the test. The brittle materials should be run till the crushing or shattering occurs (ASTM International, 2015).

Report Data

Description of the materials should be included in the final report. Sample configuration needs to be provided through a sketch. Measured dimensions of each and every sample should be included. Explanation of the test fixture and the lubricant information needs to be provided. Also, this information should include calibration data. Test speed and stress and strain diagrams should be included if possible. Yield strength or point should be included when needed and the method of determination should be discussed, including compressive strength for brittle exhibit samples. The compressive strength and total strain should be included for ductile materials. Type of failure should be discussed when possible for samples. Precision and accuracy of the data should be mentioned along the report with any anomalies that took place during the test (ASTM International, 2015).

$$Ultimate\ Compressive\ Strength(PSI) = \frac{Maximum\ laod\ (lb)}{Cross\ sectional\ area\ of\ the\ sample(SqIn.)}$$

$$Cross\ Sectional\ area\ of\ the\ sample\ (SqIn.) = \frac{\pi * Diameter^{\,2}}{4}$$

Chapter 4

Experimental Plan

This study was conducted to print a bio-composite using stainless steel and calcium phosphate tribasic with a porous structure and test the printed samples for understand its compression strength. Experiments were conducted to understand which volume mixture would print a sample of the bio-composite. The table below shows volume ratios of pre-experimental tests.

Trail	Stainless Steel Volume%	Tri Calcium Phosphate Tribasic Volume %	Results
1	50	50	Printed crumbled samples
2	60	40	Printed crumbled samples
3	70	30	Printed crumbled samples
4	80	20	Printed good samples
5	90	10	Printed good samples

Table 1 - Volume mixture selection

After performing the pre-experiments 80% of ss316 to 20% HAp were selected to continue the study. This volume ratio was selected because it was the first volume ratio that printed a complete sample without crumbling. This experiment was continued with the SS 80% and 20% to understand how the effect of changing parameters in X1 binder-jet printer affects the strength of the printed bio-composite. After printing the samples a modified version of the ASTM E9 standards were used to conduct compression tests.

The samples were printed by changing different parameters. The parameters were changed according to a partial factorial design for two levels and four factors. The parameters were changed according to the fractional factorial design of experiment template shown below.

	A	B	C	D	
Experiment No	Layer Thickness	Roller Speed	Sintering Time	Sintering Temperature	Comments
1	-1	-1	-1	-1	Crumbled
2	-1	-1	1	1	Samples printed and tested
3	-1	1	-1	1	Crumbled
4	1	1	-1	-1	Samples printed and tested
5	1	-1	-1	1	Samples printed and tested
6	1	-1	1	-1	Crumbled
7	-1	1	1	-1	Crumbled
8	1	1	1	1	Crumbled

Table 2 - Fractional factorial DOE

Parameters and experiments

The X1-Lab machine is capable of changing many parameters for binder jet printing. In this study four of those parameters have been taken into consideration to get a better understanding of how it effect the printing and the compression strength. The parameters were layer thickness, roller speed, sintering time and sintering temperature.

Layer Thickness	Roller Speed	Sintering Temperature	Sintering Time
µm	mm/s	C	Hr
50	1	1150	6
200	10	1200	8

Table 3 - samples printed parameters

Layer thickness

The layer thickness means the resolution of the printer. In an Ink jet printer resolution is monitored with pixels. In AM the layer thickness is the resolution measurement of the build component. Layer thickness can be identified as the minimum thickness of the material layer, that the printer will lay down. In material printing the lower number for the layer thickness will provide a higher resolution

for the part. Table 3 indicates the parameters that were used to print the experiments. The 50 µm is the lower end and 200 µm is the higher end of layer thickness which X1-Lab binder jet printer can print.

Roller speed

The roller speed is the speed of the powder spread across the printing bed. This is performed by moving the print bed across a spinning roller. The roller speed can be adjusted through the X1-Lab software. At lower roller speeds the material is spread more evenly across the print bed. The highest roller speed of the X1-Lab is 10 mm/s and the lowest roller speed is 1 mm/s

Sintering time

Sintering time can be adjusted on the sintering furnace. This determines how long the printed part is being sintered. The maximum sintering temperature inside the furnace will be achieved in steps. The sintering machine holds the maximum temperature for this time period. The printed samples were sintered in two different time periods. When sintering, 6 hours was the low end and 8 hours was the high end.

Sintering temperature

This is the maximum sintering temperature samples will be exposed to. Once it reached to the maximum temperature the furnace holds in that temperature over the sintering time. The printed samples were sintered in two different temperatures, 1200°C was the high end of the sintering temperature and 1150°C was the lower end

Materials and volumes

Stainless steels 316 (SS316) is the material used in this study. The sample are printed with stainless steel powder infiltrated with HAp powder. The chemical composition of SS316 is provided in the table 4

Material Composition SS316 % by weight								
C	Mn	P	S	Si	Cr	Ni	Mo	N
0.08	2.00	0.05	0.03	0.75	16.0 - 18.0	10.0 - 14.0	2.0 - 3.0	0.10

Table 4 - SS316

The average density of the stainless steel 316 is 7.99 g/cm^3. The SS316 was mixed with HAp.80% SS316 and 20% HAp by volume composition. Density of the HAp was 3.14 g/cm^3. Some information about HAp can be found in the table 5

Calcium Phosphate Tribasic (HAp)					
Molecular Formula	Molecular Weight	(Form)	Solubility	Phosphorus positive for	Calcium by ICP-atomic emission
$Ca_5(OH)(PO_4)_3$	502.31(g/mol)	Powder	50mg/ML,1 M HCL	Pass	34.0% - 40.0%

Table 5 - HAp Info

Material preparation for printing

SS 316 and HAp was mixed by 80% to 20% by volume. The density of the materials were used to calculate the mass that needed to be mixed.

$$Density\ (D) = \frac{Mass\ (m)}{Volume\ (V)}$$

Accounting the densities of these materials 200 g of stainless steel powder was mixed with the 20.2 g of HAp to achieve the 80% to 20 % volume ratio of the final mixture. Material was measured using a scale and mixed with a plastic stick. Pictures of individual materials and mixed material can be found in the appendix. Multiple batches of this mixture were used to fill the supply and the print bed.

