

**NEW DESIGN OF PURE TITANIUM METAL ON
METAL TOTAL HIP REPLACEMENT WITH ION
IMPLANTED WEAR RESISTANCE SURFACE**

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Executive Summary

Relative movements of the human body parts are enable due to the existence of joints. However, disease and injury could cause joint disorder. Nowadays, joint replacement is a widely accepted treatment for damaged joint. In Malaysia, hip joint or knee joint replacements, either as total or partial, have become a standard procedure in many hospitals. Sultanah Aminah hospital for example, conducts 4-5 joint replacement surgeries every week. A typical artificial joint consists of two different materials which have self lubricating bearing behaviour such as metal and polymer. A well perform joint must fulfil bio mechanics aspects of mobility and stability, as well as bio compatibility aspects. Despite of the smooth mobility, the main problem by metal-polymer bearing is the wear which occur in the surface contact, because friction moment will create small particles or debris which can accumulate in the surrounding human tissue and cause various problems. Many studies reported that metal-polymer pair contact is satisfactory for short term only. Therefore, a hard material couple such as metal-metal which provides better tribological properties is considered high potential. The current available metal-metal joint is made from Titanium alloy Ti-6Al-4V, but the toxic presence of Al and V ions in this alloy has been a serious issue of concern. The aim of this study is to obtain the long lifetime of the hip joint implant without significant negative effect of wear debris. Thus, development of metal-metal bearing joint made from Pure Titanium with surface modification would be an utmost expected solution.

Surface modification is conducted by using Ion Implantation Process. This process resulted the improvement in strength, surface hardness and wear resistance.

Introduction

Hip joint is an important component of the human skeletal system. It is located at the junctions between the pelvis and the upper leg bone (femur). However, under specific condition, such as disease or damage by accident, hip joint needs to be replaced.

1.1. Background of Study

Human body extremities part, such as hip and knee, are subjected to high load and impact [1]. That is why artificial extremities joints are wear easily. A new generation of joint replacements is now being intensively developed in which materials with higher hardness and toughness such as metals and ceramic composites are applied, because a conventional metal-polymer bearing is not able to provide sufficient wear resistance in hip and knee replacements [2].

Although metal on metal contact pair assures better tribological properties, design improvements of the artificial joint have to be done to compensate high impact and excessive stiffness which can influence mobility and stability of the joint [3].

In the movement of hip joint, intensive contact occurs in the surfaces of the femoral head and its socket. Wear could be reduced by increasing the surface contact

in order to prevent high stress concentration. Hence, in the design, the diameter of femoral head and the clearance between the head and the socket must be carefully determined to maintain low stress concentration and to create air damping.

Beside of the design of the joint replacement, material plays a very important role. Materials for human body implants must be bio compatible, corrosion resistant, strong and has sufficient elasticity [4]. Titanium meets these requirements to a very high degree standard. Titanium has also excellent temperature stability, wear and abrasive resistance as well as light weight [5, 6].

The use of Titanium Alloy Ti-6Al-4V is normally more frequent compared to pure Titanium in biomedical application due to its superior mechanical properties. However, the drawback in metallic implants is that the metals will corrode to some extent in body fluids thereby releasing ions that might possibly be harmful over a prolonged period of time. It is now believed that Al ions have been associated with Alzheimer's disease and V, Co, Mo, Ni and Cr ions are suspected of being toxic or carcinogenic [7]. Therefore pure Titanium is considered better solution since it will not produce toxic ions and its mechanical properties also satisfy biomedical application requirements [8].

To improve the resistance of pure titanium to abrasion and corrosion, surface treatments must be conducted. Among the techniques of surface treatments, the recently developed ion implantation has been reported to be positive potentials for bio materials [9, 10]. In the ion implantation process, nitrogen ions are implanted into the surface of the components by using kinetic energy of high energy beam, so that the ions penetrate into the crystal structures. As result, the surface is geometrically almost unchanged but contains an extremely fine dispersion of titanium nitrides. The hard nitrides improve the tribological properties by increasing surface hardness and reducing abrasion.

This research will be focused on designing pure Titanium metal on metal hip

joint replacement which satisfy bio mechanical and bio compatibility aspects. The fully functional prototype will be fabricated and surface modification will be done by using ion implantation technique to increase hardness and wear resistance of the contact surfaces between femoral head and its socket. Finally, performance evaluation will be conducted in a mechanical loading simulator.

1.2. Problem Statement

A significant number of total joint replacements fail long before their expected life causing severe traumas to the patients.

Hence, ceramic on ceramic, and metal on metal bearing pairs are interesting and potential alternatives. One of the current available metal on metal pairs is made of Titanium Alloy Ti-6Al-4V.

