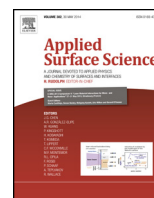




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Phase formation and mechanical/tribological modification induced by nitrogen high temperature plasma based ion implantation into molybdenum

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ABSTRACT

Transition metal nitrides present high hardness, good wear resistance and chemical stability. The formation of a surface layer of these materials on different types of substrates can improve surface properties without changing bulk characteristics. Molybdenum is used in many technological applications and the search for ways to effectively improve its properties is justified. In this work nitrogen ions were implanted into molybdenum by means of high temperature plasma based ion implantation (HTPBII), in order to produce a layer of molybdenum nitride on the surface of the material. The treatment was performed in the temperature range of 800–1200 °C, for 1 h. X-ray diffraction spectra showed the presence of the cubic-Mo₂N phase in most of the samples. The tetragonal-Mo₂N phase was also observed, depending on treatment conditions. Glow discharge optical emission spectroscopy was used to study the composition and thickness of the nitride layer. A 12 μm thick Mo₂N layer was observed for samples treated at 1100 °C, although beyond this temperature threshold, a significant amount of nitride can no longer be produced. In relation to the surface mechanical properties, a ninefold increase in surface hardness was obtained, as well as a decrease in the friction coefficient. Wear against an alumina ball was not observed.

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1. Introduction

Transition metal nitrides present high hardness, good wear resistance and chemical stability [1,2]. Due to these interesting properties, different methods have been developed in order to produce a surface layer of these materials on different types of substrates, improving surface properties without changing bulk characteristics [3–5]. In view of that, molybdenum nitrides such as Mo₂N are very interesting materials [5,6].

Molybdenum is used in many technological applications, mostly as an alloying element [7]. But the pure element is also used as a catalyst [8,9] and in aerospace applications where it is present in gridded ion thrusters [10]. In this case, the molybdenum extraction grids are struck by energetic ions, which cause sputtering and erosion of the material, limiting its lifetime [11]. Therefore, the search for ways to effectively improve the properties of molybdenum is

justified. One way in which this could be performed is by producing a layer of Mo₂N on its surface.

Plasma based ion implantation (PBII) is a very efficient technique that can accomplish this task, since it allows the attainment of surfaces with improved mechanical and tribological properties without altering bulk characteristics [12]. High temperature plasma based ion implantation (HTPBII) is an even more attractive technique for several cases, because it combines the effects of the ion implantation with diffusion of the implanted ions. The result is the formation of thicker modified layers, which can present even better physical and chemical properties. Heating of the samples during PBII can be accomplished by several different means. In this work, a technique that combines electron with ion bombardment was employed [13]. This technique has been recently applied with success to titanium alloys [14–16] and niobium [17].

Previous studies have also investigated the implantation of nitrogen into molybdenum, in temperatures up to 750 °C, but in this temperature range the obtained nitride layer was not very thick [18,19]. Our experimental setup allows the heating of samples to much higher temperatures. In view of that, the aim of this work is to

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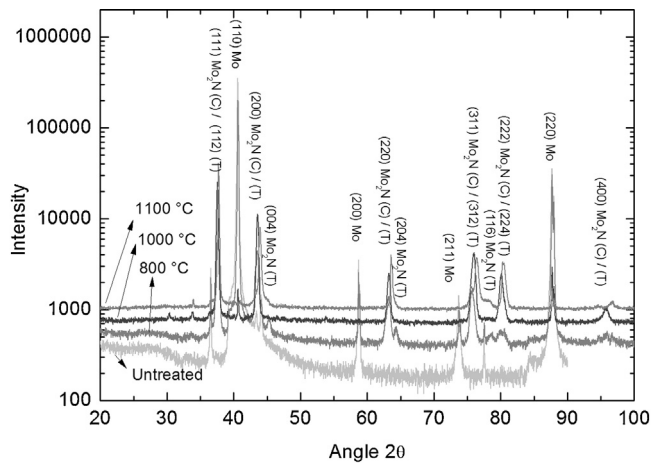


Fig. 1. XRD for untreated and treated samples between 800 °C and 1100 °C.

study the surface composition and mechanical properties (micro-hardness, friction and wear behavior) of molybdenum treated by HTPBII in temperatures up to 1200 °C, in order to improve its properties for mechanical wear resistant applications.

2. Experimental

Molybdenum samples (99.9% pure) with 2.0 cm in diameter and 2 mm in thickness were polished to a mirror-like finish. Before treatment they were cleaned in ultra-sound bath with acetone for 10 min and in isopropyl alcohol for another 10 min. Nitrogen HTPBII was performed at a working pressure of 3 mtorr. High negative voltage pulses of 10 kV/30 μs/500 Hz were applied to the samples for 1 h. In order to heat the samples to the desired temperature, during pulse-off time the sample-holder is positively biased (50–150 V), attracting electrons emitted by a low work-function (2 eV) thermionic cathode, while ion implantation takes place during pulse-on time. Details on this setup are described elsewhere [13]. Studied treatment temperatures were 800 °C, 1000 °C, 1100 °C and 1200 °C. Other treatment parameters – working pressure, treatment time and pulse parameters – were kept the same for all samples.

