QUALITY CONTROL BASE ON SURFACE ROUGHNESS CHARACTERISTIC – OXIDE LAYER ON PURE TITANIUM

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Abstract

Titanium, invariably for decades, is the most promising metallic material used in medicine (implantology). And its suitability is primarily determined by biotolerance, which depends on the preparation of the surface of the elements. This paper assesses the quality control of oxide layers produced on pure (medical) titanium in the process of thermal oxidation in an air atmosphere. The parameters characterizing surface roughness have been used as control parameters. It was noted that the titanium surface undergoes a slight change with short oxidation times, and the oxidation is relatively even in nature. However, with longer oxidation times (more than 45 minutes) roughness parameters change drastically, which is not always beneficial from the point of view of further use.

Keywords: Quality control, titanium oxidation, oxide layer, roughness

1. INTRODUCTION

Titanium and its alloys are a group of metallic materials that enjoy the greatest interest in medicine, especially in implantology [1,2]. Titanium owes this special distinction to the medical industry: relatively low specific gravity (density), very satisfactory ratio of strength to density or yield strength, very good strength in fatigue tests, among metallic biomaterials the lowest value of Young’s modulus, but most importantly biotolerance, tendency to spontaneous passivation, high local and general corrosion resistance [3–7]. Intensive development of the industry (especially medical industry), the search for new solutions, an effort to implement the principles of Industry 4.0 and sustainable development [8, 9], invariably based on the proper and thorough knowledge of mechanisms occurring spontaneously (or without human intervention) - often such processes are called special processes, of which the assessment quality is particularly difficult and requires the use of destructive testing and statistical analysis [10]. Despite the very satisfactory properties of pure titanium, research (based on basic knowledge) to improve its properties is constantly carried out. Pure titanium easily passivates with minimal oxygen or moisture content in the operating environment - the result of the spontaneous oxidation process is an oxide layer consisting mainly of TiO₂ (rutile, anatase) [11–13]. There are many technologies to improve the performance of titanium, addition in biomedical composites [14], alloying [15,16]- the most commonly used implants are made of titanium alloys. There are also a number of technologies for surface modification of Ti or alloy elements, e.g. production of conversion layers (Ti(HPO₄)₂·nH₂O), nitriding [17], application of substoichiometric oxide layers (tribofilms like γ-Ti₃O₅, Ti₅O₉ and Ti₅O₉ and Mo₉₇TiO₂₈ as well as double oxides, like β-NiMoO₄ or NiTiO₃) [18], etc. However, the most common method for surface treatment of titanium-based metals is thermal or electrochemical oxidation [4,7,13,19]. This article attempts to characterize and identify the quality of oxide layers formed on pure titanium during thermal oxidation in an air atmosphere. Cause that the main limitation in the use of pure titanium is its poor resistance to abrasive wear, as a result of oxide layer damaged or deformation [7,11], the set of surface roughness parameters has been used as parameters for verifying the quality of titanium oxide layer.
The issue discussed in this article may be of interest in many other areas, including bioceramics layers [20], behavior of the layer [21] and its corrosion resistance [22,23], what may be of significance interest in biotechnology [24,25]. Such results should be also taken into consideration in evaluation methodologies [26,27] and decision processes [28-30] as well as in the stereological analysis [31-33], laser machining of surface layers [34-36] and uncertainty analysis [37].

2. EXPERIMENTAL

As tested material commercial, technically pure titanium Grade 2 (Bibus Metals) has been used. Titanium Grade 2 is one of the most commonly used variants of titanium in implantology. The sample before exposure in oxidation atmosphere were polished on water paper with gradation to 1000, then rinsed with distilled water, degreased with methyl alcohol. 13 series of samples were prepared (10 in each series). All samples have been placed in an oven chamber (with free atmosphere access to the measured surface). Oxidation has been carried out at 700±30 °C. Oxidation time has been measured from the moment the stated temperature was reached. Pure titanium samples oxidized for 5, 10, 15, 20, 30, 35, 40, 45, 50, 60, 90 and 120 minutes. Sample 0 was used as reference (no oxidation, roughness determined immediately after grinding and degreasing)

The 2D roughness measurement has been performed using a contact profilometer (MarSurf PS 10). Measurement of roughness parameters have been carried out in accordance with the standard ISO 13565-2, the measuring section 4 mm. Five measurements have been made on each sample. Data presented in the paper is the arithmetic mean of all measurements for samples from one series. According ISO standard (ISO 13565-2) during measurements the fallowing parameters have been determined: Ra - arithmetic mean deviation of the assessed profile, Rz - maximum height of the profile, Rp - maximum profile peak height, Rv - maximum profile valley depth, Rt - total height of the profile, Rk - core roughness depth.

3. RESULTS AND DISCUSSION

The results of the parameters measurements (average value with standard deviation) have been presented in Table 1.

