Fabrication and Properties of Cobalt-Chromium Implant Composite

By

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CONTENT

LIST OF TABLE

LIST OF FIGURE

GLOSSARY

Acetabular cup	A part of a hip implant that forms the socket in the ball-and-socket structure of the hip joint.
Anecdotal	Informal
Arthroplasty	A surgery performed to relieve pain and restore range of motion by realigning or reconstructing a dysfunctional joint.
Biomaterial	A biocompatible material that is used to construct artificial organs, rehabilitation devices, or prostheses and replace natural body tissues.
Bone Grafting	A surgical procedure that places new bone or a replacement material into spaces between or around broken bone (fractures) or in holes in bone (defects) to aid in healing.
Collagen	The fibrous protein constituent of bone, cartilage, tendon, and other connective tissue. It is converted into gelatin by boiling.
Courageous	Having or characterized by courage.
Fortuitous	Happening by accident or chance/Lucky or fortunate.
Laissez faire	An economic doctrine that opposes governmental regulation of or interference in commerce beyond the minimum necessary for a free-enterprise system to operate according to its own economic laws.
Osteoblast	A cell from which bone develops; a bone-forming cell.
Osteocalcin	A noncollagenous protein found in bone and dentin.
Osteoconduction	A phenomenon in which there is an ingrowth of sprouting capillaries, perivascular mesenchymal tissues, and osteoprogenitor cells from the recipient host bed into the three-dimensional structure of an implant or graft.

Orthopedic	Medical specialty concerned with the skeleton and its associated structures.
Phosphate	A salt or ester of phosphoric acid. /A fertilizer containing phosphorus compounds.
Prosthetics	The branch of medicine or surgery that deals with the production and application of artificial body parts.
Tuberculosis	An infectious disease producing lesions especially of the lungs: consumption (no longer in scientific use).

FABRIKASI DAN SIFAT-SIFAT KOMPOSIT KOBALT-KROMIUM IMPLAN

ABSTRAK

Komposit kobalt-kromium implant telah dihasilkan secara teknik metarlugi serbuk. Aloi Kobalt-kromium telah dicampurkan bersama hydroxyapatit (HAP) dengan komposisi 0% , 5%, 10%, 15% dan 20%. Pemprosesan untuk menghasilkan produk melibatkan percampuran serbuk, pengadunan, diikuti dengan pemadatan dan proses pensinteran. Kobalt, kromium dan hydroxyapatit telah dicampurkan di dalam Pengisar Bebola Planet pada 600 rpm dalam masa 30 minit. Penghasilan CIC melalui proses pemadatan dengan menggunakkan Mesin Pengujian Sejagat, GOTECH. Tekanan yang digunakan ialah 500MPa. Proses pensinteran pada kadar 20^0C/min dalam masa 3 jam telah dijalankan pada suhu 1000^0C. Komposit yang dihasilkan telah dijalankan beberapa ujian bagi mengetahui sifat mekanikal dan mikrostruktur. Analisis mikrostruktur dilakukan dengan menggunakkan mikroskop imbasan elektron dan penganalisis imej yang disambungkan dengan mikroskop optik. Dalam analisis mikrostruktur, terdapat beberapa ciri-ciri yang perlu dikaji seperti saiz partikel, keliangan, mod bentuk partikel, kelakuan kakisan, ikatan antara campuran partikel dan mekanisma patahan dimana, bahan komposit tersebut akan menunjukkan sifatnya secara terperinci. Sifat-sifat seperti kekerasan, ketumpatan, taburan saiz partikel, ketulenan bahan mentah, kekuatan mampatan dan kelakuan kakisan, masing-masing telah dikaji melalui Vickers Micro Hardness, AccuPyc 1330 Gas Pycnometer, Penganalisis partikel (MALVERN MASTERSIZER) 2000, Pembelauan Sinar-X (XRD), ujian pemadatan dan ujian rendaman dalam cecair Natrium Klorida, (0.9%.NaCl). Analisis mikrostruktur terhadap komposit menunjukkan taburan partikel hydroxyapatit adalah seragam dalam matriks kobalt-kromium. Hasil kajian XRD, menunjukkan ketulenan setiap partikel seperti kromium, kobalt dan hydroxyapatit pada julat yang tinggi. Pada amnya, julat kobalt dan chromium antara 40 hingga 50 darjah, dan tiada tanda yang jelas tentang kehadiran HAP dalam analisa bahan komposit tersebut. Bagi analisis ketumpatan, kedua-dua data ujikaji dan teori menunjukkan graf yang serupa kerana graf tersebut mengalami penurunan ketumpatan apabila penambahan HAP dilakukan. Kekerasan komposit menunjukkan kekerasan menurun dengan penambahan HAP. Analisis modulus sonik menunjukkan hubungan salingan antara modulus dengan halaju bunyi dimana data modulus akan menurun dengan kenaikan data halaju bunyi. Analisis pada mikrostruktur dalam ujikaji mampatan menunjukkan perubahan berlaku apabila komposit berubah daripada mod mulur kepada mod rapuh apabila penambahan HAP dilakukan. Namun, corak retak menunjukkan tidak berterusan bagi kelakuan mod mulur dan retak berterusan untuk kelakuan mod rapuh. Kajian kakisan telah menunjukkan komposit lebih terkakis apabila ditambah HAP.