The phase composition was analyzed by XRD (Philips 3410 diffractometer in a standard theta–2theta Bragg – Brentano configuration with Ni-filtered Cu Kα radiation). The nitrogen concentration profiles of implanted samples were obtained by glow discharge optical emission spectroscopy (GDOES), carried out on a Jobin-Yvon GD profiler. The surface hardness was measured by microindentation with a normal load of 50 gf for all samples except the sample treated at 800 °C, which was measured with a load of 10 gf. Dwell time was 10 s. Dry friction coefficient measurements were accomplished in a CSM-Instruments pin-on-disk tribometer. A 3 mm diameter Al₂O₃ ball (99.8% purity), hardness of 19 GPa, was loaded by the force of 2.0 N against the rotating flat sample surface with 5 cm/s of linear speed and 3 mm of wear track radius. The test was performed up to a sliding distance of 190 m. The worn tracks and the surface roughness were studied with an optical profiler (Wyco NT 1100). Surface morphology was viewed by Scanning Electron Microscopy (SEM), using a JEOL JSM-5310 apparatus.

3. Results and discussion

XRD analysis for samples treated between 800 °C and 1100 °C (Fig. 1) shows very intense and clear Mo₂N peaks. For treatment temperature of 1100 °C, pure molybdenum substrate peaks do not even appear, while at 1000 °C, only small (110) and (220) Mo

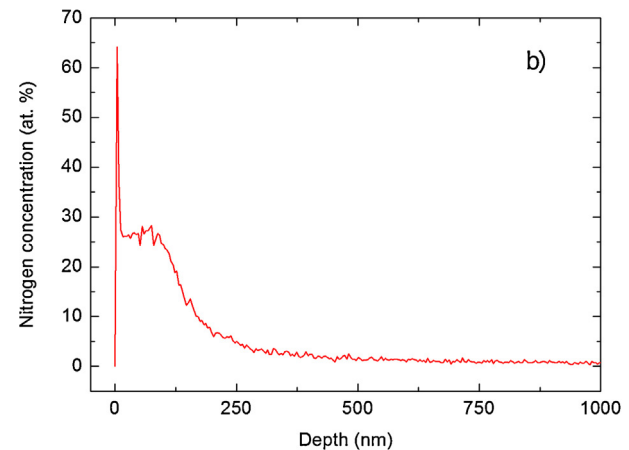
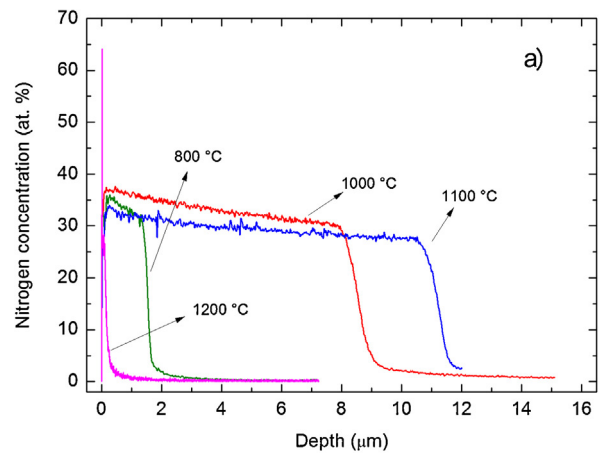


Fig. 2. Nitrogen concentration profiles as a function of depth for high temperature-implanted samples. (a) All samples, (b) magnified profile of sample treated at 1200 °C.

peaks can be detected. In as much as the analysis was not performed in grazing angles, it can be inferred that the produced nitride layer might be significantly thick. GDOES analysis (Fig. 2a) confirms that, showing the presence of nitrogen up to a depth of 12 μm for sample treated at 1100 °C, 9 μm at 1000 °C and 2 μm at 800 °C. Nitrogen concentration profiles also show a concentration plateau around 30–35 atomic percentage, which is consistent with the stoichiometry of Mo₂N. This indicates that a layer of this material is being produced. The diffusion process is clearly very important and contributes to the formation of thicker nitride layers as temperature increases.

The sample treated at 800 °C contains not only the cubic phase of Mo₂N, but also the tetragonal phase, since some secondary peaks of this phase could be detected at $2\theta = 45.3^\circ$, 64.3° and 78.6° , according to the PDF database [20]. The presence and evolution of these peaks had been previously observed as the implantation temperature increased from 450 °C to 700 °C [18,19]. This unusual transition from c-Mo₂N to t-Mo₂N was attributed to a lattice distortion due to the Jahn-Teller effect [21], coupled with a volume reduction of the lattice. However, as treatment temperature further increases, that is, above 1000 °C, only the cubic-Mo₂N phase can be detected. In addition, it should be noticed that the nitride peaks are dislocating toward higher 2θ angles as treatment temperature increases. The dislocation is more evident in the (220), (311) and (222) c-Mo₂N peaks, and is an indication of residual stress in the nitride layer, which is a common occurrence in surface modification and deposition processes. Such stress could be induced during ion implantation as well as during the fast cooling process.

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