

DEVELOPMENT OF STAINLESS STEEL FOAM WITH HA BIO ACTIVE
CERAMIC ADDITION USING SLURRY METHOD

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ABSTRACT

This present study focuses on fabricating of Stainless-Steel Foam with Bio Active Ceramic addition using slurry method. The mechanical and physical properties of stainless steel is still not perfectly adequate for medical implants requirements. Therefore, the slurry method is mainly focused on increasing the mechanical and physical properties of the stainless-steel foams with hydroxyapatite addition. The samples were produced with different compositions of SS316L and HA powders were mixed together with binders which were polyethylene glycol (PEG) and carboxymethyl cellulose (CMC) to prepare slurry solution for impregnation of polyurethane (PU) sponge as the template. SS316L-HA foam were fabricated by slurry method and sintered at various sintering parameters. The physical and mechanical properties were determined for each sample of SS316L-HA foam by analysis of shrinkage, apparent porosity, apparent density, compressive strength and microstructure analysis. Following optimization, the SS316L-HA foam produced was found to have attractive mechanical and physical properties much like human bone. The suitable percentages of HA via slurry method are 1%-5%. Notwithstanding, this included the average shrinkage occurs at the sintering temperature of 1350°C with 1wt% of Hydroxyapatite are 26.54%. The average porosity was obtained at temperature 1150°C with 59.88% of 3wt% of HA. The average density obtained for SS316L-HA foam are 3.83g/cm³, sintered at 1350°C of 1wt.% HA. The maximum value of yield strength was 18.85Mpa with 10wt.% HA, sintered at temperature 1300°C. The maximum value of modulus elasticity was 0.49GPa with 10wt.% HA, sintered at temperature 1150°C. The average pore size for all SS316L-HA foam samples are in the range of 310 µm to 386µm. These results are fulfill the objectives of development of stainless-steel foam with bio active ceramic addition using slurry method.

ABSTRAK

Kajian ini memfokuskan kepada SS316L dengan penambahan Seramik Bio Aktif menggunakan kaedah buburan. Sifat mekanikal dan fizikal keluli tahan karat masih tidak mencukupi untuk keperluan implan perubatan. Oleh itu, kaedah buburan terutamanya tertumpu pada peningkatan sifat mekanikal dan fizikal buih keluli tahan karat dengan penambahan HA. Objektif kajian ini adalah pertama, mengenal pasti peratusan HA yang sesuai melalui kaedah buburan. Kedua, optimumkan suhu pensinteran yang sesuai untuk SS316L-HA. Ketiga, pengaruh pengesanan penambahan HA dan suhu pensinteran ke atas sifat fizikal dan mekanikal buih SS316L-HA selepas proses pensinteran. Sampel telah dihasilkan dengan komposisi berbeza serbuk SS316L dan HA dicampur bersama pengikat iaitu polietilena glikol (PEG) dan karboksimetil selulosa (CMC) untuk menyediakan larutan buburan untuk impregnasi span poliuretana (PU) sebagai templat. Buih SS316L-HA telah direka dengan kaedah buburan dan disinter pada pelbagai parameter pensinteran. Sifat fizikal dan mekanikal ditentukan untuk setiap sampel buih SS316L-HA dengan analisis pengecutan, keliangan ketara, ketumpatan ketara, kekuatan mampatan dan analisis struktur mikro. Berikutan pengoptimuman, buih SS316L-HA yang dihasilkan didapati mempunyai sifat mekanikal dan fizikal yang menarik seperti tulang manusia. Peratusan HA yang sesuai melalui kaedah buburan ialah 1%-5%. Walau bagaimanapun, ini termasuk pengecutan purata berlaku pada suhu pensinteran 1350°C dengan 1wt% Hydroxyapatite ialah 26.54%. Purata keliangan diperolehi pada suhu 1150°C dengan 59.88% daripada 3wt% HA. Purata ketumpatan yang diperolehi untuk buih SS316L-HA ialah 3.83g/cm³, disinter pada 1350°C 1wt.% HA. Nilai maksimum kekuatan hasil ialah 18.85Mpa dengan 10wt.% HA, disinter pada suhu 1300°C. Nilai maksimum keanjalan modulus ialah 0.49GPa dengan 10wt.% HA, disinter pada suhu 1150°C. Purata saiz liang untuk semua sampel buih SS316L-HA adalah dalam julat 310µm hingga 386µm. Keputusan ini memenuhi objektif pembangunan buih keluli tahan karat dengan penambahan seramik.

