# ANALYSIS OF CORROSION FATIGUE FOR COMMERCIALLY PURE TITANIUM USING NITROGEN ION IMPLANTATION

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**To my beloved mother and father (the late);**

**I would like to thank you for your great affection goes to me forever.**

**To my wife Nurhanisah binti Abdul Wahab and my children Muchammad Mirwanda, Fajar Ramadhan and Nadhila Nurdin, your love and understanding had encouraged me to complete this great opportunity in my life.**

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# ABSTRACT

The objective of this research is to determine the corrosion fatigue behaviours for commercially pure titanium (CpTi) using nitrogen ion implantation. A series of studies was conducted to obtain the mechanical properties, corrosion resistance and fatigue and corrosion fatigue behaviours and to develop model prediction of corrosion fatigue life of nitrogen ion implanted CpTi (Nii-Ti). Initially, nine specimens of CpTi were implanted nitrogen ion with the energy of 80, 100 and 115 keV and dose of $0.5x10^{17}$, $1.0x10^{17}$ and $2.0x10^{17}$ions/cm$^2$ to characterize its surface properties and to obtain corrosion resistance. The result shows that energy of 100 keV and dose of $2.0x10^{17}$ions/cm$^2$ was the optimal implanted parameter. In the second study, fatigue and corrosion fatigue test were performed to investigate the fatigue and corrosion fatigue behaviours. The fatigue specimens were implanted with the energy of 100 keV and dose of $2.0x10^{17}$ions/cm$^2$. The fatigue tests were carried out for Nii-Ti specimens in saline solution and for CpTi and Nii-Ti specimens in laboratory air by means of axial loading condition at stress level between 240 and 320 MPa. The results were nitrogen ion implantation improved slightly the fatigue life of CpTi and Nii-Ti with the fatigue strength of 250 MPa and 260 MPa, respectively. Finally, the prediction of corrosion fatigue life was developed based on corrosion pit growth law. The stress amplitudes of 250, 260 and 280 MPa were selected to measure penetration rate of specimens at various elapsed times using electrochemical method in saline solution, then established the empirical model. The result shows that the expression fits the experimental data well. In conclusion, the effects of nitrogen ion implantation on surface properties and adhesion strength of nitride layers improved the fatigue and corrosion fatigue life of Nii-Ti.

# ABSTRAK

Objektif kajian ini ialah untuk menentukan tingkah laku lesu kakisan untuk titanium komersil murni (CpTi) menggunakan nitrogen ion implantasi. Satu siri kajian telah dijalankan untuk menentukan sifat-sifat mekanikal, rintangan kakisan, kelesuan dan tingkah laku lesu kakisan dan membangunkan pengiraan daripada model hayat lesu kakisan CpTi setelah ditanamkan ion nitrogen (Nii-Ti) Dalam kajian awal, sembilan spesimen CpTi yang telah ditanamkan ion nitrogen dengan tenaga 80, 100 dan 115 keV dan dose $0.5x10^{17}$, $1.0x10^{17}$ dan $2.0x10^{17}$ions/$cm^2$ untuk ciri-ciri sifat permukaan dan rintangan kakisan. Hasil kajian menunjukkan bahawa tenaga 100 keV dan dose $2.0x10^{17}$ions/$cm^2$ adalah parameter optimum penanaman itu. Dalam kajian kedua , ujian lesu dan kakisan lesu telah dijalankan untuk melihat lesu dan hayat lesu kakisan. Spesimen lesu telah pun ditanam ion nitrogen dengan tenaga 100 keV dan dose $2.0x10^{17}$ions/$cm^2$. Ujian lesu dengan dijalankan bagi spesimen CpTi dan Nii-Ti dalam persekitaran makmal dan ujian lesu kikisan untuk spesimen Nii-Ti dalam larutan masin dengan keadaan beban paksi dalam julat tegasan antara 240 MPa. dan 320 MPa. Berdasarkan kajian ini didapati bahawa penanaman ion nitrogen meningkat sedikit hayat lesu CpTi dan Nii-Ti dengan kekuatan lesu masing-masing iaitu pada 250 MPa dan 260 MPa. Dalam kajian akhir terhadap angaran hayat kakisan lesu telah dibangunkan berdasarkan hukum pertumbuhan kakisan lubang. Amplitud tegasan 250 MPa, 260 MPa dan 280 MPa telah dipilih untuk melihat kadar penusukan yang terjadi terhadap specimen Nii-Ti pada pelbagai masa berlaku mengunakan kaedah elektrkoimia dalam larutan masin; kemudian menubuhkan model empirik bagi anggaran hayat kakisan lesu. Keputusan kajian menunjukkan, pengiraan dan ujikaji boleh dibandingkan dan bersesuian juga. Sebagai kesimpulan, kesan penanaman ion nitrogen pada sifat-sifat permukaan dan ketegasan lekatan lapisan nitrida boleh meningkatkan hayat lesu dan hayat lesu kakisan Nii-Ti.

