Mechanical and Physical Properties of Titanium Alloy Ti6Al4V-2Y

by

Ahmad Sofwan Bin Md Yusoff

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

SEPTEMBER 2012

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Mechanical and Physical Properties of Titanium Alloy Ti6Al4V-2Y

by

Ahmad Sofwan Bin Md Yusoff

A project dissertation submitted to the

Mechanical Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfillment of the requirements for the

BACHELOR OF ENGINEERING (Hons)

(MECHANICAL ENGINEERING)

Approved by,

____

(AP Dr. Patthi bin Hussain)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

AHMAD SOFWAN BIN MD YUSOFF

ABSTRACT

This report will discuss the effect of pinning a foreign element, yttrium into titanium alloy using FFC Cambridge Process. Pinning of yttrium is expected to improve mechanical and physical properties of current titanium alloy grade 5. Titanium alloys are widely used in various applications such as in the aerospace industries as bodies of aircraft and in biomedical field as in implants and prosthetic limbs. This project's objectives are to determine the mechanical and physical properties of titanium alloy Ti6Al4V-2Y and to identify the effect of different yttrium concentration pinned into titanium alloy. The methodology of this project is observing the titanium alloy under the Variable Pressure Field Emission Scanning Electron Microscope (VP-FESEM) observation, Vickers Hardness Test, X-Ray Diffraction (XRD), and Optical Microscope observation. The results show that Ti6Al4V-2Y has porous surface. From observation, grain size of Ti6Al4V-2Y is smaller than Ti6Al4V-7Y and subsequently the hardness value of Ti6Al4V-2Y is higher than Ti6Al4V-7Y. It is concluded that mechanical and physical properties of Ti6Al4V-2Y has been determined and the effects of different yttrium concentration pinned into titanium alloy has been identified.

ACKNOWLEDGEMENT

First and foremost, I would like to take this opportunity to direct my utmost indebtedness to my Final Year Project supervisor, AP Dr. Patthi Hussain for his prized supervision and advices throughout the entire project. His provisions, tolerance, and willingness to assist me in the problem or complications that I confronted in my project have contributed tremendously to my project. I would also like to take this opportunity to acknowledge the Final Year Project coordinator, Dr. Hassan Fawad for his effort in guaranteeing the project is developed smoothly within the time frame specified. Last but not least, I would like to give my utmost gratitude and appreciation to my family and fellow friends for their provision and inspiration so that this project can be accomplished fruitfully.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Project Background

There are many metals produced by reduction of their own oxides. According to Fenn, the reduction can be achieved by using different type of process while taking into account relative stability of metal oxides and the oxides of impurities [1]. Reduction of relatively unstable oxides can be conducted by producing the heat until their decomposition temperature (eg. HgO) but the most common reductant is carbon (FeO). Although carbon can be used to reduce almost all metal oxide, very high temperatures are needed to accomplish the reduction which brings problem such as difficulty in stopping the back reaction (MgO) or the formation of stable carbides (Ti_2O). In addition, electrolysis of aqueous or more commonly known as molten salt solution can be used to dissolve the metal oxides.

For compounds that are very stable such as (Al_2O_3), fused salt electrolysis the best way but this technique is suitable for metals that are deposited in the melting point of the metal is less than reduction temperature of its oxide (Al) [2]. For high melting point of metal (Ti), it is basically difficult to hold out molten salt electrolysis. Furthermore, (Ti) was always polluted with significant amount of oxygen. In recent times, the FFC Cambridge process was possible to reduce solid oxide films on titanium foil by making the foil cathodic in a bath of molten calcium chloride and it is also possible to reduce solid titanium oxide pellets. In addition, this process is not only for titanium dioxide but other metal oxides as well. Metal alloys can be fused using FFC Cambridge Process too, and to be specific in this case; Titanium alloy will be pinned together with Yttrium using the stated process. There are 2 similar experiments and tests will be done to the fused alloys which differ in the yttrium concentration which is 2wt %Y and 7wt % Y.

1.2 Problem Statement

This project starts with pinning of yttrium into titanium alloy, Ti6Al4V. Yttrium is a soft, silver-metallic, lustrous and highly crystalline transition metal in group 3 in the periodic table. 2wt % yttrium will be added into the alloy. The pinning action [3] is anticipated to reduce the grain size of titanium alloy and further improving the strength. The pinning of yttrium to titanium alloy will increase workability, adds resistance to high-temperature recrystallization and ominously enhances resistance to high-temperature oxidation.

With another similar experiment will be done using a different concentration of yttrium, we are anticipating different outcomes of both experiments. According to early prediction, different concentration of yttrium will end up causing different change in the titanium alloy physical and mechanical properties.

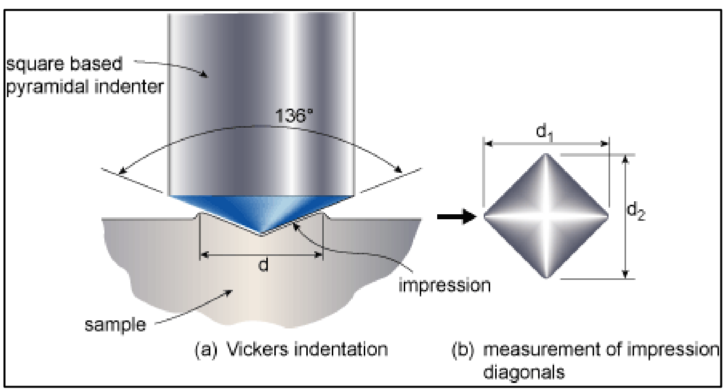

Figure 1: Schematic diagram showing the indenter in Vickers Hardness Test [10].

In this project, we will utilize the concept of determining hardness on a specific material. In this project, Vickers Hardness Test plays a massive role in order to determine the value of hardness of the alloy used, which in this case is Ti6Al4V-2Y. The Vickers Hardness Test is regularly easier to use than other hardness tests since the required calculations are independent of the size of the indenter. Besides, the indenter can be used for all materials regardless of hardness. The result will then be compared to the Ti6Al4V-7Y. The basic principle, as with all communal measures of hardness, is to discern the questioned material's ability to resist plastic deformation from a given source. The Vickers Hardness Test can be used for all metals and has one of the broadest scales among hardness tests. The unit of hardness specified by the test is known as the Diamond Pyramid Hardness (DPH) or the Vickers Pyramid Number (HV). The hardness number is determined by the load over the surface area of the indentation and not the area normal to the force, and is therefore not a pressure. Bear in mind that the hardness number is not really a true property of the material and is an empirical value that should be seen in unification with the experimental methods and hardness scale used.

Table 1.1: A sample of results from testing material using Vickers Hardness Test [10]

Table III - Vickers indentation results.
[Tabela III - Resultados da indentação Vickers.]

Material	Load P, (N)	Diagonal 2a, (μm)	Crack length c, (μm)	Hardness H_v, (GPa)	Toughness K_{Ic}, (MPam$^{1/2}$)
F	4.91	19.2 ± 0.5	21.3 ± 0.5	24.80 ± 1.5	3.16 ± 0.4
	9.81	29.8 ± 0.2	36.5 ± 0.4	19.88 ± 0.5	2.95 ± 0.4
	49.1	69.1 ± 0.5	114.5 ± 1.5	19.05 ± 0.3	2.89 ± 0.3
	98.1	95.5 ± 1.7	179.2 ± 3.7	19.97 ± 0.1	2.89 ± 0.3
	147.2	117.4 ± 0.4	235.7 ± 5. 7	19.81 ± 0.2	2.88 ± 0.2
	196.2	137.5 ± 0.7	287.6 ± 19.2	19.24 ± 0.2	2.91 ± 0.3
	245.3	152.1 ± 2.4	330.1 ± 20.8	19.66 ± 0.3	2.93 ± 0.2
M	4.91	20.3 ± 0.5	22.5 ± 0.4	22.23 ± 1.3	3.09 ± 0.3
	9.81	29.8 ± 0.2	36.6 ± 0.3	18.92 ± 0.3	2.96 ± 0.4
	49.1	70.1 ± 0.5	115.3 ± 6.6	18.51 ± 0.2	2.92 ± 0.2
	98.1	96.3 ± 0.5	179.6 ± 2.6	19.61 ± 0.1	2.90 ± 0.1
	147.2	120.1 ± 2.4	234.7 ± 7.3	18.94 ± 0.2	2.97 ± 0.4
	196.2	139.9 ± 1.2	282.2 ± 9.7	18.57 ± 0.3	3.04 ± 0.3
	245.3	154.8 ± 3.0	323.9 ± 14.5	18.98 ± 0.2	3.06 ± 0.1
C	4.91	21.89 ± 0.7	24.5 ± 0.3	19.02 ± 1.1	2.95 ± 0.4
	9.81	31.2 ± 1.1	38.7 ± 1.6	18.76 ±1.3	2.96 ± 0.4
	49.1	70.2 ± 0.5	115.1 ± 2.1	18.51 ± 0.2	2.91 ± 0.2
	98.1	98.7 ± 0.5	179.4 ± 4.2	18.67 ± 0.1	2.98 ± 0.1
	147.2	121.7 ± 0.6	238.9 ± 16.6	18.43 ± 0.2	3.01 ± 0.2
	196.2	140.9 ± 0.8	278.8 ± 18.2	18.31 ± 0.4	3.14 ± 0.2
	245.3	156.1 ± 1.2	314.6 ± 23.6	18.68 ± 0.3	3.23 ± 0.3

1.3 Objectives

The objectives of this project are:

1. To determine the mechanical and physical properties of titanium alloy Ti6Al4V-2Y.
2. To investigate the effects of different yttrium concentration pinned into titanium alloy.