There are some concerns regarding the toxicity of Al and V wear debris in the human body that might have negative side effects to the human body. Pure Titanium is therefore can be proposed as a safer alternative metal to replace Ti-6Al-4V Titanium Alloy bearings. However, as a pure metal, pure titanium tribological properties is lower than Ti-6Al-4V Titanium Alloy, that requires a special surface treatment to improve the surface mechanical properties.

1.3. Objective

The objectives of this research are:

1. To apply metal to metal contact bearing in the hip joint replacement unit and to establish the detail design layout of the joint unit which made from pure Titanium

and which fulfil bio compatibility and bio mechanical principles.

2. To fabricate the on metal bearing hip joint unit and to modify the titanium surface by using ion implantation technique in order to increase its tribological properties.
3. To evaluate the friction, wear and fatigue performance of the artificial joint under mechanical loading according to its operation conditions

1.4. The scopes of work.

The scopes of work for this project are:

1. Designing pure Titanium metal on metal hip joint replacement which satisfy bio mechanical and bio compatibility aspects.
2. Fabricating the fully functional prototype
3. Modifying surface by using ion implantation technique to increase hardness and wear resistance of the contact surfaces between femoral head and its socket.
4. Conducting performance evaluation in a mechanical loading simulator.

1.5. Importance of Study

The importance of this study are stated as the following

1. Reducing hip joint replacement in order to reduce severe trauma that exists in

such surgery

2. Eliminating the risk of toxicity of wear debris in the human body

2.1. Hip Joint Implant

Hip joint is an important component of the human skeletal system (see Figure 2.1 on page 6). It is located at the junctions between the pelvis and the upper leg bone (femur). However, under specific condition, such as disease or damage by accident, hip joint needs to be replaced. A schematic diagram of the artificial hip joint is presented in Figure 2.2a on page 7. Figure 2.2b on page 7 shows an X-ray of a total hip replacement.

Hip joint replacement is described as the greatest achievement in orthopaedic surgery in the twentieth century. Although the field has been dominated for some fifty years by implants based upon metallic femoral heads and stems and polymeric

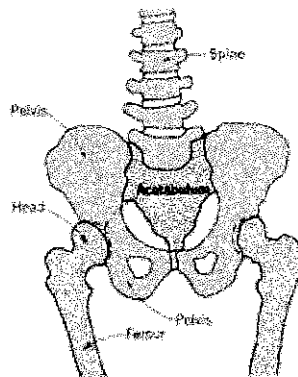


Figure 2.1.: Schematic diagram of human hip joints and adjacent skeletal components.

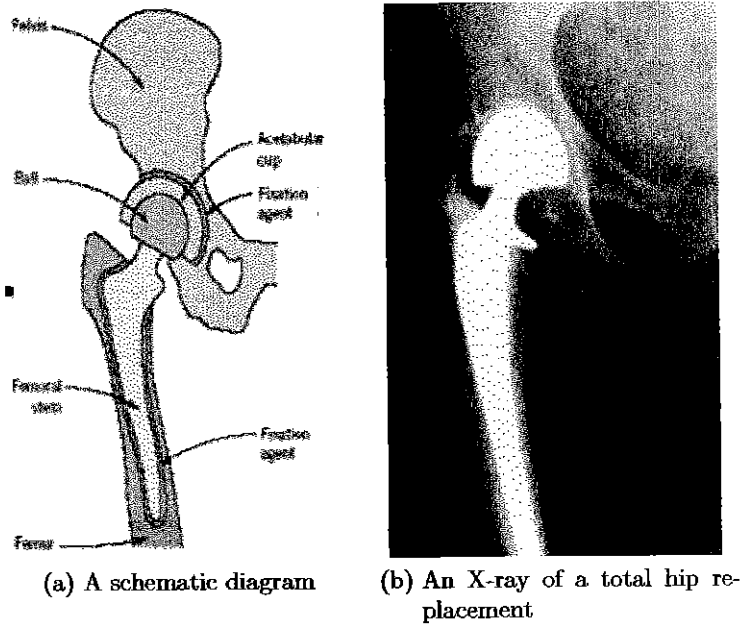


Figure 2.2.: Hip Joint Implant

acetabular cups, they suffer from high wear of the soft cup [11, 12, 13].

The wear of polymer cup in hip implants lead to loosening of the implant and osteolysis. Mass loss as a result of wear leading to osteolysis may cause dislocation and subsequently revision and/or replacement of the implant [14]. A significant number of total joint replacements fail long before their expected life causing severe traumas to the patients. Hence, ceramic on ceramic, and metal on metal bearing pairs are an interesting alternative [15, 16, 17, 18].

