Surface Nanocrystallization of Ti-6Al-4V Alloy: Microstructural and Mechanical Characterization

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In this study, microstructural and mechanical properties of Ti-6Al-4V alloy, before and after the SMA treatment (SMAT) as well as the duplex SMAT/Nitriding process at different treatment conditions, were investigated in order to deepen the knowledge of these properties for biomedical devices. For that purpose, tribological (wear resistance, coefficient of friction) and mechanical (Vickers micro-hardness) tests were performed. To carry out the microstructural and surface topographical characterization of the samples, the scanning electron microscopy (SEM) and the 3D-SEM reconstruction from stereoscopic images have been used. By means of profiles deduced from the 3D images, the surface roughness has been calculated. The obtained results allowed to find an interesting SMAT condition which, followed by nitriding at low temperature, can greatly improve tribological and mechanical properties of Ti-6Al-4V alloy. It was also shown from SEM characterization and the original method of 3D-SEM reconstruction, that SMAT can reduce the machined grooves and consequently the roughness of the samples decreases. Moreover, we demonstrated, for the first time, that instead of usual etching method, the ionic polishing allowed to reveal the grains, the grain boundaries and the twins as well as the surface nanocrystalline layer generated by SMAT. Thus, the thickness of the SMATed layer decreases with the nitriding temperature, whereas the surface grain size increases.

Keywords: Nanocrystalline Materials, Ti-6Al-4V Alloy, Surface Nanocrystallization, Surface Mechanical Attrition Treatment, Nitriding, Microstructure, Mechanical Properties.

1. INTRODUCTION

The Ti-6Al-4V alloy is intensively applied in the manufacturing of biomedical devices (used as implant material in dentistry and orthopedics), due to its well-established biocompatibility, corrosion resistance and mechanical behaviour.¹ Indeed, for most materials such as Ti-6Al-4V alloy, their failure occurs at their surface; thus, optimization of the surface microstructure and properties may effectively enhance the global behaviour and particularly the service lifetime of materials. In recent years, with the emergence of nanocrystalline materials, new ways to improve the surface nanocrystallization or mechanical properties of materials have been developed. In this context, it was shown that the process of SMAT (Surface Mechanical Attrition Treatment) is a technique quite innovative, which enhances the mechanical and surface properties of a material without altering its chemical composition.²–¹¹ On the other hand, nitriding process which is widely used to form surface nitrides, is of great industrial interest as it forms a unique composite structure with a hard surface and a tough interior, so that the global mechanical performance and wear/corrosion resistance of alloys and steels are greatly improved.¹²,¹³ These two surface engineering techniques are quite innovative. Indeed, it was recently shown that surface nanocrystallization greatly facilitates the nitriding process (by reducing treatment temperatures and enhancing nitriding processes) and provides an alternative approach to the surface modification of metallic materials.¹⁴ Due to this excellent enhancement, surface engineers and researchers wish to better understand the SMAT and/or the duplex SMAT/nitriding process effects on metallic materials widely used in many industrial and biomedical applications, by varying the treatment conditions in order to optimize these surface engineering techniques.
Great attention has been given to the possibility of using SMAT to produce bulk nanocrystalline materials, and nitriding process to modify the surface of many metallic materials. Actually in our knowledge, few studies concern the use of the duplex SMAT/nitriding process on Ti-6Al-4V alloy in order to enhance its global properties. Although studied separately, no systematic comparison between SMAT and nitriding process, in terms of their effects on materials microstructural and mechanical properties, has been reported.

The aim of this study was to investigate the microstructural and mechanical properties of Ti-6Al-4V alloy, before and after the SMA treatment as well as the duplex SMAT/Nitriding process, in order to deepen the knowledge of these properties for biomedical devices.

2. EXPERIMENTAL DETAILS

The samples of Ti-6Al-4V in form of disc (flat specimen) were subjected to different SMAT conditions (noticed SMAT1, SMAT2 and SMAT3) in order to achieve ultrafine-grained surface layers. The details of the SMAT procedure were previously described in the literature.\(^{15}\) The main parameters of the SMAT process used are: the sonotrode magnitude, the shot diameter and the treatment time (confidential parameters).