1.4 Scope of Study

This project is a laboratory-based project in which it highlights the engineering knowledge and analyzing the problem by means of practical laboratories work determine physical and mechanical properties of Ti6Al4V-2Y. This project was focused on the ability to understand the problem encountered and to use the entire necessary tests needed in order to verify the feasibility and reliability of the proposed solution of the problem. In this case, the problem is to determine the physical and mechanical properties of Ti6Al4V-2Y. In order to verify its physical properties, the samples were observed under Optical Microscope and Scanning Electron Microscope (SEM). X-ray Diffraction test was conducted to determine the orientation of a single crystal or grain and find the crystal structure of Ti6Al4V-2Y. It was also to measure the average spacing between layers or row of atoms, size, shape and internal stress of its regions. As for mechanical properties, Vickers test was conducted to determine the hardness value. Results were compared with another sample with different yttrium concentration, Ti6Al4V-7Y. By comparing both results, the effects of different yttrium concentration pinned into titanium alloy could be identified.

CHAPTER 2

LITERATURE REVIEW

2.1 FFC Cambridge Process

Fenn [1] explained that the FFC Cambridge Process is a novel electrolytic method for reducing metal oxide to metal in a molten salt. Since 1950s, millions of dollars were spent to replace the old Kroll process of reducing metal oxides into their pure form. Mohandas stated [3] that eventhough FFC Cambridge Process is very effective in fusing metal alloys; cost limitation will always be the biggest obstacle. There are also some alloys that are difficult or impossible to create by fusion process, because the melting temperature of one element may be greater than the boiling temperature of the other. Bhagat said [4] the FFC Cambridge Process works as the molten salt acts as the electrolyte for the electrochemical reduction of metal oxide to metal. The metal oxide powder is contained in the cathode, and is directly converted into metal by electro-deoxidation. Oxygen ions will carry the current across the cell, and gas is evolved at the anode, leaving pure metal at the cathode. Products of electrochemical reaction can be simply grounded to metal powder.

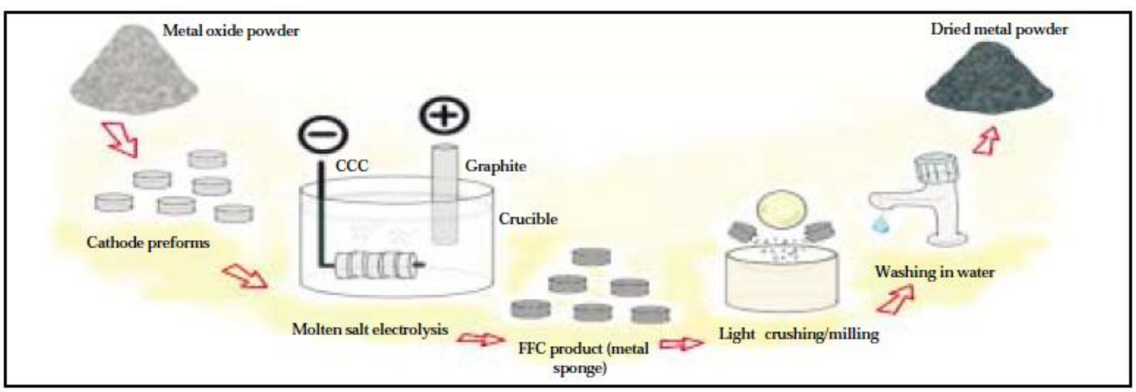

Figure 2.1: Schematic diagram showing the stages of the FFC Cambridge Process [1].

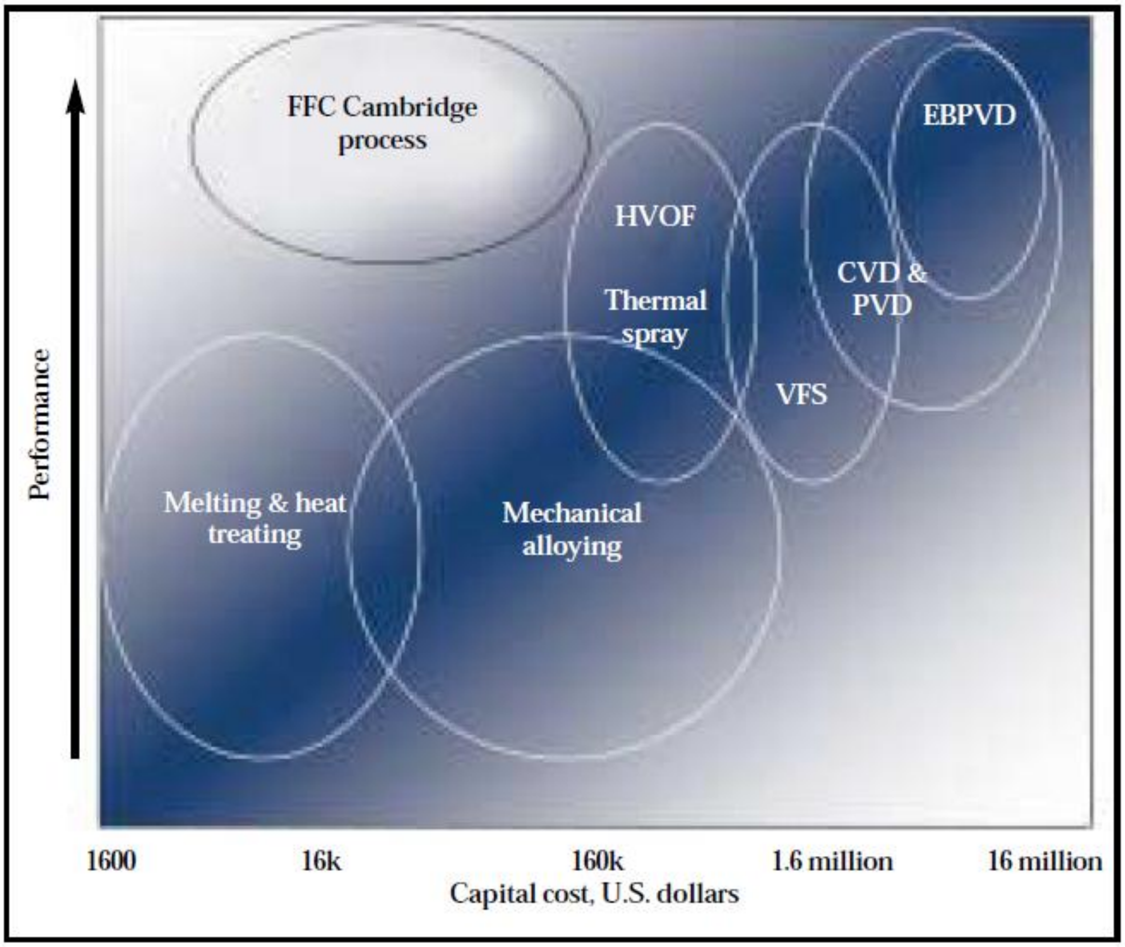

Figure 2.2: Performance and cost consideration for the FFC Cambridge Process and other alloying technologies [3].

2.2 Hydrogen Embrittlement of Aluminium

Lu proposed [2] four general mechanism; (i) formation of a hydride phase; (ii) enhanced local plasticity; (iii) grain boundary weakening and (iv) blister and bubble formation. For these mechanisms to be operational, however, a critical local concentration of H is required, either to form a hydride phase or to initiate cracking at microvoids and grain boundaries. One of the outstanding problems in the current theories of hydrogen embrittlement is the lack of a comprehensive and coherent atomistic mechanism to account for the critical H concentrations at crack tips. Moreover, it is widely observed that H-enhanced dislocation mobility is a prelude to the embrittlement and that the fracture planes coincide with the slip plane of the material, which is not the typical situation; how all these phenomena come about still remains a mystery. It is generally believed that dislocations are central to H embrittlement phenomena, and a large body of work has been dedicated to elucidate hydrogen-dislocation interaction and its consequences on embrittlement. Vacancies, being ubiquitously present in solids and having the ability to act as impurity traps, could play a central role in the embrittlement process, but detailed arguments about this role or estimates of its relative importance are totally lacking.