Figure 33 - Print head X1-Lab printed layer

After mixing the materials it were transferred in to print and the supply bed of the X1-Lab machine. 3D model for the samples were created in a CAD drawing using Creo 2.0 CAD software. The CAD

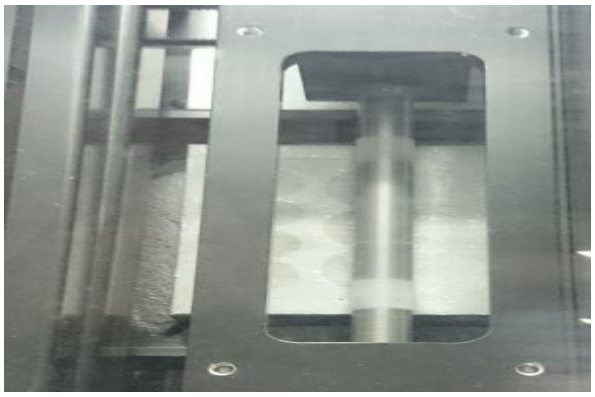


Figure 34 - Roller leveling a layer of powder

model was then loaded in to the X1-Lab™ printer software in STL format. The sample length is 30mm and the diameter is 10 mm. The parameters on the machine were changed in each batch. For an example, in the first batch the roller speed was set at 1 mm/s and the layer thickness was set at 50µm. Three samples were printed at the time. Wear samples and small implant samples were

printed at the same parameters at the same time along with the compression test samples. After each batch was printed they were cured for two hours in the X1 oven at 150°C. Then it was transferred in to the X1 sintering machine.

Pre sinter cure process

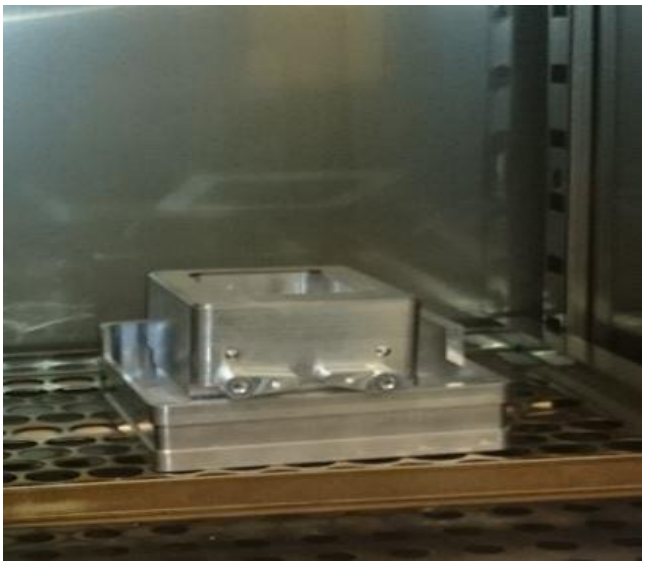


Figure 35 - Printed material in the cure oven

The printed samples were carefully transferred from the printer to the pre heated curing oven. At this stage the samples were only supported by the loose powder around the printed area. They were extremely brittle and had to be handled with care. The samples were cured at 150°C for two to four hours. Then the samples were transferred in to the sintering furnace. Heat insulated pads should be used to handle the curing assembly.

Sintering Process

Figure 36 - Sintered samples

Once the material were cured, they do not need the assistance of the powder bed. Still the samples were considerably brittle so the sample were moved with caution. Small painting brush was used to move the support powder around the sample. The sample should be transferred to the sintering bowl. The parts needs to be covered with the sand. Once the samples were placed in the sintering bowl some more sand should be poured on top. Refer to the picture in the appendix. Then the sintering bowl was placed in the baffle. The sintering machine door was closed to start the process. Then the machine evacuate the air in the sintering chamber. Machine then draw Argon gas from the compressed gas bottle. The sintering machine reaches the final temperature in increment steps. The sintering time and temperature can be set by the user. In this study, samples were sintered at 1150°C or 1200°C. The sintering time periods were 6 hours or 8 hours. Figure 36 show the sintering furnace and the figure 37 shows the printed samples. After the sintering process is completed the sample were ready to be post processed according to the needs of the experiments. After sintering some samples may indicate flaws which make the samples not suitable for post processing or for the experiments. To understand how the materials were sintered in the process, one sample from each experiment was scanned with an electron microscope. The images of this process can be found

chapter 5. Out of 8 experiments 5 of the experiments were crumbled during the printing stage or in the sintering stage. Even though the crumbled experiments were re printed, still they crumbled again. However, three of the experiments were successfully printed and sintered. The samples were then post processed to get them in proper shape and L/D ratio for compression testing.

Figure 37 - Sintering Machine

Post processing samples

Sintered materials were post processed using a lathe. The samples were machined down till it achieved a prefect cylindrical shape. Also the ends were machined till it achieved a perfect flat

Figure 38 - Post processing on the lathe

surface. Due to the porous ness of the samples a high turning speed was used to remove material.

Figure 39 - Post processed ready to test sample

The samples were machined to a Length to Diameter ratio of1.5 to 2.0. The samples were then measured for their length and diameter

Sample Number	Experiment Number	Layer Thickness	Roller Speed	Sintered Temperature	Sintered Time	Dimensions		L/D
		µm	mm/s	C	Hours	Length (in)	Diameter (in)	
1	5	50	1	1200	8	0.785	0.466	1.685
2	5	50	1	1200	8	0.898	0.471	1.907
3	5	50	1	1200	8	0.857	0.442	1.939
4	2	200	1	1200	6	0.865	0.485	1.784
5	2	200	1	1200	6	0.875	0.501	1.747
6	2	200	1	1200	6	0.994	0.496	2.004
7	4	200	10	1150	6	0.874	0.467	1.872
8	4	200	10	1150	6	0.879	0.476	1.847
9	4	200	10	1150	6	1.003	0.501	2.002

Table 6 - Dimensions and Parameters of the test samples

Test Setup

The samples were setup for compression testing on the MTS 810 material testing system. The MTS machine was set to run failure detect mode test. The trigger for the machine was set to monitor the load. When the sample reaches the maximum load it was compressed till the force drops below 85% of the maximum load of the sample and the machine will stop the test.