One of the current available metal on metal pairs is made of Titanium Alloy Ti-6Al-4V. However, there are some concerns about the toxicity of Al and V wear debris in the human body that might have negative side effects to the human body. Pure Titanium is therefore can be proposed as a safer alternative metal to replace Ti-6Al-4V Titanium Alloy bearings. Several attempts have been undertaken to improve the surface properties of titanium alloys either by plasma treatment or by appropriate coating films.

Currently, the most accurate method of analysing wear phenomenon in hip joint

is to use a hip joint simulator. Hip joint simulators have the ability to reproduce the complex kinematics and mechanics of human hip joint, and importantly, can accommodate the actual prosthetic component themselves [19, 20].

Ideally, an artificial hip that has been surgically implanted should function satisfactorily for the life of the recipient and not require replacement. For current designs, lifetimes range between 15 and 25 years.

In essence, there are four basic components to the artificial hip [21]: (1) the femoral stem, (2) the ball that attaches to this stem, (3) the acetabular cup that is affixed to the pelvis, and (4) a fixation agent that secures the stem into the femur and the cup to the pelvis.

The property requirements on the materials to be used for these elements are very stringent because of the chemical and mechanical complexity of the hip joint. Whenever any foreign material is introduced into the body environment, rejection reactions occur. The magnitude of rejection may range from mild irritation or inflammation to death. Any implant material must be biocompatible, it must produce a minimum degree of rejection. Products resulting from reactions with body fluids must be tolerated by the surrounding body tissues such that normal tissue function is unimpaired. Biocompatibility is a function of the location of the implant, as well as its chemistry and shape.

Body fluids consist of an aerated and warm solution containing approximately 1 wt% NaCl in addition to other salts and organic compounds in relatively minor concentrations. Thus, body fluids are very corrosive, which for metal alloys can lead not only to uniform corrosion but also to crevice attack and pitting and, when stresses are present, to fretting, stress corrosion cracking, and corrosion fatigue.

The bones and replacement components within the hip joint must support forces that originate from outside the body, such as those due to gravity; in addition, they

must transmit forces that result from muscular action such as walking. The material should have a good mechanical characteristics such as modulus of elasticity, yield strength, tensile strength, hardness, fatigue strength, fracture toughness, and ductility to deal with it.

Three other important material factors to consider are density, property reproducibility, and production cost.

2.2. Ion Implantation

Surface Hardening is a process used to improve the wear resistance of parts without affecting the more soft, tough interior of the part. This combination of hard surface and resistance to breakage upon impact is useful in parts that must have a very hard surface to resist wear, along with a tough interior to resist the impact that occurs during operation. One of the surface hardening process is Ion Implantation

Ion Implantation involves the bombardment of a solid material with medium-to-high-energy ionised atoms and offers the ability to alloy virtually any elemental species into the near-surface region of any substrate. The advantages of this process are it can be performed irrespective of thermodynamic criteria such as solubility and diffusivity, the possibility of low-temperature processing, the limitations of dimensional changes and the delamination possibility of conventional coatings are avoided. In almost all cases the modified region is within the outermost micro meter of the substrate, often only within the first few hundred angstroms of the surface. Maximum concentrations of several tens of atomic percent are usually achievable, although this depends on the ion-substrate combination.

Ion implantation is a high technology approach for modifying surface properties of materials. It is similar to a coating process, but it does not involve the addition of

a layer on the surface. Originally developed for use in semiconductor applications, and still used extensively in that capacity today. Ion implantation uses highly energetic beams of ions (positively charged atoms) to modify surface structure and chemistry of materials at low temperature. The process does not adversely affect component dimensions or bulk material properties.

A significant advantage of ion implantation is that the treated surface is an integral part of the work piece and does not suffer from possible adhesion problems associated with coatings. The moderate heating associated with the process virtually eliminates any risks of distortion or oxidation effects.

Nitrogen is the most common ion used for metallurgical application when the ions penetrate the surface of the specimen, some of them micro cracks, some fill lattice spaces in crystalline structures, and some react chemically to form compounds, giving new lattice properties. It has been established that nitrogen implantation into metals can alter their surface properties such as friction, wear, corrosion, etc.

Improvement of the surface hardness and corrosion resistance are depended on the used of implantation parameters such as energy, dose or time. The two key parameters defining the final implant profile are dose ϕ (usually given in *ions/cm²*) and energy, E , (in kilo electro Volt, *keV*). The dose is related to the beam current, I , by the following formula [22]:

$$\phi = \frac{It}{qiA} \quad (2.1)$$

Where t denotes implantation time, A beam area and qi is the charge per ion. Typical beam current and implantation doses range from $1\mu A - 30 mA$ and $10^{11} - 10^{19} atoms/cm^2$.

