EFFECTS OF PLASMA IMMERSION ION IMPLATATION ON FATIGUE PROPERTIES OF TITANIUM ALLOY SURFACES

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1. Introduction

Lightweight materials capable to withstand high temperatures with friction and/or oxidation are a demand imposed by several transportation and power generation systems, as in high speed aircrafts and engines, respectively. In order to comply with such requirements, the improvement of surface properties of these materials is essential, as well as the understanding of the links among their composition, processing method, microstructure and properties [1]. In addition, high temperature resistant alloys must combine two major requirements when thermal cycles are applied: low scale growth rate and adequate scale adherence [2]. Materials such as stainless steels, refractory metals and alloys, and superalloys are especially creep-resilient and commonly employed in high temperature service applications [3]. Nevertheless, modern surface functionalization is often applied. Ion implantation and its variants are still rarely commercially used for metal alloys – while they are state-of-the-art and indispensable for semiconductor applications. Especially the poor tribological properties of titanium alloys had hampered the increase of their technological applications [4], while PIII has been very effective in improving their tribological properties [5], and consequently in increasing their lifetime. PIII is especially promising because it allows the transference of laboratory processes to industrial applications. Three-dimensional characteristic is one of the reason for such possibility, i.e. the entire surface of parts with complex shapes can be treated without changing their masses [6]. Independent temperature control is another advantage, since simultaneous heating of the substrate is crucial for the treatment of Ti alloys to promote diffusion of the implanted ions [7] In this work, nanoindentation tests were employed to study the mechanical fatigue properties of titanium alloys (sintered Ti-Si-B and commercial Ti-6Al-4V) surface modified by HTPIII. The results after the plasma treatment are correlated with reciprocating wear tests and elemental depth profiles obtained by secondary ion mass spectroscopy (SIMS).

2. Experimental

High-purity elemental powders - Ti, Si and B - were used in this work to prepare four different powder mixtures: Ti-5.5Si-20.5B, Ti-7.5Si-22.5B, Ti-16Si-4B and Ti-18Si-6B. The milling process was carried out at room temperature in a Fritsch planetary ball mill under an argon atmosphere using rotary speed of 240-300 rpm, a ball-to-powder weight ratio of 10:1 and stainless steel vials (225 ml) and balls. The mechanically alloyed powders were uniaxially pressed for 2 minutes at 110 MPa and then isostatically pressed for 1 minute at 300 MPa, both operations being performed at room temperature. Subsequently, these green compacts were hot-pressed with 30 MPa and 1100oC for 20 minutes into a graphite die under argon atmosphere. Before HTPIII treatment, the samples were grounded on SiC papers and polished with colloidal silica suspension. Additional Ti-6Al-4V sample discs of 15-mm diameter and 3-mm thickness were also grounded, polished and cleaned in ultrasound acetone bath. For the present experiments, each titanium alloy sample was connected to a tungsten wire that in turn was fixed on a stainless steel rod playing the role of the discharge anode. This assembly was positively polarized to + 700 VDC in relation to the grounded chamber wall and simultaneously by negative HV pulses of -7 kV (length of 30 □s and repetition rate of 400 Hz). This process was operated in vacuum environment in nitrogen atmosphere at a working pressure of 10-3 Torr for 60 min. A thermionic oxide cathode generates primary electrons to ignite the glow discharge and to heat the substrate up to 800oC. The implantation of nitrogen ions takes place when the high negative voltage pulses are applied. The nitrogen depth profiles implanted into Ti alloys were measured using time-of-flight secondary ion mass spectrometry (ToF-SIMS). Tribological evaluations of the sample surfaces were conducted with a CSM tribometer with measurements of the dry friction coefficient accomplished in an oscillating ball-on-disk tribometer. The following parameters were used: load of 1 N with a 4.76-mm diameter alumina ball as a counterpart material, maximum speed of 10 cm/s and total track length of 2 mm. The afflicted surfaces after the wear test were examined using a scanning electron microscope employing the

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secondary electron detector mode to find out more information about the wear mechanisms. Roughness and surface profiles after wear test were measured with an optical profilometer. Additionally, dynamic nanoindentation was employed for surface characterization with conventional nanohardness being measured using a quasi-continuous stiffness measurement (QCSM). The employed indenter was a Berkovich three-sided pyramidal diamond. An average of 10 single measurements was used to determine the average hardness. Furthermore, the nanofatigue tests were conducted in the same nanoindentation equipment, however now with a spherical diamond indenter (tip radius of 10 µm). The samples were subjected to cyclic contact tests by repeatedly indenting the same area at maximum loads of 100-750 mN with minimum loads of 1.0-7.5 mN, respectively. A larger number of cycles were performed at the same position by repeating this 300-cycle experiment several times. Using the same spherical indenter, nanowear experiments were performed with loads of 10, 50 and 100 mN to make tracks with 80-µm length.

3. Results and Discussions

For the Ti-6Al-4V sample after HTPIII the SIMS experiments identify a nitrogen-rich layer with a thickness of about 1 µm. The untreated samples show a barely visible nitrogen signal below the surface oxide, indicating a large nitrogen uptake and retention during the HTPIII process. The roughness (Ra) is always increased after nitrogen implantation: for the commercial Ti alloy from 40 nm to up to 220 nm, as verified by optical profilometry. The sintered Ti-16Si-4B alloys present higher roughness in untreated condition (around 110 nm) and the HTPIII further increases this value to around 160 nm. Reducing the lengthscale of the experiments, nanowear experiments with a diamond tip also confirm that the HTPIII treatment significantly increased the wear resistance of the Ti-6Al-4V surface. Thus, a combination of resistance to plastic deformation (i.e. higher hardness) and resilience (increased wear resistance) can be inferred for both HTPIII treatments and Ti-Si-B alloys.The nitrogen implantation increased the hardness of the Ti-6Al-4V alloy three times, which is evidence of the presence of a titanium nitride phase in this surface region. However, the Ti-Si-B alloys had almost no increase of such property as the original hardness is already similar to the hardness of the nitrided Ti-6Al-4V alloy. It is concluded the process of nitrogen plasma immersion ion implantation at high temperature generates a surface layer rich in nitrogen that reduces the coefficient of friction and wear rate of a conventional Ti-6Al-4V alloy. This treatment also increases hardness, whilst it decreases the fatigue resistance due to surface embrittlement. In sintered Ti-Si-B alloys has a much smaller effect as a lower nitrogen amount is retained than in the commercial Ti-6Al-4V alloy, since in such alloy there are phases of high hardness and oxidation resistance, making difficult the implantation of the nitrogen at similar temperatures. These new alloys already present high hardness and wear resistance, but they also show low fatigue resistance, even without nitrogen addition at the surface. The results indicated that both HTPIII treated alloys showed improvements.

4. References

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