CORROSION FATIGUE CRACK PROPAGATION OF AISI 316L BY NITROGEN ION IMPLANTATION IN SIMULATED BODY FLUID

A thin layer was formed on the surface of the AISI 316L substrate by the process of nitrogen ion implantation at a vacuum pressure of 10^{-6} torr, a current of 50 mA for 90 minutes with the implantation energy of 20, 40 and 60 keV. The purpose of this study was to investigate the effect of nitrogen ion implantation on corrosion fatigue resistance in Simulated Body Fluid solution as a corrosion medium. The results show that the nitrogen ion implantation could increase corrosion resistance by reducing the corrosion rate and increasing the number of fatigue cycles.

Keywords: AISI 316L, corrosion, fatigue, nitrogen ion implantation, simulated body fluid

INTRODUCTION

Fatigue failure caused severe fatigue in 19 cases of bone surgery out of 30 cases that were studied. In order to choose the right procedure, the fracture pattern, the bone interaction, and the indication of infection must be considered [1]. Most medical implants fail because the fracture mechanism is dominated by ductile characteristic with the presence of nonmetallic inclusions resulting in an increase of stress [2]. Initial tensile stress on the plate screws on the bone are caused by natural activities of the human body due to cyclical load/unload. This causes fractures after four months of use, so orthopedic implant design is very important in addition to the fixation procedures and compatibility of bone morphologies [3].

Corrosion rates can be enhanced by increasing the bioactivity of metal surfaces. The form of the oxide layer of the implant surface can increase corrosion resistance and biocompatibility [4]. Atomic diffusion in metal processing can affect the formation and properties of metals as a passive layer [5].

A thin layer of Physical Vapor Deposition (PVD) on the surface of austenitic stainless steel can improve corrosion behavior and enhance the resistance of crack propagation on the austenitic stainless steel surface. The amplitude of the plastic strain affects the fatigue resistance of AISI 316L on the surface by ion implantation. Surface crack initiation at low amplitude is inhibited by layering on ion implantation, where the initial crack starts from below the surface [6]. Nitrogen implantation can improve metal properties such as corrosion and fatigue. The implantation of nitrogen into titanium and its alloys can improve the biological, chemical, and mechanical properties [7]. The corrosion rate of nitrogen ion implantation is affected by modifications in the current density of corrosion and corrosion resistance, where the increase in corrosion potential thermodynamically can reduce the intensity of corrosion [8]. When the values of corrosion current density are related to the rate of corrosion, this means the surface of AISI 316L has a good corrosion resistance. In this case, the ion implantation on the surface of AISI 316L has a high corrosion resistance [9]. The corrosive media can decrease the fatigue life. Corrosive solution can propagate the rate of fatigue crack [10]. Surface treatment can improve the surface characteristics of AISI 316L on surface morphology, roughness, hardness and corrosion resistance [11, 12].

The purpose of this study is to investigate the effect of nitrogen ion implantation on corrosion resistance and fatigue corrosion characteristics in a Simulated Body Fluid solution.

EXPERIMENTAL PROCEDURES

AISI 316L stainless steel was applied as the specimen of corrosion and fatigue with composition as shown in Table 1 and microstructure as shown in Figure 1.

The nitrogen ion implantation treatment was applied on the surface of AISI 316L at different energies of implantation: 20, 40 and 60 keV. Variables in this study were a current of 50 mA for 90 minutes at a vacuum pressure of 10^{-6} torr.

VersaSTAT was used as a corrosion test and VersaStudio was used to analyze the data with parameters as shown in Table 2. Simulated Body Fluid (SBF) was used in this research with composition as shown in Table 3.

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Table 1 **Composition of AISI 316L/wt.%**

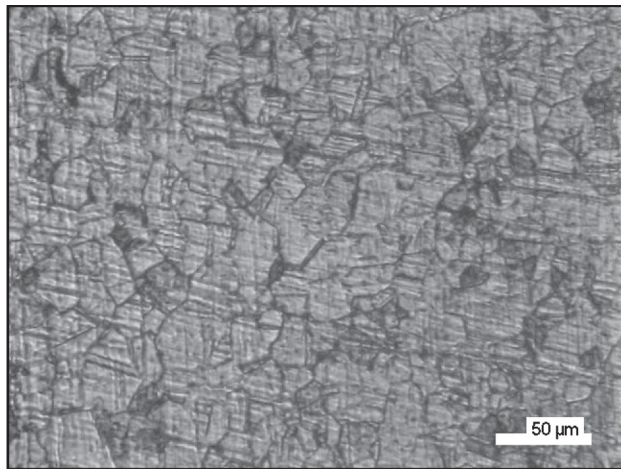| C	Si	P	Mn	Ni	Cr	Mo	Fe
0,03	0,96	0,03	1,07	10,86	16,78	1,89	67,78