Plasma nitriding was performed in BMI vacuum furnace at temperatures of 375 and 730 °C.

Tribological tests were carried out to appreciate the wear behaviour of the various treated samples and consequently to retain the best condition of nanostructures generation. Vickers hardness variation as a function of the load (from 0.3 kg to 10 kg) was measured on the sample top surface.

Fig. 1. Evolution of the mass loss of samples as function of the different treatments applied.

Fig. 2. SEM micrographs of the top surfaces of the untreated Ti-6Al-4V specimens (a) and those subjected to SMAT1 (b), duplex SMAT1/nitriding at 375 °C (c) and duplex SMAT1/nitriding at 730 °C (d).
For each measurement, five repetitions were carried out in order to get the mean value.

To investigate the microstructural properties of Ti-6Al-4V alloy samples subjected to various SMAT and/or nitriding treatments, Scanning electron microscopy (SEM) observations were performed using a JEOL 5400 LV microscope operating at 15 kV. The samples were embedded in an electrically conducting cold-setting resin and then mirror polished by standard mechanically metallographic techniques followed by ions polishing with an ion-beam milling system (RES 101, Leica Mikrosysteme GmbH, Hernalser Hauptstrasse, Vienna). Typical plane view SEM observations of the top treated surfaces and the cross-sectional SEM observations were performed.

Three-dimensional (3D) reconstruction was carried out using stereoscopic images obtained by tilting the sample holder. Next, commercial software (MeX, Alicona, France) was used to give a Digital Elevation Model (DEM) of the surface specimen from which 3D-SEM images and roughness profiles were extracted. For each DEM, several measurements were carried out in order to get a mean value of the rough-mean-square surface roughness.

3. RESULTS AND DISCUSSION

The wear resistance using evolution of the mass loss of samples before and after SMAT and/or nitriding, at various treatment conditions, is shown in Figure 1. One can notice that for the SMAT without nitriding, the mass loss of the samples is less important for SMAT1 compared to the other SMAT conditions for which mass loss remains higher than that of the untreated material, taken as a reference.

Fig. 3. 3D-SEM micrographs of the surface topography of the treated Ti-6Al-4V specimens (first raw) together with the profiles (second raw) deduced from these micrographs. Samples subjected to SMAT1 (a1 and a2), duplex SMAT1/Nitriding at 375 °C (b1 and b2), duplex SMAT1/Nitriding at 750 °C (c1 and c2).
reference. For the corresponding SMAT treatments followed by nitriding at 375 and 730 °C, one observes that only the duplex SMAT1/nitriding at 375 °C presents the lower mass loss compared to all the other treatment conditions. It was also shown that at this SMAT condition, the coefficient of friction of the samples was slightly reduced in comparison to the other treatment conditions (results not shown). From these results, it appears that SMAT1 treatment condition followed by nitriding at 375 °C improved more significantly the resistance to wear and the coefficient of friction of the Ti-6Al-4V alloy samples; consequently this condition was chosen for structural and mechanical characterization in order to better understand its influence on these properties.

Figure 2 shows the micrographs of the samples surface subjected to SMAT1, and the duplex SMAT1/Nitriding at 375 °C and 750 °C compared to the untreated sample. A drastic change in the microstructural morphology of the top surface sample after treatments was clearly noticed. This revealed that the substrates surface topography varies with the treatment conditions. Indeed, the micrograph obtained for the samples subjected to SMAT1 followed by nitriding at 375 °C, shows that the structure of the top treated surface is homogeneous and regular compared to the images of the samples subjected to SMAT1 and SMAT1/nitriding at 730 °C, where microstructural morphology is not quite homogeneous.

In order to have full information on the surface topography of the treated samples, 3D-SEM micrographs have been reconstructed.

Figure 3 shows the 3D-SEM micrographs of the surface topography obtained for flat specimen substrates subjected to SMAT1, and the duplex SMAT1/Nitriding process at 375 °C and 750 °C together with the profiles deduced from these micrographs. The surface roughness Ra variation obtained from these profiles is presented in Figure 4. It can be noted initially that the surface roughness of the substrate decreases after SMAT treatment, and this decrease becomes more pronounced when SMAT treatment was followed by nitriding at 375 °C; while, for the nitrided samples without SMAT, the surface roughness increases with the nitriding temperature. This result is in agreement with the observations of Chen and Jaung\(^{16}\) which highlighted a significant increase of the surface roughness of the Ti-6Al-4V alloy nitrified without SMAT, when the nitriding temperature is increased from 500 to 900 °C. However, in the case of the SMATed samples, the surface roughness increases when the nitriding temperature reaches 730 °C.