Figure 41 - Compression setup for a sample

Figure 40 - Compression test

The MTS 810 records load in every 50lb increments. At the same interval it records the stroke and the time. The force was applied to the material in a 1000lb/sec rate. The MTS 810 also records the maximum load for each sample. The bearing surface was cleaned using acetone before placing the sample on the center of the load axis. Then the top bearing surface was brought down to touch the top of the test sample. The safety shield was used as cover for the area between the machine and the user. The test was conducted according to the above sequence.

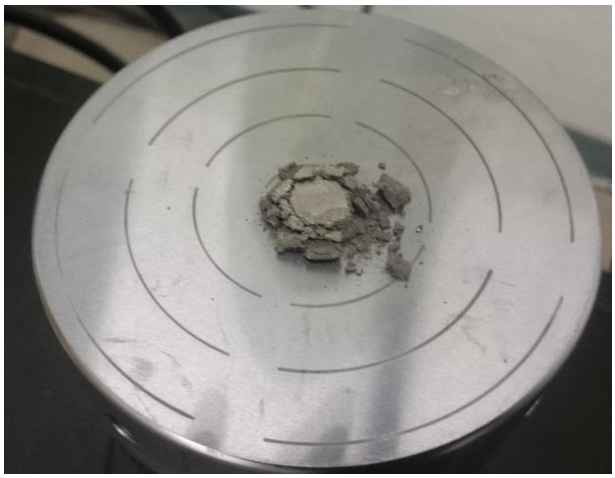


Figure 42 - Tested Sample

Chapter 5

Results

Conversion	
1 KSI	1000PSI

Conversion	
1 KSI	6.89475728MPA

Conversion	
1 MPA	145.037738 PSI

Table 7 - Conversion table

Experiment 2

Sample Length	Diameter of the sample	Area of the sample	L/D
In.	In.	In^2	
0.898	0.471	0.175	1.970

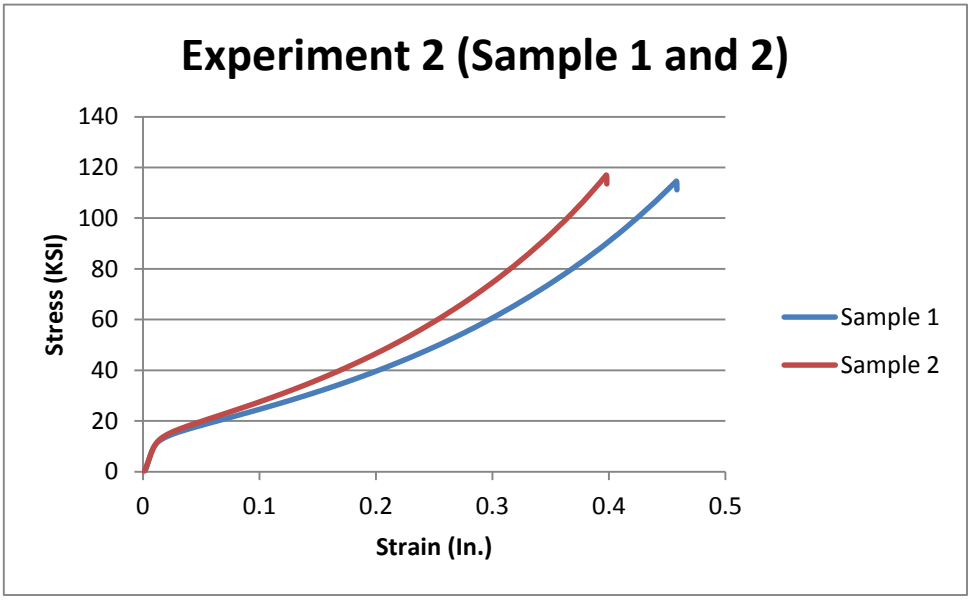
Table 8 -Experiment 2 - Sample Number 1 – Dimensions

Sample Length	Diameter of the sample	Area of the sample	L/D
In.	In.	In^2	
0.785	0.466	0.171	1.685

Table 9 - Experiment 2 Sample Number 2- Dimensions

Layer thickness	Roller speed	Sintering time	Sintering temperature
µm	mm/s	Hours	°C
50	1	8	1200

Table 10 - Parameters of Experiment 2



Graph 1 - Experiment 2 Stress strain plots

The samples in the experiment was crushed at 116.991 KSI and the sample 1 failed at 114.596 KSI. Ultimate compressive strength is calculated using average maximum loads of the failed samples. The experiment two samples were crushed at around 115.844 KSI (798.713 MPA).

Experiment 3

Sample Length	Diameter of the sample	Area of the sample	L/D
In.	In	In^2	
0.874	0.467	0.171	1.872

Table 11 - Experiment 3 Sample number 1

Sample Length	Diameter of the sample	Area of the sample	L/D
In.	In	In^2	
0.879	0.476	0.178	1.847

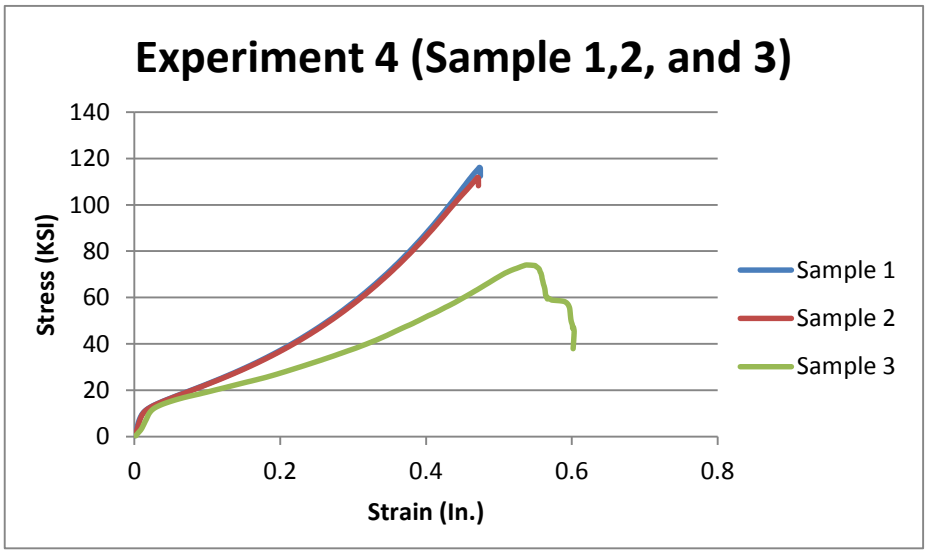
Table 12 - Experiment 3 Sample number 2

Sample Length	Diameter of the sample	Area of the sample	L/D
In.	In	In^2	
1.003	0.501	0.197	2.002

Table 13 - Experiment 3 Sample number 3

Layer thickness	Roller speed	Sintering time	Sintering temperature
µm	mm/s	Hours	°C
200	10	6	1150

Table 14 - Parameters of experiment 3



Graph 2 - Experiment 3 Stress Strain plots

The sample 1 was crushed at 116.225 KSI. Sample number 2 was crushed closer to the sample 1 at 111.923 KSI the sample 3 crushed at 70.577 KSI deviating from the first two samples.

