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FATIGUE LIFE PREDICTION OF COMMERCIALLY PURE TITANIUM AFTER NITROGEN ION IMPLANTATION

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ABSTRACT

Prediction of fatigue life has become an interesting issue in biomaterial engineering and design for reliability and quality purposes, particularly for biometallic material with modified surfaces. Commercially pure titanium (Cp-Ti) implanted with nitrogen ions is a potential metallic biomaterial of the future. The effect of nitrogen ion implantation on fatigue behavior of Cp-Ti was investigated by means of axial loading conditions. The as-received and nitrogen-ion implanted specimens with the energy of 100 keV and dose of 2×10^{17} ions cm^{-2}, were used to determine the fatigue properties and to predict the life cycle of the specimens. The effect of nitrogen ion implantation indicated revealed improved the tensile strength due to the formation of nitride phases, TiN and Ti_2N. The fatigue strength of Cp-Ti and Nii-Ti was 250 and 260 MPa, respectively. The analytical results show good agreement with experimental results.

Keywords*:* Cp-Ti; fatigue; fatigue life; biomaterial; ion implantation.

INTRODUCTION

In addition to the design of joint replacements, the material selected plays a very important role. Materials for human body implants must be biocompatible, corrosion resistant, strong, and display adequate elasticity (Pompe et al., 2004). Titanium meets these requirements to a very high degree; it has excellent temperature stability, wear and abrasion resistance, and is lightweight (Qu et al., 2005, Kustas and Misra, 1992). The use of titanium alloy Ti-6Al-4V in preference to pure titanium in biomedical applications is because of its superior mechanical properties and moderate modulus, i.e., 100–110 GPa (Elias et al., 2008). However, the drawback of this alloy is that corrodes to some extent in body fluids, thereby releasing ions that might possibly be harmful over a prolonged period. It is now believed that aluminium ions have been associated with Alzheimer’s disease and V, Co, Mo, Ni, and Cr ions are suspected of being toxic or carcinogenic (Rogers et al., 1997). Therefore, pure titanium is considered a better solution because it will not produce toxic ions and its mechanical properties satisfy biomedical application requirements (Silvaa et al., 2004). Titanium has excellent corrosion resistance, good biocompatibility, and a good specific strength-to-weight ratio, but poor wear resistance. If it were utilized as a metal-to-metal artificial hip replacement, in which wear resistance is property of interest, surface modification of Cp-Ti would be necessary in order in to improve its surface wear properties.

Surface modification methods, such as anodic oxidation treatment, sandblasting, carbide coating, plasma nitriding, electrochemical treatment, and nitrogen ion implantation (Velten et al., 2002; Kapczinski et al., 2003; Guilherme et al., 2005; Jagielski et al., 2006; Jiang et al., 2006; Song et al., 2007; Ali et al., 2011), have been proposed to increase the corrosion resistance and wear resistance of the material. The technique of nitrogen ion implantation has been explained well and shown to be a good method by which to enhance passivity and to reduce corrosion rate. This is due to the formation of TiN and Ti_2N phases, which avoids the migration of ions and stabilizes the TiO_2 film growth on the surface of Cp-Ti (Arenas et al., 2000). Nitrogen ion implantation modifies the Cp-Ti surface to produce wear resistant species like nitrides (TiN; Ti_2N) other than TiO_2 on the surface. The ion implantation technique is now available as an industrial-scale process that allows medical engineering to improve hardness, corrosion resistance, and achieve an accuracy of form necessary for biomedical devices. Additionally, the ion implantation technique is also employed to enhance fatigue properties for some critical components. A detailed explanation of the ion implantation process is given by Nastasi and Mayer (2006).

Almost all of the surface treatment processes, including ion implantation, are prone to roughness and contamination because surface voids can cause an increasing risk of the modified surface being susceptible to corrosion. Electrochemical investigations of the corrosion behavior of Cp-Ti and titanium alloys have almost always demonstrated a very good passivity condition of the surface. However, studies focusing on ensuring the reliability of medical implants are still insufficient. Therefore, the fatigue life prediction of pure titanium as an implant material, following nitrogen ion implantation, will be a valuable contribution to ensuring the sustainability of the implanted devices. The objectives of this study are to evaluate the fatigue properties of the Cp-Ti implanted with nitrogen ions and to predict the fatigue life time of the Cp-Ti implanted with nitrogen ions (Nii-Ti), based on experimental data.

MATERIAL AND METHODS

The material used in this study was Cp-Ti in the form of a round 30-mm-diameter bar, originally supplied by Fiko Ltd., Ukraine with the code of VT1-0 grade. The chemical composition and mechanical properties of the investigated material are listed in Tables 1 and 2. The microstructure of the Cp-Ti in original condition (as-received Cp-Ti) is shown in Figure 1.

