Surface nanostructural modifications of Ti implanted by N⁺ ions as a function of energy

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Surface nanostructural modifications of Ti implanted by $N^+$ ions as a function of energy

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The surface modification of titanium foil/sheet samples (0.5 mm thickness) implanted by nitrogen ions of different energy and fluence of $1 \times 10^{18} N^+ \text{cm}^{-2}$ was studied using X-ray diffraction (XRD), atomic force microscopy (AFM), scanning electron microscopy and secondary-ion mass spectrometry (SIMS). XRD patterns showed the development of titanium nitride with different compositions in the implanted samples, and the presence of different titanium compositions such as titanium oxides was also observed. AFM images at 16 and 20 keV showed the formation of grains, which were attributed to the initial sputtering of grain boundaries. The morphology of the surface changed at 25 keV showing granular structure with an almost uniform background and lowest surface roughness relative to lower and higher implantation energies. A correlation was obtained between all results for XRD, SIMS and AFM except the titanium nitride maximum intensity at 25 keV $N^+$ implantation. In order to achieve more detailed information about the role of $N^+$ energy in this kind of work it is proposed that a further investigation is needed on both $N^+$ energy and substrate temperature as well as some theoretical studies.

Keywords: ion implantation; depth profile; surface modification; titanium

1. Introduction

In order to understand the mechanisms that lead to thin film property modification caused by the so-called sub-stoichiometric condition, a number of researchers have devoted their work to this subject [1–4]. Vaz et al. [5] studied structural, physical and mechanical properties of sub-stoichiometric TiN$_x$ thin films. In our earlier work [6] on the nanostructural modification of Ti implanted with $N^+$ ions as a function of substrate temperature we reported on the development of titanium nitride with different compositions in the implanted sample as well as the presence of different titanium compositions such as titanium oxides. As mentioned in [6], although a great amount of literature on TiN exists, to the best of our knowledge, there has been no study on the stoichiometric changes made during the $N^+$ ion implantation of bulk titanium (sheet/foil) samples prior to our work [6]. Hence, in order to extend this line

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of investigation it was decided to study the dependence of the nature of sub-
stoichiometric nitride formation in titanium (TiN$_x$) during nitrogen ion implantation
and its relationship with the nanostructural changes as a function of N$^+$ ion energy,
using X-ray diffraction (XRD), atomic force microscopy (AFM), scanning electron
microscopy (SEM) and secondary-ion mass spectrometry (SIMS).

2. Experimental details

The samples used were cut in two different sizes of 3 × 3 mm (for SIMS analysis),
10 × 5 mm (for XRD, SEM and AFM analysis) from 0.5 mm thick polycrystalline
Ti foil (Goodfellow Metals, 99.6+\% purity, including about 300 appm C, 150 appm
N and 2000 appm O). Neither mechanical nor chemical polishing treatments were
applied to the samples surfaces.

The nitrogen ion implantation of the samples was carried out at a substrate
temperature of 300 K using a fluence of $1 \times 10^{18}$ N$^+$ cm$^{-2}$ for 1600 s and with four
different N$^+$ ion energies of 16, 20, 25 and 30 keV. The ion current density during
implantation was 100 $\mu$A cm$^{-2}$. The base pressure of the chamber was $2 \times 10^{-5}$ mbar,
which during the implantation process increased to $5 \times 10^{-5}$ mbar.

A substrate holder system with controlled heating system was designed and
constructed. A detailed description of this is given in our earlier work [7]. On the
copper disk substrate holder, two of each size of the substrates can be fixed by a
stainless steel mask.

The temperatures of substrate were controlled by programmed thermostats and
thermocouples fixed inside a hole on the surface of copper disk substrate holder.