Experiment 5

Sample Length	Diameter of the sample	Are of the sample	L/D
In.	In	In^2	
0.865	0.485	0.185	1.784

Table 15 - Experiment 4 Sample number 1

Sample Length	Diameter of the sample	Are of the sample	L/D
In.	In	In^2	
0.874	0.501	0.197	1.747

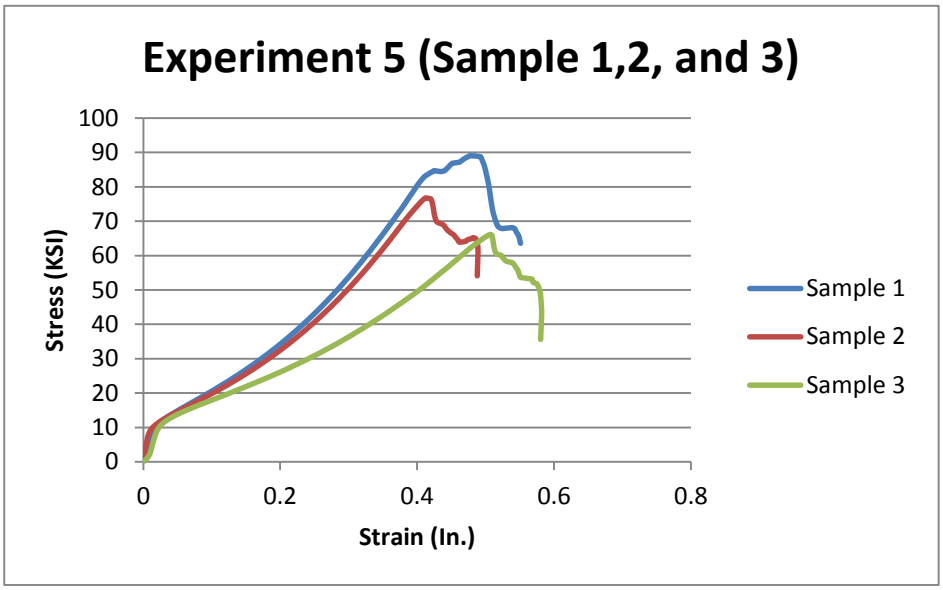
Table 16 - Experiment 4 Sample number 2

Sample Length	Diameter of the sample	Are of the sample	L/D
In.	In	In^2	
0.994	0.496	0.193	2.004

Table 17 - Experiment 4 Sample number 3

Layer thickness	Roller speed	Sintering time	Sintering temperature
µm	mm/s	Hours	°C
200	1	6	1200

Table 18 – Parameters of experiment 4



Graph 3- Experiment 4 Stress Strain plot

The sample 1 crushed at 89.027 KSI. Sample number 2 crushed closer to the sample 1 at 76.810 KSI the sample 3 crushed at 66.141 KSI different from the first two samples.

Chapter 5

Scanning Electron Microscopy (SEM) images of the sintered samples

Experiment 1

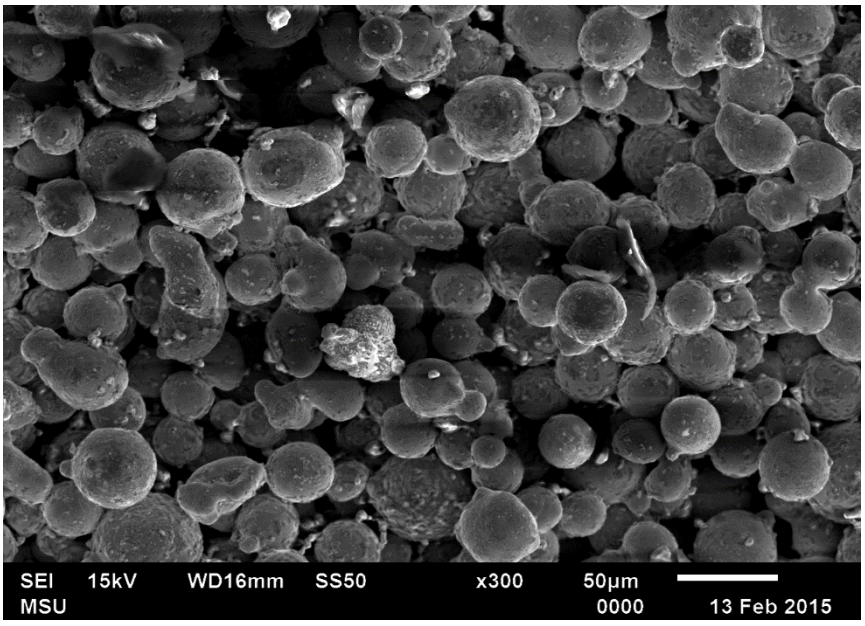


Figure 43 - SEM Experiment 1 (300 Zoom)

