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Effect of laser shock peening on the high temperature oxidation resistance of titanium

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ABSTRACT

The effect of laser shock peening on the high temperature oxidation resistance of commercial pure titanium at high temperature (700 °C) was studied in long-time (3000 h) exposure under dry air. A reduction of the gain mass by a factor 4 was found for laser-shock peened (LSP) samples compared to untreated titanium, which supports the interest of laser-shock treatment for the improvement of high temperature resistance. Short-durations (10 h and 100 h) oxidation experiments, devoted to investigate the influence of the LSP treatment on the first stages of the oxidation process, were also carried out by TGA. Several techniques as scanning electron microscopy, hardness and roughness measurements, X-ray diffraction and X-ray photoelectron spectrometry, micro-Raman spectroscopy, nuclear reaction analysis and electron backscattered diffraction were used to characterize the sample after laser treatment and oxidations. The formation of a continuous nitrogen-rich layer between the oxide layer and the α-case area in LSP samples appears to be the key factor to explain the reduction of oxygen diffusion, and thus the improvement of the oxidation resistance of laser shocked titanium. Moreover, the grain-texture of LSP samples after oxidation can also explain the improvement of the high temperature oxidation resistance after long times exposures.

1. Introduction

Titanium alloys are widely used materials in the aerospace field due to their high mechanical resistance combined with low density and excellent resistance to corrosion [1]. Hence, titanium alloys are very attractive for compressor section components in gas turbine engines working at temperatures between 300 and 600 °C [2]. Beyond that temperature, a deterioration of the passivation layer which covers titanium components and then an acceleration of the oxidation rate becomes possible. Moreover, flaws and cracks initiated at the surface can lead to fatigue and failure of these components.

Many surface treatments have been tested to develop suitable oxidation-resistant coatings for titanium alloys. Most of the treatments used for oxidation protection are chemical processes as pack cementation coatings [6], ion implantation [7], and PVD ceramic coatings [8]. Another interesting way to improve the oxidation resistance, and also the fatigue performances of titanium alloys, can be a mechanical surface treatment. Indeed, large compressive stresses and surface hardening induced by mechanical treatments, such as shot-peening (SP) with WC balls, has been reported to play a positive role in the oxidation resistance of zirconium for instance [9–10]. A beneficial effect of SP treatments on the high fatigue performance of several titanium alloys has also been reported [11]. However, shot-peening can induce surface contamination due to the wear of the balls during the treatment [12]. In this context, laser surface treatments have also been proposed as a competitive alternative technology to improve fatigue, corrosion and wear resistance of metals [11,13–17]. The high pressure shock wave generated in the interaction of a nanosecond pulsed laser beam (with power density in the GW/cm^2 range) with the metal surface can produce deep compressive residual stresses with less cold work compared to SP [11]. Thus, the thermal relaxation of these residual stresses at high temperatures is lower after LSP treatments.

In the high temperature oxidation process of titanium, besides the rutile oxide layer formed on the surface, the inward diffusion of oxygen gives rise to the formation of a rich oxygen-content area, called α-case [3–4]. In the case of oxidation in air, the presence of atmospheric nitrogen together with oxygen reduces the oxidation rate of titanium as shown by Coddet and Chaze [5]. Nitrogen is progressively incorporated in the rutile layer which grows from the beginning of the oxidation process. The higher diffusion coefficient of nitrogen in rutile compared to that of oxygen, leads to a progressive accumulation of nitrogen near

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the metal-oxide interface, its dissolution in the metal being slower than that of oxygen [5]. This nitride layer slows down the oxygen diffusion and then brings a protection in terms of oxidation.

The aim of this work is to study the effect of laser shock peening on the high temperature oxidation resistance of commercially pure titanium. A comparison between treated (LSP) and untreated samples (US) was led in terms of oxidation resistance at 700 °C under dry air. Long-time (3000 h) oxidation experiments under atmospheric air were done by furnace exposure, whereas Thermo-Gravimetric Analysis (TGA) under synthetic air was used for short-time (10 h and 100 h) oxidation experiments devoted to investigate the influence of LSP treatment on the first stages of the oxidation process. Surface and cross-section characterizations by different techniques (X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) coupled with Energy Dispersive Spectroscopy (SEM/EDS), micro-Raman spectroscopy, Nuclear Reaction Analysis (NRA), Electron Backscattered Diffraction (EBSD), X-ray Photoelectron Spectrometry (XPS), micro-hardness and roughness measurements were mainly used to investigate the composition and the microstructure of untreated and LSP treated samples before and after the oxidation experiments. After the discussion of the results obtained by all these techniques, we will propose a model to explain the observed improvement of the oxidation resistance of titanium by LSP treatments.

2. Experimental details

2.1. Material

The material used in this study was a commercially pure titanium, 1 mm thick plate (grade I, certified purity 99.6%, Goodfellow). The material was manufactured by cold-rolling and annealing. The actual composition of the metal was measured by Inductively Coupled Plasma Mass Spectrometry. The measured composition (in wt%) is the following: Ti bal., Fe 0.06, O 0.05, Co 0.03, Ni 0.023, Cr 0.018, C 0.011, Sn 0.01, Mo 0.01, Si 0.01, Al 0.005, Zr 0.005, Mn 0.005, Cu 0.002. The size of the samples used for laser treatments was 25x50x1 mm^3. For TGA purposes, the samples are cut in small pieces (8x10x1 mm^3). For simplicity purposes, the untreated samples are named US (untreated sample) while the laser-shock peened samples are called LSP in the following sections.