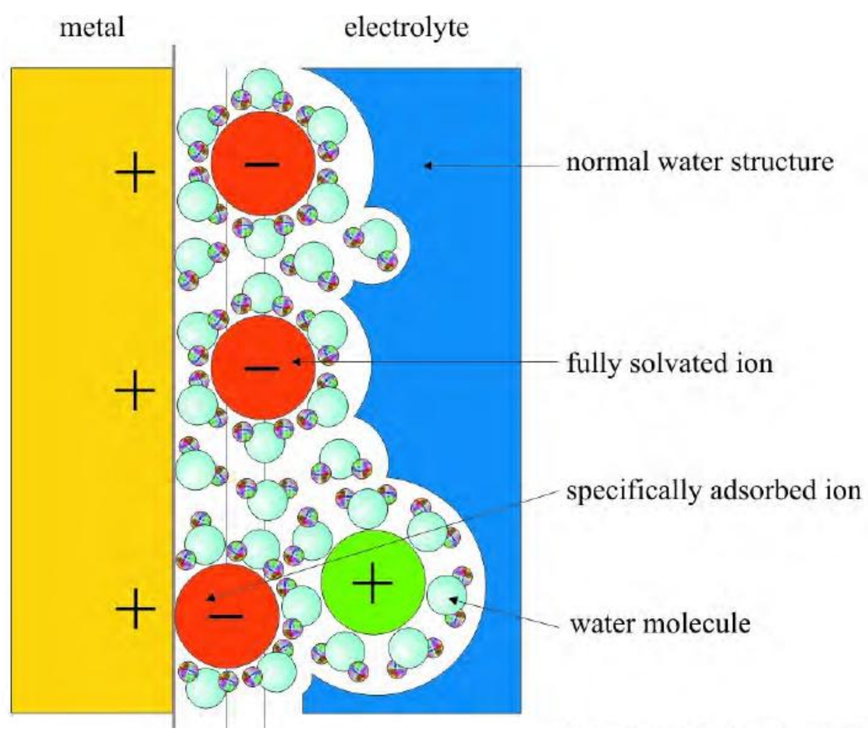

Figure 2.3: Schematic diagram of metal/electrolyte interface [2]

2.3 Ti-6 Al-4 V Titanium Alloys

General characteristics

Titanium alloy have high strength, low weight ratio and outstanding corrosion resistance. That's why it has been used in a wide range of successful applications which demand high levels of consistent routine in medical as well as in aerospace, automotive, chemical plant, and other major industries. In addition, titanium has replaced heavier, less serviceable or less cost effective materials. Designing with titanium while consider all factors into account has resulted in consistent, economic and more durable systems or components. There are many different grade of titanium available. Ti6Al4V is defines as grade 5. It is the most frequently used alloy and stronger than pure titanium but have same stiffness and thermal properties. Furthermore, it is heat treatable. This grade is an outstanding mixture of strength, resistance of corrosion, weld and fabric-ability. Therefore, it is used broadly in wide range of major industries.

Special characteristics

Ti6Al4V is the most common used titanium alloy. It features good machine-ability and excellent mechanical properties. The Ti6Al4V alloy offers the excellent performance for a multiple of weight reduction applications in major industries. It is also has numerous applications in the medical industry and has excellent biocompatibility [7].

Applications

Ti6Al4V is typically used for [9]:

- Direct Manufacturing of parts and prototypes for racing and aerospace industry
- Biomechanical applications, such as implants and prosthesis limbs
- Marine applications
- Chemical industry
- Gas turbines

a **CHEMICAL SPECIFICATION**

	Arcam Ti6Al4V, Typical	Ti6Al4V, Required*	Ti6Al4V, Required**
Aluminium, Al	6%	5,5–6,75%	5,5–6,75%
Vanadium, V	4%	3,5–4,5%	3,5–4,5%
Carbon, C	0,03%	< 0,1%	< 0,08%
Iron, Fe	0,1%	< 0,3%	< 0,3%
Oxygen, O	0,15%	< 0,2%	< 0,2%
Nitrogen, N	0,01%	< 0,05%	< 0,05%
Hydrogen, H	0,003%	< 0,015%	< 0,015%
Titanium, Ti	Balance	Balance	Balance

*ASTM F1108 (cast material) **ASTM F1472 (wrought material)

b **MECHANICAL PROPERTIES**

	Arcam Ti6Al4V, Typical	Ti6Al4V, Required**	Ti6Al4V, Required***
Yield Strength (Rp 0,2)	950 MPa	758 MPa	860 MPa
Ultimate Tensile Strength (Rm)	1020 MPa	860 MPa	930 MPa
Elongation	14%	>8%	>10%
Reduction of Area	40%	>14%	>25%
Fatigue strength* @ 600 MPa	>10,000,000 cycles		
Rockwell Hardness	33 HRC		
Modulus of Elasticity	120 GPa		

*After Hot Isostatic Pressing **ASTM F1108 (cast material) ***ASTM F1472 (wrought material)
The mechanical properties of materials produced in the EBM process are comparable to wrought annealed materials and are better than cast materials.

Figure 2.4: (a) Chemical specification comparison between Arcam Ti6Al4V, cast Ti6Al4V and wrought Ti6Al4V and (b) Mechanical properties comparison between Arcam Ti6Al4V, cast Ti6Al4V and wrought Ti6Al4V [7].

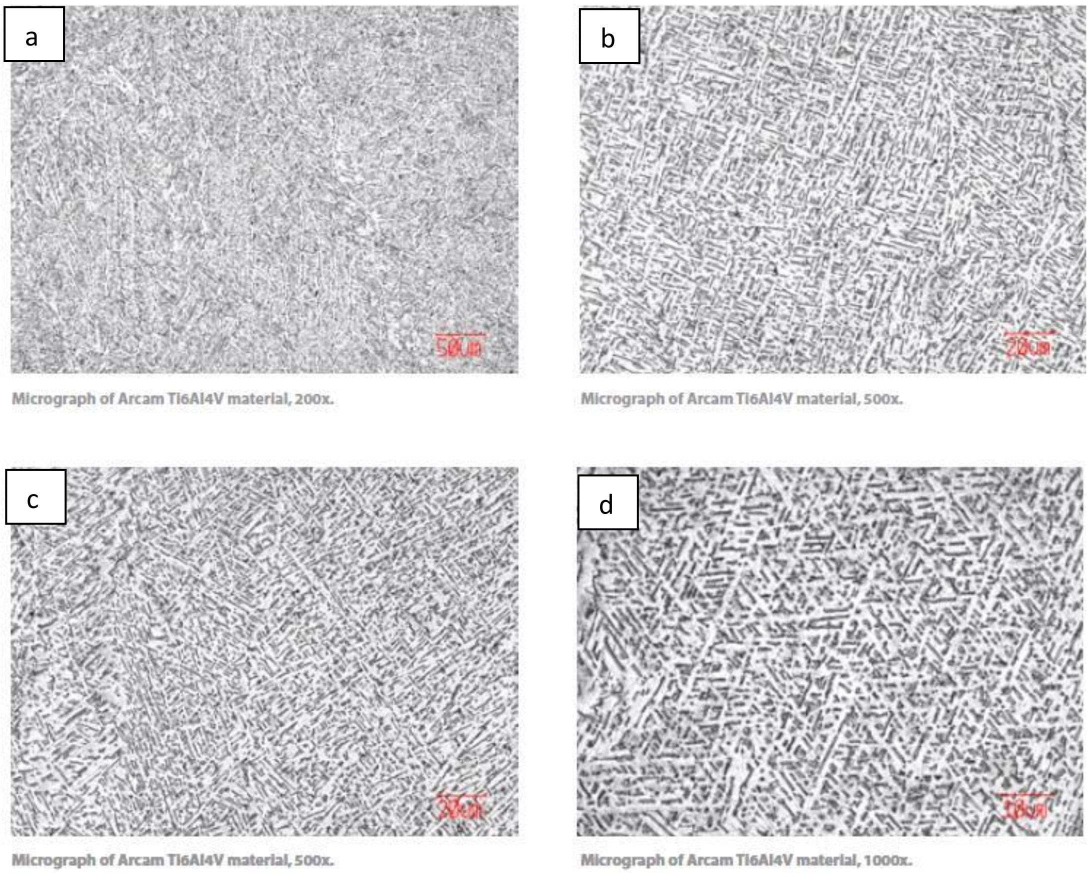

Figure 2.5: Micrograph of Arcam Ti6Al4V material: (a) 200X zoom, (b) 500X zoom, (c) 500X zoom and (d) 1000X zoom [7].

Arcam stated that their Ti6Al4V parts manufactured in the EBM process have a microstructure better than cast Ti6Al4V comprising a lamellar α-phase with larger β-grains, and with a higher density and significantly finer grain, thanks to the rapid cooling of the melt pool. The build chamber is kept at a raised temperature throughout the entire build, and the material thus comes out of the EBM process in a naturally aged condition [7].

2.4 X-ray Diffraction

According to pdx [11], X-ray diffraction techniques are a non-destructive test which reveals information about the crystal structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident beam that will be diffracted and transmitted. The diffraction pattern will be recorded.

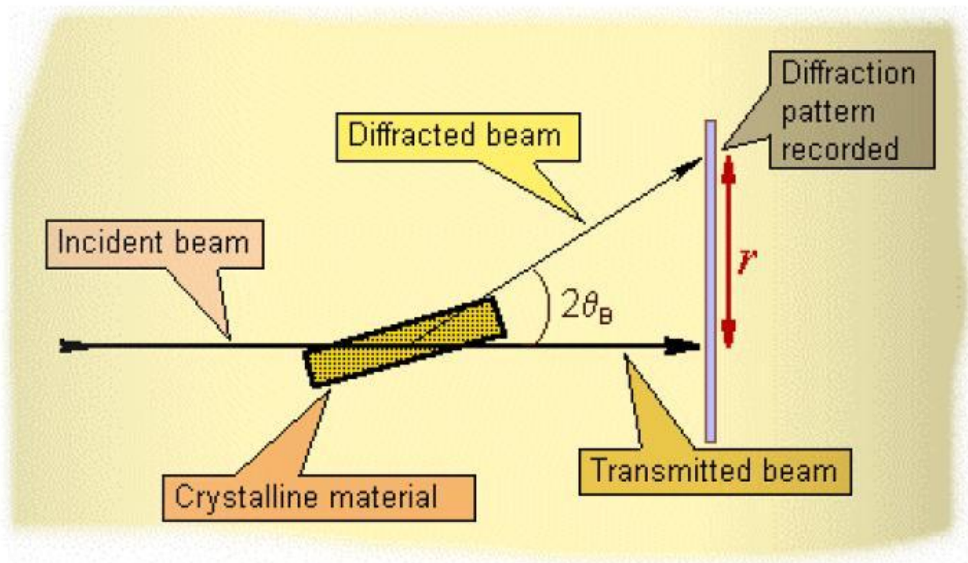

Figure 2.6: Operation of X-ray Diffraction [11].