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## LIST OF SYMBOLS AND ABBREVIATIONS

| | | |
|---|---|---|
| A | - | Area |
| ASTM | - | American Society for Testing and Materials |
| Al | - | Aluminium |
| $Al_2O_3$ | - | Aluminium Oxide/ Alumina |
| At% | - | Atomic Percentage |
| a.u. | - | Auxiliary unit |
| BATAN | - | Badan Tenaga Nuklir Nasional (National Nuclear Energy Agency of Indonesia) |
| bcc | - | Body centred cubic |
| cm | - | Centimetre |
| C | - | Paris's material constant |
| CE | - | Counter Electrode |
| CpTi | - | Commercially pure titanium |
| $\frac{da}{dN}$ , $\frac{da}{dt}$ | - | Fatigue crack growth rate |
| EW | - | Equivalent weight |
| $E_{corr}$ | - | Corrosion potential |
| f | - | Frequency |
| F | - | Corrosion degradation factor |
| fcc | - | Face-centred cubic |
| h | - | Hour |
| Hz | - | Hertz |
| HF | - | Hydrofluoric acid |
| $HNO_3$ | - | Nitric acid |
| $H_2O$ | - | Water |
| $I_{corr}$ | - | Total anodic current |

| | | |
|---|---|---|
| $i_{corr}$ | - | Corrosion current density |
| $K$ | - | Stress Intensity Factor |
| $k$ | - | Strength coefficient |
| $Kc$ | - | Critical stress intensity factor |
| $K_{IC}$ | - | Fracture toughness |
| $K_{pc}$ | - | Stress intensity factor at instance of crack initiation |
| $K_t$ | - | Stress concentration factor |
| keV | - | Kilo electro Volt |
| $\Delta K$ | - | Stress intensity factor range |
| $\Delta K_{th}$ | - | Fatigue threshold stress intensity factor range |
| $l$ | - | Liter |
| $m$ | - | Paris's material constant |
| mV/s | - | Milivolt per second |
| mg | - | Miligram |
| mm | - | Milimetre |
| MPa | - | Mega Pascal |
| mpy | - | Mil per year |
| mV | - | Milivolt |
| n | - | Number of element |
| N | - | Number of cycle |
| $2N_f$ | - | Reversals to failure |
| nA | - | nanoampere |
| NaCl | - | Sodium chloride |
| $N_2$ | - | Nitrogen |
| Nii-Ti | - | Nitrogen ion implanted Cp Ti |
| $O_2$ | - | Oxygen / Air |
| $R$ | - | Stress ratio, $(R = S_{a_{min}}/S_{a_{max}})$ |
| RE | - | Reference Electrode |
| $S_a$ | - | Stress amplitude |
| $S_{cf}$ | - | Fatigue strength in corrosive medium |
| $S_f$ | - | Fatigue strength in laboratory air |
| $S_m$ | - | Mean stress |
| $S'_f$ | - | Fatigue strength coefficient |

