FABRICATION OF 316L STAINLESS STEEL (SS316L) FOAM VIA POWDER COMPACTION METHOD

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To my beloved husband Shamsulhafizi bin Ahmad,

My son Muhammad Mujahid bin Shamsulhafizi,

This is for you…

## ACKNOWLEDGEMENT

With the name Allah s.w.t, that gives me the strength to finish up all my research work following the time that has been set. Verily Allah s.w.t almighty and creatures is powerless.

I would like to express my greatest appreciation to my supervisor Assoc. Prof Dr. Sufizar bt. Ahmad for her generous guidance, support with constructive advice, motivation and trust throughout this research.

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# ABSTRACT

Metal foam is the cellular structures that made from metal and have pores in their structures. Metal foam also known as the porous metals, which express that the structure has a large volume of porosities with the value of up to 0.98 or 0.99. Porous 316L stainless steel was fabricated by powder metallurgy route with the composition of the SS316L metal powder as metallic material, polyethylene glycol (PEG) and Carbamide as the space holder with the composition of 95, 90, 85, 80, and 75 of weight percent (wt. %). The powders were mixed in a ball mill at 60 rpm for 10 minutes and the mixtures were put into the mold for the pressing. The samples were uniaxially pressed at 3 tons and heat treated by using box furnace at different sintering temperature which are 870°C, 920°C, and 970°C separately. The suitable sintering temperature was obtained from the Thermal Gravimetric Analysis (TGA). There are several tests that have been conducted in order to characterize the physical properties of metal foam such as density and porosity testing, and the morphological testing (Scanning Electron Microscopy (SEM)), and Energy Dispersive X-ray (EDX). From the result, it can be conclude that, the sintering temperature of 920°C was compatible temperature in order to produce the metal foams which have large pores. Other than that, the composition of 85 and 75 wt. % is the best compositions in order to creates the homogenous mixture and allow the formation of large pore uniformly compared to other compositions which in line with the objective to produce foams with low density and high porosity which suitable for implant applications. The average pore size was within range 38.555µm to 54.498 µm which can be classified as micro pores.

## ABSTRAK

Logam berbusa adalah struktur sel yang diperbuat daripada logam dan mengandungi liang-liang di dalam strukturnya. Logam berbusa yang juga dikenali sebagai logam berliang yang mempunyai sejumlah besar keporosan pada strukturnya dengan nilai sehingga 0.98 atau 0.99. Struktur berliang 316L keluli tahan karat telah difabrikasi dengan kaedah metalurgi serbuk dengan komposisi serbuk logam SS316L sebagai bahan logam, Polyethylene Glycol (PEG) sebagai penguat dan Baja Urea sebagai pemegang ruang dengan komposisi bahan 95, 90, 85, 80, dan 75 peratus berat (wt. %). Kesemua serbuk dicampur dan diadun menggunakan pengisar bebola pada kelajuan 60 rpm dalam masa 10 minit dan campuran dimasukkan ke acuan untuk ke proses penekanan. Sampel dipadatkan dengan tekanan 3 tan dan di sinter menggunakan relau kotak pada suhu yang berbeza iaitu 870°C, 920°C, dan 970°C secara berasingan. Suhu sinter yang sesuai diperolehi daripada Analisis Gravimetrik Haba (TGA). Terdapat beberapa ujikaji dijalankan untuk mencirikan sifat fizikal logam berbusa seperti ujikaji ketumpatan dan keliangan, ujikaji morfologi dengan menggunakan Pengimbas Mikroskopi Elektron (SEM) dan Tenaga Serakan Sinar-X. Daripada keputusan, dapat dirumuskan suhu sintering 920°C adalah paling sesuai untuk digunakan untuk menghasilkan sampel dengan saiz liang yang besar. Selain itu komposisi 85 dan 75 wt. % adalah komposisi terbaik, di mana komposisi ini menghasilkan campuran yang homogen yang membenarkan pembentukan liang yang besar secara seragam di mana selaras dengan objektif asal kajian untuk menghasilkan logam berbusa yang mempunyai ketumpatan yang rendah dan keliangan yang tinggi di mana sesuai untuk aplikasi implan. Purata saiz liang adalah dalam julat 38.555µm hingga 54.498µm dan boleh diklasifikasikan sebagai liang mikro.