In order to highlight the nanostructured layer generated by SMAT treatment and the global microstructural characteristics of the specimen after treatment, typical cross-sectional SEM observations were performed. Figure 5 shows the cross-sectional SEM micrographs of the Ti-6Al-4V samples subjected to SMAT1 and the duplex SMAT1/nitriding at 375 °C and 730 °C. It appears that ionic polishing allowed to reveal the grains, the twins and the grain boundaries as well as the surface nanocrystalline layer. One may mainly observe that the SMATed layer thickness decreases with the nitriding temperature, whereas the surface grains size increases. Indeed, the thickness of the surface nanocrystalline layer is approximately 70 µm for the SMATed samples, 15 µm for the duplex SMAT/nitriding process at 375 °C, and practically less than 1 µm at 730 °C. These observations corroborate those of Avelar-Batista and al\(^{17}\) which showed that nitriding at 750 and 800 °C of Ti-6Al-4V alloy involves an increase in the grain size. Moreover, the microstructural evolution along the depth of the SMATed sample (Fig. 5(a)) indicated that much larger grains were observed with a further increase in the depth, evidencing the substrate grains refinement induced by surface mechanical attrition treatment. This is in agreement with previous results obtained with SMAT treatment on metallic materials.\(^{5}\)

From these results, we may state that the SMAT1 treatment followed by nitriding process at 375 °C presents an interesting result with a lower surface roughness obtained in comparison to all the other treatment conditions. It appeared clearly that SMAT treatment at this condition can reduce the machined grooves and consequently the roughness of the samples decreases.

Figure 6 shows the variations of the Vickers microhardness with the load at the top surface of the samples subjected to SMAT1, and the duplex SMAT1/Nitriding at 375 °C and 750 °C compared to the untreated samples. Whatever the load applied, an increase of the
microhardness of the samples after SMAT1 treatment can be observed; this increase is more pronounced when the nitriding temperature increases from 375 °C to 750 °C. Indeed, Avelar-Batista and al17 highlighted an increase in the hardness of Ti-6Al-4V alloy nitried without SMAT, when the nitriding temperature was increased from 400 to 800 °C. It is also noticed that when the nitriding temperature is increased to 730 °C, the microhardness of the samples decreases with the load. This could be related to the increase in the grains size of the surface nanocrystalline layer during the nitriding highlighted by the cross-sectional SEM observations.

To summarize, the surface treatment of Ti-6Al-4V alloy by means of SMAT1 followed by nitriding at low temperature (375 °C) appears to be the best treatment which may lead to improvement of the studied properties. Further work based on rotating bending fatigue tests and nanoscale characterization by high resolution transmission electron microscopy (HR-TEM) are currently in progress with a view of more optimizing the SMAT conditions.

4. CONCLUSIONS

The microstructural and mechanical properties of Ti-6Al-4V alloy, before and after the SMA treatment as well as the duplex SMAT/Nitriding process at different treatment conditions, were investigated in order to deepen the knowledge of these properties for biomedical devices. This study allowed us to optimize the SMAT condition. Indeed, we have shown that plasma nitriding at low temperature (375 °C) after surface nanocrystallization (SMAT) leads to an important improvement of the wear resistance and the coefficient of friction. It was also shown at this optimized treatment condition that the Vickers microhardness of the treated samples was improved. In addition, we highlighted, from SEM observations, that SMAT can standardize the machined grooves and consequently reduce the roughness of the samples.

Finally, we demonstrated for the first time, that instead of usual etching method, the ionic polishing allowed to reveal the grains and the grain boundaries as well as the surface nanocrystalline layer and the twins. Thus, the SMATed nanocrystalline layer thickness decreases with the nitriding temperature, whereas the surface grain size increases.
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References and Notes

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