During implantation, ions come to rest beneath the surface in less than 10-12 s. This rapid stopping time produces an ultra fast quench rate in the wake of the stopping ion. This allows many novel surface alloys or compounds unattainable by conventional (equilibrium) processing techniques to be produced at room temperature. These include substitutional solid solutions of normally immiscible or low-solubility elements. Such highly metastable and amorphous alloys often possess unique physical and chemical properties.

On a commercial scale, the applications for ion implantation of metals continue to increase, at present mainly for anti wear treatment of high-value components. A large number of industrial trials have involved the implantation of nitrogen for improving the wear resistance of coated and uncoated tools and other precision components. Implantation appears to be an attractive technique for treating industrial components by stabilisation of the micro structure (preventing a change in wear mode), by transformation to a wear-resistant mode, or by chemical passivation to prevent a corrosive wear mode

Figure 2.3 on page 12 shows a schematic view of the path of an individual ion as it loses energy in a material, thereby forming a shallow surface-modified region. As indicated in the figure, the ion does not travel in a straight path to its resting place, due to collisions with the target atoms. Target atoms are displaced from their lattice sites with sufficient energy that they can themselves displace additional target atoms, resulting in a collision cascade. These individual collisions with lattice atoms within a single collision cascade are shown in the insert at the bottom of Figure 2.3 on page 12.

Ion implantation into metals was intensively studied since many years. The possibility of new phase synthesis, formation of supersaturated solutions or surface layers characterised by high stress level are well established [10].

Tsyganov et.al[23] investigated Ion Implantation of Ca and/or P into Ti or Ti alloys is of interest in order to enhance mechanical properties and biocompatibility

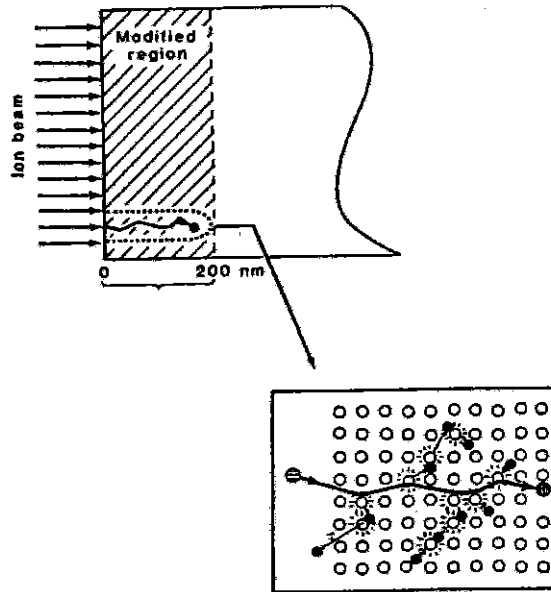


Figure 2.3.: Schematic view of ion implantation process (top) and depiction of the interactions with substrate atoms in a single collision cascade (bottom).

for medical applications. In this work, the micro structural changes of the implanted surface layer were studied. For deeper calcium implantations, precipitation of the metastable hexagonal modification of calcium has been observed. Besides these new phases, partial amorphization is observed.

High dose implantation of phosphorus leads mainly to amorphization of the implanted layer. High dose double implantation with P followed by Ca also leads to partial amorphization. Diaz et.al [24] modified surface of Ti-6Al-4V and Co₂₈Cr₆Mo by Conventional Ion Implantation and Plasma and Plasma Immersion Ion Implantation of N and O. This study resulted that the wear performance was only slightly improved due to a thin layer thickness, whereas, in contrast, the corrosion rate was significantly reduced. Ikeda et.al[25] confirmed that the improvement of wear and corrosion resistance of Co-Cr alloy modified by The Plasma-Based Ion Implantation, as well as high pulse voltage and cooling of the substrate for The Plasma-Based Ion Implantation were the most effective treatments.

2.3. Titanium in Biomedical applications

Titanium is present in the earth's crust at the level of about 0.63% by mass [26] and is therefore the fourth most abundant structure metal after aluminum, iron, and magnesium. It is recovered from TiO_2 -rich deposits of rutile and ilmenite, FeTiO_3 , that are found on every continent [27].