ABSTRACT

Cobalt implant composite (CIC) was produced by powder metallurgy technique. Composition of 0% ,5%, 10%, 15% and 20% of hydroxyapatite was mixed with cobalt-chromium alloy. The fabrication technique is mixing, blending, pressing and sintering of the final product. Cobalt, chromium and hydroxyapatite powders were mixed in planetary ball mill at 600 rpm for 30 minutes. The consolidation method for CIC was uni-axial compacting using Universal Testing Machine (UTM) Gotech. The pressure used was 500 MPa. The CIC was sintered at 1000^0C temperature with 20^0C/min for 3 hours. The composites then were evaluated and tested to evaluate the microstructure and mechanical properties. The microstructure analysis is carried out by using the Scanning Electron Microscope and Image Analyzer attached to the optical microscope. In microstructure analysis, there are several characteristics need to observe i.e., particle sizes, porosities, mode of shapes, corrosion behaviours and bonding between mixed particles and fracture mechanism, which these can describe the composites material in details. The properties such as hardness, density, and particle sizes distribution, purity of raw materials, compressive strength and corrosion behaviours are analyzed by using Vickers Micro Hardness, AccuPyc 1330 Gas Pycnometer, MALVERN MASTERSIZER 2000 particle analyzer, X-Ray Diffraction (XRD), Compression test and Immersion Fluid test in Natrium Chloride (0.9%.NaCl), respectively. From the microstructure analysis of the composite, the microstructure indicates the homogenous distribution of the chromium particles, and HAP particles are distributed homogenously in the matrix cobalt chromium. From the X-ray diffraction (XRD) the high peak of the x-ray analysis is indicating the purity of each powder such as chromium and cobalt. In general cobalt and chromium peaks occur at the range of 40 to 50 degree and there is no obvious sign of HAP signal in XRD analysis of the composites. Both experimental and theoretical density graphs have shown a similar pattern line which both experienced the density gradually decreased when percentage of HAP increased. The hardness of the composites decreases slightly with the increasing weight percent of HAP. The sonic modulus analysis, indicating that there is a reciprocate relationship between modulus and sound velocity, whereby modulus will be decreased when the sound velocity increases. The microstructure analysis on compression test, indicated the deformation behavior of the composite started to change from the ductile mode to the brittle mode resultant with the added of HAP. Besides, the crack pattern showed non continuously for the ductile mode behavior and had a continuous line for the brittle mode behavior. Corrosion study indicated that composites experienced more corrosion when the HAP was added.