Figure 1 Microstructure of AISI 316L

The fatigue specimens refer to the standard of ASTM-E647 [14]. The polynomial incremental method was used to analyze the fatigue characteristics indicated by Paris constants (C and n) in $da/dN = C (\Delta K)^n$ [15]. The plotting of (da/dN) vs (ΔK) indicated the area of the

Table 2 **Parameters of corrosion test [12]**

Endpoint properties	Initial potential (V)	-0,1
	Vertex potential (V)	1,2
	Final potential	-0,1
Scan properties	Step high (mV)	2
	Step time (s)	1
	Scan rate (mV/s)	2
Threshold properties	Start level (V)	0,2
	Threshold (mA)	1
Cell properties	Leave cell on	No
	Cell to use	External
Instrument properties	Current range	Auto
	Voltage range	+/- 6 V
	Acquisition mode	Auto
	Electrometer mode	Auto
	E filter	Auto
	E2 filter	Auto
	I/E stability	Fast
	Bandwidth limit	Auto
	LCI bandwidth limit	Auto
	iR compensation	Disable

Table 3 **Composition of SBF [13]**

Reagent	Quantity
NaCl	8,0350 g
$NaHCO_3$	0,3550 g
KCl	0,2250 g
$K_2HPO_4 \; 3H_2O$	0,2310 g
$MgCl_2 \; 6H_2O$	0,3110 g
1,0M-HCl	39 ml
$CaCl_2$	0,2920 g
Na_2SO_4	0,0720 g
Tris $((HOCH_2)_3CNH_2)$	6,1180 g
1,0M-HCl	0-5 ml

fatigue crack propagation rate in Region II. The Paris constant value of C can be determined by plotting the line to intersect the axis at $\Delta K = 1$ MPa m$^{1/2}$. The value of n represents a line gradient of the curve [16].

RESULTS AND DISCUSSION

Corrosion resistance

The corrosion test aims to determine the corrosion resistance characteristics of 316L SS material after being coated by Nitrogen, obtained from the ion implantation process. Corrosion test results appeared in the form of a relationship between current and potential as shown in Figure 2. Figure 2 shows that the greater the potential
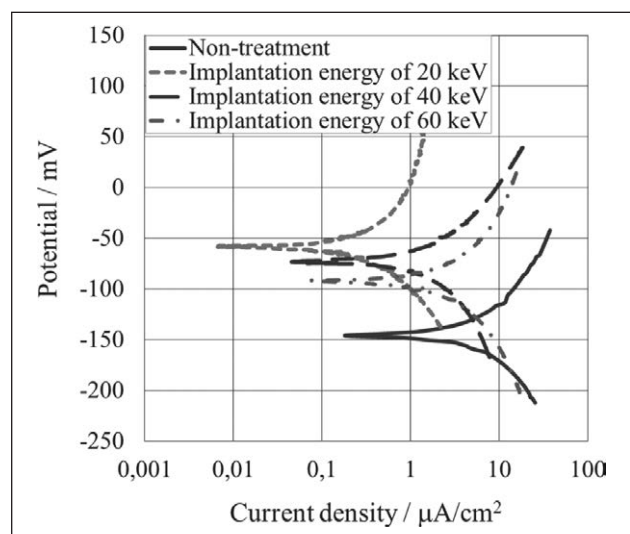

Figure 2 Current density and potential of AISI 316L specimens by nitrogen ion implantation
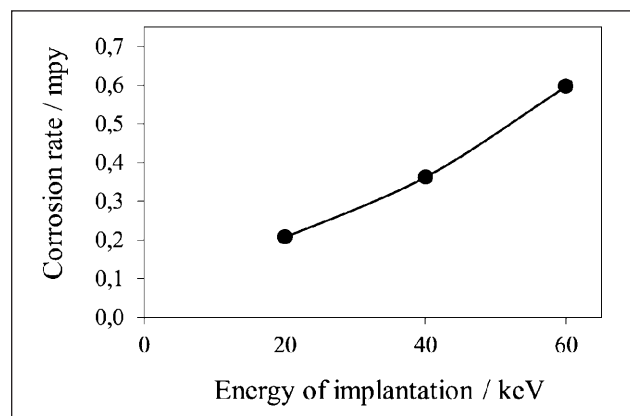

