Evaluation of fatigue property of titanium wires by rotating-bending testing in 1 mass% lactic acid solution

Narushima T 1, Ueda K 2, Yamashita M 2, Murakami T 2, Ouchi C 2, Iguchi Y 2

1 Tohoku University Biomedical Engineering Research Organization (TUBERO), Sendai, Japan.
2 Department of Materials Processing, Tohoku University, Sendai, Japan.

Email: narut@material.tohoku.ac.jp

Abstract

The fatigue properties of the wire made of C.P.Ti and the new β-type titanium alloy, Ti-14Mo-3Nb-1.5Zr, were investigated in the rotating-bending testing. The wire made of the new β-type titanium alloy exhibited the fatigue strength higher than the C.P.Ti wire. The elution of titanium ion from the wire into the 1 mass% lactic acid solution and distilled water was suppressed at very low level during the fatigue testing. The DLC coating on C.P.Ti wire improved the ratio of fatigue strength to tensile strength.

1 Introduction

Titanium and its alloys are mainly used for implantable biomaterials because of their excellent properties such as lightweight, corrosion resistance, mechanical strength and shape memory/superelasticity. Various kinds of new titanium alloys have been developed for biomedical applications in last two decades [1]. The present authors developed the FES electrode which was made from a helically coiled rope wound from 19 strands hard drawn wires of the austenitic stainless steel such as SUS316L type [2] or Fe-22Cr-10Ni-6Mn-2Mo-0.5N [3]. The diameter of the wire is 25 µm. Titanium is a candidate for a source material of the FES electrode due to their excellent biocompatibility. The titanium materials have to be cold-drawn to an extremely fine wire for application to an FES electrode. The present research group developed the new β-type titanium alloy with high strength and excellent cold workability without containing toxic alloying elements [4].

In the present work, fatigue properties of the cold drawn titanium wire made of commercially pure (C.P.) Ti and the new β-type titanium alloy were investigated in lactic acid solution and distilled water. Since the surface modification of the titanium is important in biomedical applications, the effect of diamond like carbon (DLC) coating on the fatigue properties of the titanium wire was examined.

2 Methods

The titanium wires made of C.P.Ti (Nilaco Corp.) and the new β-type titanium alloy, Ti-14Mo-3Nb-1.5Zr (mass%), were used for fatigue testing. The diameter (d), Vickers hardness (Hv) and tensile strength (σU) of the as cold drawn wires are summarized in Table 1. The source materials of the C.P.Ti wires with 100 µm and 200 µm in diameter were grade 1 and grade 2, respectively, but the oxygen content analyzed in the wires increased up to around 0.5 mass%. This increase of oxygen content in the wires probably occurred during the drawing process.

Figure 1 shows the cross section and side view of the wires made of C.P.Ti and the new β-type titanium alloy. The DLC layer on the C.P.Ti wire with 100 µm in diameter was formed by unbalanced magnetron sputtering. The coating layer consisted of four layers, Cr, Cr+WC, WC+DLC and DLC, and the total thickness of the coating layer was 10 µm or 20 µm.

Fatigue property of the wires in 1 mass% lactic acid solution and distilled water at 310 K was evaluated using a dual-driven rotating-bending fatigue machine. The maximum bending stress induced to the wire ranged from 200 to 800 MPa. The detailed procedure of the fatigue test has been reported elsewhere [3]. After fatigue testing, the wires were observed by scanning electron microscopy (SEM), and the concentrations of metal ions eluted from the wires into the 1 mass% lactic acid solution and distilled water were measured by inductively coupled plasma (ICP) atomic emission spectroscopy.
Table 1: Diameter and mechanical properties of titanium wires

<table>
<thead>
<tr>
<th>Materials</th>
<th>d/µm</th>
<th>Hv</th>
<th>σ/MPa</th>
</tr>
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<tbody>
<tr>
<td>C.P.Ti</td>
<td>100</td>
<td>237</td>
<td>970</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>143</td>
<td>450</td>
</tr>
<tr>
<td>Ti-14Mo-3Nb-1.5Zr</td>
<td>174</td>
<td>304</td>
<td>1000</td>
</tr>
<tr>
<td>C.P.Ti (DLC coating)</td>
<td>120</td>
<td>117</td>
<td>290</td>
</tr>
<tr>
<td></td>
<td>140</td>
<td>134</td>
<td>330</td>
</tr>
</tbody>
</table>

3 Results

The fatigue curves of the titanium wires in rotating-bending testing are shown in Figure 2. The arrow mark inside the figure indicates that no failure up to $10^7$ cycles was observed. The wire made of the new β-type titanium alloy exhibited the fatigue strength higher than the C.P.Ti wire. A significant difference of fatigue curves between in 1 mass% lactic acid solution and distilled water was not observed. Figure 3 shows the concentration of titanium ion released from the C.P.Ti wire into the solutions as a function of number of cycles. The ion elution was suppressed at very low level even in the 1 mass% lactic acid solution due to the high corrosion resistance of titanium.

The effect of DLC coating on fatigue curve in the 1 mass% lactic acid solution is shown in Figure 4. It is suggested that the fatigue life depends on the tensile strength of the wire. The DLC coating suppressed the elution of titanium into the solutions, while the elution of Cr and W ions, which were in the coating layer, was detected in the 1 mass% lactic acid solution after the fatigue testing.
4 Discussion and Conclusions

The fracture surface of the C.P.Ti wire with 100 µm in diameter under maximum bending stress of 345 MPa is shown in Figure 5. The fracture surface consisted of the two distinct regions, i.e., the fatigue crack propagation region and the final overload region [5]. The crack origin is observed on the fracture surface in Figure 5. It is likely that the crack originated from surface defects formed in cold drawing process. Therefore, the optimisation of the cold drawing process is needed to improve the fatigue properties of the titanium wires.

Figure 6 summarized the relationship between tensile strength and fatigue strength of the wires in the 1 mass% lactic acid solution. It is empirically known that the fatigue strength (σf) is around 50 to 60 percent of the tensile strength (σt). The value of the σf/σt was less than 0.33 in the C.P.Ti wire with 100 µm in diameter. The presence of the surface defects which might act as crack origins seems to decrease the value of σf/σt. On the other hand, the C.P.Ti wire with DLC coating exhibited relatively high σf/σt values. The tensile strength of the wire with DLC coating was decreased by the recovering or recrystallization during the coating process (see Table 1). However, it is suggested that the DLC coating could improve the smoothness of wire surface.

In conclusion, the present work has clarified the fatigue properties of titanium wire in the 1 mass% lactic acid solution and distilled water for its application to an FES electrode. The wire made of Ti-14Mo-3Nb-1.5Zr had excellent fatigue properties compared with the C.P.Ti wire. The fatigue strength was affected by the conditions of wire surface, and the DLC coating is effective to improve the ratio of fatigue strength to tensile strength.

References


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