CHAPTER 1

INTRODUCTION

1.1 Research Background

The overwhelming success of orthopedic biomaterials is perhaps best exemplified by their worldwide market, which dominated biomaterial sales at approximately $14 billion in 2002 with expected growth rate of 7% to 9% annually. Global sale of fracture management products only totaled approximately $15 billion in 2000, whereas the other approximately $12 billon was spent to joint replacements. Global sale of knee implant product approximately $2.5 billion in 2002, representing approximately 700,000 knee replacement surgeries, which include first-time joint replacement procedures and revision procedures for replacement, repair, or enhancement of an implant product or component from a previous procedure. Revision procedures are growing at an accelerated rate of approximately 60% in the United State (Buddy et al., 2004).

Due to the clinical success of total joint replacement procedures for knee and hips, the demand for total joint replacement procedures of over joints, such as the shoulder and elbow, continues to grow. Approximately 55,000 shoulder and elbow implant procedures were conducted in 2000 (Buddy et al., 2004).

For generations various materials so-called biomaterials are used in medicine and dentistry with a purpose to replace or repair a body feature, tissue, organ or function. The performance of biomaterial in direct contact with living tissue is controlled by two sets of characteristics: biofunctionality and biocompatibility. When metals and alloys are considered the central aspects of biocompatibility are the corrosion resistance of materials and the influence of corrosion products on the surrounding tissue. Corrosion resistance of the most common used metallic materials such as austenitic stainless steels, cobalt–chromium, titanium and titanium based implant alloys relies on their passivity by a thin surface layer of oxide (Tanja et al., 2003).

Many alloys had been studied for surgical and dental implant. According to Marti (2000), the first medical use of cobalt-base alloy was in the first cast dental implants, due to its excellent resistance to degradation in the oral environment. Various in vitro and vivo test have shown that the alloys are biocompatible and suitable for use as surgical implants. From observation by Walker et al. (1989) the most biocompatible metallic implant and widely used is titanium alloys due to their light weight, biocorrosion resistance and mechanical properties. However the release of Aluminums and 4V ions from the alloy might induce some long term health problems. Meanwhile, the low wear resistance of titanium alloys could accelerate the release of such harmful ions (Wang, 1996).

Conventionally, fabrication of implant alloys has been established using casting technique. However, the temperature in melting of the implant alloys is very high especially for cobalt, molybdenum and titanium alloys, which has prompted other method

of fabrication namely powder metallurgy technique. It is necessary to fabricate by using powder metallurgy technique because it is effectives cost and easier to fabricate. Powder metallurgy technique also can be used to produce implant materials of controlled porosity and better mechanical properties.

Many studies also reported based on alloys without reinforced with hydroxyapatite (HAP). It can be found that the hydroxyapatite powder (HAP) has been studied to be used as tissues and bone replacement. However, there is no research reported for cobalt chromium alloys with HAP composite. Therefore, this research is focused on the fabrication and properties of cobalt implant composites produced by powder metallurgy route.

1.2 Research Objectives

The objectives of this research are:

- To fabricate the implant composite of cobalt-chromium with HAP by powder metallurgy technique.
- To study the microstructure and the properties of the composite such as density, hardness, compressive strength and corrosion behaviour.
- To investigate the fracture mechanism in the composites.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Biomaterial

According to Ikada (2002), materials of biological origin, synthetic polymer, glass ceramics and metal have been used as biomaterials which can be defined as materials to be used in contact with human living cells. Each material has its own advantages and disadvantages. For instance, advantages of polymeric materials over the others are their excellent flexibility in physical properties varying from viscous fluid to tough solid.

A serious drawback of polymers is their insufficient mechanical strength when used as medical devices in orthopedic and oral surgeries. In such cases, ceramics and metals are the first choice for the biomaterials, although their modulus is too high in comparison with that of bone and tooth tissues, occasionally resulting in stress shielding to the tissues in contact.

Biomaterials can be defined as any substances or combination of substances synthetic or natural in origin, which can be used for any period of time, as a whole or as a part of a system which treats, augments or replaces any tissue, organ or function of the body.