Table 1. Chemical composition of original Cp-Ti.

N	C	H	Fe	O	Al	Si	Ti	Res
0.04	0.05	0.003	0.13	0.11	0.49	0.03	base	0.30

Table 2. Mechanical properties of original Cp-Ti.

Ultimate Tensile Strength (MPa)	Elongation (%)	Reduction Area (%)	Impact Strength
430	29	56	16

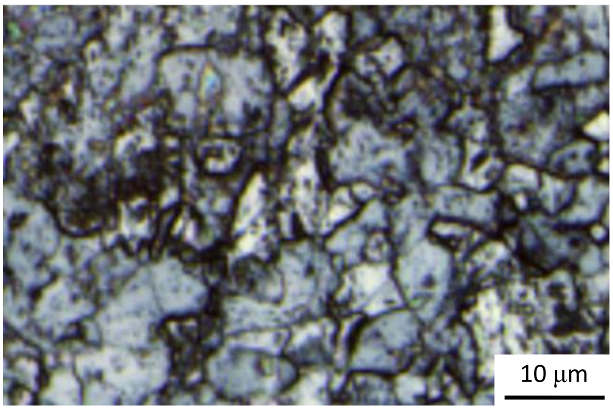

Figure 1. Microstructure of original Cp-Ti, etching of 10% HF and 5% HNO_3; 200× (Falazzaky et al., 2012).

Fatigue specimens are prepared based on ASTM E466 and E 468. Firstly, Cp-Ti was prepared in lengths of 90 mm, and then cut in to four parts by using a wire-cutting machine. Secondly, the machining processes were carried out to form the standard specimens by using an automatic lathe. Finally, the specimens were ground with abrasive paper and then polished by using 1 μm diamond paste to achieve surface roughness values of 0.06–0.1 μm. Fatigue tests were performed on specimens with the shape and dimensions shown in Figure 2. Polished specimens were rinsed with water, and then ultrasonically cleaned with ethanol over 30 minutes and dried at room temperature prior to the nitrogen ion implantation process. They are subjected to implantation of nitrogen ions with the energy of 100 keV and dose of 2×10^{17} ions cm^{-2}, which were reported as the optimum parameters by Fulazzaky et al. (2012).

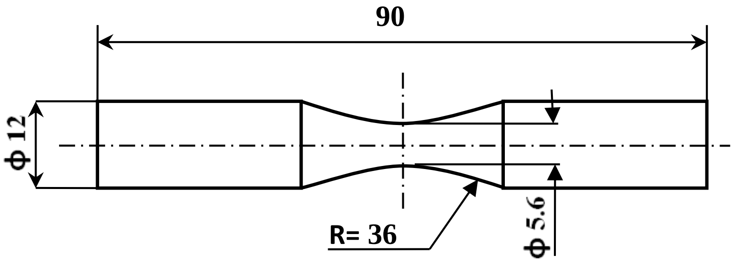

Figure 2. Shape and dimensions of the fatigue specimen (mm).

RESULTS AND DISCUSSION

Nitride Phases and Tensile Properties

Preliminary studies on the mechanical properties show that nitrogen ion implantation improves the tensile properties, wear resistance, and corrosion behavior (Fulazzaky et al., 2012). Results of the tensile test exhibit an improvement of tensile properties of Cp-Ti following nitrogen ion implantation, as shown in Table 3. The improvement of those properties was due to the formation of a new phase in the Cp-Ti system called titanium nitride, e.g., TiN and Ti_2N. X-ray diffraction (XRD) observations exhibited that these phases have the unit cell of fcc (TiN) and tetragonal (Ti_2N), whereas the Cp-Ti itself has a hexagonally spaced lattice. Figure 3 shows the XRD pattern of Nii-Ti. The combination of phases of Ti, *TiN* and *Ti_2N* in the subsurface of Cp-Ti, following implantation with nitrogen ions, enhances the surface strength, wear and pretting resistance, and introduces positive changes to the ductility.

Table 3. Tensile properties of original Cp-Ti and Nii-Ti.

Measurement	Unit	Cp-Ti	Nii-Ti
Ultimate tensile strength	MPa	497	539
0.2% offset yield strength	MPa	385	440
Elongation in 25 mm	%	33.3	36.6