Since the discovery of titanium in 1791, and up until Kroll's innovative process development in 1932, there had been no practical methods to recover titanium metal from these ores because of its pronounced affinity for oxygen. Modern ore extraction, beneficiation and chemical processes have since then enabled the large-volume manufacturing of high-grade TiO_2 , an important pigment for paints and commercial products, and of titanium metal for the production of the Commercially Pure Titanium grades, titanium-based alloys and other alloys systems.

In 1948 the Dupont Company was the first to produce titanium commercially. Today, beside of aerospace as the primary consumer, the other market such as medicine is gaining increased acceptance [28]. Commercially Pure Titanium is unalloyed titanium. At service temperatures it consists of 100% hcp phase. As a single-phase material, its properties are controlled by chemistry (iron, oxygen and interstitial impurity elements) and grain size. Commercially Pure Titanium is classified into Grades 1 through 4 depending on strength and allowable levels of the elements iron, carbon, nitrogen, and oxygen. CP Titanium ASTM Grade 2 has the yield strength of 275 *MPa*[27, 29].

In the galvanic series of metals, titanium has a standard potential of -1.63 *V* which is close to aluminum. Therefore, titanium is very active in Electro-motif force (Emf) series about 1.2 *V* more active than iron [30]. The excellent resistance of titanium to general corrosion in most environments is well-known. This is the result of stable protective surface film, which consists basically of TiO_2 . This thin oxide film passivates

titanium as long as the integrity of film is maintained, generally caused by which most oxidizing environment, for example in salt solution or in nitric acid and chromic acid solution. On the other hand, titanium is not corrosion resistant under reducing condition, where the protective nature of oxide film breaks down such as in sulfuric, hydrochloric and phosphoric acid is not good [27].

The use of titanium in the biomedical field has become a well established area due to the property requirement better than any competing material such as stainless steel and Co-based alloy. The properties of interest for biomedical application are corrosion resistance, biocompatibility, bioadhesion, modulus of elasticity, fatigue strength, and good workability including joining and casting.

Titanium has excellent corrosion resistance and biocompatibility and moderate strength. However, its disadvantage as biomedical application materials is its poor wear resistance [31]. To overcome this problem surface modification is necessary. Several surface modifications have been carried out to improve wear and corrosion resistance of pure titanium such as carbide coating [32], plasma nitriding [33], electrolytic polishing [34], sand blasting [35] spark anodizing treatment [36] and ion implantation [37, 38, 39, 40, 10, 41].

Among the techniques of surface modifications, the recently developed ion implantation has been reported to be positive potentials for biomaterials [31, 42, 43]. Ion implantation is beneficial for surgical material because it has a good adhesive between the modified surface layer and the substrate [31].

2.4. Wear of Metal

Wear will exist when there is a contact between two material, Holm assumed that the real area of contact is formed by the plastic deformation of contacting asperities and

considered wear as an atomic process. On this basis, it can be shown that the worn volume per unit sliding distance W is given by Eq. (2.2) [44]

$$W = ZP/p_m \quad (2.2)$$

where Z is the number of atoms removed per atomic encounter and p_m is the flow pressure of the material.

Assuming that the total real area of contact consists of N circular " a spots," each of radius a , and considering the worn volume as consisting of ξ -atomic layers removed from contacting " a spots," Holm shows that

$$Z = \frac{\xi\alpha}{2a} \quad (2.3)$$

where α is the interatomic spacing.

Archard modified Eq.(2.2) as can be seen in Eq.(2.4). This is similar to the equation of Holm, and is obtained essentially by replacing Holm's concepts of removal of atoms by removal of wear particles [44].

$$W = KP/3a \quad (2.4)$$

2.5. Fatigue of Metal

Most mechanical components and structures made of metal and alloy are subjected to cyclic loading. Some those machine components such as super-heaters, propeller shafts, turbines and pump elements, drilling equipment in petroleum industry severely

suffer from corrosion fatigue problem. Once cyclic loading occur in inert environment, the structures or components suffer to fatigue failure.

In the case that the components and structure subjected to cyclic loading and corrosive environment even fresh water or atmospheric air, the corrosion fatigue can occur [45, 46, 47]. The first study of metal fatigue is believed to have been conducted around 1829 by German mining engineer W.A.J. Albert. The detailed research effort into metal fatigue was initiated in 1842 following the railway accident in France. The cause of this accident was traced to fatigue failure. A systematic investigation of fatigue failure was conducted by A. Woehler during the period 1852-1869 in Berlin. His work led to characterization of fatigue in term of Stress-life (S-N) curves and to the concept of fatigue endurance limit. Another well known fatigue researcher of this era was Fairbairn, W., (1864), Geber, H.,(1872), Goodman (1899), Erwin & Rosenhain (1900), Erwin & Humfrey (1903). The development of metal fatigue research came in new era was begun by Griffith (1921) and Paris et.al (1963). They applied the fracture mechanic concept to solve fatigue problem of notch specimen.

