TEMPERATURE-DEPENDENT TRIBOLOGICAL IMPROVEMENTS IN LOW-ENERGY NITROGEN ION IMPLANTED VANADIUM-TITANIUM ALLOYS

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ABSTRACT
A detailed tribological characterization of low-energy, nitrogen implanted V5at. %Ti alloy is presented. Samples were nitrogen-implanted at an accelerating voltage of 1.2 kV and 1 mA/cm², up to a dose of 1E19 ions/cm². The tribological properties of the alloys: microhardness, friction coefficient and wear resistance, have improved after ion implantation and this improvement increases as the implantation temperature increases. The microstructure of the alloys were analysed by transmission electron microscopy. A direct correlation between structural modifications of the nitrogen implanted layer and the improvement in their tribological properties is obtained. For samples implanted at 848 K a nanocomposite layer where the reinforcement particles are TiN precipitates forms. TiN precipitation appears as the responsible of the improvement in the tribological properties.

INTRODUCTION
Active research on low-energy high-fluence ion implantation into metallic targets has been developed in the last years to improve their tribological properties. The main advantage of low-energy high-current-density ion implantation is the high depth of ion penetration, >1μm, as compared with the conventional high-energy low-current-density implantation, 0.1-0.3μm. Beam energy, flux and implantation temperature determines the final depth of the implanted layer1. Recent results have proved that high and low-energy nitrogen implantations are an effective tool to improve the tribological properties of VTi alloys2,3. In this research the role of the implantation temperature on the tribological properties in low-energy nitrogen ion implantation of V5at%Ti produced by arc melting was analyzed. Transmission electron microscopy (TEM) and energy-dispersive x-ray spectroscopy (EDX) have been used to characterize the structural modifications induced by the implantation.

RESULTS
The tribological properties of the alloys have been measured using as a reference the measurements made in the unimplanted area of the samples, the back side of each sample. The tribological properties are summarized in table1.

<table>
<thead>
<tr>
<th>Implantation Temperature</th>
<th>Universal Hardness (N/mm²) at final load of 2 mN</th>
<th>Friction Coefficient at contact pressure of (GPa)</th>
<th>Wear Coeff (m²/N) at load 1 N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unimplanted 1900±100</td>
<td>0.9</td>
<td>0.6</td>
<td>3.0 E13</td>
</tr>
<tr>
<td>Implanted 3000: 300</td>
<td>0.25 →0.9</td>
<td>0.42 →0.9</td>
<td>1.4 E13</td>
</tr>
<tr>
<td>Unimplanted 1900±200</td>
<td>0.6</td>
<td>0.75</td>
<td>3.5 E13</td>
</tr>
<tr>
<td>Implanted 3000: 400</td>
<td>0.15</td>
<td>0.2 →0.65</td>
<td>1.3 E13</td>
</tr>
<tr>
<td>Unimplanted 1900±300</td>
<td>0.6</td>
<td>0.75</td>
<td>3.5 E13</td>
</tr>
<tr>
<td>Implanted 5200: 800</td>
<td>0.15</td>
<td>0.2 →0.65</td>
<td>9.7 E14</td>
</tr>
<tr>
<td>Unimplanted 1800±200</td>
<td>0.6</td>
<td>0.75</td>
<td>3.5 E13</td>
</tr>
<tr>
<td>Implanted 9000: 2000</td>
<td>0.15</td>
<td>0.25</td>
<td>---</td>
</tr>
</tbody>
</table>

Microindentation tests exhibited a noticeable increase in hardness. A clear enhancement of the hardness with the...
implantation temperature was found. The test for a final load of 2 mN are presented in figure 1 for samples implanted at different temperatures. For a temperature of 753 K the penetration of the indenter was ≈100 nm, however for the sample implanted at 848 K the penetration of the indenter is reduced until ≈40 nm. The thickness of the implanted layer active for the hardness increase can be estimated from the microindentation tests only for an implantation temperature of 673 K and was close to 1 µm.

Figure 1. Microindentation load-unload curves for a final load of 2 mN

The measured friction and wear coefficients for implantations at different temperatures are presented in table 1. In samples implanted at T=673 K, for low contact pressure, ≤0.60 GPa, two stages in the friction coefficient are observed. The initial stage having a low friction coefficient of μ=0.1, only for few cycles is followed by a sharp transition to a second stage, rising stage, μ=0.25, where the friction coefficient rises to the value measured for the unimplanted samples. For samples implanted at T=673 K the friction coefficient μ=0.15 and no sharp transitions between low and high friction coefficient were observed. For high contact pressure, ≥0.65 GPa, it is possible to observe the transition from high to low friction regime except for the samples implanted at T=848 K. The observed wear decrease, see table 1, is expected after the hardness increase measured in the microindentation tests. No wear was measured for the samples implanted at T=848 K when the same contact pressure than for samples implanted at lower temperature is used, even after 10^5 cycles.

Unimplanted alloys appear free of defects and only isolated non-stoichiometric titanium carbide precipitates were observed in TEM images. For samples implanted at T=673 K, up to 0.25 µm deep, a heavily contaminated layer with carbon and other impurities was observed; follow by a distribution of nanocrystals embedded in an amorphous matrix, from 0.25 µm to 0.8 µm deep, and from 0.8 µm to 1 µm deep, precipitates are imaged. The EDX spectra indicate that some of the nanocrystals and all the precipitates analysed are rich in Ti. The alloys implanted at T=848 K are polycrystalline and show an inhomogeneous distribution of precipitates. Figure 2 is a typical cross-sectional bright field image from a region with precipitates. Precipitates have needle shape and it is possible to observe these until the limit of transparency for the TEM images, close to 9 µm deep. The crystal structure of the precipitates were analysed by electron microdiffraction, and was identified as FCC with a lattice parameter of 0.42 nm. In the EDX spectra from the precipitates and in very thin areas of the sample N and Ti were detected and can be unambiguously identified as TiN.

Figure 2. Bright field cross-sectional image of a sample implanted at 848 K. TiN precipitates are indicated by an arrow. Inset: Detail of a precipitate.

SUMMARY AND CONCLUSIONS

Tribological properties are improved after low energy N-implantation. An accurate selection of the implantation temperature determines the tribological performance of the V-Ti alloys and the thickness of the layer modified by implantation. For samples implanted at T=673 K the active thickness of the implanted layer was estimated ~1 µm. In samples implanted at T=848 K the active layer is an order of magnitude bigger, > 9 µm. The last is an indication that ballistic and diffusive processes operate simultaneously for lower implantation temperature, T ≈ 753 K whereas diffusion is the main active process for T ≈ 848 K. TiN precipitation appears to be responsible for the improvement in the tribological properties. For implantation at 848 K a nanocomposite layer containing TiN precipitates forms at the sample surface.

ACKNOWLEDGMENTS

Research supported by CICYT project ESP2002-04509-C04-01. TEM work has been made at the IABMET of Univ. Carlos III. The help of Dr. J.P. Rivère with samples implanted at low temperature and the facilities given by the Laboratoire de Metallurgie Physique, Univ. de Poitiers are acknowledged.

REFERENCES