Implementation

XRD users must be trained in operation and basic radiation safety in order to use the XRD machine. There are strictly no exceptions to ignore this rule. In Universiti Teknologi PETRONAS (UTP), students did not have the qualification to use the XRD machine. The entire test will be conducted by technician who has the authority to use the machine. Basically, there are 3 steps to use XRD machine which are sample preparation, XRD log and operation.

2.5 Vickers Hardness Test

According to Instron [10], Vickers test methods are defined in the following standards:

- ASTM E384 – micro force ranges – 10g to 1kg
- ASTM E92 – macro force ranges - 1kg to 100kg
- ISO 6507-1,2,3 – micro and macro ranges

All Vickers use a 136° pyramidal diamond indenter that forms a square indent [10].

- The indenter is pressed into the sample by an accurately controlled test force.
- The force is maintained for a specific dwell time, normally 10 – 15 seconds.
- After the dwell time is complete, the indenter is removed leaving an indent in the sample that appears square shaped on the surface.
- The size of the indent is determined optically by measuring the two diagonals of the square indent.
- The Vickers hardness number is a function of the test force divided by the surface area of the indent. The average of the two diagonals is used in the following formula to calculate the Vickers hardness.

HV = Constant x test force / indent diagonal squared

Because of the wide test force range, the Vickers test can be used on almost any metallic material. The part size is only limited by the testing instrument's capacity [10].

Strengths

1. One scale covers the entire hardness range.
2. A wide range of test forces to suit every application.
3. Nondestructive, sample can normally be used.

Weaknesses

1. The main drawback of the Vickers test is the need to optically measure the indent size. This requires that the test point be highly finished to be able to see the indent well enough to make an accurate measurement.
2. Slow. Testing can take 30 seconds not counting the sample preparation time.

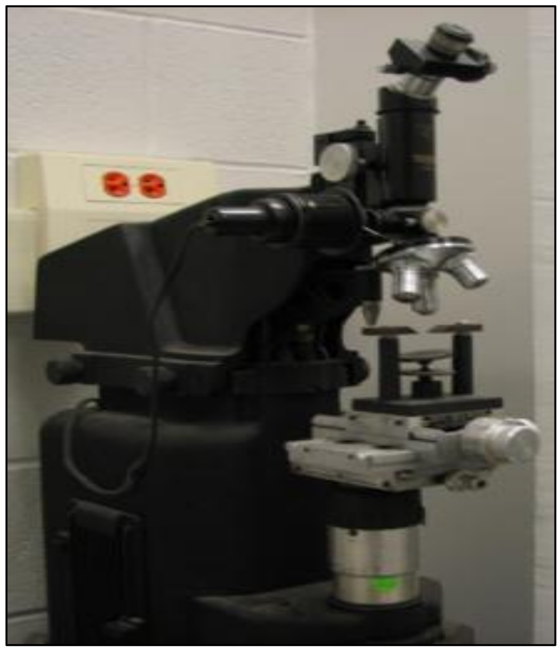

Figure 2.7: Vickers Hardness Tester Machine [10]

2.6 Field Emission Scanning Electron Microscope

History

FESEM is the acronym for Field Emission Scanning Electron Microscope. It was Ernest Ruska (1906 - 1987) whom in his PhD. thesis mentioned that the potential for electrons to be used in a microscopy area. In 1933 Ruska and Knoll constructed the first electron microscope and in 1935 Knoll wrote the first work describing the concept of a SEM. In 1938 Von Ardenne built a scanning transmission microscope (STEM) adding coils to a transmission electron microscope.

According to Infohost[12] the first SEM used to study a solid surface was described by Zworykin et al (1942) working for the RCA laboratories in the United States. As a practice in the early days the gun was situated in the bottom so the specimen chamber and column were high enough for the operator but then again the specimen might fall down the column. A resolution of 50 nm was accomplished with this microscope. The first micrographs displaying the striking three-dimensional imaging capability were obtained in Cambridge at the Engineering Department in 1952 by Dennis McMullan who was continuing the work by Ken Sander (both under C. W. Oatley supervision). The next important step was also in Cambridge when Oatley enhanced the secondary electron detector by adding a scintillator to convert electrons to photons, and let the way for advancement in signal to noise ratio.

Nowadays, three-dimensional features can be observed due to the large Depth of Field available in the FESEM. The addition of energy dispersive X-ray detector combined with digital image processing is a powerful tool in the study of materials, allowing good chemical analysis of the material. The FESEM is a major tool in materials science research and development.

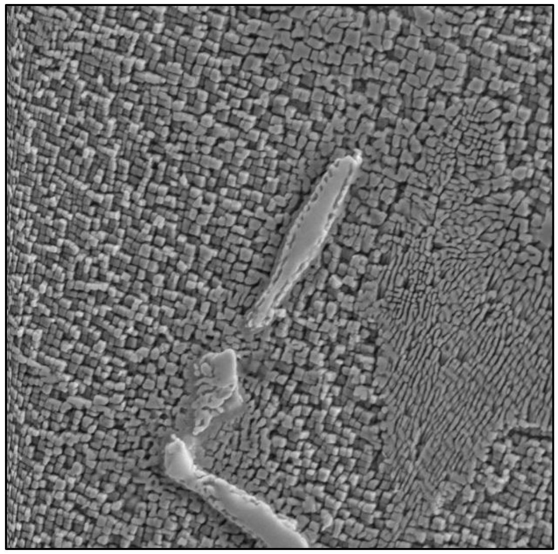

Figure 2.8: A FESEM image showing a super alloy microstructure [12]

Principles

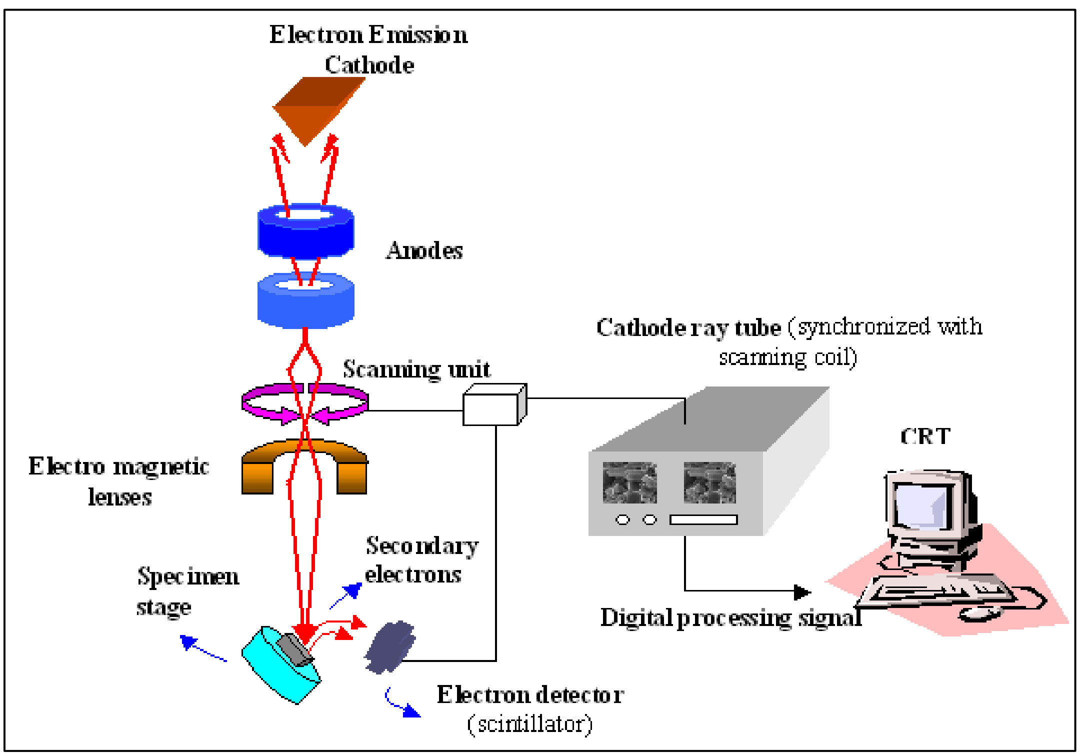

Figure 2.9: FESEM principle [12]

As stated by Infohost[12] under vacuum, electrons generated by a Field Emission Source are accelerated in a field gradient. The beam passes through Electromagnetic

Lenses, converging onto the specimen. As result of this bombardment different types of electrons are emitted from the specimen. A detector catches the secondary electrons and an image of the sample surface is constructed by comparing the intensity of these secondary electrons to the scanning primary electron beam. Finally the image is displayed on a monitor.

Specimen preparation

Some materials can be examined in the Field Emission Scanning Electron Microscope, with virtually no specimen preparation (e.g. metals). Specimen thickness is not a consideration, unlike transmission electron microscopy (TEM). But the sample preparation is often necessary [12]. This preparation is function of the material (biological, ceramic, metal) and it is highly dependent on the type of information we are attempting to derive. The specimen must be representative of the population if the analysis results are to be extrapolated to the entire population.

