Modification of titanium surface by magnetic resonance signals and gamma radiation

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INTRODUCTION

Titanium and titanium alloys are widely used in biomedical devices and components, especially as hard tissue replacements because of their desirable properties, such as relatively low modulus, good fatigue strength, formability, machinability, corrosion resistance and biocompatibility[1-5]. However, titanium and its alloys cannot meet all of the clinical requirements. Therefore, in order to improve the biological, chemical and mechanical properties, surface modification is often performed. Most surface modifications of clinically available oral implants employ techniques that increase the roughness of the surface, compared with different forms, shapes and sizes. Most of these roughened surfaces are produced either by blasting, abrading and coating methods using different material particles and/or by chemical methods. Surface roughness plays an important role in cell adhesion to the surface. Quality of materials used for orthopedic prostheses and dental implants does depend on their surface properties. That is why treatment leading to modification of surface topographical properties shows potential for improving osteointegration of orthopedic prostheses and dental implants[6-9]. Magnetic resonance is nonionizing radio frequency signals. It uses magnetic properties of the hydrogen nucleus excited by radiofrequency radiation transmitted by a coil surrounded by the body part[10]. Gamma radiation is electromagnetic radiation of high frequency (very short wavelength). Exposure to gamma rays and electrons beam is most commonly used to sterilize medical de-

KEYWORDS

Titanium biomaterial; Gamma radiation; MRI; Hardness; Roughness; Microstructure.

ABSTRACT

Many attempts were made to improve roughness of titanium implants which is important for early fixation and long-term mechanical stability of the prosthesis. In the present work microstructure, surface roughness and hardness of titanium biomaterial were investigated before and after exposure to magnetic resonance imaging (MRI) and gamma radiation. It was found that microstructure, (crystallinity, crystal size and orientation), of titanium biomaterial was changed after exposure to magnetic resonance imaging (MRI) and gamma radiation. Roughness parameter (Ra) was significantly increased after exposure to magnetic resonance imaging and gamma radiation. However, Vickers hardness number of titanium was significantly decreased. © 2013 Trade Science Inc. - INDIA
vices. The capability of ionizing radiation to kill micro-organisms was established early in the past century. These sterilization techniques are based on the penetrating ability, and the ease of delivery of the required doses\[^{[1]}\]. Gamma radiation and magnetic resonance imaging may affect physicochemical surface properties, such as the surface chemistry, charge and the topography, of titanium implants. So, the aim of this study was to modify surface roughness of titanium plates after exposure to magnetic resonance signals and gamma radiation.

**MATERIALS AND METHODS**

Sixty specimens of cp Ti plates \((10\text{mm} \times 10\text{mm} \times 1\text{mm})\) (ASTM, Grade IV, Modern Techniques and Materials Engineering Center, Egypt) were machined, ground on a 600-grit silicon carbide paper (Leco Co., St. Joseph, MI, USA) under running water and then cleaned in an ultrasonic (Bandelin, Sonorex, Germany) bath filled with distilled water for 5 min. These specimens were divided into six groups:

- **Group 1**: control machined group.
- **Group 2**: specimens subjected to MRI non-ionizing radio frequency (RF) signals 1.5 T for 15 minutes.
- **Group 3**: specimens subjected to MRI non-ionizing radio frequency (RF) signal 1.5 T for 30 minutes.
- **Group 4**: specimens subjected to 10 kGy of gamma radiation.
- **Group 5**: specimens subjected to 20 kGy of gamma radiation.
- **Group 6**: specimens subjected to 30 kGy of gamma radiation.

Microstructure of each specimen was performed on the flat surface of all specimens using an Shimadzu X–ray Diffractometer (Dx–30, Japan) of Cu–K\(\alpha\) radiation with \(\lambda=1.54056\) Å at 45 kV and 35 mA and Ni–filter in the angular range 20 ranging from 0 to 90° in continuous mode with a scan speed 5 deg/min.

Surface roughness was measured by using a portable surface texture measuring instrument, Surftest SJ-201 P (Mitutoyo Corporation, Japan). It is a portable solution for precise, effective and easy surface measurements in a different environment. It has oversize characters which are displayed on the large easy-to-view LCD and equipped with differential inductance detector. A diamond stylus with tip radius 5 \(\mu\)m is used in the measurements. The measured roughness parameter is the average roughness height of the surface \(R_a\) which is one of the first parameters used to quantify surface texture.