| | | |
|---|---|---|
| SBF | - | Simulated Body Fluid |
| SEM/EDS | | Scanning Electron Microscopy/Energy Dispersion Spectroscopic |
| $\Delta S$ | - | Stress range ($\Delta S = S_{max} - S_{min}$) |
| Ti | - | Titanium |
| $TiO_2$ | - | Titanium Dioxide |
| $Ti_xN$ | - | Titanium Nitride |
| WE | - | Working Electrode |
| $\varepsilon_p$ | - | Plastic strain |
| $\varepsilon_e$ | - | Elastic strain |
| ε | - | Epsilon |
| $\sigma'_f$ | - | Fatigue strength |
| κ | - | Kappa |
| ϴ | - | Theta |
| α | - | Alpha |
| ρ | - | Rho (density) |
| γ | - | Gamma |
| **χ** | - | Chi |
| μA | - | Microampere |
| μm | - | Micrometer |
| *W* | - | Atomic weight of the element |
| XRD | - | X Ray Diffraction |
| *Y* | - | Geometry correction factor |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

This chapter begins with the background of the research and important elaboration, followed by problem statements, objective, scope, contributions of the research and organization of the thesis.

## 1.1 Background of research

Metallic biomaterials are the most appropriate implant materials to replace failed hard tissue at present. Stainless steels, cobalt based alloys, titanium and its alloys are the three most used metals for biomaterials in fabrication of medical devices.

Recently, titanium and titanium alloys are getting much more attention as biomaterials because they have high specific strength, low density, good resistance to corrosion, moderate elastic module of 100-110 GPa (Leyens & Peters, 2003; Majumda *et al*., 2008), no allergic problems and good biocompatibility (non-toxic and not rejected by the human body) among other metallic biomaterials. They exhibit a high corrosion resistance due to the formation of a stable passive layer ($TiO_2$) on its surface.

Therefore, they have been extensively used in the last several decades as materials for dental implants, and medical devices. Nowadays, they are also considered by medical engineering as the material of choice for medical application (Elias *et al*., 2008; Niinomi, 1998; Van Noort, 1987), i.e. the prosthetics, internal fixation, inner body devices and instrumentation.

Since significant benefit of titanium for patients, surgeons and engineers, the use of the titanium would steadily increase in the near future. Besides the increasing of titanium used as implant materials are due to the increase in the olders generation or aging population worldwide, the trend toward more active lifestyles, and the ability to control health care cost.

The population ratio of the aged people has grown rapidly, so the number of the elder demanding and replacing failed tissue with artificial devices from biomaterials is also increasing, particularly, the amount of usage of instruments for replacing failed hard tissues such as artificial hip joints, and dental implants.

Pure titanium and Ti–6Al–4V are still the most widely used among the titanium alloys where they meet a demand almost the market of titanium biomaterials. Basically, they are developed as structural materials particularly for aerospace structures (Niinomi, 2003; Luetjering & Williams, 2003).

Therefore, the development of titanium targeted for biomedical applications are highly required. Accordingly, the research and development on titanium composed of non-toxic elements were started (Silvaa *et al*., 2004), and are under development which increase continuously. Although commercially pure titanium (CpTi) exhibit several advantages as biomaterial, but its resistance to wear is lower than Ti alloy. It is therefore necessary of surface treatment of commercially pure Titanium (CpTi) to enhance the resistance to wear by ensuring no decline in corrosion resistance.

Some surface processing, such as sandblasting, induces rough and contaminated surfaces and it might be an increasing in risk to failure due to this surface condition results in higher corrosion susceptibility. Electrochemical investigations of the corrosion behaviour of CpTi and Ti alloys have always demonstrated very good passivity condition of the surface. However, the study about ensuring reliability of medical implant is still insufficient. Therefore, the prediction of the corrosion fatigue life of pure titanium with nitrogen ion implanted surface as the implant material would be a valuable contribution on ensuring the sustainability of the implant devices.

## 1.2 Problem statements

Nitrogen ion implantation was introduced onto CpTi to modify the surface condition for more reliable performance and to increase its surface resistance to wear and corrosion. However, the problem with ion implantation is crystallographic damage, produces implantation damage on the surface (Rautray *et al*., 2011) and point defect in target crystal on impact resulting imperfection in lattice crystal such as vacancies and interstitials known as point defects. Ion implantation introduces both a chemical change in the target surface and a structure change in the crystal structure that could be surface defect in the form of a void or micro crack those can cause premature failure of the implanted device.