Variation of temperature during N$^+$ ion implantation was recorded using the
abovementioned system, as shown in Figure 1. Although the implantation for all
samples started at 300 K, as can be seen in Figure 1, the temperature of the substrate
as a result of heat transfer from ion beam to the samples rose to 600–700 K
depending on the N$^+$ energy at the end of implantation process. Although the
change of temperature during implantation can play a role in the nucleation and

![Figure 1. The evolution of Ti foil temperature during N$^+$ ion implantation with fluence of $1 \times 10^{18}$ N$^+$ cm$^{-2}$ and different ion energies.](image_url)
post-nucleation stages of the surface modification, we could not take this into account, and on the other hand in most cases did not change dramatically from one sample to another. In Figure 1 it can be observed that the time required to reach almost the final temperature for all initial temperatures is about 800 s (i.e. one half of the implantation time).

Prior to installation of substrate in the implantation chamber, all Ti samples were ultrasonically cleaned in heated acetone then ethanol. A Philips, X’pert MPD diffractometer (Cu Kα, radiation) with a step size of 0.02° and a count time of 2 s per step was used to obtain the microstructure of the samples, and an atomic force microscope (Auto probe PC, Park Scientific Instrument, USA; in contact mode, with low stress silicon nitride tip of less than 200 Å radius and tip opening of 18°) was employed in order to obtain the surface physical morphology and surface roughness (root mean square, \( R_{rms} \), and average, \( R_{ave} \)). The density of implanted N\(^+\) ions in Ti samples was obtained and depth profiling was carried out using SIMS (Cameca, IMS 6 F) analysis. Cs\(^+\) ions were used as primary ions with +5 keV impact energy on the surface of the sample and positive secondary ions (N\(^+\), Ti\(^+\), O\(^+\) and C\(^+\)) were detected. The spot size for SIMS measurements was 5 μm. Prior to each real sample analysis, the stability of the ion source was checked and during the process of SIMS analysis of each sample, in addition to the depth profile analysis of N\(^+\), the Cs\(^+\)Ti\(^+\) and Ti\(^+\) were also analyzed and no instability in the ion source was observed.

The details of Ti samples produced for investigation in this work are given in Table 1.

### 3. Results and discussions

#### 3.1. SIMS depth profile analysis and X-ray diffraction (XRD)

The distribution of N\(^+\) ions of energies 16, 20, 25 and 30 keV implanted in Ti foils was simulated using SRIM2000 code. The nominal N\(^+\) ion average projected range (\( R_p \)), straggling (\( \Delta R_p \)), maximum/peak nitrogen concentration (\( C_p \)) and the expected sputtering rate (\( R_s \)) at the end of implantation process (i.e. after 99,000 ions were implanted), obtained from SRIM2000 calculations, are given in Table 2. With respect to the results of the sputtering rate it should be noted that since the binding energy of the surface atoms for titanium is not available, the heat of sublimation is not

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>N(^+) energy (keV)</th>
<th>( R_{rms} ) (Å)</th>
<th>( R_{ave} ) (Å)</th>
<th>Grain diameter (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>16</td>
<td>21.7</td>
<td>17.4</td>
<td>157.0</td>
</tr>
<tr>
<td>Ti(_1)</td>
<td>20</td>
<td>62.5</td>
<td>44.9</td>
<td>153.0</td>
</tr>
<tr>
<td>Ti(_5)</td>
<td>25</td>
<td>63.9</td>
<td>50.7</td>
<td>150.0</td>
</tr>
<tr>
<td>Ti(_9)</td>
<td>30</td>
<td>32.5</td>
<td>24.1</td>
<td>141.0</td>
</tr>
<tr>
<td>Ti(_13)</td>
<td>30</td>
<td>90.8</td>
<td>70.4</td>
<td>148.0</td>
</tr>
</tbody>
</table>
used instead in the SRIM calculation, which apparently does not predict the realistic values and should be considered as a rough estimate. It should also be noted that the role of temperature, ion flux and some other parameters such as surface roughening, hence change of surface binding energy, are not included in SRIM2000 computer simulation code.