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PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF SYMBOLS AND ABBREVIATION

SS316L	-	Stainless Steel
HA	-	Hydroxyapatite
PU	-	Polyurethane
PEG	-	Polyethylene Glycol
CMC	-	Carboxymethyl Cellulose
SEM	-	Scanning Electron Microscope
UTHM	-	University Tun Hussein Onn Malaysia
CaP	-	Calcium phosphate
CDA	-	Calcium-deficient apatite
β -TCP	-	Beta-tricalcium phosphate
Ti	-	Titanium
W_w	-	Wet weight
W_d	-	Dry weight
W_s	-	Soak weight
ρ_w	-	Density of water
l_o	-	Length before sintering
l_i	-	Length after sintering
ϵ	-	Strain
σ	-	Stress

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Stainless steel (SS316L), like titanium and titanium combinations, is generally utilised as the most well-known loading-bearing implant for its high corrosion resistance, low cost, mechanical properties, and excellent biocompatibility (Thanh *et al.*, 2013). Since 1660, implants have been used for several purposes. The current use of implants in dentistry and medicine began in 1960 (Kurgan *et al.*, 2012). Stainless steel 316L (SS316L) remains widely used and extends from cardiovascular to otorhinology and dental. SS316L is used as an orthopaedic bone fixation implant, orthodontic wire, cardiovascular stent implant and prostheses in dentistry. SS316L is well known for producing biomedical applications (Hermawan 2011; Ding *et al.*, 2010; Roguska *et al.*, 2011).

The concept of bioactive material is intermediate between inert and resorbable materials. Although several definitions of biological activity have been reported, one is particularly simple Shi *et al.* (2021) describing the properties of an implanted material which allows it to bind to living tissues. Bio ceramics describe a host of ceramics (including bioactive glasses) specially designed and manufactured for the repair and reconstruction of diseased, damaged, missing or worn-out parts of the body” (Fu *et al.*, 2020). Calcium phosphate (CaP) bioceramics include an intimate mixture of calcium-deficient apatite (CDA), hydroxyapatite (HA), beta-tricalcium phosphate (β -TCP) and biphasic calcium phosphate (BCP) – an intimate mixture of HA and β -TCP. Bioactive bioceramics are available as powders, granules, pellets, and blocks

(dense or porous), as cement (CPC), as composites (CaP/polymer), and as coatings on orthopedic and dental implants (Bala *et al.*, 2017).

Biomaterial research, according to Williams, is a study of the structure and properties of biomaterials, the mechanisms that interact with biological systems, and their clinical outcomes. Biomaterial are defined as materials intended to interface with biological systems for evaluating, treating, improving, or replacing body tissue, organ, or function (Manam *et al.*, 2017). Hydroxyapatite (HA) is a ceramic made of calcium phosphate, which has a similar shape and composition to human bones and teeth. Hydroxyapatite is commonly used as a bone replacement or dental implant because of its good properties, such as high biocompatibility, osseo conductivity, and bio affinity to living tissues. HA's low mechanical properties, such as its brittleness and low fracture durability, limit its use in load-bearing applications. Therefore, metallic materials are used to enhance the mechanical properties of metal-ceramic composites (Ramli *et al.*, 2014).

In order to replace the cancellous bone, there are specific features that generally need to be fulfilled by metal implants that include interconnecting pores of about 30-90 percent with a pore size of 100-600 μm to provide room for cell migration and new growth tissue, and a young module that is close to the cancellous bone, $<3 \text{ GPa}$ (Cachinho *et al.*, 2007). If a hard implant is inserted into hard tissues (e.g., bones), the bones is ultimately exposed to a minimized mechanical stress that contributes to bone absorption. Therefore, this phenomenon is known as a stress shielding effect that results in the death of bone cells (Leel *et al.*, 2013).