The important concept of stress-life was proposed by Woehler. The method characterizes the total fatigue life in terms of nominal stress amplitude and cyclic number (S-N) curve [48]. The total fatigue is defined as accumulation crack initiation, short crack, long crack and critical fracture or final failure [49]. The fatigue life equation is written as Eq. 2.5:

$$\sigma_a = \alpha - \beta \log N \quad (2.5)$$

Basquin modified Woeler formula in term of correlation log-log scale, a linear relationship is commonly observed, as seen in Eq. 2.6 and Eq. 2.7. The stress-life curve characterizes the contribution of crack initiation and crack propagation processes to total fatigue life in nominally smooth specimen

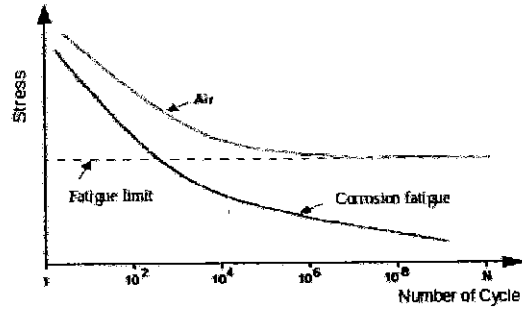


Figure 2.4.: S-N curve for fatigue and corrosion fatigue

$$\ln \sigma = \alpha - \beta \ln N \quad (2.6)$$

or

$$\frac{\Delta \sigma}{2} = \sigma_a = \sigma'_f (2N_f)^b \quad (2.7)$$

In the presence of corrosive medium, the S-N curve changes in form of no endurance limit appearance. Figure 2.4 on page 17 shows S-N curve fatigue in lab-air and corrosive environment.

Fatigue life reduces significantly due to corrosion environment and the difference becomes more pronounced at higher life ($>2 \times 10^5$ cycles) when corrosion plays a greater role in reducing the fatigue life [50]. The reduction in fatigue life is caused by pitting as a result of premature crack initiation.

A good prediction in corrosion-fatigue life can be made by determining the relevant fatigue parameters via a few simple fatigue tests on the smooth specimen in the environment at the frequency of interest. Researcher reported that the reduction in fatigue strength due to aggressive medium can quantitatively be expressed by the corrosion degradation factor F [45] as can be seen in Eq. 2.8:

$$F = \frac{\sigma_F}{\sigma_{CF}} \quad (2.8)$$

where σ_F is fatigue strength in lab-air and σ_{CF} is fatigue strength in corrosive medium.

3.1. Research Plan

Research Plan can be seen in Figure 3.1 on page 21. This research will be carried out in 4 phases as described below:

In the first phase, a complete defined detail design from the pure titanium artificial hip joint unit will be prepared for product fabrication. The unit consists of 3 parts, i.e. a socket in form of a spherical cup to replace acetabulum, a metal ball to replace the femoral (thigh bone) head and a metal stem to replace the removed femur area. The most importance design parameters in metal on metal hip joint are the diameter of the femoral head, the clearance between the head and the socket and geometry accuracy. These parameters must be carefully determined.

Next, the detail design layout will be sent to manufacturing facilities. Machining process is considered suitable to obtain the basic geometry. Machining and polishing process will be then carried out to achieve the expected geometrical accuracy and surface roughness. From the second phase, a fully functional prototype of the pure titanium metal-metal joint replacement unit will be obtained which satisfy the requirements for dimension tolerance, geometrical accuracy and surface roughness which are crucial in the wear behaviour of the metal-metal bearing.

In the third phase, the prototype will be sent to surface modification facility. By using plasma ion implantation technique, the nitrogen ions will be implanted to the surface of the femoral head and the socket of the prototype to increase surface hardness and wear resistance. Scanning electron micrograph will be used to investigate the morphology and quantity of the Titanium nitrides.

Finally, the fully functional prototype will undergo performance assessment for various mechanical loadings according to its operation condition. Tribology investigation and fatigue testing will be conducted. From the data comparison, it is expected that the prototype will achieve a better performance than the existing joint replacement. The results from this evaluation phase will be used to improve the design and the manufacturing processes.

3.2. Material

Titanium alloys are commonly used in the application of biomechanics parts. One of the applications is for hip joint prostheses. When wearing is one parameter that mostly considered, the Titanium alloy is much more superior compared to that from polyethylene derivatives. Although the wear of existing Ti-6Al-4V Titanium Alloy on Ti-6Al-4V Titanium Alloy hip joint prostheses is much lower than the more widely used polyethylene on metal bearings, there are some concerns about the toxicity of Al and V wear debris in the human body that might negative side effects to the human body. For this reason, the use of Pure Titanium is a potential metal and safer to replace Ti-6Al-4V Titanium Alloy bearings.