Figure 2 shows the SIMS profiles for $N^+$, $Ti^+$, $O^+$ and $C^+$ from untreated and implanted Ti foil for four different $N^+$ ion energies of 16, 20, 25 and 30 keV. The density of the implanted nitrogen ions and $O^+$, $C^+$ and $Ti^+$ sputtered from the Ti samples may be estimated from the area under the corresponding curves in Figures 2, as shown in Figure 3a [8]. In order to obtain a quantitative value for the ion penetration depth, the sputtering time should be translated to depth, which usually needs some means of crater depth measurement, such as large-scan AFM or a suitable profilometer. Since such an instrument was not accessible to us, the data presented here can only be considered as qualitative results.

Results show that there exists a minimum for $Ti^+$, $N^+$, $O^+$ and $C^+$ curves at about 20 keV, and a maximum at higher energies, which is more distinguishable in case of $C^+$. The penetration depth of $N^+$ in Ti samples (Figure 3b) also shows a minimum about 20 keV.

Figure 4 shows the XRD patterns obtained from untreated titanium, and those of $N^+$-implanted Ti at four different energies of 16, 20, 25 and 30 keV.

If we compare the XRD patterns of the unimplanted and implanted titanium foils in Figure 4, it can be observed that the following Ti nitride crystallographic orientations of different phases are formed after $N^+$ ion implantation with different energies:

- $TiN(200)$, $TiN(400)$ (cubic) (with respect to JCPDS card No: 38-1420);
- $Ti_2N(103)$, $Ti_2N(111)$ (tetragonal) (with respect to JCPDS card No: 17-0386);
- $TiN_{0.26}(102)$, $TiN_{0.26}(103)$ (hexagonal) (with respect to JCPDS card No: 44-1095);
- $\eta$-$Ti_3N_2$ (1024), $\eta$-$Ti_3N_2$ (1022) (rhombohedral) (with respect to JCPDS card No: 40-0958);

and intensity changes occur in the following Ti oxide diffraction lines:

- $Ti_3O_3(131)$, $Ti_2O_3(226)$, $Ti_2O_3(404)$, (rhombohedral) (with respect to JCPDS card No: 43-1033);

Table 2. Results for different implantation parameters from SRIM2000 simulation program.

<table>
<thead>
<tr>
<th>$N^+$ ion energy (keV)</th>
<th>$R_p$ (nm)</th>
<th>$\Delta R_p$ (nm)</th>
<th>$C_p$ (atoms cm$^{-3}$)</th>
<th>$R_s$ (atoms ion$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>16</td>
<td>30.8</td>
<td>14.8</td>
<td>$2.35 \times 10^{23}$</td>
<td>0.5828</td>
</tr>
<tr>
<td>20</td>
<td>37.4</td>
<td>17.5</td>
<td>$2.00 \times 10^{23}$</td>
<td>0.5418</td>
</tr>
<tr>
<td>25</td>
<td>45.6</td>
<td>20.5</td>
<td>$1.75 \times 10^{23}$</td>
<td>0.4969</td>
</tr>
<tr>
<td>30</td>
<td>53.5</td>
<td>23.3</td>
<td>$1.50 \times 10^{23}$</td>
<td>0.4804</td>
</tr>
</tbody>
</table>
As is evident, the intensities of these peaks vary with N\(^{+}\) ion energy, whereas some of them are very weak, which adds to the complexity of the analysis. Due to the reactive nature of titanium with oxygen (i.e. the gettering property of titanium) different diffraction lines from different phases of titanium oxide (i.e. Ti\(_2\)O\(_3\) and anatase (A) and brookite (B) phases) are also present in the XRD pattern of the unimplanted Ti sample. In order to present such a number of diffraction lines

- A(105), A(400), A(411), A(228) (with respect to JCPDS card No: 21-1272);
- B(160), B(332), B(024), B(125) (with respect to JCPDS card No: 29-1360).

Figure 2. SIMS profiles of implanted titanium with N\(^{+}\) ions with a fluence of 1 \times 10^{18} N\(^{+}\) cm\(^{-2}\) and different energies: (a) N\(^{+}\) profiles; (b) Ti\(^{+}\) profiles; (c) O\(^{+}\) profiles; (d) C\(^{+}\) profiles.
obtained for different phases of titanium nitride and titanium oxides, we had no choice but to divide the XRD patterns into three ranges, namely 30–60, 60–90 and 90–120° for presentation in Figure 4.