# TABLE OF CONTENT

# LIST OF TABLES

# LIST OF FIGURE

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

At present, high requirement of lightweight constituent make metal foams extremely attractive as an industrial technology for biomedical application which is demanding on weight reduction (Gauthier, 2008). The physicality of metal foam is high porosity make metal foams very lightweight. Basically, metal foams are artificial porous medium that has solid matrix structure of metal consist of empty or fluid-filled voids. Metal foams have been characterized into two types which are open-cell and closed-cell. The foams is called open-cell when the voids are connected via open pores, but when the foams are separated by the solid walls and not connected via open channel are described as closed-cell (Dukhan, 2013).

In order to achieve the metal foams which suitable in orthopedic application and have good properties such as low density, high strength-to-weight ratio, excellent mechanical properties, biocompatibility and corrosion resistance, the suitable materials have to choose carefully. Metals are the most suitable material to fabricate the metals foam proportionate to the ceramic and polymer. Even though the ceramic material have excellent corrosion resistance but ceramic cannot being employed as load bearing implants due to their brittle properties, whereas polymeric systems cannot sustain the mechanical forces present in joint replacement surgery (Ryan, Pandit & Apatsidis, 2006). There are various types of metal that have been used as main materials to fabricated metal foams includes titanium, titanium alloys, nickel, aluminum, magnesium, and stainless steel(Rosip *et al.*, 2013).

Since early 1960s, Stainless steel widely used in orthopedic application such as fabrication of femoral stems, balls and acetabular cups, fabrication of knee and femoral components and tibial trays because of its biocompatibility and inexpensive (Davis, 2003). Porous Stainless steel is compatible to use as a coating on Stainless steel implants. The methods that use to fabricate these coating are by sintering beads or thermal spray techniques. The oxides that formed on the surfaces of stainless steel is more stable than the oxides formed if using titanium and titanium alloys and leads to the crevice corrosion and degradation of the implants. Because of that, Stainless steel is choosing to replace other materials over the year. Stainless steel has been approved in terms of mechanical properties and clinical trials by the US Food and Drug Administration (FDA). Its mechanical properties are often used as bench mark criteria to evaluate other alloys for stent applications (Chen *et al.*, 2014).

There are large varieties of fabrication techniques for metal foams or similar porous structures but usually favorable technique is liquid phase or powder metallurgy process. By using compaction method, metal powders are mixed with foaming agent and then compacted by using hot pressing, cold pressing, hot extrusion, or co-extrusion. The final product of the compaction process is a dense foamable material that can be worked into sheets and profiles (Banhart & Baumeister, 1998) Slurry method are commonly used to produce metal foams by providing metal powder, blowing agent and a binder that mix together and the mixture poured into mould and left to the elevated temperature until melting temperature(Rosip *et al.*, 2013). Casting process also has been used to produce metal foams around inorganic hollow spheres with a liquid melt or using open porosity polymer foams as starting points.

This research is to fabricate the 316L Stainless Steel (SS316L) foam prepared by Compaction technique and to study and characterize the properties of SS316L foam after sintering process. The SS316L have used as a raw material and Polyethylene glycol (PEG) and Carbamide are used as a binder and space holder respectively. The material will be mixed by using ball milling machine to get the homogenous mixture. After that the compaction process will be held by using conventional axial pressing. This process is known as powder metallurgy technique. The Properties Characterization will be measured by doing density and porosity test, Thermal Gravimetric Analysis (TGA), Scanning Electron Microscopy (SEM), and Energy Diffraction X-ray (EDX).

## 1.2 Problem Statement

The major challenges that need to focused while producing metal foam is the mismatch of the properties between bones and the metallic material. Due to this mechanical mismatch, bone is insufficiently loaded and become stress shielded, which eventually leads to bone resorption (Ryan et al., 2006).

Thus, there are factors need to be considered includes the interconnecting pores that suitable with bone, the pores of the implants same with the pores of bone, the shape and the density of implants is same with the shape and density of bones.

## 1.3 Objectives

The objectives of this research are:

i) To fabricate the 316L Stainless Steel (SS316L) as metallic cell prepared by Compaction technique.

ii) To characterize the properties of fabricated SS316L foam after sintering process.

## 1.4 Scope of Study

The scope of this research includes:

i) The percentage of the composition for the SS316L powder are 95, 90, 85, 80 and 75 of weight percent (wt. %) respectively, for the Polyethylene glycol is 1 of weight percent (wt. %) and balance is for the Carbamide composition.

ii) The sintering temperatures that have been choosing are 870$^{O}$C, 920$^{O}$C, and 970$^{O}$C.

iii) The characterization for the properties of metal foam will be study by conducted the Thermal Gravimetric Analysis (TGA), Density and porosity test, Scanning Electron Microscopy (SEM), and Energy Diffraction X-ray (EDX).

