IMMERSION ION IMPLANTATION ON SS304

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1. Introduction

In practical engineering applications, many failures of parts begin frequently from their surface through wear, fatigue, corrosion, and so on. Therefore, durability against damages of components can be improved by strengthening their surface region 0. Failure as thread galling commonly happens in oil country tubular goods (OCTG) connections. Galling has also been observed in products and tools of sheet metal forming processes, as well as in some medical, aerospace and aeronautical parts 0]. Friction between local contact points produces cold welding because of the severe plastic deformation as well as the local high temperature 0. Galling is a severe adhesive wear that occurs under conditions of non- or poor lubrication and high contact pressure when relative motion exists between the contacting surfaces 00. In extreme cases, galling can be accompanied by cold welding of the mated parts 0. Galling occurred on Ti-6A1-4V/ SS304 and SS304/SS304 couples contacts after vibration tests of the CBERS-3, when no additional surface treatment was applied to such contacts. For this reason, an N-PIII was carried out and this problem was successfully solved. Other methods were previously employed – *e.g.* heat treatments and plasma nitriding – without success to solve the galling problem. The present study aims to describe how PIII affects the wear behavior of SS304, but also presents a detailed study on the formed phases produced by this technique.

2. Experimental

The SS304 samples used in this work has the following composition: 19.00 Cr, 11.00 Ni, 0.03 C, 1.50 Mn, 2.00 Si and 66.47 Fe (wt.%). For PIII treatment, the samples were polished using silicon carbide emery papers starting from 300 to 1200 grade and finished up using alumina powder of 0.25 µm. The samples were ultrasonically cleaned before loading into the PIII apparatus. The experimental set-up for the present N-PIII processing of SS304 is discussed in more detail elsewhere 0. The substrate was negatively biased to a voltage of 12 kV, and the implantation was carried out with pulses of 50 µs at a frequency of 300 Hz, for 180 and 270 minutes. The sample temperatures were kept around 350°C according a Raytec infrared pyrometer. The current density presented values around 7 mA/cm² during the high voltage pulses. Samples were characterized by X-ray diffraction (XRD) using a Philips PW1830 diffractometer with CuKa radiation and a high-resolution Philips X'Pert MRD X-ray diffractometer in the Bragg-Brentano Grazing Incidence (GIXRD) mode. The pristine samples were etched with acqua regia reagent and analyzed by scanning electron microscope for grain size measurement. Surface morphology was verified by atomic force microscopy (AFM) operating a Shimadzu SPM-9500J3 in the dynamic mode. Auger electron spectroscopy (AES) depth profiling was used in order to obtain the depth dependence of the chemical composition from the surface region. AES profiles were obtained by using argon ions for sputter etching in a Microlab 310-F spectrometer. The dry wear behavior of the SS304 was studied by means of a CMS Tribometer in ball-ondisk mode, where a tungsten carbide (WC) ball of 3-mm diameter was scratched against the SS304 samples surface with velocity of approximately 10 m/s at a constant load of 2 N. Surface hardness and modulus were measured using a MTS nanoindenter. The SS samples used in these analyses have 15-mm diameter per 2mm thickness. Pin-pull components of the camera were treated together with the mentioned samples and submitted to qualification procedures, which include vibration tests using frequencies from 20 to 2000 Hz and functional tests.

3. Results and Discussion

SS304 samples have the average grain size of ASTM 4.5 and after PIII treatments they exhibited a slight changing in their apparent color. The untreated alloy exhibits the fcc γ -austenite phase and the bcc α -martensite phase (only at surface areas). On the other hand, PIII treatment yielded a nitrogen-rich expanded austenite (γ_N) phase at surface, which is very similar with N-PIII for 4.5 hours at different conditions 0. The CrN phase was not identified even in a low omega angle of 1°. After the plasma treatment, roughness

increased from 1.8 nm to 5.5 nm (measured area of 1 μ m²) and from 3.2 nm to 8.1 nm (measured area of 25 μ m²). The first atomic layers from the surface were slightly contaminated by species such as oxygen and carbon. These elements were confined to less than 25 nm while the most important element present in the subsurface of treated layer is nitrogen. The maximum nitrogen depth was about 700 nm, and a maximum concentration of 30 at.% could be achieved at a depth of about 20 nm. The samples treated for 3h showed better resistance (volume loss of 144 mm³) than the sample treated for 4.5h (176 mm³) and the untreated one (231 mm³).

Assembled CBERS-3 imaging camera was submitted to extensive testing in a simulated space environment performing the qualification tests similar to that described by Tosetto et al. 0. The vibration test was carried out with a random frequency (20-2000 Hz) for 2 minutes per axis (xyz), where the rigid fixation components without the layer produced by PIII suffered galling. After the PIII treatment with nitrogen, these parts surpassed the aforementioned tests, because a perfect functioning of the remotely controlled camera focusing mechanism could occur afterwards. The crucial reason for this success is the suppressing of galling in such components. These components compose a part of an anti-vibration stopper that avoids any damage on the focusing system of the camera during the severe stage of launching. It must be pointed out that even such shallow nitrided layer can be adopted to improve dramatically the metallic surfaces on some applications with successful results. N-PIII treatment on SS304 using 12-keV energy was successfully used to implant nitrogen to a depth of approximately 700 nm. XRD results revealed the existence of expanded austenite in the SS304 surface subjected to such treatment. Hardness and wear resistance of the treated SS304 has been found to increase significantly after such implantation. Corrosion resistance was maintained, which can be corroborated by the very low content of CrN. Even this relatively shallow layer avoided galling on components of CBERS-3 camera during simulation tests of pin-pull elements, which are actuated remotely (as in the real time satellite operation in space). It is noteworthy that a relative simple configuration was used to produce such protective layer, where only nitrogen gas was implanted rather than using gas mixtures or deposition schemes. This is a new option to solve the galling problem in extremely sensitive components as the ones used in space or medical grounds, besides other solutions such as wet lubrication, Zn-Ni coatings or the changing of materials. The little modification of the dimensions of the parts is another important advantage of the PIII method used here, specially for applications in space components.

4. References

[1] S. K. Ghosh, P. K. Limaye, B. P. Swain, N. L. Soni, R. G. Agrawal, R. O. Dusane, A. K. Grover, Surf. Coat. Techn. 201 (2007) 4609-4618.

[2] Y. Guangjie, Y. Zhenqiang, W. Qinghua, T. Zhentong, Eng. Failure Analysis, 13 (2006) 1275-1284.

[3] S. R. Hummel, Tribology Int., 41 (2008) 175-180.

[4] K. Miyoshi, J. H. Sanders, C. H. Hager Jr., J. S., Zabinski, R. L. V. Wal, R. Andrews, K. W. Street Jr., B. A. Lerch, P. B. Abel, Tribology Int., 41 (2008) 24-33.

[5] K. Gurumoorthy, M. Kamaraj, K. P. Rao, S. Venugopal, Mater. Design, 28 (2007) 987-992.

[6] M. P. Pereira, W. Yan, B. F. Rolfe, Wear 268 (2010) 1275-1284.

[7] M. Ueda, L. A. Berni, J. O. Rossi, J. J. Barroso, G. F. Gomes, A. F. Beloto, E. Abramof, Surf. Coat. Technol. 136 (2001) 28-31.

[8] C. B. Mello, M. Ueda, C. M. Lepienski, H. Reuther, Appl. Surf. Sci., 256 (2009) 1461-1465.

[9] I. Tosetto, R. Araujo, C. Gonçalves, J. A. Rodrigues, Microwave and Optoelectronics Conference, 1 (2003) 513-517.

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