To generalize the following fundamentals must be taken in consideration:

1. Because the signal detection is influenced by the surface topography, it is highly recommended to polish the sample surface.
2. For fracture studies, the sample should be in an unaltered state.
3. Foreign substance can be deposit at the specimen surface (usually carbonaceous material derived from breakdown of a hydrocarbon. This phenomenon is called contamination. To avoid it is necessary to ensure that the specimen is properly clean and thoroughly degreased and dried.
4. If the material is an insulator it must be coated, providing a path to release the absorbed electrons in the specimen.
5. When any kind of mounting is used, although the specimen itself may be a conductor an appropriate connection must be created to avoid the charging.
6. In case of non-conductive material, the recommended coating is carbon if you are planning to do an energy dispersive spectroscopy (EDS).
7. The specimen size is limited by considerations of accommodation in the specimen stage.

CHAPTER 3

METHODOLOGY

3.1 Planned Progress Flow of the Project

This section consists of the planned progress flow for this "Mechanical and Physical Properties of Titanium Alloy Ti6Al4V-2Y" project. After the project title selection, a preliminary research work was done including the consultation with the supervisor to get the overview and basic understanding of the selected project. The details and information of the project which was given by the supervisor was studied in order to understand the project background, problem statement, objectives and scope of study for this particular project. After fully understood the basics of the project, a more thorough research work was done for the literature review of this project. During this stage, all related materials and information including the previous researches and journals were retrieved as much as possible for a more thorough and deep understanding on the effects and significance of yttrium addition into titanium alloy.

Then, after completing all the preliminary research works, the main stage of this project was inaugurated. Firstly, the sample of a complete pinned titanium alloy was acquired. In this case, the sample was acquired by the reducing process of TiO_2 into pure titanium known as FFC Cambridge Process; this process was reported to be done at Cambridge University as the local universities do not have the necessary equipment to undergo the process. After a fresh sample was acquired, sample was observed under optical microscope with 150X magnification. The images received by optical microscope were then compared with images from FESEM observation.

Once images from FESEM were developed, microstructure analysis was done. From the analysis, grain size was determined. From FESEM images, element distribution across the surface was also possible to be measured. After the surface of sample has completely been analyzed, Vickers hardness test was done to the surface in order to get the hardness value of both samples, Ti6Al4V-2Y and Ti6Al4V-7Y. After completing microstructure analysis, the sample was put under XRD inspection in order to analyze the diffraction pattern, along with all the raw sample of each element involved in the forming of the alloy. All specimens were processed to powder form. After each diffraction pattern has been acquired, patterns were analyzed, whether new peak was formed or was there any peak loss within the new pattern of Ti6Al4V-2Y.

Finally, the final report was produced and compiled based on the overall study and the results from each test and analysis. All details regarding the planned progress flow for this project are shown in the chart in Figure 3.2 and Project Key Milestone Chart as in Table 3.1 and Table 3.2 respectively.

3.2 Research Methodology

The study was divided into 2 phases for FYP I and FYP II as follows:

3.2.1 FYP I

The experimental study was divided into 2 phases:

3.2.1.1 Literature Review

- FFC Cambridge Process
- Hydrogen Embrittlement of Aluminium
- Ti6Al4V Titanium Alloy

3.2.1.2 Preparation of equipment, tools and facilities.

- Laboratory manual (Guideline) and preparation of the experiment and practical work
- Utilize the equipment provided by Mechanical Engineering Department to commence the tests and observation.
- Variable Pressure Field Emission Scanning Electron Microscope (VP-FESEM), Optical Microscope, X-Ray Diffraction (XRD) machine and Vickers Hardness Tester.

3.2.2 FYP II

- Experimental studies on the mechanical and physical properties of titanium alloy were carried out during the semester of September 2012.
- Based on observation results, further analysis on the effect of yttrium addition into titanium alloy was conducted.

3.3 Tools and Software Required

1) Field Emission Scanning Electron Microscope

1) Vickers Hardness Test

2) Inverted Microscope

3) X-Ray Diffraction

Figure 3.1: VP FESEM used for capturing samples' images [12]

3.4 Steps Taken

The chart below shows the steps taken throughout the course of this project.

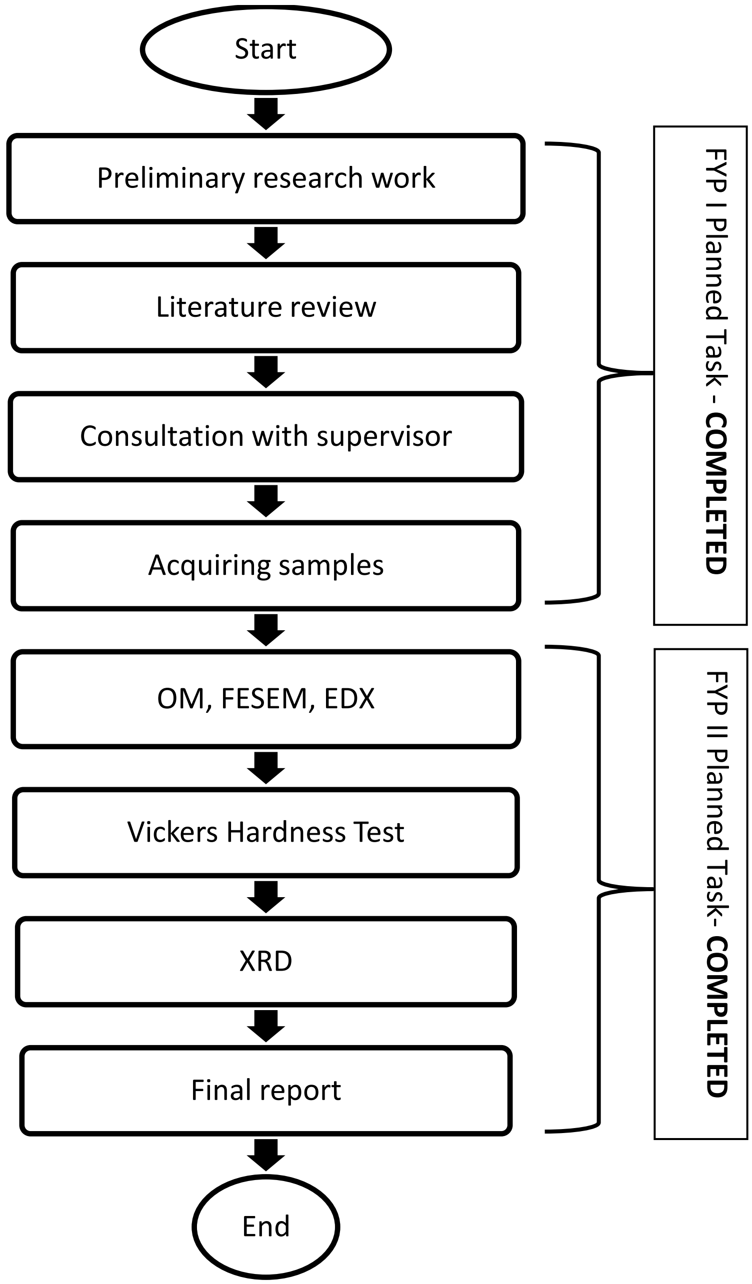

Figure 3.2: Steps taken throughout the project

3.5 Project Timeline

Several targets had been set for the FYP I. Table 3.1 shows the project activities and key milestones for FYP I and Table 3.2 shows project activities for FYP II.

Table 3.1: Project Activities and Key Milestones for FYP I

No.	Detail/ Week	1	2	3	4	5	6	7	MID SEM BREAK	8	9	10	11	12	13	14
1	Selection of Project Topic	■														
2	Preliminary Research Work		■	■	■	■	■									
3	Submission of Extended Proposal							○								
4	Proposal Defense										○					
5	Execution of project											■	■	■	■	
6	Submission of Interim Draft Report														○	
7	Submission of Interim Report															○

Legends:

■ Project Activity

○ Key Milestone

Table 3.2: Project Activities and Key Milestones for FYP II

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project work								MID SEM BREAK								
2	Submission of progress report																
3	Project work continues																
4	Submission of Draft report													○			
5	Submission of dissertation (soft bound)														○		
6	Submission of Technical paper														○		
7	VIVA															○	
8	Submission of dissertation (hard bound)																○

Legends:

Project Activity

○ Key Milestone

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Data Collection

This project is aimed to determine the mechanical and physical properties of titanium alloy Ti6Al4V-2Y. These properties and analysis could be obtained using tools such as Optical Microscope, Field Emission Scanning Electron Microscope (FESEM), X-Ray Diffraction and Vickers Hardness Test. All data gathered was then compared to titanium alloy Ti6Al4V-7Y observation results to identify how different concentration of yttrium would affect these two alloys in any way.