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Introduction

This chapter justify about literature review of the research to gather information and knowledge of the material that need to be used in this research, the method that have been used in previously study and the method that has been select in this research. This section focused on the Stainless Steels and its properties, the binder and space holder that has been used and the fabrication method that involved.

### 2.1.1 Metal Foam

Basically, metal foam can be described as the cellular structure that made from metal and have pores in their structures. Metal foam also known as the porous metals, which express that the structure has a large volume of porosities with the value of up to 0.98 or 0.99. The high porosity contributes lightweight to the metal foams. Besides, the terms foamed metal or metallic foam illustrate the fabrication or forming process of the metal foam.

Metal foams have been characterized into two types of structure which are open cell and closed cell. The structure of the porous metals influences the applications of the metal foams. Closed cell foam can be described as the pores is fill with gas and separated from each other by metal cell walls, have good strength and usually used in structural application. Open cell foam contain a continuous network of metallic struts and the enclosed pores in each strut frame are connected, the strength are weaker than the closed cell and are mainly used in functional applications where the continuous nature of the porosity is exploited (Kennedy, 2012). Figure 2.1 show the example of the microstructure of closed cell foam and open cell foam for metal foams.

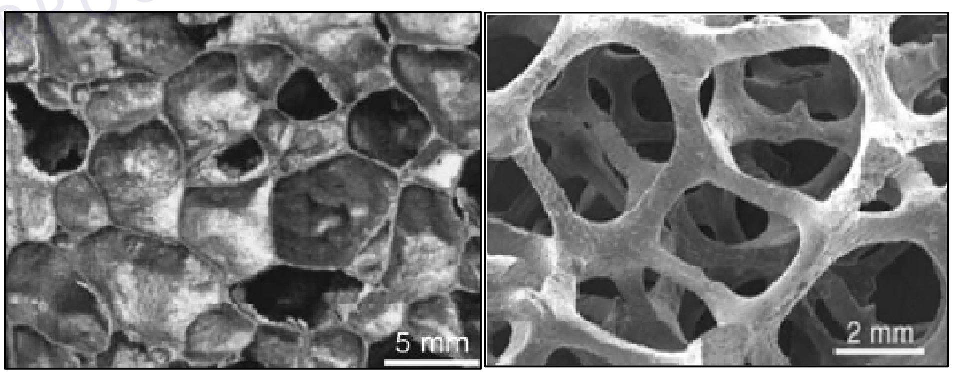


Figure 2.1: Microstucture of (left) a closed cell foam and (right) an open cell foam (Kennedy, 2012)

There are other application of metal foams such as energy absorber, heat exchangers, mechanical damping and in filters system. Nowadays, metal foams has been use in biomedical application as bone implants. Metal foams have excellent potential for implant application due to its low density and good combination of properties because of the reduced stiffness mismatches. Other than that, it is important to make sure the bone ingrowth which possible by metal foams and greatly improve the bone implant interface and may allow for efficient soft tissue attachment supplementing the stability of the implant by biological fixatation (Mariotto *et al.*, 2011).

## 2.2 Metallic Material

The excellent of electrical and thermal conductivity and mechanical properties make metal used as biomaterials which suitable used as biomedical materials. Biomaterial can be expressed as any material used to make devices to replace a part or a function of body in a safe, reliable, economic, and physiologically acceptable manner (Park & Lakes, 2007). Metals offer excellent strength and resistance to fracture, which suitable for the medical application that requiring load bearing.

There are number of metallic material that good biocompatibility which are not cause serious toxic reaction in human body such as Stainless Steels, Cobalt Alloys, Titanium Alloys and noble metals. These metals are suitable for the structural application in the body such for implants for hip, knee, ankle, shoulder, wrist, finger, or toe joints. These metals have the ability to bear significant loads, withstands fatigue loading, and undergo plastic deformation prior to failure, made these metals are popular chosen as material for the implant application (Shi, 2006).

Some metals have high corrosion resistances which made its suitable used as passive substitutes for fracture healing aids as bone plates and screws, spinal fixation devices, and dental implants. Corrosion can be defined as the unwanted chemical reaction of a metal with its environment, resulting in its continued degradation to oxides, hydroxides, or other compound. It is important to choose metallic materials which have high corrosion resistance due to its biocompatibility in human body.

Other than that, metallic material play active roles in devices such as vascular stents, catheter guides wires, orthodontic arch wires, and cochlea implants (Park & Lakes, 2007). Figure 2.2 show the typical implant applications for orthopedic purposes.