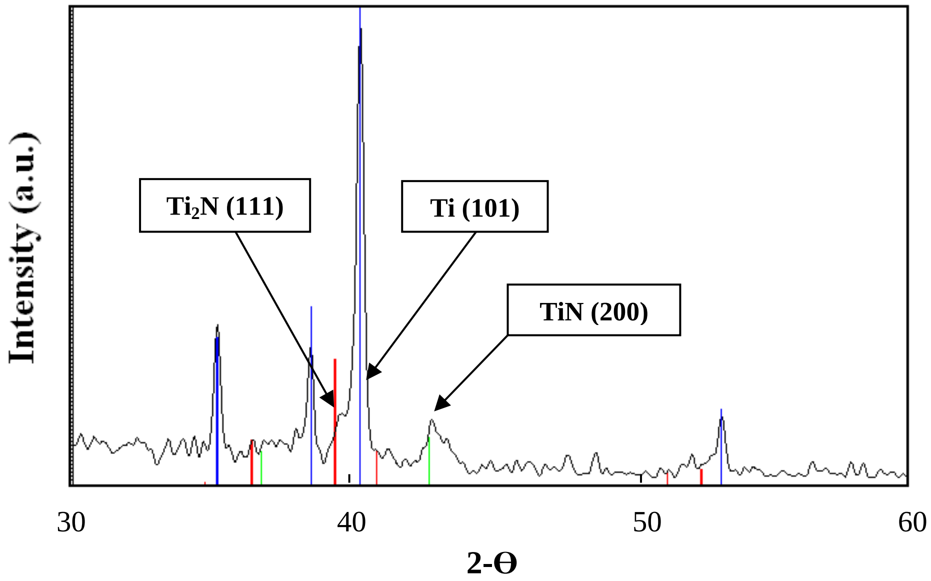

Figure 3. XRD pattern of Nii-Ti implanted with energy of 100 keV and dose of 2 × 1017 ions cm^{-2}.

As reported by Yu et al. (1993), the corrosion fatigue (C-F) endurance of Ti-6Al-4V was also increased effectively by nitrogen ion implantation. Because this material is prepared for hip joint replacement applications, the fatigue behavior as well as CF of Nii-Ti is also expected to be better than Cp-Ti.

Stress-life (S-N) Curves

Fatigue can be defined as the sequential stages of metal damage that evolve with accumulated load cycling in a laboratory environment. Material loaded cyclically below its yield stress will also fail following a specific number of cycles. An aggressive environment can lower the fatigue life of a material significantly, which is known as corrosion-fatigue. Figure 4 shows S-N curves for Cp-Ti and Nii-Ti determined in this work and compared with the work of Fleck and Eifler (2010). The graph shows that the endurance of Cp-Ti and Nii-Ti in the current study is at the stress of 250 and 260 MPa, respectively, with 1×10^7 cycles. It is verified that the fatigue test program in the present work could be fatigued by a stress of 250–320 MPa.

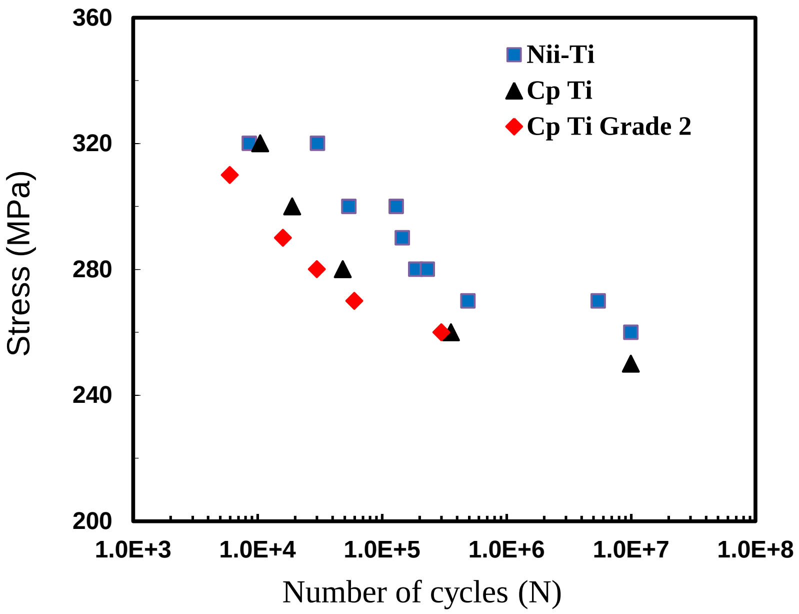

Figure 4. S-N curves of Cp-Ti and Nii-Ti compared with Cp-Ti Grade 2 (Fleck and Eifler, 2010).