4.2 Microstructure

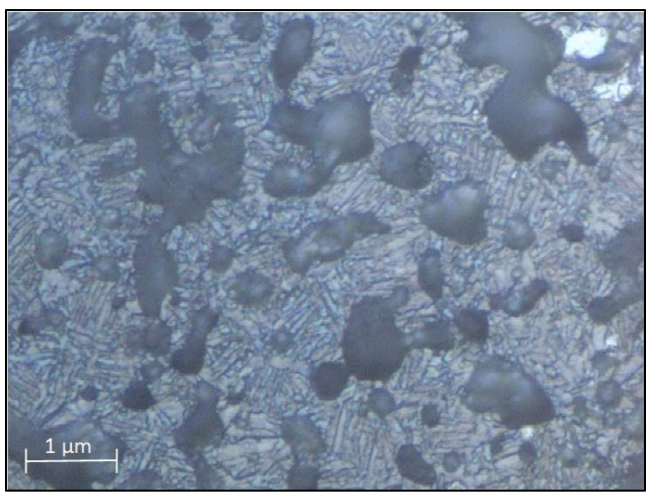

Figure 4.1: Image of alloys' surface using optical microscope under 150X magnification

As seen in Figure 4.1, the surface of this alloy contains quite a number of porosities. This is due to its suggested biomedical application such as implants, prosthetic limbs and various medical usages [13]. Processed under the FFC process is also a contributing factor to the buildup of the porosities on the alloy surface [1]. Arcam stated that [7], porosity in titanium alloy is used to lower the hardness and tensile strength but in the same time increasing the elasticity of the material making it very suitable for medical purposes.

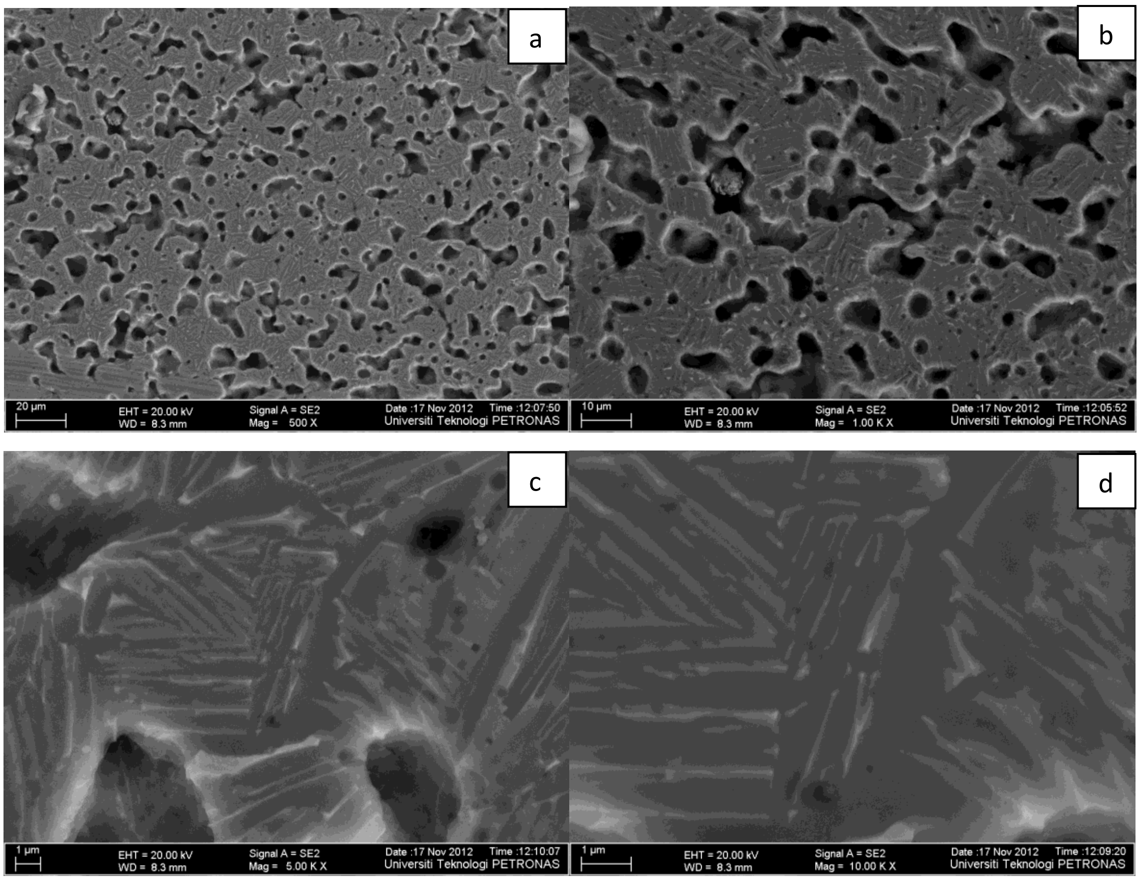

Figure 4.2: FESEM images of Ti6Al4V-2Y under: a) 500X magnification b) 1000X magnification c) 5000X magnification d) 10000X magnification

Based on images a), b), c) and d) in Figure 4.2, titanium alloy Ti6Al4V-2Y has two major portions consisted of dark and bright areas. Dark zones had been determined as the porosities and the bright zones were the grain boundaries of alloy. Grain size was determined using the intercept technique. In simple terms, a random straight line was drawn through the micrograph image. Then, the numbers of grain boundaries intersecting with the line were counted. The average grain size was determined by dividing the number of intersections by the actual line length.

The estimation of grain size can be calculated using the following method;

Average grain size =1/(number of intersections/actual length of the line).
where actual line length = measured length divided by magnification

From calculation, it is decided that the average grain size of titanium alloy Ti6Al4V-2Y is **1.44μm** and the average grain size of Ti6Al4V-7Y is **2.53μm.**

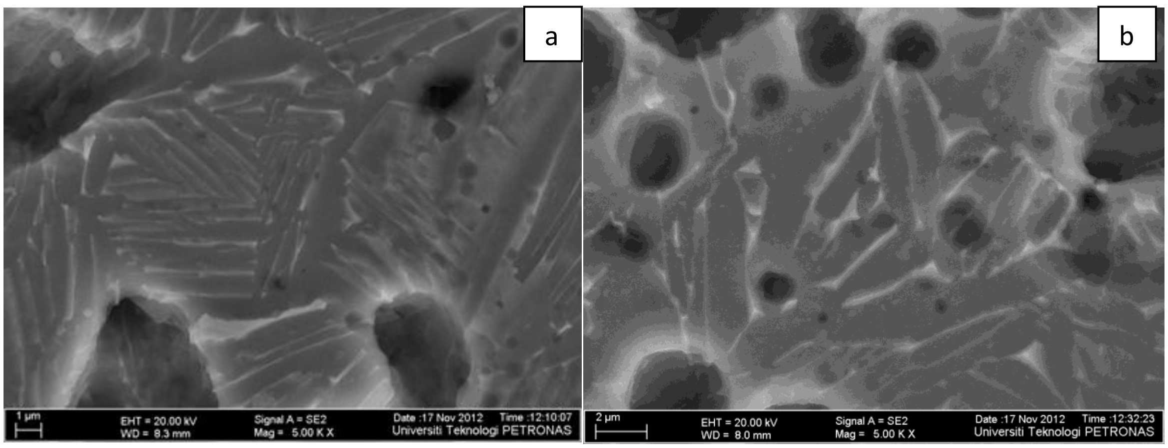

Figure 4.3: Comparison of grain boundaries of both alloys : a) Ti 6Al 4V-2Y at 5000X magnification b) Ti6Al4V-7Y at 5000X magnification

Image a) and b) in Figure 4.3 shows the comparison between both alloys in the grain arrangement and grain boundaries size. Raw image analysis shows that Ti6Al4V-2Y exhibits a neater, tight grain arrangement which leads to an early conclusion that Ti6Al4V-2Y has higher hardness.

Hardness test were done to both specimens in order to acquire the hardness value of both titanium alloys. Test was done under the load of 1000gf and dwell time of 15 seconds for every run. Acquired value of hardness is shown in Table 4.1.

Table 4.1: Hardness Value of tested alloys

ALLOYS	Ti6Al4V-2Y	Ti6Al4V-7Y
Value 1	165.0 HV	186.5 HV
Value 2	160.3 HV	145.0 HV
Value 3	159.7 HV	136.8 HV
Average value	161.7 HV	156.1 HV

4.3 Energy Dispersive X-Ray Analysis

4.3.1 Broad Spectrum Analysis

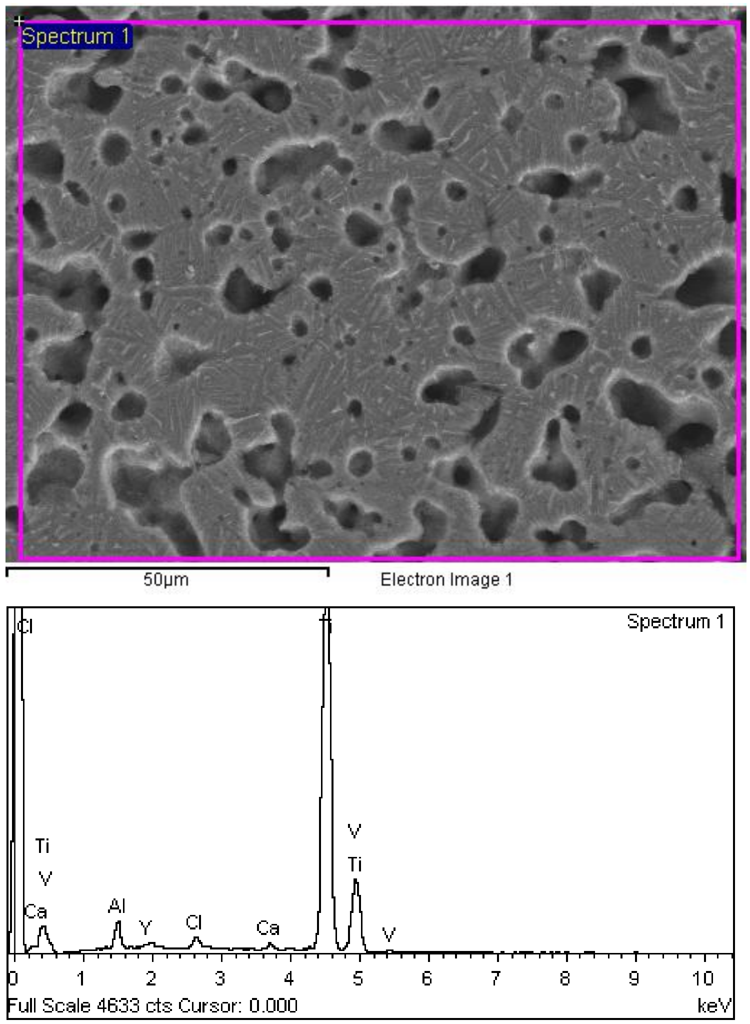

Figure 4.4: EDX analysis on spectrum 1

Table 4.2: Element distribution on spectrum 1

Element	Weight %	Atomic %
Al	3.31	5.74
Cl	1.06	1.40
Ca	0.73	0.85
Ti	88.68	86.67
V	5.25	4.82
Y	0.98	0.52