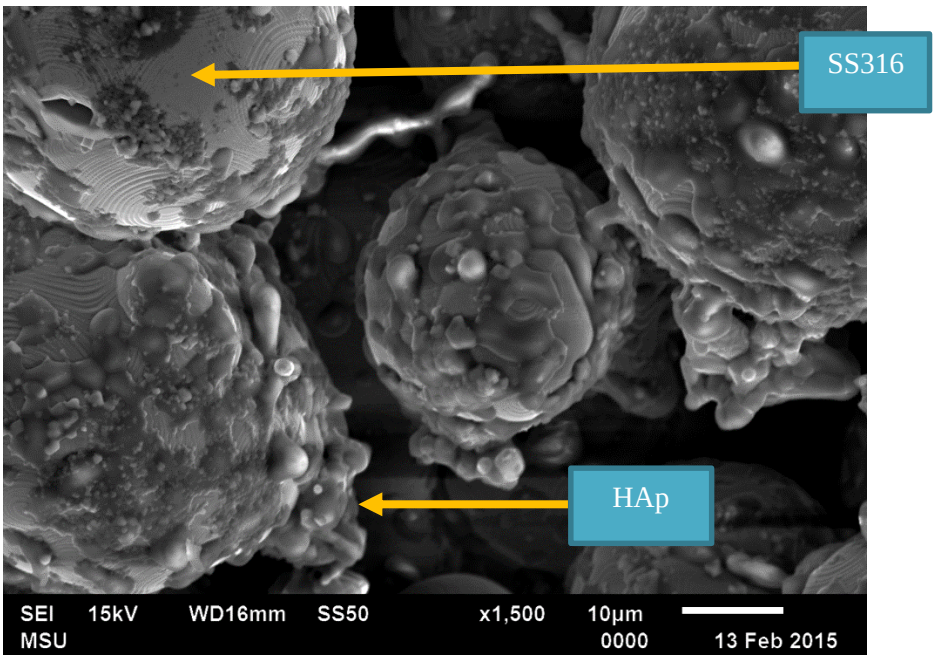


Figure 44 - SEM Experiment 1 (1500 Zoom) HAp is coated around the SS 316

Experiment 2

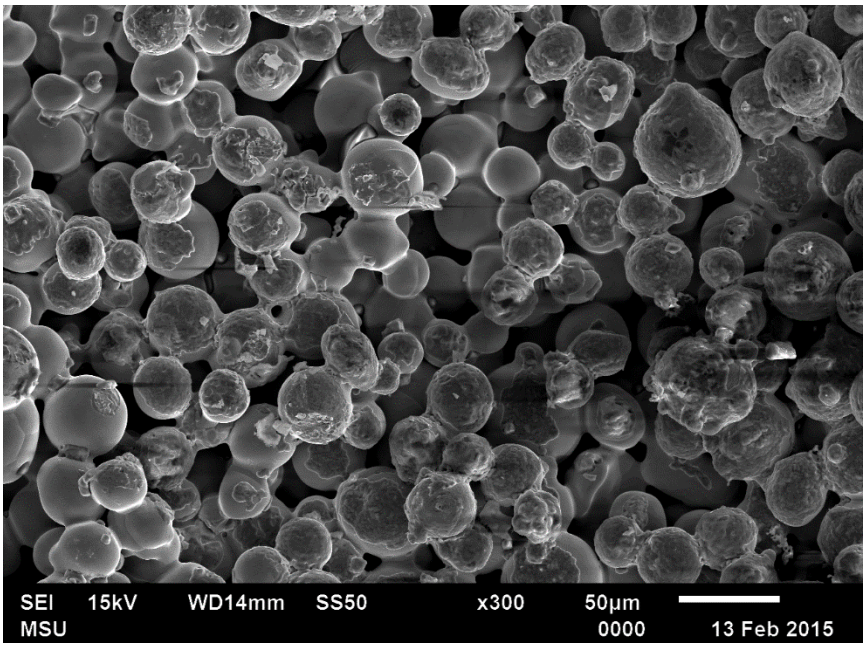


Figure 45 - SEM Experiment 2 (300

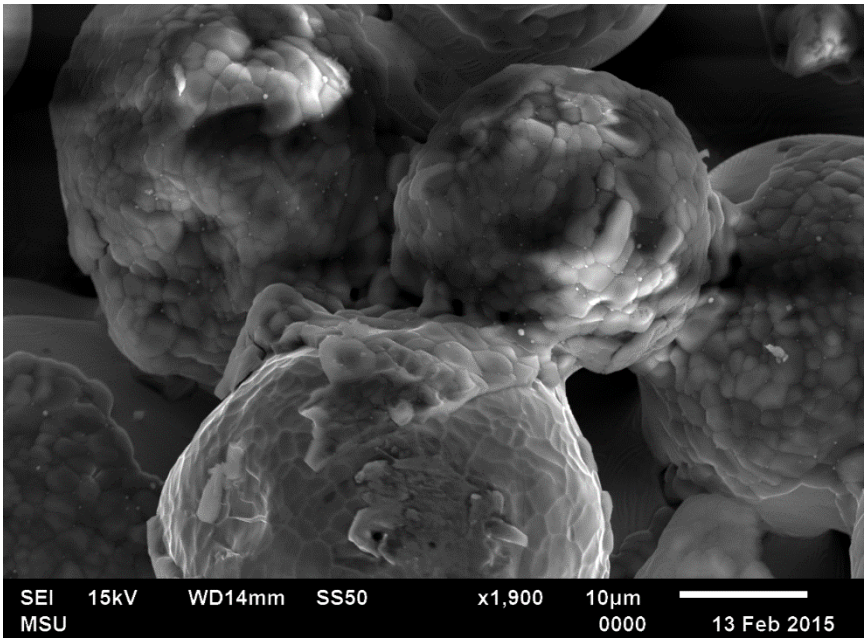


Figure 46 - SEM Experiment 3 (1900 Zoom) Sintered SS316 and HAp