Several composites of metal-hydroxyapatite were tested. Particular attention is paid to composites materials such as titanium and austenitic stainless steel. Based on an analysis of the results obtained, it can be assumed that the application of SS316L stainless steel to hydroxyapatite results in an increase in stiffness and strength compared to pure bioceramic hydroxyapatite. However, it resulted in improved biocompatibility, corrosion resistance, and stainless-steel hardness and wear resistance (Szewczyk-Nykiel, 2015).

For a wide range of applications, metal foams with visible, and interconnected pores is very attractive. For example, in the automotive industry, as a catalytic converter, as a heat insulator in an industrial furnace, and as a bone implant in biomedical. Aluminium foam with open pore structures can be produced using several manufacturing methods, including the space holder process, combustion replication

methods, liquid deposition, and rapid prototyping. The sponge replication process has been shown that in all ways foam with open interconnected holes close to the spongy bone structure can be produced (Noor *et al.*, 2019).

Replication, or slurry dipping, is a simple and common way to produce porous and interconnected foam using a ceramic slurry. With the presence of the central space in the struts and microspores and micro-cracks due to uneven ceramic slurry coating, this process creates scaffolding with weak mechanical properties (Muthutantri *et al.*, 2018). The preparation of stainless-steel foam from the slurry of stainless steel is not simple. During the slurry preparation, there are many parameters are involved that affect the viscosity of the slurry, such as the composition of the material, the mixing method, the type of binder, and the type of solvent used. Particularly when using the sponge replication method to fabricate the foam, it is important to produce a stainless slurry with sufficient stability and viscosity. Full impregnation of stainless-steel slurry on PU foam templates could only be achieved with specific slurry viscosity. It is very difficult to produce stainless-steel slurry with low viscosity and high solid quality. A low viscosity slurry is required to allow the slurry to penetrate easily into the pores of the PU foam and require a high solid content is required to improve mechanical stability during sintering. (Noor *et al.*, 2019).

1.2 Problem Statement

Stainless steel was used as an important material in surgical field. Stainless steel is a generic term for many different steels mainly used for their resistance to a wide range of caustics (Patel *et al.*, 2012). Metals have generally been used in large orthopedic load-bearing applications (Park *et al.*, 2002). Some non- metallic materials have also been proposed as artificial bone candidates, but a wide range of applications have not been identified.

In the production of metal foam structures, there are many methods that can be used to produce metal foams. However, there is no detailed method that can be used to produce foam metal depending on the type of metal used. Some methods are not suitable for the use of substances. Therefore, it is essential to select a method suitable for the production of foam metal. The formation of cavities in the density, size, shape, and properties of metal foams has been one of the often-noted problems in metal foam production. Slurry method is a commonly used technique because it is one of the

techniques that has many advantages in the production of metal foams. This way you can produce good quality products. According to Yu *et al.* (2021), slurry method is one of the methods for producing metal foams. This method is very suitable for producing uniform bubbles and open cells. This slurry method is expected to produce high-quality products in the formation of SS316L-HA.

Figure 1.1 shows the elastic modulus of most materials that are currently used in biomedical applications. The diagram clearly shows that the elastic modulus of cancellous and cortical bone is very low compared to the elastic modulus of metal implants, especially in stainless steel. This causes stress shielding that occurs on interfaces that affect the long-term stability of the implant (Shah *et al.*, 2019). In addition, porous metal can provide space for bone ingrowth to achieve biological fixation. Nowadays, several metallic implants have been replaced by ceramic and polymer implants due to their superior biocompatibility properties compared to metallic implants.