Table 1 Selected, most important surface roughness parameters determined for titanium after thermal oxidation in air atmosphere (700 °C) with exposure time range from 0 to 120 minutes

<table>
<thead>
<tr>
<th>Time, min</th>
<th>Ra</th>
<th>Rz</th>
<th>Rmax</th>
<th>Rp</th>
<th>Rv</th>
<th>Rt</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.146</td>
<td>1.465</td>
<td>2.090</td>
<td>0.701</td>
<td>0.764</td>
<td>2.090</td>
</tr>
<tr>
<td>5</td>
<td>0.177</td>
<td>1.440</td>
<td>1.739</td>
<td>0.686</td>
<td>0.754</td>
<td>1.739</td>
</tr>
<tr>
<td>10</td>
<td>0.201</td>
<td>1.978</td>
<td>2.405</td>
<td>0.708</td>
<td>1.269</td>
<td>2.507</td>
</tr>
<tr>
<td>15</td>
<td>0.202</td>
<td>1.802</td>
<td>2.172</td>
<td>0.634</td>
<td>1.168</td>
<td>2.192</td>
</tr>
<tr>
<td>20</td>
<td>0.244</td>
<td>2.430</td>
<td>2.960</td>
<td>0.751</td>
<td>1.679</td>
<td>3.201</td>
</tr>
<tr>
<td>30</td>
<td>0.325</td>
<td>2.874</td>
<td>3.728</td>
<td>0.784</td>
<td>2.090</td>
<td>3.728</td>
</tr>
<tr>
<td>35</td>
<td>0.356</td>
<td>3.019</td>
<td>3.581</td>
<td>1.019</td>
<td>2.001</td>
<td>3.835</td>
</tr>
<tr>
<td>40</td>
<td>0.639</td>
<td>5.461</td>
<td>8.155</td>
<td>1.457</td>
<td>4.004</td>
<td>8.299</td>
</tr>
<tr>
<td>45</td>
<td>0.863</td>
<td>7.277</td>
<td>9.660</td>
<td>1.522</td>
<td>5.755</td>
<td>9.660</td>
</tr>
<tr>
<td>50</td>
<td>0.858</td>
<td>7.749</td>
<td>9.128</td>
<td>1.430</td>
<td>6.319</td>
<td>9.391</td>
</tr>
<tr>
<td>60</td>
<td>0.823</td>
<td>7.860</td>
<td>9.170</td>
<td>1.440</td>
<td>6.016</td>
<td>10.328</td>
</tr>
<tr>
<td>90</td>
<td>0.963</td>
<td>7.777</td>
<td>9.120</td>
<td>1.502</td>
<td>6.075</td>
<td>9.860</td>
</tr>
<tr>
<td>120</td>
<td>0.938</td>
<td>7.749</td>
<td>9.280</td>
<td>1.453</td>
<td>6.318</td>
<td>9.481</td>
</tr>
</tbody>
</table>
In **Figure 1** the most representative surface roughness profiles for a titanium after surface oxidation have been shown. In **Figure 1** are presented the representative profiles for a sample 0 (no treatment, after grinding and polishing), and then for the samples after thermal oxidation during 5, 40 and 120 minutes of exposure. Profile images have been created using standard compliant filters (ISO 16610-21 and ISO 13565-2).

![Profile images](image)

**Figure 1** The representative profiles for a sample after thermal oxidation:
 a) 5, b) 40 and c) 120 minutes of exposure

As is easily seen, on the roughness profiles in the initial oxidation phase (at short times up to 20 minutes), the surface roughness changes significantly. The profile has numerous peaks, hills, valleys and pits - and the oxidation is even. However, with longer spontaneous thermal oxidation times, considerable surface variation is observed and visible valleys and pits appear on the profile - much less peaks (than pits) are observed (Rp), which may mean that the oxide layer no longer increases, oxidation processes occur in deeper part of the surface layers, which causes damage and cracks in the topmost oxide layer. This also may be evidence of a gradual peeling of the oxide layer (Rv).

Based on the data presented in **Table 1** the graphs representing changes of surface roughness parameters have been prepared - **Figure 2**. The most important parameter Ra of the assessed profile – has been used to
define the random surface roughness (stochastic) in some measurable space. Ra is used as a global evaluation of the roughness amplitude on a profile.

![Graphs showing changes in surface roughness parameters](image)

**Figure 2** Change in surface roughness parameters as a function of thermal oxidation time: a) Ra, b) Rz, c) Rp, d) Rv, e) Rt, f) Rk – surface of pure titanium after thermal oxidation

![Graph showing Rmax change](image)

**Figure 3** Change in Rmax (surface roughness parameter) as a function of thermal oxidation time – surface of pure titanium after thermal oxidation

As can be easily seen, the short exposure times (5, 10 minutes) of the pure titanium in oxidizing atmosphere have only a slight effect on the change in surface roughness. Clear changes are observed at times above 30
minutes. In this case, the Ra parameter value increases even 3 times. Longer exposure times (over 50 minutes) strongly change the parameter, but it is observed that running the process for more than 50 minutes does not make sense, as there are clearly no major changes on the metal surface. The value of the Ra parameter is at a comparable level. In addition, it can be observed that the Rmax value (maximum deviation from the average line) increases significantly, which may indicate a selective effect of the oxidation or the formation of defects on the oxide layer and surface cacking (Figure 3).

4. CONCLUSION

Based on the presented results about the assessment of the quality of the oxide layer on titanium (after thermal oxidation in the air atmosphere) it can be stated that with short oxidation times, the titanium surface undergoes a slight change, and the oxidation is relatively even in nature - the value of most parameters (including mainly Ra) slightly increases. However, with longer oxidation times (more than 45 minutes), roughness parameters change drastically - all parameters increase drastically, but there are definitely more valleys than the peaks visible on the roughness profile - the Rv parameter increases significantly, which may indicate a selective effect of the oxidation or the formation of defects on the oxide layer and surface cracking.

REFERENCES