Microhardness test of each group was conducted by using a digital Vickers microhardness tester, (Model FM–7, Tokyo, Japan), applying a load of 100 g for 5 seconds via a Vickers diamond pyramid.

SEM analysis of titanium plates

Thirty six square specimens of cp Ti plates (10 mm ×10 mm × 1 mm) were prepared and divided into six groups according to surface modification procedures as mentioned before (n = 3). The surfaces of the specimens were imaged using the SEM at magnifications of 1000×.

**RESULTS**

The X-ray diffraction patterns of titanium before and after exposure to MRI for 15 and 30 minutes and gamma radiation doses (10, 20 and 30 kGy) are shown in Figures 1 and 2. Sharp lines of hexagonal Ti phase is found in untreated material, also the feature of titanium phase (intensity, broadness and position) changed after exposure to MRI and gamma radiation.

The microhardness number was conducted using a digital Vickers microhardness tester, applying constant load for 5 s, for titanium. Vickers hardness number, applying at 100gf for 5 s, of titanium decreased after exposure to gamma radiation doses (10, 20 and 30 kGy) and MRI signals as shown in Figure 3(a and b).

Mean hardness values of titanium before and after exposure to MRI for 15 and 30 minutes are present in TABLE 1. It was found that the highest mean value of the hardness was for control group (662.10±61.38). One way ANOVA test showed statistical significant difference between groups (\(F=332.4, p=0.000\)). Interaction between groups revealed that there were significant differences between control and 15 min of exposure, also between control and 30 min. However, there was no significant difference between 15 and 30 min of exposure.
Figure 1: X-ray diffraction patterns of titanium before and after exposure to MRI for 15 and 30 minutes.
Figure 2: X-ray diffraction patterns of titanium before and after exposure to gamma radiation doses (10, 20 and 30 kGy).
Figure 3: Vickers hardness number of titanium (a) before and after exposure to gamma radiation (b) before and after exposure to MRI for 15 and 30 minutes respectively.
Figure 4(a) shows the roughness profiles of titanium biomaterial implant before and after exposure to MRI for 15 and 30 minutes.

Statistical analysis of roughness (Ra) of titanium before and after exposure to MRI for 15 and 30 minutes revealed that the highest mean value was found after exposure to MRI for 30 min (1.1688±4.991E-02). One-way ANOVA showed statistical significant difference between groups (F=192.775, p=0.000) as shown in TABLE 2. Interaction between groups showed that there were significant differences between control and 15 min of exposure, also between control and 30 min. However, there was no significant difference between 15 and 30 min of exposure to MRI.

The roughness profiles of titanium biomaterial implant before and after exposure to gamma radiation doses (10, 20 and 30 kGy) are shown in Figure 4b. Mean roughness values of titanium after exposure to gamma radiation (10, 20 and 30 kGy) showed increasing in Ra values and the highest mean value was after exposure for 20 kGy (0.4125±5.120E-02). One-way ANOVA showed statistical significant difference between groups (F=61.522, p=0.000) as shown in TABLE 3. Interaction between groups showed statistical significant differences between all groups.

Exposure of titanium to gamma radiation (10, 20 and 30 kGy) affect its hardness; where there was significant reduction in the hardness and the highest mean value was for control group (676.1125±45.4587). One-way ANOVA showed statistical significant difference between groups (F=674.155, p=0.000) as shown in TABLE 4. Interaction between groups showed that there was significant difference in the hardness between control group and after exposure to gamma radiation.
at 10, 20 and 30 kGy. However, there were no significant differences between (10, 20), (10, 30) and (20, 30) kGy.

SEM examination of titanium surfaces of different groups showed that more grooves were found in groups modified with MRI signals for 30 min and gamma radiated group at 30 kGy in comparison with machined control group (Figure 5 (a, b and c)). In the control group the grooves are shallow; however in modified groups the grooves are more irregular, deep and coarse.

Figure 4b: Roughness profile of titanium before and after exposure to gamma radiation.
TABLE 1: Mean microhardness values of cp Ti with different time of exposure to MRI.