Buddy et al. (2004) had study that human body has been introduced to the non-biological materials since the prehistory. Due to the poor understanding of biocompatibility and sterilization, most implant prior to 1950 faced with a low probability of success. Now as there will be an elaborated upon throughout the textbook therefore the factors that contribute to biocompatibility include the chemistry of the implant, shape, mechanics and design. Early studies, especially with metals, explored primarily chemistry ideas to explain the observed bioreaction.

According to Buddy et al. (2004) possibly the first study assessing the in vivo bio-reactivity of implant was performed by Levert in 1829. The specimens of gold, silver, lead, and platinum were studied in animals such as dogs. Platinum was found to be well tolerated. Copper, magnesium, aluminum, alloy, zinc and nickel found to be discolored the surrounding tissue while gold, silver, lead and aluminum were tolerated but inadequate mechanically. Borthe et al. (1940) discussed the possible use for titanium and alloys for medical implants.

In the early 1940s, papers appeared discussing the reaction to implant poly (methyl methacrylate) and nylon. Ingraham et al. (1947) published the first paper on polyethylene as a synthetic implant material. The paper pointed out that polyethylene production using a new high-pressure polymerization technique began in 1936. This process enabled the production of polyethylene free of initiator fragments and other additives. Ingraham et al. (1947) demonstrated good results on implantation (i.e., a mild foreign body reaction) and attributed these results to the high purity of the polymer they used. Later, in 1949 paper

commented on the fact that addition too many plastics had a tendency to "sweat out" and this may be responsible for the strong biological reaction to those plastics by Le Veen and Barberio (1949). A vigorous foreign body reaction to cellophane, lucite, and nylon but extremely mild reaction to "a new plastic," Teflon, was explored. Both authors incisively concluded, "Whether the tissue reaction is due to the dissolution of traces of the unpolymerized chemical used in plastics manufactured or actually to the solution of an infinitesimal amount of the plastic itself cannot be determined." The possibility that cellulose might trigger the severe reaction by activating the complement system could not have been imagined because the complement system was not yet discovered.

By end of the World War II, high-performance metal, ceramic and especially polymeric materials transitioned from wartime restricted to peacetime available. The possibilities for using these durable, novel, inert materials immediately intrigued surgeons with needs to replace diseased or damage body parts. The surgeons have made a big changes by applied the materials which is originally manufactured for airplanes and automobiles, to medical problems. These early biomaterials include silicones, polyurethanes, teflon, nylon, methacrylates, titanium, and stainless steel.

A historical context helps us to appreciate the contribution made primarily by medical and dental practitioners. There was a little precedent for surgeons to collaborate with scientists and engineers, after World War II. As the life of their patient was at stake, the medical and dental practitioners of this era felt that it was appropriate to invent on their own. The physician had much more freedom than is seen today to take heroic action to

the patient where other options were exhausted (Kolff, 1998). These medical practitioners had read about the post-World War II marvels of materials science. Looking at a patient open on the operating table, they could imagine replacements, bridges, conduits, and even organ systems based on such materials. Many materials were tried on the spur of the moment. Some by chance succeeded. It is really high-risk trials, but usually took place where there are no other options. The surgeon often had a life at stake and was willing to take a huge technological and professional leap to repair the individual. This tolerant biomaterials era quickly led to a new order characterized by scientific/engineering input, novel procedures and a sharing of decisions prior to attempting high-risk (Clark et al. 1976). Meanwhile, a foundation of ideas and materials for the biomaterials field was built by courageous, intensely committed, creative individuals and it is important to look at this foundation to understand many of the attitudes, trends and materials common today.

2.2 Fundamentals

2.2.1 Prerequisites of Biomaterials

Different kinds of artificial tissues and organs are clinically used or under investigation. However, any materials are not always applicable to the medical systems, because materials to be used as biomaterials should meet several basics that are very different from those for the materials in non-medical use. The basics necessary for biomaterials are given in Figure 2.1. The most important one is non-toxicity, in other words, safety to the human body. The toxicities which foreign materials will cause include hemolysis, sustained inflammation and allergy. It is needless to say that every material for any practical purpose should be effective and durable over the period of their use.