Experiment 3

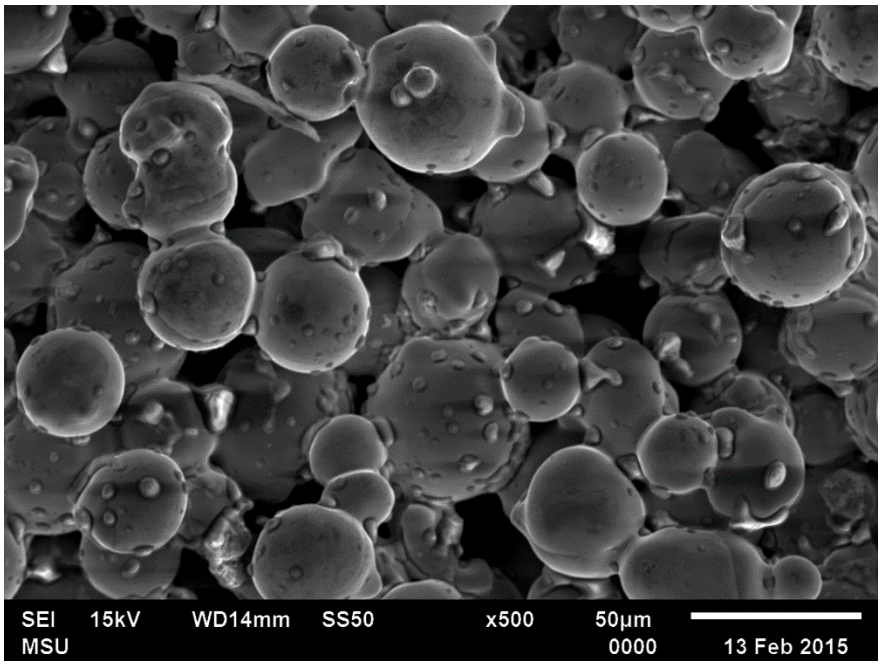


Figure 47 – SEM Experiment 3 (500

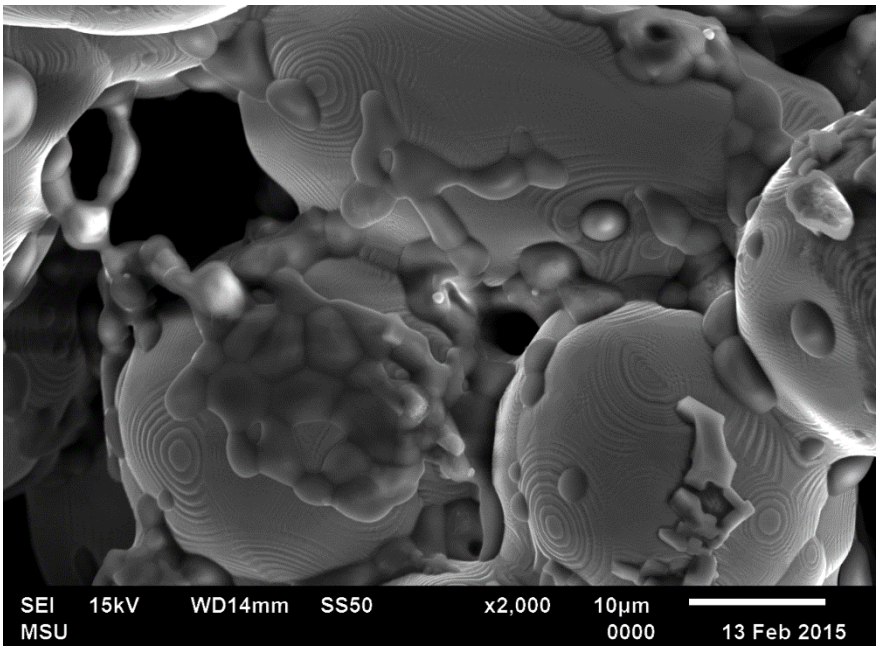


Figure 48 - SEM Experiment 3 (2000 Zoom)

Experiment 4

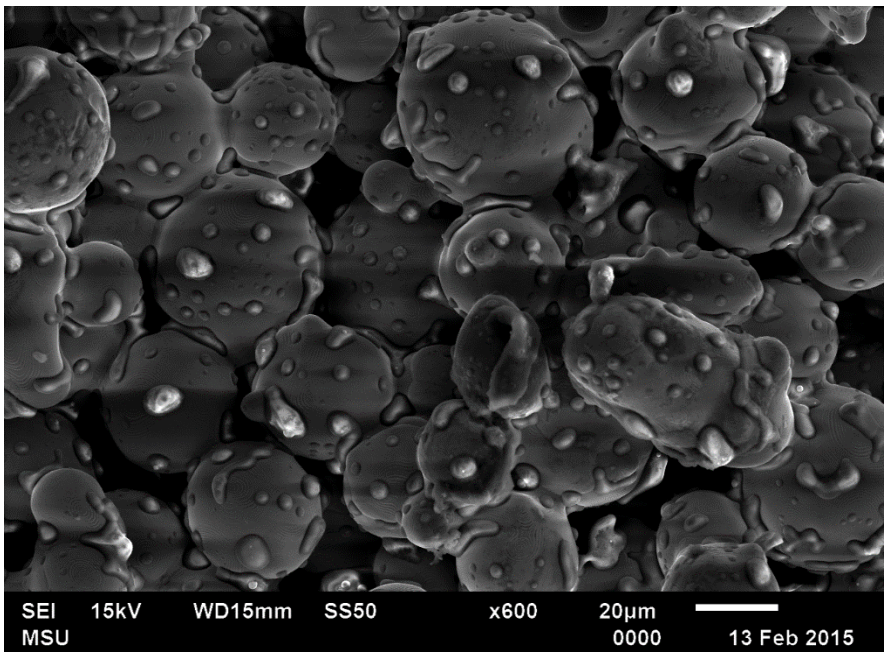


Figure 49 - SEM Experiment 4 (600 Zoom)