The d-spacing of the titanium nitride lines obtained from the XRD patterns are listed in Table 3. Variation of all the data in Table 3 with $N^+$ ion energy is almost negligible. Crystallite size (coherently diffracting domains), $D$, was obtained by applying the Scherrer formula [9] to measure the full width at half maximum (FWHM) of the dominant peaks of different titanium composites (i.e. TiN(111), Ti$_2$N(111),...

![Graph 1](image1.png)

**Figure 3.** (a) N$^+$, O$^+$, C$^+$ and Ti$^+$ ion densities and (b) penetration depth of N$^+$ ions in Ti versus N$^+$ energy for N$^+$ ions with a fluence of $1 \times 10^{18}$ N$^+$ cm$^{-2}$ implanted in a titanium sample.
The results are given in Table 4. The results show that the crystallite size decreases with increasing $N^+$ energy to a minimum at 25 keV, then again at 30 keV it increases. This behavior is in agreement with the AFM results discussed in Section 3.2.

In order to resolve the twin and triplet peaks we had to fit Voigt’s functions plus a level background to the experimental peaks [10], as discussed in our earlier work [6]. Similar to the procedure implemented in [6] the area under the peak was considered as the intensity of diffracted X-ray according to the following list:

- titanium peaks Ti(002) and Ti(101) (Figure 4a);
- titanium nitride peaks TiN(400), TiN(200), Ti$_2$N(111) and Ti$_3$N$_{2-x}$(2014) (Figure 4b), and Ti$_2$N(103), TiN$_{0.26}$(102), TiN$_{0.26}$(103), Ti$_3$N$_{2-x}$(1022) (Figure 4c);
the brookite phase of titanium oxide $B(125)$, $B(160)$, $B(332)$ and $B(024)$ (Figure 4d);

- the anatase phase of titanium oxide $A(105)$, $A(400)$, $A(411)$ and $A(228)$ (Figure 4e);

- and Ti$_2$O$_3$(404), Ti$_2$O$_3$(131) and Ti$_2$O$_3$(226) (Figure 4f).

In Figure 5 it can be observed that, apart from TiN(400), Ti$_3$N$_{2-x}$(2014), TiN$_{0.26}$(102), TiN$_{0.26}$(103) and Ti$_3$N$_{2-x}$(1022), which only show a maximum at about 25 keV energy, the rest of the diffraction lines, irrespective of being nitride or oxide, show a minimum at about 20 keV energy and a maximum at about 25 keV energy. One may correlate the variations obtained for the area (density) and the penetration depth for $N^+$, $O^+$, $Ti^+$ and $C^+$ in Figures 3a and 3b to the variations in the intensity of the diffraction lines observed in the XRD results in Figure 5.
Table 3. The $d$ spacing of different titanium nitride lines in Å.

<table>
<thead>
<tr>
<th>Sample</th>
<th>TiN (111)</th>
<th>TiN (200)</th>
<th>TiN (400)</th>
<th>Ti$_2$N (103)</th>
<th>Ti$_2$N (111)</th>
<th>TiN$_{0.26}$ (102)</th>
<th>TiN$_{0.26}$ (103)</th>
<th>Ti$<em>3$N$</em>{2-x}$ (1022)</th>
</tr>
</thead>
<tbody>
<tr>
<td>16 keV</td>
<td>2.5458</td>
<td>2.1089</td>
<td>1.0659</td>
<td>2.3558</td>
<td>2.2925</td>
<td>1.7356</td>
<td>1.3355</td>
<td>0.9889</td>
</tr>
<tr>
<td>20 keV</td>
<td>2.5451</td>
<td>2.1063</td>
<td>1.0667</td>
<td>2.3530</td>
<td>2.3019</td>
<td>1.7284</td>
<td>1.3309</td>
<td>0.9887</td>
</tr>
<tr>
<td>25 keV</td>
<td>2.5461</td>
<td>2.1095</td>
<td>1.0667</td>
<td>2.3569</td>
<td>2.3024</td>
<td>1.7302</td>
<td>1.3334</td>
<td>0.9899</td>
</tr>
<tr>
<td>30 keV</td>
<td>2.5467</td>
<td>2.1097</td>
<td>1.0664</td>
<td>2.3593</td>
<td>2.2911</td>
<td>1.7381</td>
<td>1.3377</td>
<td>0.9898</td>
</tr>
</tbody>
</table>

Table 4. The crystallite size, $D$, for different titanium composites.