However, the use of metal implants is essential for implants that require high strength and durability. Until now, stainless steel grade 316L, cobalt alloy, pure commercial titanium, and Ti-6Al-4V alloy were the most widely used metal biomaterials in implant devices. These materials have high corrosion resistance, biocompatibility, and excellent mechanical properties, in particular titanium and titanium alloys (Joy Anne *et al.*, 2019). However, titanium and titanium alloys are more expensive than stainless steel and have less wear resistance than others. In addition, compared to Ti and Co-Cr alloys, SS316L has the longest history of biomedical implant applications, has good workability, fracture toughness, low cost, and easy availability (Nauri *et al.*, 2021). In fact, the success of the application of stainless steel 316L as an implant material in human life has long been proven.

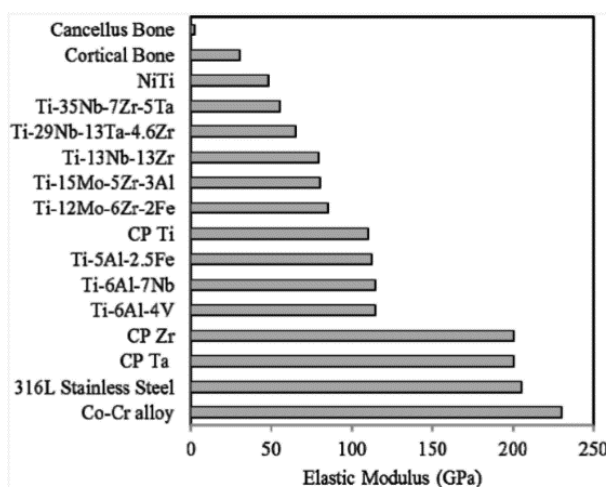


Figure 1.1 shows the elastic modulus of most materials currently used for biomedical applications (Li *et al.*,2014).

Metal implant is stronger than the human bone based on the modulus elasticity as shown in Fig. 1.1. The large difference of the modulus elasticity between the bone and metal implant can result in stress shielding effect and metal implant failure. Therefore, slurry method with HA addition has been explored to improve the properties.

1.3 Objective of Study

The main purpose of the research is to fabricate stainless-steel foam by adding bio-active ceramics using the slurry method. The objectives of this research are:

- i. To identify the suitable percentage of bioactive ceramic HA via slurry method.
- ii. To optimize the suitable sintering temperature to produce SS316L-HA foam.
- iii. To verification the influence of HA addition and sintering temperature on the physical and mechanical properties of SS316L-HA foam after sintering process.

1.4 Scope of Study

Various ranges and parameters are analyzed and defined to conduct laboratory studies for foam manufacturing. The scope of this study can be divided into several points that are material use, fabrication, and eventually characterization of SS316L-HA foam; the scope of this study is as follows.

- i. The raw material used in the study consisted of stainless steel (SS316L), Hydroxyapatite (HA), Polyurethane (PU) foam, Polyethylene Glycol (PEG), Carboxymethyl Cellulose (CMC) as binders and distilled water.
- ii. The method of producing the SS316L-HA foam is a slurry method of 60wt.% and HA compositions with 1wt.%, 3wt.%, 5wt.%, 7wt%, and 10wt%. A composition for binders where PEG and CMC is 2.5 wt.% and distilled water. Sintering temperatures are performed at 1150°C, 1200°C, 1250°C, 1300°C and 1350°C.
- iii. The physical properties of the foams SS316L-HA studied in the Archimedes principle on porosity and density according with the standard ASTM C20-00.
- iv. The mechanical properties of the SS316L-HA foam were studied by compressive strength, yield strength and modulus elasticity testing by universal testing machine according standard ISO 13314:2011.
- v. To characterize the microstructure and morphology of SS316L-HA using SEM.

1.5 Significant of Study

There are two types of orthopaedic implants that are temporary implants and permanent implants. Plates and screws are examples of temporary implants. Usually accompanied by replacement of the knee, shoulder, spine, hips, fingers, and feet for permanent implants. In the case of permanent implants, it is very important to ensure that the binding between the implant material and the living tissue is sufficient and safe. This strong bonding can be achieved by the internal growth of the tissue of the open and interconnected pores of the implant material (Ryan *et al.*, 2006). There are many implants made with the SS316L, but it is temporary. However, there are still some weaknesses in current metal implants. Therefore, the addition of hydroxyapatite (HA) helps to enhance the mechanical and physical properties that can be used for permanent implants.