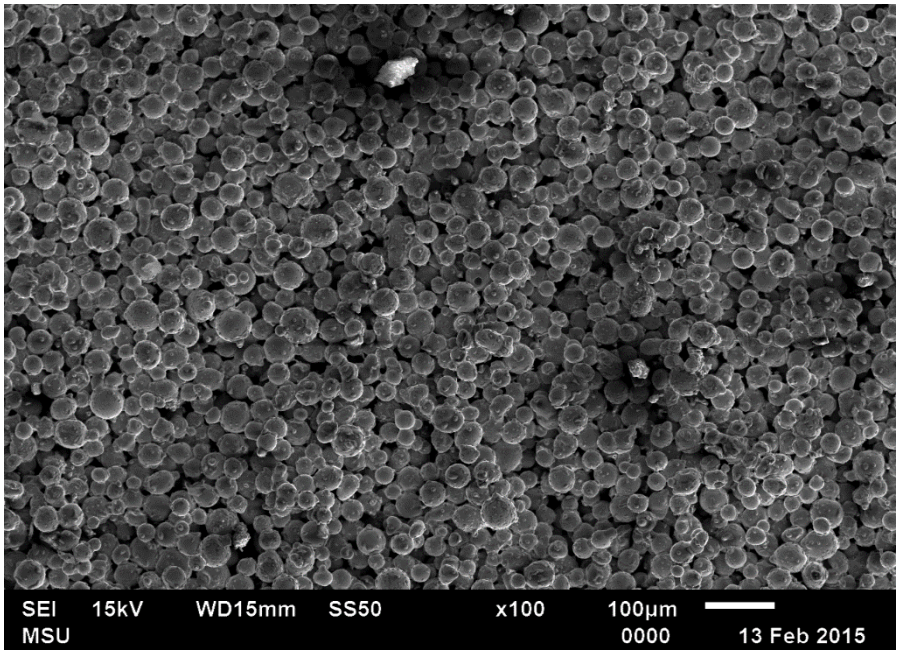
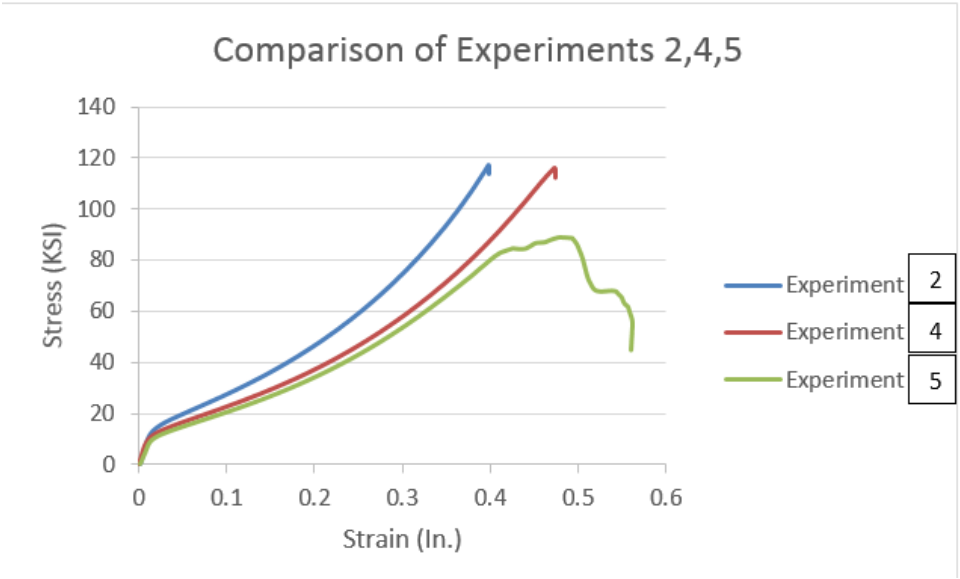


Figure 50 - SEM Experiment 4 (100 Zoom)

Discussion of results

Collected data from three experiments were further analyzed. In experiment 2 both samples showed similar behaviors. The average failure point was at 115.844KSI (798.716MPA). The sample 3 in experiment 4 indicates a higher deviation compared to the other two samples. Due to this reason the sample 3 data was included to calculate the average compressive strength of the experiment 4. The average failure point for experiment 4 was calculated at 114.074KSI (786.513MPA). Sample 3 from the experiment 5 indicates a significant deviation from the data of the other two samples. The sample 3 data was not included when calculating the average failure point in experiment 5 where the average was 82.919 KSI (571.706MPA).

In order to contrast data from different experiments the collected data was plotted in a graph. The sample with the highest failure point was chosen from each experiment for this purpose.



Graph 1 - Experiments 1 to 4 Stress Strain plots

Experiment 2 and 3 show similar compressive strengths even the parameters of the samples were different. The layer thickness, roller speed, sintering time and temperature is different from each other. The experiment 5 indicated a lower failure strength comparing to the experiment 2 and 4.

Comparing the averages of the experiments 2 and 4 with the experiment 5 indicates around 28% reduction of strength. The experiment 4 and 5 have different roller speeds and different sintering temperatures from each other. The experiment 2 and 5 have different layer thickness and sintering times from each other. Out of 8 experiments only 5 experiments were tested from the data obtained from the study is not enough to collude which parameters effected the compressive strength. This study shows parameters of X1-lab binder-jet printer dose effect the overall compression strength of the bio-composite.

Conclusions of the study

A bio-composite can be created with a porous structure using stainless steel316 and calcium phosphate tribasic by additive manufacturing binder-jet printing process. The printed samples were tested for compression strength. The parameters of the binder jet printing. X1-Lab binder-jet printing machine does effect the compression strength and the porous structure of the bio-composite. Identification of which parameters effect the compression strength was inconclusive due to the amount of data from the study.

Further Research

Four further research experiments are suggested by isolating individual parameters to pin point which of them makes the most effect on the ultimate compressive strength. Conducting wear experiments would help to understand how it might affect manufacturing biomedical implants. Also the samples should be tested for bio compatibility to understand rejection factors and tissue growth inside the porous structure of the material. Studies could be conducted by changing the HAp

volume ratio with SS316 to understand how the volume of HAp change the strength of the bio composite.

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Appendix

MTS Calibration Data

Load Cell

```
Sensor File Name: 100kN.scf                                    4/2/2015 3:48:30 PM
Items preceded by an asterisk (*) have been modified.

Application Information
  Name                                                         : Station Manager
  Version                                                      : 5.0E 2325

Conditioner Type                                               : 493.25 DC
Sensor Name                                                    : 661_20E-03
Sensor Serial #                                                : 75293
Conditioner Serial #                                           : 02010709
Dimension                                                      : Force
Last Calibration Date                                          : 10-22-07
Hardware Resource                                              :
General Information                                            :
Polarity                                                       : Normal
Cal Type                                                       : Gain/Linearization
Number of Ranges                                               : 1

  100 kN
    Fullscale Min                                              : -22.481 (kip)
    Fullscale Max                                              : 22.481 (kip)
    Pre-amp                                                    : 240.0
    Excitation (p-p)                                           : 17.998 V
    Post-amp                                                   : 1.0407 (none)
    Fine Zero                                                  : 0.000 V
    DeltaK                                                     : 1.0000 (none)
    Shunt Reference (+)                                        : 17.731 kip
    Neg. Compression                                           : -2.22453 mV/V
    Pos. Tension                                               : 2.22453 mV/V
```