Biomaterials are not exceptional; hemodialyser should effectively remove renal toxins and excessive water from the patient's blood while artificial joints should replace the defective joint of patients with high durability.

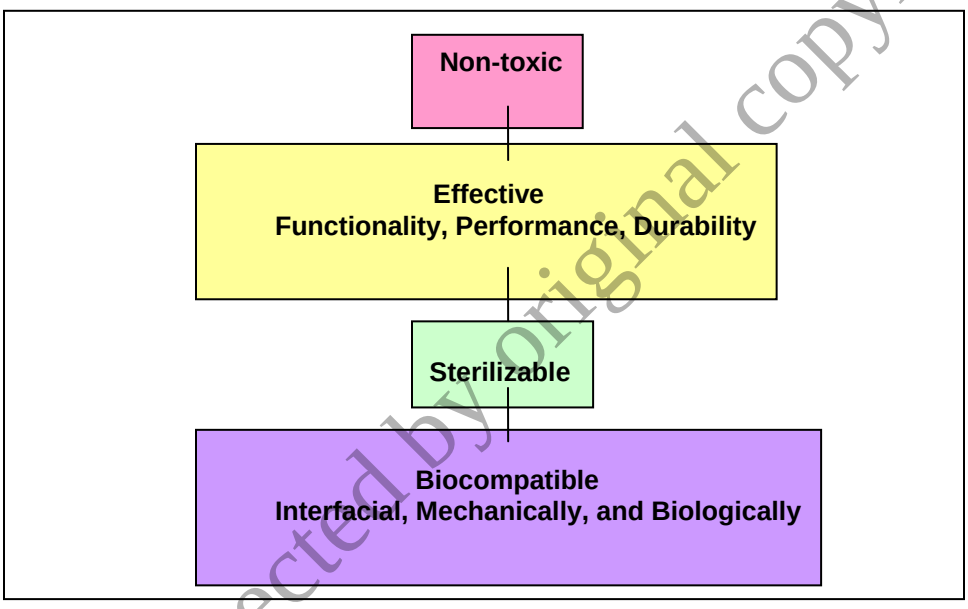

Figure 2.1 Prerequisites of materials as biomaterials (Ikada, 2002)

Sterilizability is also the minimum condition that biomaterials should satisfy. Gamma-irradiation, ethylene oxide gas (EOG) exposure, and autoclaving are the current major sterilization means. In some cases, a small amount of EOG, a toxic gas, remains in the sterilized material even after aeration, while irradiation with high-energy radiation and autoclaving often deteriorate the sterilized biomaterials. It should be kept in mind that water-swollen hydrogels as well as materials with immobilized proteins and cells are not easy to access to the conventional sterilization methods because of their high resistance to such harsh conditions.

2.2.2 Causes of Toxicity

Very often, even biomaterial scientists do not distinguish non-toxicity from biocompatibility which will be described below. It is helpful for us in understanding the nature of biomaterials to make clear distinction between non-toxicity and biocompatibility.

The toxicity of biomaterials is mostly caused from the material surface or inside. Most of them are water-soluble or water-dispersible substances as listed in Figure 2.2. Medical devices free of these eventually toxic substances can be fabricated if much care is paid to the manufacturing process. In addition, one should be always careful in removing microorganisms from biomaterials, although they will be killed by sterilization, because the dead body of microorganisms might contain biological fragments.