1.6 Organization of Thesis

This thesis is divided into three chapters: introduction, literature review, and research methodology. Chapter 1 reviews the background of the study, problem statement, objectives, scope of study, significance of study, and finally the structure and organisation of the thesis.

Chapter 2 explains more details about Chapter 2: explores and describes what biomedical implants are, common features of natural bones, and the application of porous materials towards biomedical implants. Describe the technique of the foam replication method. This report discusses the properties of stainless steel, the use of stainless steel as an implant material, and methods used in previous works to process stainless-steel foam. The characteristics of hydroxyapatite and the uses of hydroxyapatite as implant materials. The parameters used throughout the sintering process are also discussed.

Chapter 3 further discusses the preparation of samples and the international standards used in the testing of samples. All of the raw materials used in this study have explanations. The details of the parameters studied and analysed in the experiments are also described in this chapter.

In Chapter 4, the results, and discussions of the mechanical and physical properties of SS316L-HA foam produced by the replication method are presented. Also, the microstructural analysis was conducted. Finally, chapter 5, which presents the results and findings that are summarized along with the suggestions for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Mechanical disorders in the human body can often be repaired by surgical implantation of synthetic replacement parts called biological implants. Clinicians and engineers have spent a lot of research over time studying the physical and mechanical properties of all types of human bones and implants to treat various injuries with bone replacement. Many factors influence the success of bone replacement surgery with implants, including the physical and mechanical properties of implant materials, biocompatibility with human body and implant materials, patient health, and the expertise of surgeons performing surgeries. Currently, implant materials only survive in the human body for about 12-15 years and cannot function well. This condition causes re-surgery to monitor the condition of the implant, the patient's health, and to replace the implant. Re-surgery and replacement of the implants will involve additional cost to the patient. The causes of implant failure depend on mechanical, chemical, biocompatible, implant design, surgery, tribology, etc.

2.2 Biomedical Implant

According to Williams, biomaterials study is an analysis of the nature and properties of biomaterials, the processes by which they communicate with biological systems and their success in clinical applications. Biomaterial is described as material intended to interact with biological systems for the evaluation, diagnosis, enhancement, or

replacement of any tissue, organ, or feature of the body (Barrere *et al.*, 2008). Biomaterials are also implanted biomedical materials. Biomaterials can be either naturally existing or artificial materials, such as cotton and bone (Abdalla, 2013). A biomedical implant is a particular tool, system, or component that is implanted into the body. The human body is responsible for removing or supporting some portion of the internal body that does not function and for improving the patient's capacity and health, Domb & Khan (2014).

Thus, the biocompatibility of medical devices covers both the materials used and the design of the device (e.g., geometry, mechanics, and electrical control) of the device. In fact, many clinical failures of joint replacements, for example, are due to suboptimal mechanics of the device, rather than problems with the material properties (Liu *et al.*, 2015). Non-material issues are beyond the scope of this review article as well as this journal. As for the biocompatibility of implant materials, not only the chemical interactions of the implanted material with the host physiological system (e.g., corrosion of alloys and toxicities of metal ions) but also the physical impacts of the implanted material on the surrounding tissues (the mechanical properties of the material), although the former is the more common and primary concern.

Conception of implants for the purpose of bone surgery is strongly affected by the advances achieved in biomaterial engineering. Attempts were made to obtain biomaterials that would meet stringent requirements for biofunctionality, mechanical ability, degradation resistance, or biocompatibility (Vaccaro & A.R. 2002; Dewidar *et al.*, 2006; Mudali *et al.*, 2003). However, the word "biomaterial" is used with a number of meanings in the authorised and scientific population. Medical devices in the legal field mean any apparatus, instrument, machine, implement, implant, appliance, in vitro reagent calibrator, material, software, or other related or similar articles intended for use in production, in combination or on their own, according to Huebsch & Mooney (2009). Figure 2.1 shows the types of biomaterials.