Figure 3 Effect of implantation energy on the corrosion rate of AISI 316L

Table 4 **Corrosion rate of AISI 316L by Nitrogen ion implantation**

Energy of implantation/ keV	Corrosion potential/ mV	Corrosion current density/ mA/cm^2	Corrosion rate/ mpy
0	-146	2,123	0,888
20	-57	0,495	0,207
40	-71	0,866	0,362
60	-91	1,426	0,597

value, the more cathodic the specimen is. The corrosion rate was obtained from Figure 2 by determining the intersection between the oxidation and reduction Tafel lines. Thus, the corrosion potential results were: E_{corr} -57 mV at 20 keV, -71 mV at 40 keV, -91 at 60 keV, and the corrosion current density of I_{corr} 0,495 mA/cm^2 at 20 keV, 0,866 mA/cm^2 at 40 keV, 1,426 mA/cm^2 at 60 keV.

Figure 3 and Table 4 show that ion implantation can reduce the rate of corrosion by 77 % from 0,888 mpy to 0,207 mpy at implantation energy of 20 keV.

The implantation energy affects the corrosion rate, where the greater the implantation energy, the corrosion rate will increase 75 % from 0,207 to 0,362 mpy and then increase 65 % from 0,362 to 0,597 mpy. This is because the greater the implantation energy needed to coat the surface of the substrate, the deeper the diffusion of N ions entering the surface of SS 316L substrate, so that the compaction of the implantation dose occurs at lower energy. This is also due to the increased corrosion current density which increases with the implantation energy needed to coat the surface of the AISI 316L substrate.

Every time an impact occurs at high energy, the ions will lose energy, so that at a certain time, the atoms will run out of energy and eventually stop occupying space between the target atoms. The ions which diffuse into the target surface will experience energy loss, eventually ceasing to occupy the lattice space between the densities of the atoms and will be able to enhance the corrosion resistance at a lower energy. Opportunities for dopant ions to occupy a larger gap cause a greater possibility of dopant ions to be distributed evenly. An even distribution of dopant ions will cause the arrangement of the target atoms to become dense. In ion implantation, a high-energy ion beam shot onto the surface of a material can damage the structure of the material. The target atoms which are pushed by the ions will push the neighboring target atoms so that it will damage the structure of the target atoms. The atoms that first shift or bounce will be able to collide with the next atoms. If there is enough energy, it will be able to shift or bounce other target atoms.

Corrosion fatigue characteristics

Fatigue testing on AISI 316L coated in nitrogen specimens obtained from the ion implantation process aims to determine the fatigue propagation characteristics that occur after the specimen is coated by a nitrogen ion implantation process. This test aims to determine the effect of nitrogen ion implantation on the rate of fatigue resistance properties, especially phenomena that occur during the fatigue propagation process. The results of fatigue testing in Simulated Body Fluid (SBF) media in the relation of a crack length and the number of cycles is shown in Figure 4.

Figure 4 shows that the specimen treated with nitrogen ion implantation has broken in the number of cycles
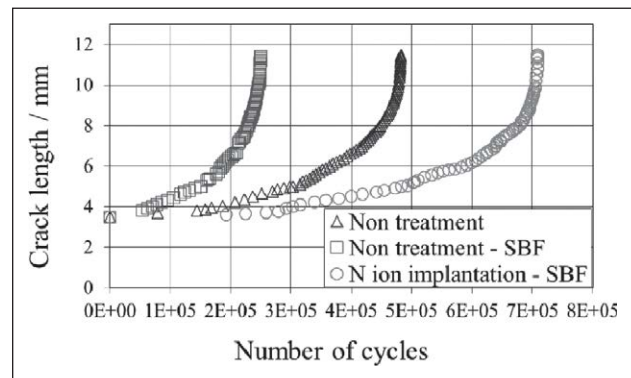

Figure 4 Fatigue crack growth properties of AISI 316L specimens

of 708 880. The Nitrogen layer affects inhibiting crack initiation in SS 316L specimens in the SBF solution, resulting in an increase of 184% in the number of fatigue cycles from 249 972 to 708 880. The ion implantation process is very important to improve the AISI 316L fatigue properties because the thin nitrogen layer can inhibit crack initiation which can increases the fatigue resistance of AISI 316L material.