Several research works studied on corrosion behaviour of CpTi and Titanium alloys (Fukumoto *et al*., 1999; Sundararajan, & Praunseis, 2004; Raman *et al*., 2005), however more work are necessary in the area of fatigue as well as corrosion fatigue for the nitrogen ion implanted CpTi (Vardiman & Kant, 1982).

This study focuses more comprehensive on the analysis of corrosion fatigue for commercially pure titanium using nitrogen ion implantation. The nitrogen ion implantation might create surface damage and lattice disorder in the near surface of the material. Therefore, the nitrogen ion implanted CpTi, with surface damage, when it is applied a cyclic stress in body fluid or saline solution environment could be the potential factors caused the corrosion fatigue failure for the proposed biomedical material. Intensive research is needed to prove the fatigue behaviour of CpTi after implantation with nitrogen ion.

## 1.3 Objective

The objective of this research is to analyze corrosion fatigue behaviours for commercially pure Titanium using nitrogen ion implantation. A series of studies was conducted to achieve specific objectives as follows:

1. To obtain the optimum parameter of CpTi after nitrogen ion implantation.
2. To investigate the influence of the nitrogen ions implantation on fatigue and corrosion fatigue properties.
3. To develop an empirical model based on experimental data for corrosion fatigue life prediction.

## 1.4 Scope of research

The scopes of this research are as follow:

1. Introducing the nitrogen ion onto surface of CpTi by ion implantation method to modify the surface hardness that can improve the wear and corrosion resistance. Beam energy of 80, 100 and 115 keV and dose of $0.5x10^{17}$, $1.0x10^{17}$ and $2.0x10^{17}$ ions/$cm^2$ are used as the variables proposed for nitrogen ion implantation process.
2. Specimens that had been implanted with nitrogen ion were analyzed for the formation of nitride phase. Surface hardness and corrosion rate of the Nii-Ti specimens were tested to verify the optimal parameter of implantation process. In addition, the tensile and wear resistance tests were also to be carried out on the nitrogen ion implantation specimens.
3. The nitrogen ion implanted specimens were tested the fatigue in laboratory air and in saline solution with the frequency of 10 or 20 Hertz and stress ratio, R, of -1.
4. Based on the experimental results, the empirical model was developed for estimation of corrosion fatigue life for CpTi using nitrogen ion implantation.

## 1.5 Contributions of research

Corresponding to the above objective, some important points could be expressed as contribution to the knowledge and professional usage as well as industrial application. There are three prominent contributions that can be provided from the result of this research.

1. Improvements in corrosion resistance and mechanical properties for Ti used biomedical applications have practical importance. Different energies at different doses for implanting nitrogen ion are a good approach.
2. The use of nitrogen ion implantation is a good technique for the improvement of fatigue strength of Ti base materials.
3. The penetration growth law for Nii-Ti was established for contribution to the service life estimation of Ti base materials in acidic environments.

## 1.6 Thesis organization

The present thesis comprised of five chapters that were organized in order to address the objectives referred to in section 1.3 which are:

- Chapter 1: The description of research overview was discussed and the investigations performed in this area was briefly reviewed. The knowledge gap for significant corrosion behaviour and mechanical properties of CpTi with surface modification is extracted from the state-of-art to define the research objectives. The problem statements, research objective, scope of the research and the research contributions are described. The overall contents of the thesis are also summarized in this chapter.
- Chapter 2: The basic theory to support the implementation of the whole research is discussed in this chapter.
- Chapter 3: The details of the experimental investigations are presented. The properties of the CpTi, the fabrication process and equipment used in the research activities are described. The loading set-up, experimental conditions and measuring systems employed to collect the experimental data are explained.
- Chapter 4: The achieved results of the research are presented and discussed following the objective of the research. The most important findings are also described.
- Chapter 5: The conclusions derived from experimental and theoretical investigations are presented. The future works as recomendations are also stated in this chapter.

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