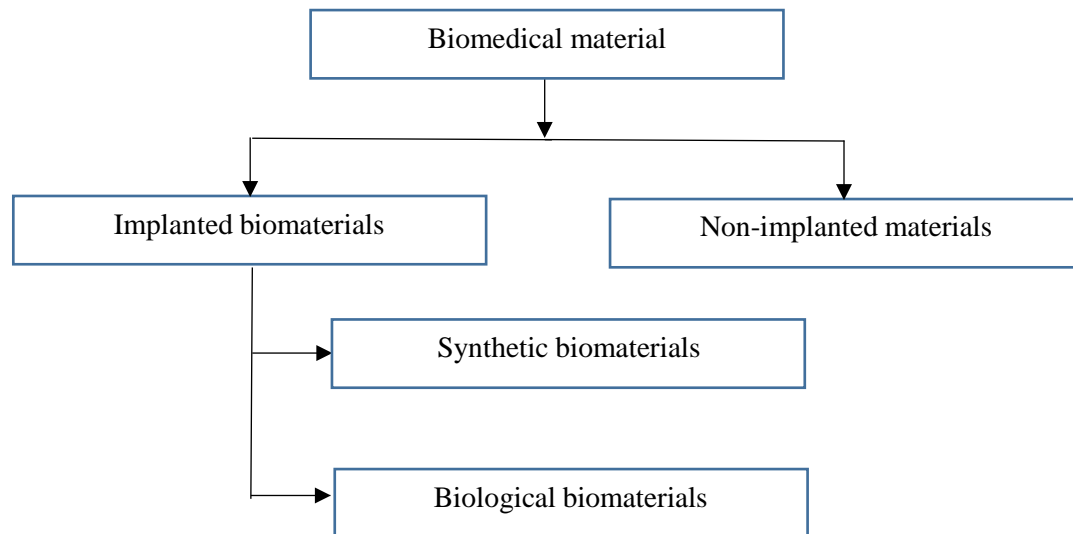


Figure 2.1: Shows the definition of biomaterials. (Chen *et al.*, 2015)

Compared to the other materials, bioactive glasses are highly biocompatible and have a greater chance of integrating with human tissue than the metal implants mentioned above, making them a good option for improving the biocompatibility and bioactivity of these metals. Bioactive glass offers the following benefits: replacing damaged bone and tissue that will integrate well with the body's environment, facilitating tissue regeneration, and degrading at a similar rate of tissue regeneration (Jie *et al.*, 2021).

2.3 Properties of Natural Bone

The bone form classification is based on bone density, porosity, and metabolic function (Polo-Corrales *et al.*, 2014). In addition, Lu stated that the pores can be divided into three groups. The first type of grouping is in the inner area of the bone, the second is in the wall of both the macrospores, whereas the third group is in the cortical bone's dense exterior layer, which is sometimes referred to as cortical bone. Compact skeleton, whereas the elastic inner centre is known as a cancellous, which is sometimes recognised as a trabecular and spongy tissue (Lu *et al.*, 2003). It performed a microstructure study of natural bone using a scanning electron microscope (SEM) where the findings are seen in Figure 2.2.

The physical and mechanical properties of the natural bone vary depending on size of the bone. Subject, i.e., age, gender, health status, bone types, and different regions of the same bone (Kato *et al.*, 2013). The nature of the bone, consisting of bone

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APPENDIX A

LIST OF PUBLICATIONS

Ahmad, S., Rani, R. Y., Yusop, U. A., Muda, R., Rahman, H. A., Ismail, A., & Noor, F. M. (2022). EFFECT OF COMPOSITION AND SINTERING TEMPERATURES ON THE PROPERTIES OF STAINLESS STEEL (SS316L)-HYDROXYAPATITE (HAP) FOAM. *Malaysian Journal of Microscopy*, 18(2), 164-173.

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