2.2. Laser shock peening (LSP) treatments

LSP treatments were done with a GAIA HP laser source operating at a 532 nm wavelength. All treatments presented in this study were done by using a focused beam with a typical 3 mm spot diameter. The laser shot frequency was 0.5 Hz and the pulse duration was 7 ns. The laser irradiance was 9.1 GW/cm^2. The laser scanned the sample surface with a velocity of 1.1 mm/s. This leads to an overlapping area of 30% between two successive impacts. LSP treatments were performed on both sample faces. The laser path is snake-shaped by describing successive displacements along parallel lines going and coming. The sample is covered during the laser shock by an aluminium adhesive coating film in order to prevent the oxidation during the shot. For the laser irradiance values used here, each impact generates a plasma plume over the target which is confined near to the surface of the sample by both the confinement media. Here, water was used as confinement media to increase the shock amplitude. The use of a laser wavelength at 532 nm reduced laser beam absorption in the confinement media for increasing the LSP treatment effect [18]. When the plasma blows off, a back shock-wave is generated into the target. Then a large residual compressive stress with small amount of work hardening is obtained [13].

2.3. Mass gain measurements

The oxidation rates of US and LSP samples at 700 °C in dry air were studied in two different time exposure ranges.

- Long-time (3000 h) oxidation experiments were carried out within a SETNAG furnace at 700 °C under atmospheric air. The samples were extracted and weighed regularly.
- Short-time (10 h and 100 h) oxidation experiments were performed by isothermal exposure in thermo-gravimetric analyzer (TGA) using a SETARAM SETSYS EVOLUTION 1750. A heating rate of 10 °C/min was used from ambient to 690 °C, then the heating rate was reduced to 1 °C/min up to 700 °C.

2.4. Characterization techniques

The cross sections of the samples were resin-coated and mirror-polished. Cross-sections were observed by SEM coupled with an EDS microprobe analyzer (TESCAN VEGA 3) for determining the thickness of the oxide scale and the diffusion layer (named α-case) size.

The surface roughness was measured using a surface roughness meter VEECO WYCO NT 9100. Micro-hardness measurements were made before and after LSP treatments using a ZWICK/ROEL indenter (Vickers's diamond pyramid) with a load of 50 gf. For quick alpha-case measurements purposes we used micro-hardness techniques in cross-section profile using 25 gf load, applied for 10 s.

X-ray photoelectron spectrometry (XPS) was used to investigate the possible surface-contamination of samples after LSP treatments. A PHI Versaprobe 5000 working with 41.6 W AlKα_1 radiation (1486.6 eV) was used. The surface was cleaned before analysis by Ar ion beam bombardment for 1 min at 5 keV.

XRD and Raman spectroscopy were used to study the micro-phases formed in the oxidation layer. XRD patterns were obtained with a Bruker D8-A25 DISCOVER using Cu-Kα radiations at grazing incidence ($\theta_{inc} = 2°$). Raman spectra were obtained with an InVia Renishaw set-up working in backscattering configuration. The wavelength was 532 nm and the excitation power was relatively low (about 0.5 mW) to avoid heating the samples. Both sample surfaces and cross-sections were studied in this way.

The elemental composition of the samples was studied by ion beam analysis. In particular, the content and the distribution of light elements were analyzed by nuclear reaction analysis (NRA), which allows quantitative analysis of nitrogen and oxygen without influence of the chemical environment and a low influence of the roughness. This method has very good accuracy and is not sensible to the surface contamination during polishing since the measurements can be made under the sample's surface. The method used to analyze NRA spectra is extensively described in [19]. Here, a deuteron beam with a kinetic energy of 1900 keV was used to detect reactions involving nitrogen atoms, mainly the $^{14}N(d,\alpha_1)^{12}C$ nuclear reaction [20]. Cross-section maps of nitrogen distribution in oxidized samples were obtained in this way.

Grain orientation was measured by Electron Backscattered Diffraction (TSL EDAX OIM X4M EBSD system) coupled with a field-emission scanning microscope (FE-SEM, JEOL JSM-7600F). The working distance was 20 mm, the tension 20 keV, magnification ×70 and a step of 1 μm.

3. Results

3.1. Characterization of as-prepared (non-oxidized) US and LSP samples

Cross-section images, 3D–surface profilometry and micro-hardness profiles of US and LSP samples are shown in Fig. 1.

SEM images (Fig. 1.a and 1.b) show grains morphology and size. In US samples the grains size is about 40 μm. In LSP samples, the effect of the laser treatment is shown through the whole sample depth. The high density of twins induced by the LSP treatment prevents the observation of the grains shape and size.

3D–surface profiles (Fig. 1.c and 1.d) show the different surface roughness of US and LSP samples. It was found that both R_a and R_t values, respectively 0.4 and 8.3 μm, increase due to LSP treatment up