Figure 51 - General sensor data

```
Linearization                                                  : Full Range
Single Range Data                                              : Standard((kip)) Conditioner((kip))
                                                               :       -22.470         -22.481
                                                               :       -15.732         -15.737
                                                               :        -8.997          -8.992
                                                               :        -4.503          -4.496
                                                               :        -2.253          -2.248
                                                               :        -1.803          -1.798
                                                               :        -1.353          -1.349
                                                               :        -0.902          -0.899
                                                               :        -0.451          -0.450
                                                               :         0.000           0.000
                                                               :         0.451           0.450
                                                               :         0.900           0.899
                                                               :         1.351           1.349
                                                               :         1.801           1.798
                                                               :         2.251           2.248
                                                               :         4.503           4.496
                                                               :         9.004           8.992
                                                               :        15.756          15.737
                                                               :        22.508          22.481
```

Figure 52 - Linearization calibration data

LVDT sensor

```
Sensor File Name: LVDT.scf                                  4/2/2015 3:59:28 PM
Items preceded by an asterisk (*) have been modified.

Application Information
  Name                                                      : Station Manager
  Version                                                   : 5.0E 2325

Conditioner Type                                            : 493.25 AC
Sensor Name                                                 : LVDT
Sensor Serial #                                             : 0231477
Conditioner Serial #                                        : 02016888
Dimension                                                   : Length
Last Calibration Date                                       : 10-22-07
Hardware Resource                                           :
General Information                                         :
Polarity                                                    : Normal
Cal Type                                                    : Gain/Linearization
Number of Ranges                                            : 1

  5 inch
    Fullscale Min                                           : -5.0000 (in)
    Fullscale Max                                           : 5.0000 (in)
    Pre-amp                                                 : 1.0
    Excitation (peak)                                       : 10.000 V
    Phase                                                   : 53.0 deg
    Post-amp                                                : 1.1807 (none)
    Fine Zero                                               : 0.000 V
    DeltaK                                                  : 1.0000 (none)
    Neg. Compression                                        : -846.95164 mV/V
    Pos. Tension                                            : 846.95164 mV/V
```

Figure 53 - General sensor data

```
Linearization                                               : Full Range
Single Range Data                                           : Standard((in))  Conditioner((in))
                                                            :        -1.993          -2.000
                                                            :        -0.995          -1.000
                                                            :        -0.496          -0.500
                                                            :        -0.397          -0.400
                                                            :        -0.297          -0.300
                                                            :        -0.198          -0.200
                                                            :        -0.099          -0.100
                                                            :         0.000           0.000
                                                            :         0.099           0.100
                                                            :         0.198           0.200
                                                            :         0.297           0.300
                                                            :         0.397           0.400
                                                            :         0.496           0.500
                                                            :         0.995           1.000
                                                            :         2.000           2.000
```

Figure 54 - LVDT linearization calibration data

Pictures of materials

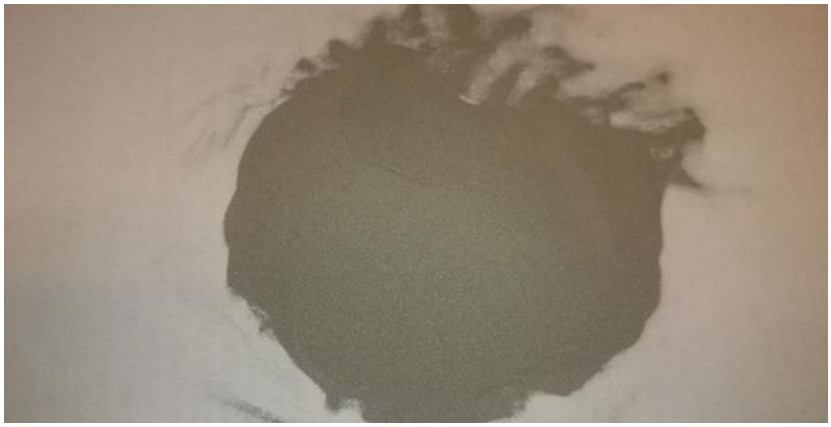

Figure 55 - Stainless steel powder

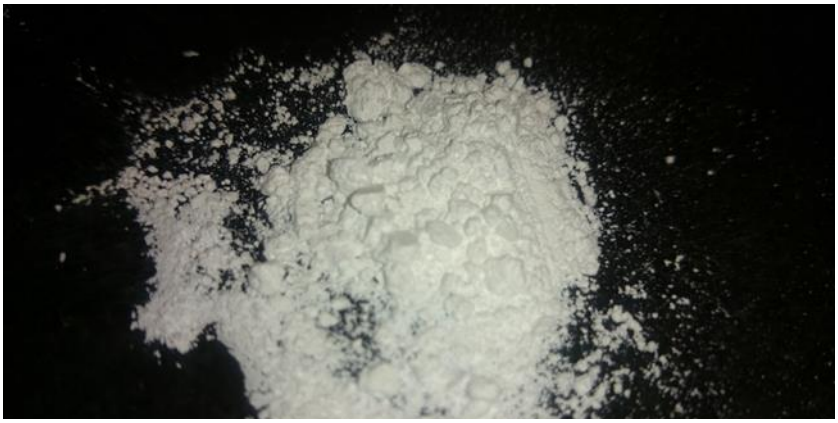

Figure 57 - Calcium Phosphate Tribasic powder

Figure 56 - Calcium Phosphate Tribasic mixed with stainless steel

Cad drawing of the printed sample

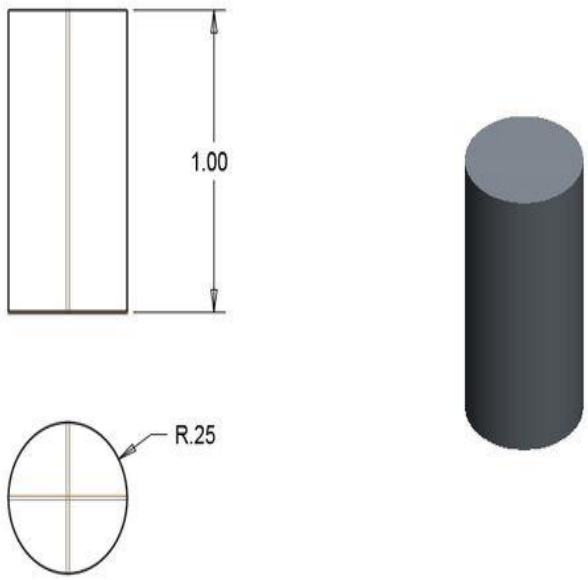

Figure 58 - CAD drawing of the printed sample