EDX analysis on spectrum 1 is shown in Figure 4.4. The spectrum was captured using FESEM image under 1000X magnification. Based on Table 4.2, Ti populates the alloy with the highest percentage followed by Al, V, Cl, Ca, and Y. The existence of Ca and Cl are believed to come from the FFC Cambridge Process which uses Ca and Cl as their main electrolyte in producing titanium alloys [1].

4.3.2 Localized Analysis

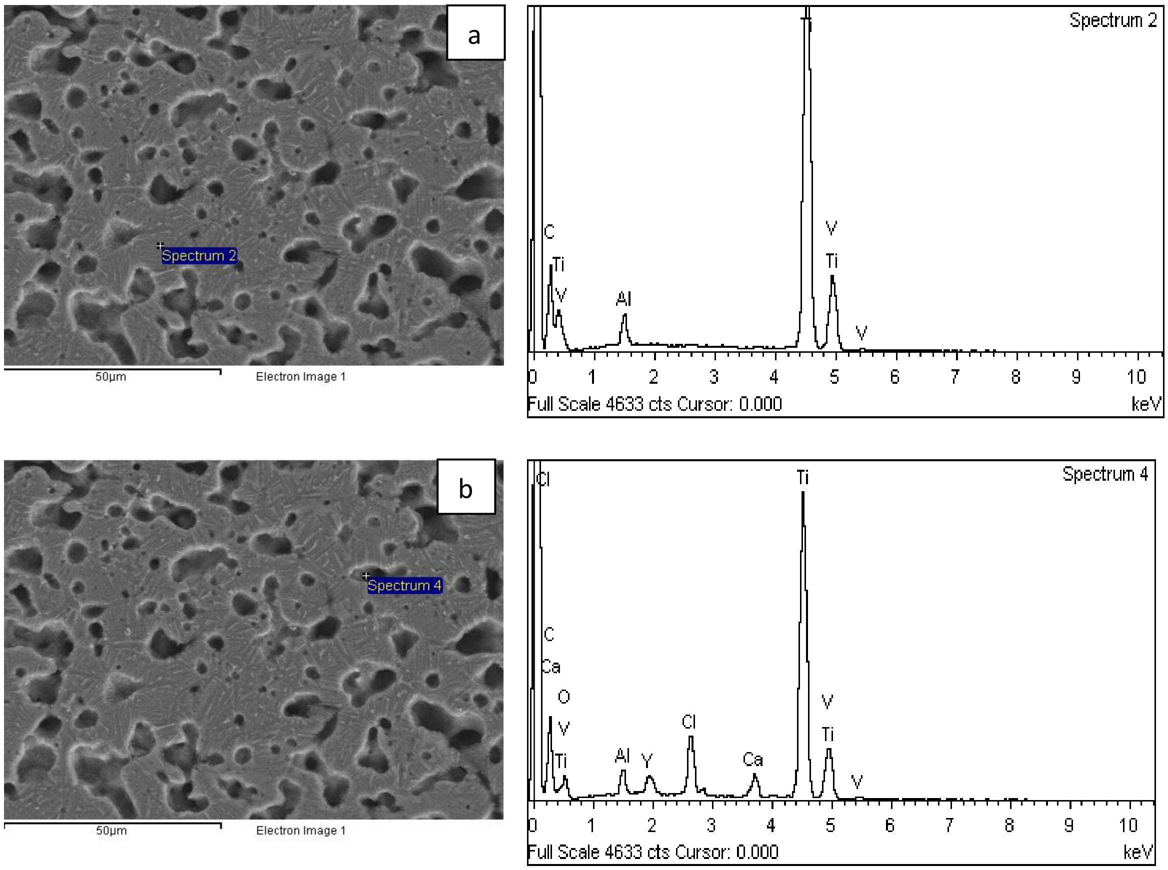

Figure 4.5: EDX analysis on: a) spectrum 2 b) spectrum 4

Different results were obtained from different spot of the EDX analysis. As in images a and b in Figure 4.5, and referring to Table 4.3 and Table 4.4 as references, there was no trace of yttrium in the grain boundaries zones. Most of the yttrium is located at the dark zones which is the porosities of the alloy’s surface. As mentioned before, traces of Ca and Cl were expected to be seen as they were the residue from the FFC Cambridge Process. Traces of C and O were identified and it is decided that they were from the oxidation process to the alloy during the FFC Cambridge Process.

Table 4.3: Element distribution on spectrum 2

Element	Weight %	Atomic %
Al	2.11	1.83
Ti	59.84	29.23
V	3.48	1.60
C	34.57	67.34

Table 6: Element distribution on spectrum 4

Element	Weight %	Atomic %
Al	1.54	1.25
Cl	4.28	2.65
Ca	0.73	0.85
Ti	41.57	19.05
V	2.33	1.00
Y	2.85	0.70
C	25.90	47.33
O	19.68	27.00

4.4 Element Mapping

Ti6Al4V-2Y Element Mapping

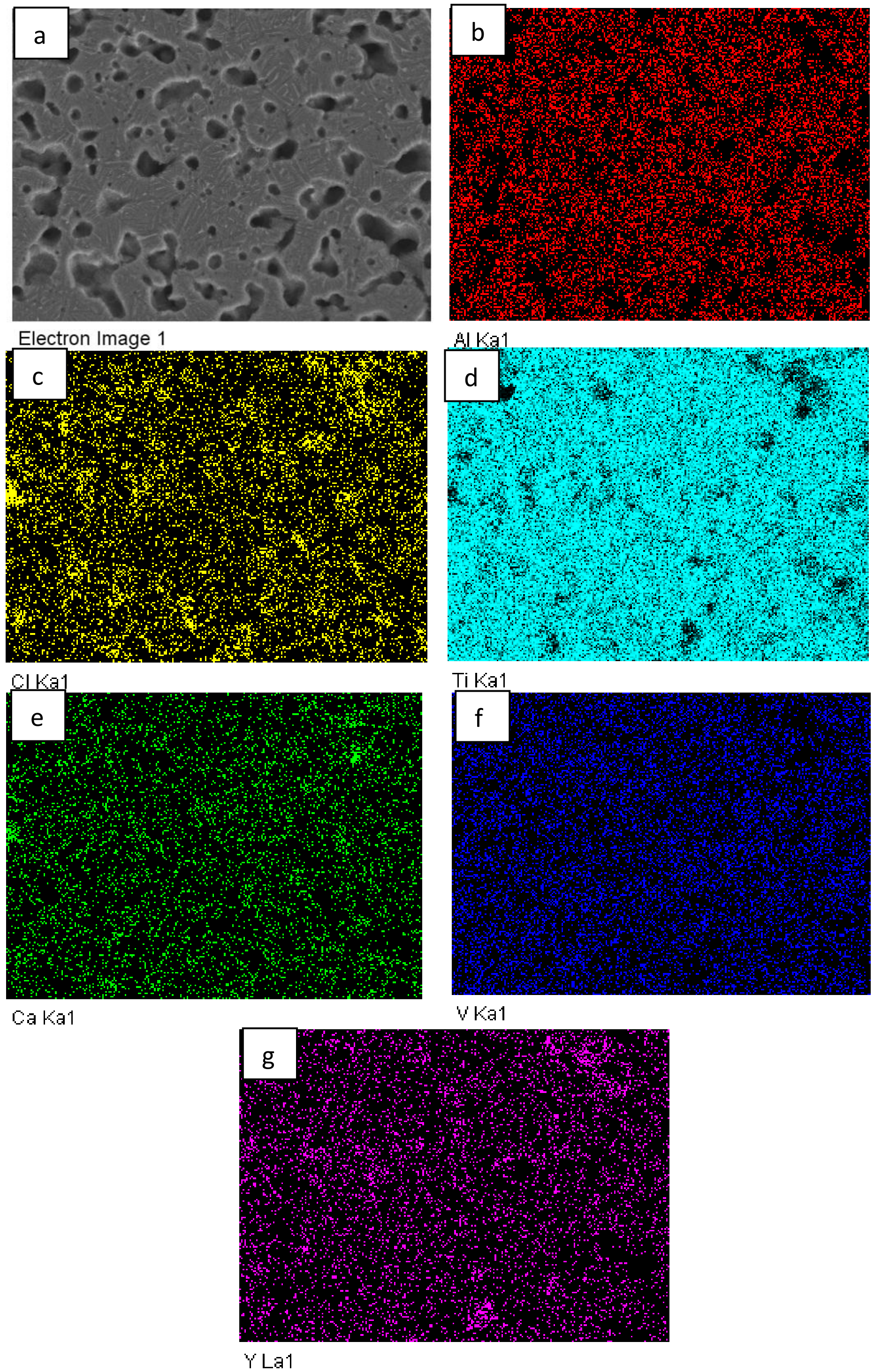

Figure 4.6: Element mapping of Ti 6Al 4V-2Y

Elements distributions are portrayed in image b) to g) in Figure 4.6, where all elements are distributed uniformly across the alloy. Yttrium in image g can be seen populating around the surface uniformly which means that the pinning action of yttrium into titanium alloy was successful. Furthermore, the uniform distribution would make sure that the alloy strength is well distributed within the alloy.