<table>
<thead>
<tr>
<th>Sample</th>
<th>TiN(111) (Å)</th>
<th>Ti$_2$N(111) (Å)</th>
<th>TiN$_{0.26}$(103) (Å)</th>
<th>Ti$<em>3$N$</em>{2-x}$(1022) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>16 keV</td>
<td>1750</td>
<td>1721</td>
<td>850</td>
<td>1640</td>
</tr>
<tr>
<td>20 keV</td>
<td>790</td>
<td>820</td>
<td>520</td>
<td>1160</td>
</tr>
<tr>
<td>25 keV</td>
<td>530</td>
<td>450</td>
<td>290</td>
<td>530</td>
</tr>
<tr>
<td>30 keV</td>
<td>1600</td>
<td>1630</td>
<td>470</td>
<td>850</td>
</tr>
</tbody>
</table>
The minima and maxima for O$^+$ and Ti$^+$ in Figure 3a can be correlated to the corresponding minima and maxima obtained for titanium oxides of different nature in Figures 5d–5f and titanium in Figure 5a, whereas one should admit that the maxima in Figure 3a are broader and may not be clearly distinguishable. In case of N$^+$ ions in Figures 3a and 3b, it can be observed that after minimum at about 20 keV the density of N$^+$ (area) increases, whereas in Figure 5 for the nitride intensities a group of nitride composites (i.e. TiN(200), Ti$_2$N(111), and Ti$_2$N(103)) show almost
similar behavior (i.e. a minimum at about 20 keV and a maximum at about 25 keV) to that observed in Figure 3a (i.e. a correlation exists), the other group (i.e. TiN(400), Ti$_3$N$_{2-x}$(2014), TiN$_{0.26}$(102), TiN$_{0.26}$(103), and Ti$_3$N$_{2-x}$(1022)) only show a maximum at about 25 keV. The contribution/sum of these two groups produces a maximum at about 25 keV (Figure 6), which apparently does not correlate with the SIMS results presented in Figure 3.

In order to carry out further analysis, the contribution of C$^+$ ions as a contaminant was also taken into account and the ratios of N$^+$/Ti$^+$, O$^+$/Ti$^+$, and C$^+$/Ti$^+$ (obtained from Figure 3a) are plotted (Figure 7). In Figure 7, it can be observed that C$^+$/Ti$^+$ ratio shows a minimum at about 20 keV and a maximum at 25 keV, whereas the behavior of O$^+$/Ti$^+$ and N$^+$/Ti$^+$ curves is exactly, as expected,
opposite to C⁺/Ti⁺. This clearly shows the role of C⁺ contamination in this work, which exhibits the expected correlation between these elements. However, none of these provide a clear evidence for the maximum obtained for the second titanium nitride group mentioned above.

In our earlier work on the role of temperature on modifications of titanium during N⁺ ion implantation [6] we were able to clearly correlate the XRD results to those of SIMS. This work shows that the role of N⁺ energy in the process of implantation is more complex than that for temperature and may require a further and profound investigation on the role of N⁺ energy at higher temperatures too as well as theoretical study of the possible interactions between the chemicals involved in these processes. This cumbersome work (i.e. a matrix made of energy and temperature) is planned in our group and the results will be reported in the near future.