Fatigue crack propagation characteristics are obtained by the incremental polynomial method, as shown in Figures 5 and 6.
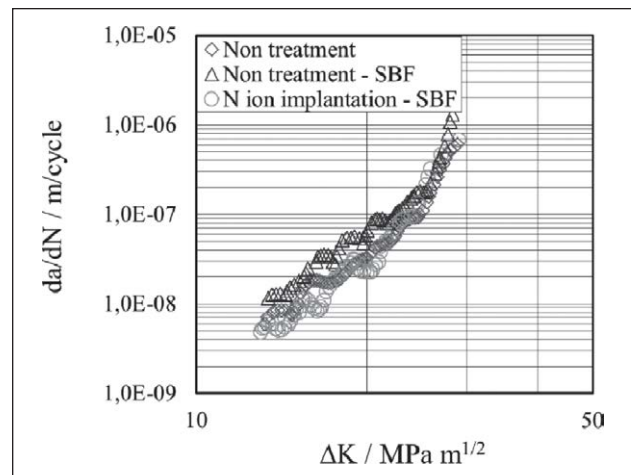

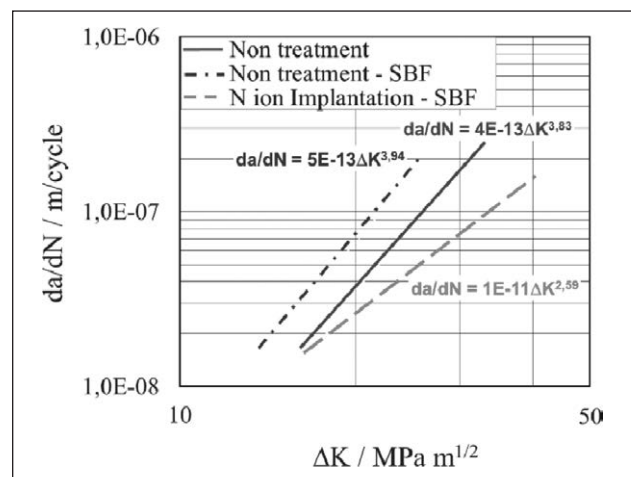**Figure 5** Curve of DK - da/dN of AISI 316L

Figure 6 Trend-line of DK - da/dN of AISI 316L by N ion implantation

Table 5 **Paris constants**

Specimens	Number of cycles	Paris constants	
		C	n
Non-treatment	482 276	4×10^{-13}	3,83
Non-treatment-SBF	249 972	5×10^{-13}	3,94
N ion implantation-SBF	708 880	10^{-11}	2,59

Figure 6 shows the effect of nitrogen ion implantation on the characteristics of fatigue crack propagation. Nitrogen ion implantation can inhibit crack initiation so that the rate of fatigue crack propagation decreases. This is shown by a slower graph marked by Paris constant values $C = 10^{-11}$ and $n = 2,59$, whereas specimens without nitrogen ion implantation have Paris C constant values $= 5 \times 10^{-13}$ and $n = 3,94$ as shown in Table 5.

A comparison of n values in specimens after treating with nitrogen ion implantation is lower than in specimens without ion implantation. This shows that nitrogen ion implantation can decrease the fatigue crack propagation. Fatigue corrosion initiation will initiate in micro-porous. The initiation of cracks in the subsurface is more difficult than on the surface. The SBF solution decreases the number of fatigue cycles, because the SBF solution can accelerate the rate of propagation of fatigue cracks. The nitrogen ion implantation layer can protect the surface of the AISI 316L in a corrosion environment by inhibiting the initial crack and increasing the fatigue resistance.

CONCLUSIONS

The results show that nitrogen ion implantation can increase the corrosion resistance by reducing the corrosion rate, delay crack initiation, reducing the fatigue crack growth rate, and increasing the number of fatigue cycles. The corrosion rate decreased by 77 % from 0,888 to 0,207 mpy in the implantation energy of 20 keV. The number of fatigue cycles increased by 184 % from 249 972 to 708 880 cycles and the rate of fatigue crack propagation decreased with a decrease in Paris n constant by 34 % from 3,94 to 2,59.

Acknowledgments

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