From Vickers Hardness Test, it is proven that Ti6Al4V-2Y has a higher value of hardness than Ti6Al4V-7Y. This means that Ti6Al4V-2Y is a stronger alloy in terms of hardness. Microstructure analysis of grain size also shows that Ti6Al4V-2Y has smaller grain size which will result in less slip in between the grain boundaries when load is applied. This will results in a more resilient material that have a bigger elasticity value. This shows that the less concentration of yttrium element pinned to the titanium alloy would provide better physical and mechanical properties of the alloys.

Each and every one image that has been retrieved from either OM or FESEM shows clearly that the surface of the titanium alloy Ti6Al4V-2Y contains porosities. This condition exists as the alloy itself is made from titanium foam in powder foam. Titanium foam itself is porous; with highly interconnected pores and wall struts that contains micron scale interconnected porosities [13]. These porosities are acting as binder to the grain in order to increase their elasticity and permeability and make this alloy very suitable for biomedical purpose. In this instance, prosthetic limbs and implants are mostly made from titanium alloy which uses titanium foam as the basic ingredient.

4.5 X-Ray Diffraction

Phase Identification

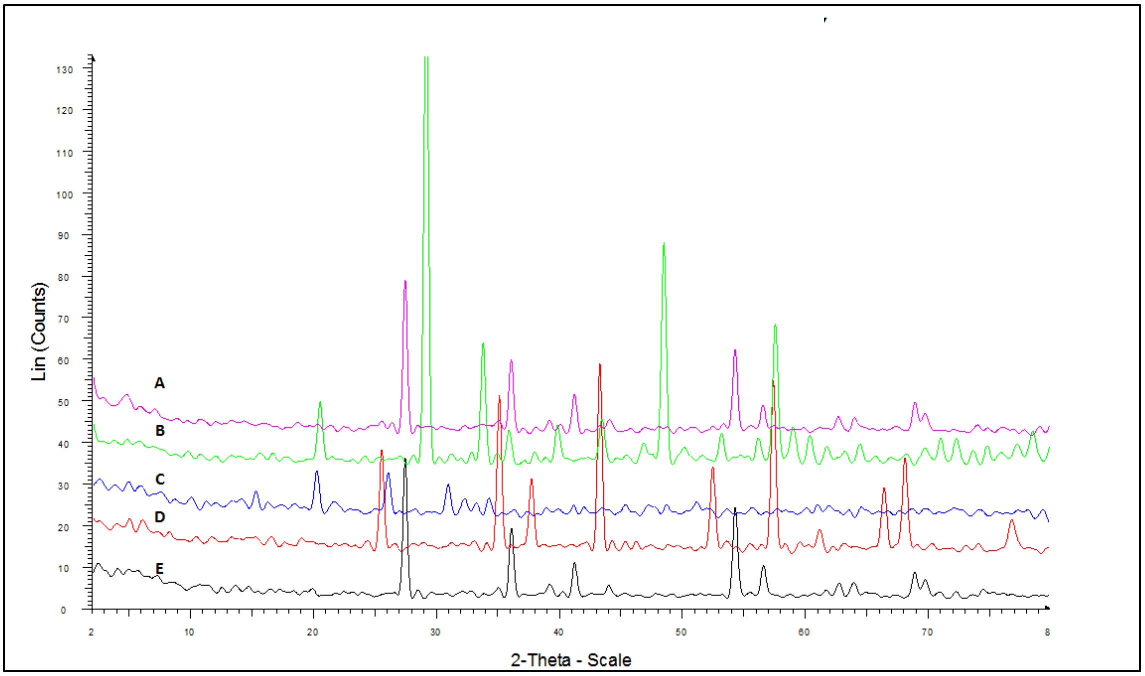

Figure 4.7: XRD peaks for: (**A**) Ti6Al4V-2Y compressed powder (**B**) Y_2O_3 powder (**C**) V_2O_5 powder (**D**) Al_2O_3 powder (**E**) TiO_2 powder

Five specimens were examined using XRD which are; Ti6Al4V-2Y compressed powder, Y_2O_3 powder, V_2O_5 powder, Al_2O_3 powder and TiO_2 powder. Comparing TiO_2 diffraction pattern and Ti6Al4V-2Y pattern, they are almost identical with some introduction of new peaks and the loss of some peaks too which means Ti6Al4V-2Y and TiO_2 have the same crystal structure. The as-received Ti6A4V-2Y powder was mild orange in appearance, indicating a mixture of V_2O_5, Al_2O_3, TiO_2, and Y_2O_3. As V_2O_5 is also orange in appearance, and the others element were white in appearance, it is logical that the end product will become orange too. Addition of 2wt % Y in titanium alloy has little effect in changing the peak formed for TiO_2 as per Figure 4.7.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the objectives, it is determined that Ti6Al4V-2Y has porous surface. Calculation shows that the average grain size of Ti6Al4V-2Y is 1.44µm and the average grain size of Ti6Al4V-7Y is 2.53µm. From element mapping using FESEM images, all elements were well distributed across the alloy. On the other hand, yttrium distribution was more focused on the porosity areas. From Vickers hardness test, Ti6Al4V-2Y has an average hardness value of 161.7HV while Ti6Al4V-7Y has an average hardness value of 156.1HV. By comparing both results, it is proven that lower concentration of yttrium decreases the average grain size and increases the hardness value of titanium alloy.

5.2 Recommendation

For the suggested future works, further research on Ti6Al4V-2Y is greatly encouraged as it serves a huge role in maintaining health and helping patients who are in need. The process of FFC Cambridge is also recommended to be introduced more in other countries in order to produce more titanium alloys in a faster yet more efficient way than the traditional method, Kroll process. Eventhough titanium alloys had already been used in the biomedical field, introducing them as the replacement for stainless steel would be a great alternative, as it will reduce the risk of contamination to the patients after the transplant as stainless steel possesses a more prone to corrosion than titanium alloys.

REFERENCES

[1] Fenn, Cooley, Fray, Smith (2004). *Exploiting the FFC Cambridge Process*, Advanced Materials & Processes, Metalysis Ltd. [p.51-53].

[2] Lu, (2005). *Hydrogen Embrittlement of Aluminum: The Crucial Role of Vacancies*, The American Physical Society [p. 1-4].

[3] Mohandas, Fray (2004). *FFC Cambridge Process and Removal of Oxygen from Metal-Oxygen System by Molten Salt Electrolysis: An Overview*, Trans. Indian Inst. Met. Vol. 57:6 [p. 579-592].

[4] Bhagat, Jackson, Inman, Dashwood (2008). *The Production of Ti-Mo from Mixed Oxide Precursors via the FFC Cambridge Process*,Journal of The Electrochemical Society.

[5] Cao, Min, Wu, Xian, Shang (2006). *Pinning of grain boundaries by second phase particles in equal-channel angularly pressed Cu-Fe-P alloy*, Material Science & Engineering, Elsevier B. V [p. 86-91].

[6] Obasi, Birocsa, Fonseca, Preuss (2011) *Effect of β growth on variant selection and texture memory effect during α -> β -> α phase transformation in Ti 6Al-4V*, Acta Materialia, Elsevier [p. 1048-1058]

[7] Ti6Al4V Titanium Alloy *(retrieve from http://www.arcam.com/CommonResources/Files/Materials/Arcam-Ti6Al4V-Titanium-Alloy.pdf on 29/6/2012)*

[8] Bhagat, R. et al (2010). *In-Situ Synchrotron Diffraction of the Electrochemical Reduction Pathway of* TiO_2, Acta Materialia, vol. 58:15, [p. 5057-5062]

[9] Alloy Data Titanium Alloy Ti 6Al-4V *(retrieve from http://www.cartech.com on 27/6/2012)*

[10] Vickers Test *(retrieved from http://www.instron.us/wa/applications/test_types/hardness/vickers.aspx on 11/8/2012)*

[11] X-ray Diffraction (XRD) *(retrieved from http://*web.pdx.edu/~pmoeck/phy381/Topic5a-XRD.pdf*)*

[12] FESEM History and Principles *(retrieved from http://infohost.nmt.edu/~mtls/instruments/Fesem/FESEM% 20history.htm on 28/11/2012)*

[13] Egement Akar (2005). *Processing and mechanical testing of Ti6Al4V foams for hand tissue implant applications.* Izmir Institute of Technology.