The abovementioned analyses of XRD and SIMS showed the formation of titanium nitride by N⁺ ions implantation into Ti foils, whereas Ti oxide is also formed due to strong reactivity (gettering effect) of Ti with different gases (residual gases) in the experimental chamber. Titanium oxide exists on the sample surface in its different composite forms and can form during implantation more vigorously, as can be recognized from the XRD results in Figure 4. The oxides can perturb the surface crystallography of the sample, whereas implantation of nitrogen ions in the sample (again as can be seen in the XRD results) causes the formation of different titanium nitride composites with different crystallographic structures/orientations. Hence, in this case, there exists a complex and amalgamated situation involving different structures/morphologies (sub-stoichiometric conditions).

3.2. SEM micrographs

Figure 8 shows typical micrographs obtained from untreated and 16 and 30 keV N⁺-implanted Ti samples. Similar to our earlier work [6], comparison of SEM micrographs of untreated and N⁺ implanted Ti samples shows the inadequacy of the SEM in resolving the structural changes to the degree obtained by AFM. As a result, there is no point in further discussing SEM results.

3.3. Surface morphologies obtained by AFM

Figure 9 shows the AFM images of both untreated and N⁺ ion-implanted Ti samples. The untreated Ti showed a granular morphology (Figure 9a). The Ti sample implanted with 16 keV N⁺ shows no significant change from the untreated sample, perhaps due to low ion energy (Figure 9b). At higher energy of 20 keV (Figure 9c) grains are formed with sharp edges and valleys between them. This can be due to the initial preferred sputtering of the grain boundaries, which has become clearly distinguishable at this energy. However, when the N⁺ energy is increased to 25 keV the whole surface structure is changed and a somewhat granular structure (smaller in size when compared with grain sizes in Figure 9c) on an almost uniform background can be observed (Figure 9d). This film with regard to the nitride formation using both XRD (Figure 5) and SIMS (Figure 3a) analyses showed the maximum
concentration of nitride. Hence, it may be concluded that the change in the surface morphology of this sample is due to formation of titanium nitride with high concentration. Figure 9e shows the three-dimensional (3D) image of sample produced using N$^+$ with 30 keV energy. It can be observed that large, distinguishable and columnar grains are formed on the surface of this sample. This may be again due to the sputtering of the titanium nitride grain boundaries by these high energy N$^+$ ions. From the simulation results using SRIM2000 Code it was also obtained that at this energy maximum/peak nitrogen concentration is reduced to its minimum.

The root mean square and the average roughness values, $R_{rms}$ and $R_{ave}$, obtained from AFM measurements are shown in Figure 10. Both $R_{rms}$ and $R_{ave}$ follow a
Figure 9. AFM images of untreated and N\(^+\) ion (fluence of \(1 \times 10^{18} \text{N}^+ \text{cm}^{-2}\)) implanted titanium with different \(\text{N}^+\) energies: (a) untreated; (b) 16 keV; (c) 20 keV; (d) 25 keV; (e) 30 keV.

Figure 10. Root mean square roughness and average roughness of Ti samples produced with different \(\text{N}^+\) energies.
similar trend and are consistent with microscopic observations. The variation of the surface roughness of the samples with energy is also consistent with the above mentioned description of 3D AFM images.

4. Conclusions

The results of XRD, AFM, SEM and SIMS analysis of the nanostructure of the surfaces of titanium foil/sheet samples (0.5 mm) modified by implantation of nitrogen ions of different energy showed the formation of different Ti–nitride and Ti–oxide composites. The AFM images of the samples at lower energies of 16 and 20 keV showed the formation of grains due to initial sputtering of the grain boundary. At a higher energy of 25 keV the topography of the film surface changed, indicative of higher titanium nitride concentration and consistent with the XRD and SIMS results.

Apart from the maximum obtained for the titanium nitride formation at 25 keV energy in the XRD results, a correlation between the XRD, SIMS and AFM results is achieved. The former may require further profound investigation on both N⁺ energy and substrate temperature as well as some theoretical studies such as interaction of particles with matter by including of ion energy and in particular the substrate temperature hence kinetic energy of substrate atoms during implantation (interaction). This may provide a more detailed computation program than SRIM or TRIM (which are lacking these effects/parameters) for experimental researchers.

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