Prediction of Fatigue Life

Fatigue failure occurs when the material experiences cyclic stresses and strains that produce permanent damage. Two major components of the crack phase will occur during the fatigue failure process: the initiation, and the propagation of fatigue cracks under cycling load (Xiang et al., 2010; Domínguez Almaraz et al., 2010; Kamal et al., 2012; Zulkifli et al, 2011). The summation of these components in number of cycles is called the fatigue life, and the stress or strain under which the fatigue life occurs is defined as the fatigue strength. Fatigue life prediction for Cp-Ti following ion implantation is needed for the design and reliability of surgical implants. Two methodologies are available for the prediction of fatigue life: one based on the material stress-life curve (Figure 4) or strain-life, and a damage accumulation model, based on the fracture mechanics and crack growth analysis approach. The second method of fatigue life prediction is used to calculate the number of cycles for failure of a component by taking into account the equivalent initial flaw size (EIFS). The basic equation for calculating the EIFS using a short crack has been proposed by several researchers (El Haddad et al., 1979; Xiang et al., 2010). The equation expresses the fatigue endurance $\Delta\sigma_f$ by using the fatigue threshold stress intensity factor ΔK_{th}, crack length a, and a geometry correction factor Y=0.73, which is crack configuration dependent (Nan et al., 2008):

$$\Delta K_{th} = K_t \sigma_f \sqrt{\pi a} Y \tag{1}$$

where K_t is the stress concentration factor specimen K_t = 1.04 (Nan et al., 2010). The crack length can be estimated from Eq. (1) as follows:

$$a_c = 1/\pi \left(\frac{\Delta K_{th}}{K_t \sigma_f Y}\right)^2 \tag{2}$$

An empirically assumed crack length, such 0.25–1 mm for metal (Liu and Mahadevan, 2009) can also be considered in solving the problem of life prediction based on fracture mechanics. Substituting those values obtained from Eqs. (1) and (2) into Paris's equation, the fatigue crack growth of a material can be expressed as follows:

$$\frac{da}{dN} = C(\Delta K_{th})^{\mathrm{m}} \tag{3}$$

By integration of the fatigue crack growth (Eq. 3) for the entire cycle of the performed fatigue life, Eq. (3) can be rewritten as:

$$\int_0^N dN = \int_{a_i}^{a_c} \frac{1}{C(\Delta K_{th})^{\mathrm{m}}} da \tag{4}$$

where C, m, and ΔK_{th} are material constants of fitting parameters. The constants C and m for pure titanium with a yield tensile strength of 380 MPa are 1.95×10^{-11} and 3.41 (Carpinteri and Paggi, 2007), respectively. Fatigue life of each stress level could be predicted using Eq. 5. Figure 5 shows the S-N curves of the experimental and analytical results for Nii-Ti.

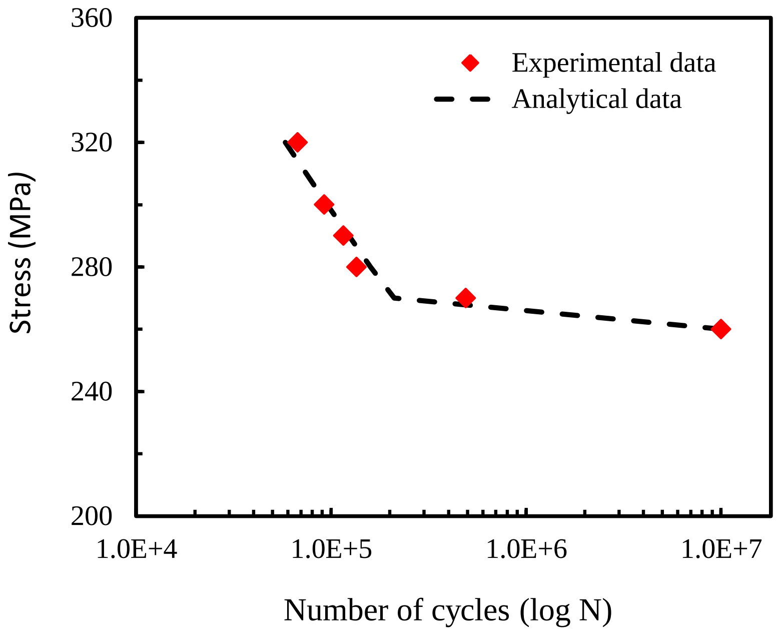

Figure 5. S-N curves of experimental and analytical results for Nii-Ti.

The experimental data used to construct the graph was the averaged value of experimental results under the assumption that both the analytical and the experimental results have the fatigue endurance of 260 MPa with the number of cycles of 10^7. The graph (Figure 5) shows the goodness-of-fit result of the analytical prediction and experimental observation, particularly at high stress levels. For lower stress levels, slightly over the fatigue limit, the fatigue life of the analytical result is lower than the experimental results. This is because of the use of Paris's constant for pure titanium.

Further study is required to determine the Paris's constant for nitrogen-ion implanted Cp-Ti.

CONCLUSIONS

The effect of nitrogen ion implantation indicates an improvement in the tensile strength due to the formation of nitride phases, *TiN* and Ti_2N. The fatigue strength of Cp-Ti and Nii-Ti was 250 and 260 MPa, respectively. The analytical results were validated by using the average value of the experimental results of Nii-Ti. The analytical results show good agreement with the experimental results. However, the new model of fatigue life prediction needs further study.

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