During the twentieth century, the first metallic material was successfully used in orthopedic applications were Stainless Steels and Cobalt-Chrome Based Alloys (Navarro *et al.*, 2008). The Stainless Steels and Cobalt-Chrome Based Alloys have high corrosion resistance based on the presence of Chromium in their properties make it has the ability to render the alloys passive. In this research, the metallic material that has been used is Stainless Steels which will be explained later.

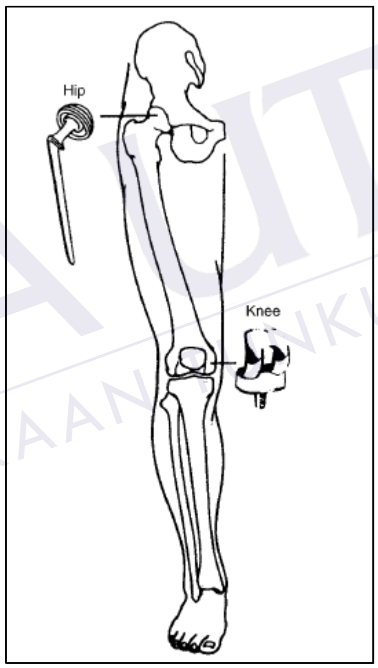


Figure 2.2: The typical implant applications for orthopedic purposes (Davis, 2003)

### 2.2.1 Stainless Steels

Stainless Steels are widely used in modern metallurgy. Historically, Stainless Steel was discovered in early 18$^{th}$ and 19$^{th}$ centuries; when the identification of Chromium as an element was begin. Harry Brearley (1871-1940) who first produced the first commercial cast of martensitic Stainless Steel, which used for cutlery area (Cobb, 2010).

Different from other alloy systems that has widely used such as Carbon Steels, Alloy Steels, and Aluminum Alloys, that are relatively dilute solution of several element in the parent matrix. Stainless Steel is one of alloys system that it is not a dilute solution. There are several percent of alloying element that contained in Alloys steels includes Carbon, Manganese, Nickel, Molybdenum, Chromium, and Silicon, Impurities Sulfur, Oxygen, And Phosphorus. Typically, Alloy Steels contain very small of amounts of Titanium, Niobium and Aluminum. In contrast, in about 11% of Chromium that contain alone in Stainless Steel (McGuire, 2008). Usually, the shape of Stainless Steels particles is irregular. Figure 2.3 show the basic shape of metal powder (Kalpakjian & Schimid, 2006).

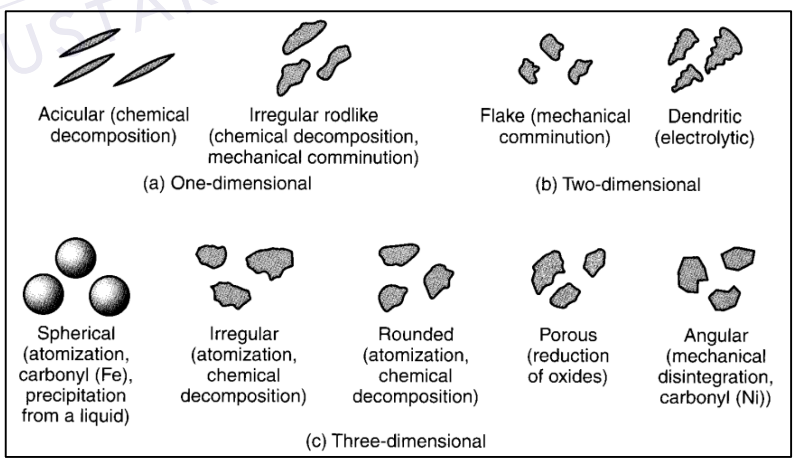


Figure 2.3: Basic shape of Metal powder (Kalpakjian & Schimid, 2006)

Basically, Stainless Steel is Iron-Base Alloys that contain a minimum of approximately 11% of Chromium (Cr), amount that needed to prevent the formation of rust in unpolluted atmosphere. There are few Stainless Steels contain more than 30% Cr or less than 50% Ferum (Fe). They fulfill the Stainless Steel characteristics condition by the formation of an invisible and adherent Chromium rich oxide surface film. The process is happen when the oxide forms and heals itself in the presence of oxygen (Davis, 1994).

There is development of Stainless Steels that used in implant application. The first Stainless Steel that has been recorded that utilize as implant fabrication was the 18-8 type which is stronger and more resistance to corrosion than the Vanadium Steel. There are some improvement has been done to Stainless Steel by attached a small percentage of Molybdenum to improve the corrosion resistance in Chloride solution which next the Stainless Steel has known as type 316 Stainless Steels. During 1950s, they upgrade the Stainless Steel by reducing the amount of Carbon content from 0.08 to a maximum amount of 0.03% to increase corrosion resistance and minimize the sensitization and become as type 316L Stainless Steels. The Chromium content also function as the reduction of Carbon content in which to impart corrosion resistance in Stainless Steel which the concentration of Chromium is 11% (Wong & Bronzino, 2007).

