

**TRIBOLOGICAL BEHAVIOR OF NIOBIUM TREATED BY HIGH TEMPERATURE NITROGEN PLASMA BASED ION IMPLANTATION**

Lilian Hoshida<sup>1\*</sup>, Michel Felipe Lima de Araújo<sup>1</sup>, Rogério de Moraes Oliveira<sup>1</sup>  
*INPE – National Institute for Space Research - São José dos Campos, SP, Brasil.*

**1. Introduction**

Niobium is a refractory metal that exhibits excellent properties such as high melting point (2468°C), high electrical conductivity, good corrosion resistance, malleability, high hardness and low density in comparison to other refractory metals and a wide variety of applications, such as in components of nuclear reactors, rockets, missiles, space systems engineering [1]. A major disadvantage of niobium is that in its pure state, it promptly oxidizes at high temperatures (above 400°C) in the presence of oxygen. Nevertheless, the incorporation of nitrogen to its surface may have the effect of improving the oxidation resistance of the bulk and improve its mechanical and tribological properties [2]. In this work, niobium was treated by high temperature nitrogen plasma based ion implantation (HTPBII), in order to produce a thick niobium nitride layer on its surface. The modified surface layer was characterized in relation to its tribological properties.

**2. Experimental**

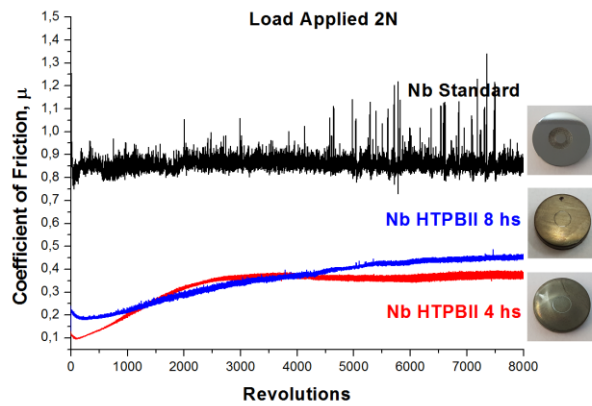
The niobium samples (99.4% purity) were cut in discs with 13 mm in diameter and 3mm in thickness. These samples were polished until a mirror like surface finish had been obtained. After polished the samples were cleaned by ultrasound bath with acetone for 10 minutes. Before treatment they were cleaned by argon sputtering for approximately 10 minutes. Nitrogen PBI was run at a working pressure of  $6 \times 10^{-3}$  mbar, and high voltage pulses of 7kV/30 $\mu$ s/500Hz were applied in two different conditions, for 4 and 8 hours. For both cases the samples were heated up to 1200°C during treatments.

Dry friction coefficient measurements were accomplished in pin-on-disk tribometer using a 3mm diameter Al<sub>2</sub>O<sub>3</sub> ball, which was loaded by the force of 2 N against the rotating flat niobium surface with 5 cm/s of linear speed and 2.06 mm of wear track radius. The worn tracks were examined by scanning electron microscopy (SEM) and the volume loss and wear rate were calculated according to ASTM G-99.

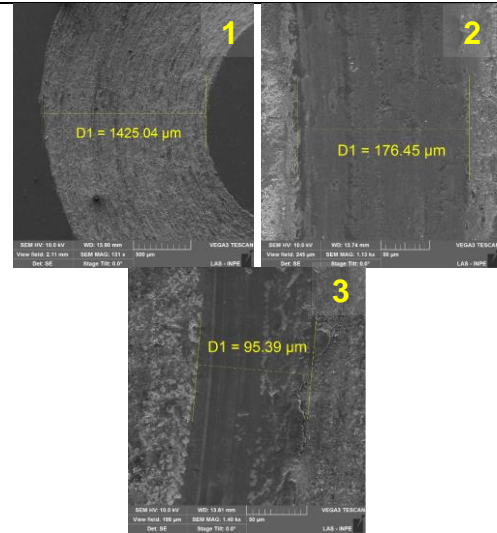
**3. Results and Discussions**

The pin-on-disc test revealed that the samples treated by HTPBII presented lower coefficients of friction (CoF) in relation to untreated sample, as shown in Figure 1. The measurements were monitored up to 8,000 revolutions and, for this case, slightly lower  $\mu$  values were attained for the sample treated during 4 hours. The worn tracks of all the samples were examined by SEM, as shown in Figure 2. The width of the tracks ( $D$ ) was measured at five different points, revealing a significant reduction of respective mean values in favor of the treated samples. In fact, while  $D \sim 1430 \mu\text{m}$  for untreated sample, a tenfold reduction was measured for the sample treated during 4 h ( $D \sim 171 \mu\text{m}$ ). For the sample treated during 8 h,  $D \sim 95 \mu\text{m}$ , almost half the value measured by the sample treated during 4 h. The respective volume loss ( $V$ ) was calculated taking into account the mean width of worn tracks, sliding distance and number of revolutions. Once again the longer treatment time led to very significant reduction of  $V$ . In fact,  $V \sim 2,27 \text{ mm}^3$  for untreated sample,  $3,60\text{E}^{-3} \text{ mm}^3$  for 4 hour sample and  $6,17\text{E}^{-4} \text{ mm}^3$  for 8 hour sample. It seems that thicker modified layers attained by the treatments performed for longer times are the main responsible for additional improvement of tribological behavior of Nb samples.

Further analysis will be carried out, as the comparative study of tribological performance by increasing the number of revolutions during pin-on-disk tests. Additionally, EDS analysis will be also performed to evaluate nitrogen peaks present on the worn tracks.



**Fig. 1.** Coefficient of friction for untreated and treated samples.



**Fig. 2.** Worn tracks viewed by SEM for (1) untreated sample, (2) 4 hours treated and (3) 8 hours treated.

#### 4. References

- [1] R. M. Oliveira, A. C. Oliveira, F. C. Carreri, G. F. Gomes, M. Ueda. *App.Surf. Science*, 283, 382-388, (2013).
- [2] C. Borcz, C. M. Lepienski, S. F. Brunatto. *Surf. and Coatings Technol.*, 224, 114-119, (2013)