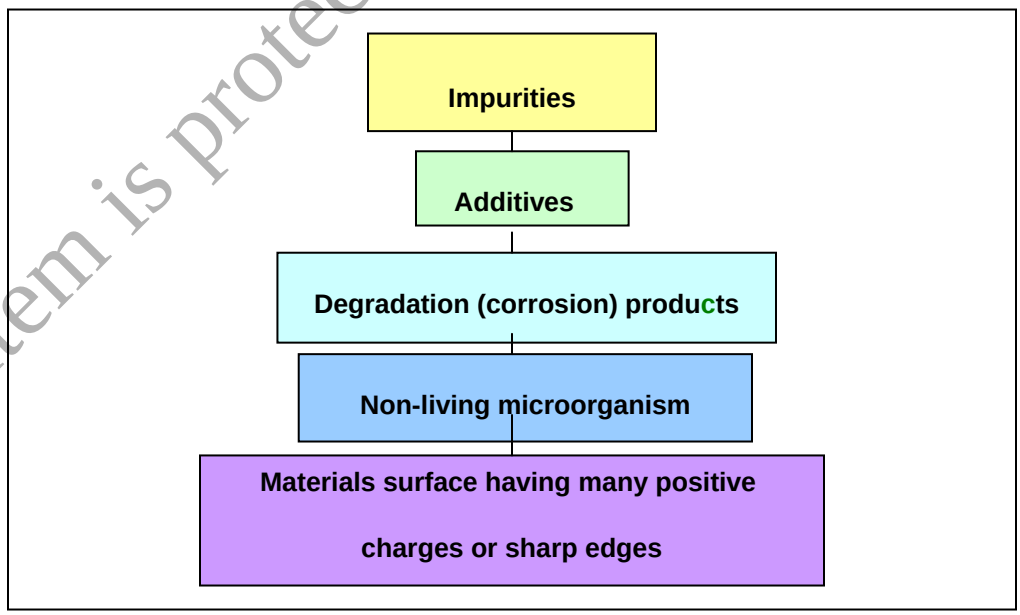

Figure 2.2 Possible toxic substances (Ikada, 2002)

2.3 Biocompatibility

As biocompatibility is influenced by a large number of factors, it is not easy to give an unmistakable definition to biocompatibility (Ikada, 1994). Generally, when foreign materials come into direct contact with the living structure, they induce so-called foreign-body reactions such as complement activation, blood coagulation, thrombus formation, encapsulation by collagen tissue, and calcification. Biomaterial which evokes less foreign-body reactions is better in biocompatibility. However, as thrombus formation is a preceding event necessary for the formation when a large-caliber vascular graft is implanted, less foreign-body reaction does not always mean to lead to better biocompatibility. Biocompatibility can be divided into two subgroups; interfacial biocompatibility and mechanical biocompatibility.

2.3.1 Interfacial Biocompatibility

If a material surface has a profound effect on the performance of the biomaterial, consider its biocompatibility as interfacial. The well-known foreign-body reactions related to interfacial biocompatibility include thrombus formation and encapsulation. As these biological events are intimately associated with protein adsorption and the subsequent platelet adhesion which occur as the initial biological response to the biomaterial coming into contact with the living body, protein adsorption and cell adhesion onto biomaterials surface have been most extensively studied in the biomaterials science. Depending on the application purpose of biomaterials, either the minimum or the maximum cell adhesion is generally required as a truly biocompatible surface.

2.3.2 Mechanical Biocompatibility

Even if a material surface is effectively modified to exhibit good blood compatibility, the material will fail in producing such a vascular graft that is applicable for long-term use. This is because most of man-made materials tend to have much higher rigidity than natural blood vessel walls, when high tensile strength comparable to that of blood vessels is given to synthetic materials.

This variance of mechanical rigidity or modulus between a biomaterial and a natural tissue induces deterioration of the tissue bonded to the biomaterial. This is called a stress shielding effect, especially in metal/bone combination. Therefore, attempts have been made to improve the mechanical biocompatibility of biomaterials by reducing their rigidity or raising their compliance to the level of natural tissues (Ikada, 2002).

2.4 Types of Biomaterials

Katti (2004), had been reported that materials scientists have investigated metals, ceramics, polymers and composites as biomaterials. The general criteria for materials selection for bone implant materials are highly biocompatible that does not cause an inflammatory or toxic response beyond an acceptable tolerable level, it has appropriate mechanical properties, closest to natural bone and the manufacturing and processing methods are economical. Here some of the key classes of the materials and their evolution from commodity materials to engineered/synthesized biomaterials are explained.