The type of Stainless Steels that most widely used for implant fabrication is the austenitic Stainless Steels which is type 316 and 316L. These groups of steel are nonmagnetic and have better corrosion resistance than others because the content of Molybdenum that enhances the resistance while attach in the salt water. The American Society of Testing and Material (ASTM) recommend type 316L rather than type 316for fabrication for implant. The standard composition of 316L Stainless Steel that follows American Society for Testing and Materials is show in Table 2.1 (Wong & Bronzino, 2007).

Table 2.1: Standard composition of 316L Stainless Steel (Wong & Bronzino, 2007)

| **Element** | **Composition (%)** |
|---|---|
| Carbon | 0.03 max |
| Manganese | 2.00 max |
| Phosphorus | 0.03 max |
| Sulfur | 0.03 max |
| Silicon | 0.75 max |
| Chromium | 17.00-20.00 |
| Nickel | 12.00-14.00 |
| Molybdenum | 2.00-4.00 |
| Ferum | Balance |

Mariotto *et al.*, (2011) in the study of fabrication metal foam for biomedical application have used Stainless Steel type 316L as main material. Ammonium Carbonate and Ammonium Bicarbonate is chosen as foaming agents that will mix with the Steel powder. The technique that has been applied to fabricate the metal foam is by using powder metallurgy techniques and the metal foam vacuum heat treated in 2 stages, the first stage at 200°C for 5 hours to decompose the carbonate and the second stage at 1150°C for 2 hours to sinter the steel. From the research, the density of the metal foam is in about 0.3 g.cm$^{??}$ and 0.5g.cm$^{??}$ for Ammonium Carbonate and Ammonium Bicarbonate foaming agents respectively. The microstructure of the metal foam is heterogeneous pore structures, composed by irregular and isolated pores. The result show that the metal foam produced can be viable in orthopedic implants (Mariotto *et al*., 2011).

The previous researchers produce of 316L Stainless Steels foam via slurry method. In the research, the 316L Stainless Steel is chosen as metallic material, where the Polyethylene Glycol (PEG) and Methylcellulose (CMC) were used as binder. The result shown that, the SS316L metal foams had been successfully produced by using slurry method. The microstructure observation show that the metal foams had closed pore than open pore and the powder particles did not well growth to combine each other after sintering process. At the end of the research, the suitable composition of SS316L for metal foams fabrication is gained with the suitable sintering temperature which resulted better strength of metal foams (Rosip *et* *al.*, 2013). Gauthier (2008) in his study of structure and properties of open-cell 316L Stainless Steels foams produced by a powder metallurgy-based process used type 316L Stainless Steel as main material, a polymeric binder, and chemical foaming agent. All the material was dry mixed, then the mixture was molded and heat treatment is applied to the metal foam. From the result of the investigation, the 316L Stainless Steels foams was produced and the heterogeneous foams feature a mix of open and closed pores depending on processing conditions. The mechanical properties that get from the test show that the value is following the scaling for metal foams and was related to foam density (Gauthier, 2008).

In this research, the 316L Stainless Steels is choosing as main material. The characteristic of the 316L Stainless Steel will be elaborate later in the next topic.

### 2.2.2 Designation of Stainless Steels

The designation of Stainless Steels can be divided into three different designation systems in principle which are the designation of wrought Stainless Steels, cast Stainless Steels and P/M Stainless Steels. Basically, in United States the American Iron and Steel Institute (AISI) numbering system and the Unified Numbering System (UNS), are design the wrought grades of Stainless Steels. Other major industrial have also been established other designation system of Stainless Steels but it also similar to the AISI. Basically for wrought Stainless Steels, the UNS designation consist of the letter “S”, follow by a five-digit number whereas for those alloys that have an AISI designation, the first three digit of the UNS designation usually correspond to an AISI number. The UNS designation consists of the letter “N” followed by a five-digit number when Stainless Steels that contain high Nickel content (>25 to 35%) (Reardon, 2011).

There are institutions which call as High Alloy Product Group of the Steel Founder’s Society of America, also known as the Alloy Castings Institute (ACI), has designated standard cast Stainless Steels grades. The composition and properties of the Stainless Steels similar to the wrought grades, and some cast Stainless Steels are modified in order to improved cast ability and the higher silicon levels are typically

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