The material used for this work is Commercially Pure Titanium. The chemical composition of Commercially Pure Titanium is as follows: N: 0.04%, C: 0.05%, H: 0.003%, Fe: 0.13%, O: 0.11%, Al: 0.49%, S: 0.03, Ti: balance. The mechanical properties of the material are as follows: tensile strength: 430 *MPa*, elongation: 29% and

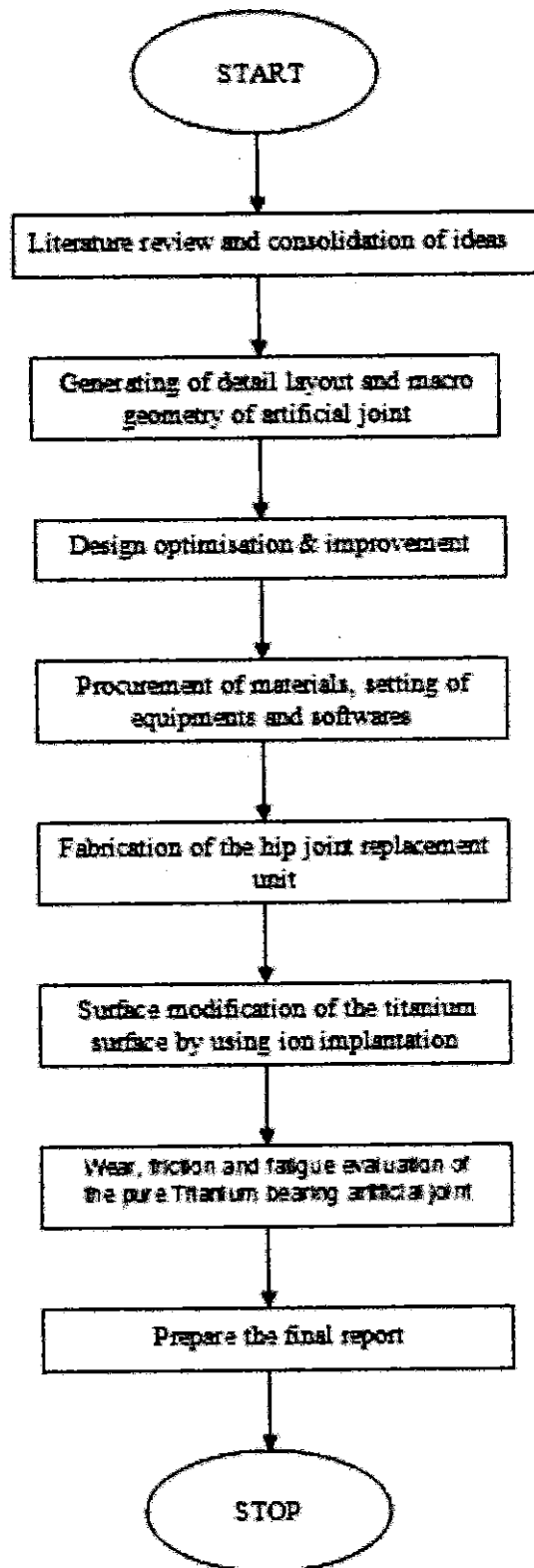


Figure 3.1.: Research Plan

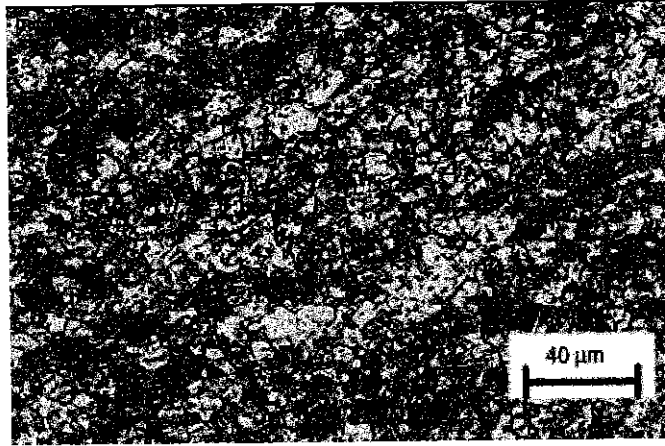


Figure 3.2.: Micro structure Commercially Pure Titanium as the received (Etchant:10% HF-5%HNO₃)

reduction area: 56%

The micro structure of the material is shown in Figure 3.2 on page 22

However, as a pure material, a wear resistant property of Pure Titanium is still needed to be improved by surfaced hardening.