<table>
<thead>
<tr>
<th>Type</th>
<th>TIME of exposure</th>
<th>Mean</th>
<th>Std. Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td></td>
<td>662.1</td>
<td>61.1</td>
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<tr>
<td>cp Ti</td>
<td>15 min</td>
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<td>36.8</td>
</tr>
<tr>
<td></td>
<td>30 min</td>
<td>216.1</td>
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TABLE 2: Mean roughness values of cp Ti with different time of exposure to MRI.

<table>
<thead>
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<th>TYPE</th>
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<th>Mean</th>
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<tr>
<td>cp Ti</td>
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<td>0.86</td>
<td>0.1</td>
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<tr>
<td></td>
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TABLE 3: One way anova for roughness of cpTi.

<table>
<thead>
<tr>
<th>Type</th>
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<th>df</th>
<th>Mean Square</th>
<th>F value</th>
<th>P</th>
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<tbody>
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<td>2</td>
<td>2</td>
<td>192.029</td>
<td>.000</td>
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DISCUSSION

Implants with microstructured surfaces have been reported to have a more intensive bone implant contact than implants with smooth machined surfaces resulting in higher mechanical retention when implanted in humans. Those are the reasons why most of the dental systems nowadays have a micro-rough surface. In a majority of those implants, the micro-rough surfaces is obtained by grit blasting and/or acid-etching the implant[12].

The effect of magnetic resonance signal and gamma radiation on the structure and physical properties of dental materials have been recently developed and introduced[13,14].

In this study x-ray diffraction pattern of titanium implant before and after exposure to MRI for 15 and 30 minutes showed sharp lines of hexagonal Ti phase of unmodified material. However, the shape of titanium phase (intensity, broadness and position) changed after exposure to MRI for 15 and 30 minutes. This may be due to the effect of magnetic field on aggregation of matrix atoms. Also, the effect of gamma radiation on structure showed that the shape of titanium hexagonal phases (intensity, broadness and position) changed after exposure to different doses of gamma radiation, due to the interaction of high energy gamma radiation with the titanium matrix.

The average surface roughness parameter (Ra) of titanium along the total sliding distance was increased after exposure to MRI for 15 and 30 minutes and after exposure to gamma radiation doses (10, 20 and 30 kGy) because MRI signals and gamma radiation may cause some inclusions and discontinuity spots (weak spots) or irregularities in the amorphous mixture of titanium oxides and hydroxides on the surface or may form nucleation sites for cracks which affect surface roughness. This is clearly seen in the SEM examination were the grooves found on the surfaces of modified groups were more irregular, deep and coarse than in unmodified control group.

Vickers hardness number of titanium was decreased after exposure to MRI for 15 and 30 minutes because
titanium is slightly paramagnetic, so the magnetic field may cause little movements of titanium atoms causing point or line defects. Also titanium biomaterial has some impurities such as iron, oxygen, nitrogen and other elements which affected by magnetic field causing defects in the matrix. However, after exposure to gamma radiation (10, 20 and 30 kGy), Vickers hardness number of titanium was decreased because high energy gamma radiation caused a softening effect of matrix alloy and changed matrix microstructure, (crystal size, and bonding strength). Also the interaction of high energy gamma radiation with the matrix caused point defect, missing atoms from lattice, or line defects, aggregation movement of atoms which lead to decreasing the hardness of titanium.

In this study there were marked changes in the microstructure, roughness and hardness of titanium surfaces after exposure to MRI signals (15 and 30 minutes) and gamma radiation (10, 20 and 30 kGy). As the exact role of surface chemistry and topography on the early events of the osseointegration of dental implants remain poorly understood, so, in vitro study should be done to evaluate whether these modified surfaces could provide adequate osseointegration process of dental implants for their immediate loading and long-term success or not.

CONCLUSION

Within the limitation of this study, it was found that:
1. Matrix microstructure and amorphous mixture of titanium oxides and hydroxides on titanium surface were changed after exposure to MRI and gamma radiation.
2. Significant increase in surface roughness of titanium was detected after exposure to MRI and gamma radiation.
3. Significant decrease in titanium Vickers hardness was found after exposure to MRI and gamma radiation.

REFERENCES