3.3. Ion Implantation

This study is proposed to obtain the optimal parameter of implantation introduced to Commercially Pure Titanium ASTM grade 2. Then, the result is used to treat the surface of femoral head and its socket. The specimen used is in disk-shaped pieces about 30 mm in diameter and 6 mm in thickness. The samples are mirror polished and rinsed with detergent and cleansed in acetone. Experimental design of the sample treatment is shown in Table 3.1 on page 23, and the shape of the sample for ion implantation is shown in Figure 3.3 on page 24

Improvement of the surface hardness and corrosion resistance are depended on the used implant parameter such as energy and dose. Some studies on Nitrogen-ion

Table 3.1.: Experimental design for N ion Implantation

Energy (keV)	Dose (Ion/cm ²)		
	5.00E+16	1.00E+17	2.00E+17
80	1	1	1
100	1	1	1
115	1	1	1

implantation on commercially pure titanium and alloy found an improvement in the electrochemical behavior of the passive film. The materials are subjected to implantation of nitrogen ions in various dose of $5E16$ to $2E17$ *ions/cm²* and at energy of 80 to 120 *keV*. The ion implantation process is performed by the Ion Implantor 200 *kV/2mA* located at BATAN Yogyakarta, Indonesia. The surface modification of titanium was studied with varied doses through nitrogen ion implantation controlled by beam times of the accelerator. The formation mechanism of a nanonitrogenizing layer and its influence on characteristics are studied. The most importance relationship to beam times with implanting energy is dose of nitrogen ions as the experimental basis.

Commercially pure Titanium is cut from a bar of 30 *mm* diameter to form discs 6 *mm* thick. All the specimens is polished using diamond paste of 2 μ *m* before ion implantation. Nitrogen ion is implanted on the polished specimens in target chamber of ICS-SP 1104 200 *keV/2 mA* particle accelerator for different doses, i.e. 0.5×10^{17} , 1.0×10^{17} and 2.0×10^{17} *ions/cm²* at energy of 80, 100 and 115 *keV*. During ion implantation, the vacuum at the target chamber is maintained below 10^{-6} *torr*.

The specimens are polished before settled on the target chamber for ion implantation processing. The N_2 are first introduced to the ion source system equipped having filament which producing electron. These electron strike the nitrogen gas and ionization the N_2 (gas Nitrogen) to (Nitrogen ion), then, selected nitrogen ions are accelerated to the desired energies and bombarded the titanium surface. These outgoing nitrogen ions collided with the atoms of the titanium surface. Some atoms on

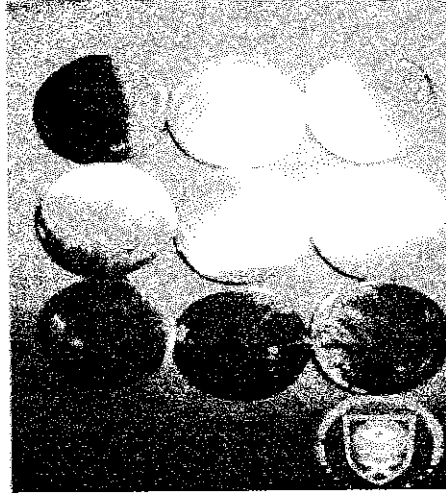


Figure 3.3.: Form and shape of specimen

the lattice of titanium are pushed and the atoms left their original balance positions, called damages and defects in lattices. Actually, most nitrogen ions penetrate into whole surface layer of titanium via ion implantation. The results are that a little of crystalline is converted to amorphous structure on the surface of the titanium and a lot of nitrogen ions occupied the vacancy among lattice or grain boundaries. The ion implantations are performed with the 80, 100 and 115 *keV* energies with 50, 100 and 200 $\mu A/cm^2$ current beam. The dose of implanting ions is give by Eq. 2.1 on page 10. The implanted doses are 2×10^{16} , 4×10^{16} , 6×10^{16} , 8×10^{16} and 1×10^{17} *ions/cm²*. According to the relevant literature, the required dose is 10^{17} *ions/cm²* for ion implantation into titanium.

Using Eq. 2.1 the time required is calculated for each dose ion implantation process. For example, data requirement for time calculation is as follows:

For:

$$D = 1 \times 10^{17} \text{ions/cm}^2$$

$$e = 1.6 \times 10^{-19} \text{coloumb (} q = 1 \text{)}$$

$$i = 100 \mu A$$

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