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# A comparative study of surface modification of TIMETAL 834 in ambient and argon atmosphere by pulse Excimer laser

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#### Abstract

Surface modification of Ti alloy by pulse excimer laser is gaining importance as it yields a beneficial effect on material performance, longevity and safety level. A pulse Excimer laser is one of the ideal source in surface modification technique as it delivers higher energy at the metallic samples and can achieve a cooling rate of  $10^8$ - $10^{10}$  K/s. In the present study, the TIMETAL 834 surface is irradiated by the Excimer laser in two different environments, argon or ambient at pulse energy 5.4 Jcm<sup>2</sup> for a single pulse. The optical microscopy was used for the microstructural characterization, whereas fretting wear test was undertaken for tribological behavior. The observation indicates that the initial microstructure ( $\alpha$  and  $\beta$  phase) getting refined after laser treatment, and the surface achieved microstructural and compositional homogeneity. The surface ripples were found in fine as well as a coarse scale for air (ambient) and argon processed sample, respectively. The surface craters are also observed in localized regions and its density was higher for argon processed sample as compared to the sample treated in Air. The average friction coefficient for argon treated sample remained unchanged, whereas a marginal decrement was observed in air processed sample compared to untreated one, indicating the substantial improvement in wear resistance properties after laser treatment.

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# Introduction

Nowadays, Titanium and its alloys are gaining more importance in many engineering fields like aerospace, automotive, medical and nuclear power plant due to its light weight, high specific strength, effective toughness and corrosion resistance<sup>1-3</sup>. However, the thermal conductivity of Ti alloy is very low which makes its tribological property fairly poor <sup>4-5</sup>. Since, the wear behavior is the surface dependent phenomena, by proper surface modification, the wear resistance properties of Ti alloy can be improved. Many methods has been attempted by several researchers to enhance the surface properties, like thin film deposition, anodizing, electrodeposition etc. which could result with the adhesion problem, film degradation and high porosity. Likewise, many conventional techniques are very expensive, time consuming and are bound by equilibrium limitations. The advantage of using laser over other techniques is, it is a fast and easy process, cost effective, non-equilibrium route and can achieve extremely fast cooling rate  $10^8$ - $10^{10}$  K/s <sup>6-7</sup>, which leads to the surface refinement, solid solution extension, microstructural homogenization and metastable phase formation <sup>6, 8-9</sup>. A few literatures are available on the surface modification of Ti alloy by laser technique. The correlation between the microstructural changes associated with the processing environment and its effect on tribological properties is still lacking and needed to be addressed further.

In this present study, the Ti alloy (TIMETAL 834) is exposed to Excimer laser for 1 pulse in argon or ambient atmosphere and the associated microstructural change is evaluated to establish its correlation with the wear properties changes.

# Experimental

#### Materials

The composition of TIMETAL 834 used in the study was found to be Ti - 5.72 %Al - 3.97 %Sn - 3.82 %Zr - 0.69 %Nb - 0.57 %Mo - 0.36 %Si (values are in wt. %). It is a near  $\alpha$ -Ti alloy with low  $\beta$ -stabilizer. The samples of dimension ~ 1 cm × 1 cm × 0.25 cm were cut by the diamond saw cutter (Buehlers make Labcut 1010) operating at a speed of ~ 100 rpm from the metal bulk. They were initially ground and polished with the emery sheet. Final polishing was carried out by 1  $\mu$ m alumina slurry to obtain a scratch free and mirror finished surface. All samples were ultrasonicated in acetone before the laser exposure.

#### Laser surface treatment

The Excimer laser equipment of Coherent GmbH make model -Micro LAS COMPex Pro 205 was used in this study. A laser with pulse width ( $\tau$ ) 20 ns and wavelength ( $\lambda$ ) 248 nm was used. The laser treatment was done under the following processing parameter: pulse energy density (fluence) 5.4 J/cm<sup>2</sup> and pulse repetition frequency 1 Hz and surface is exposed for single laser pulse in argon or ambient atmosphere. The shrouding gas (atmosphere) argon used was 99.9 % pure. The gas pressure was maintained at ~ 400 kPa.

## Methods

The microstructural investigation was done by the optical microscope (Lieca LM 6000). The fretting wear test was carried out by using a ball on flat configuration with a counter body 6 mm diameter WC ball. The parameter used for the fretting test was: fretting cycles 1000, fretting frequency 5Hz and slip amplitude 100  $\mu$ m.

#### **Results and Discussion**

## Optical microscopy

Figure 1 depicts the microstructure of the initial (untreated) Ti alloy. It indicates the presence of  $\alpha$  and  $\beta$  grains. The morphology of  $\alpha$  grains is equiaxed whereas,  $\beta$  grains are found as lamella form. These  $\alpha$  grains are fairly coarse and found in the size range  $\sim$  25 - 45 µm.



Figure 1: Optical micrograph of untreated TIMETAL 834.

The laser treated sample is illustrated in the Fig. 2. The optical image appearance shows the surface microstructural changes occurred fairly differently in air and argon atmosphere treated sample. The coarse grain of  $\alpha$  and  $\beta$  is no more present in the treated sample. It is more likely to be a single phase structure due to rapid solidification effects associated with laser treatment. In addition, it shows the surface ripples and craters. The surface ripples are more frequently found in air treated sample, which contribute to the surface roughness. However, in case of argon treated sample, the coarse ripples are observed in fairly less density. The craters are found to be in the size range  $\sim 10-60 \ \mu m$ for argon treated sample, whereas for air treated sample it is  $\sim 30$ -75 µm. The average crater size for argon and air treated sample is found to be ~ 47 and 55  $\mu$ m, respectively. Comparing the morphology, the air treated samples shows circular rings inside the crater region whereas for argon black dimples are observed inside the crater. Also, the crater density is found to be higher  $(25/cm^2)$  in argon processed sample (Fig.3), whereas the crater size is higher for the air treated sample. These craters are formed due to the melt eruption from the melt pool during the laser exposure. A similar result was also found by Xu et al <sup>10</sup> for electron beam treatment of steel sample. The authors reported that the craters were formed more likely by the carbide particles, during the electron beam exposure. The carbide particles first melt and later erupted from the surface. In the present work, it is believed that the silicide particles, surface impurities, surface defect or  $\alpha/\beta$  boundaries may serve as the nucleation sites for the crater formation leading to the surface compositional homogeneity. The laser treated sample attained surface microstructural homogeneity in spite of the presence of craters and surface waves. The degree of homogeneity attained is higher for sample treated in air compare to argon. The ripple formation at the surface is based on the fact that when the laser beam is incident on the sample, an intense energy is transferred to the surface by its absorption. With this input energy, the surface melts and immediately re-solidifies with rapid cooling. Once the molten liquid surface is frozen off, the signature of molten liquid character in the form of metastable structure and ripples remains on the surface. It is also obvious that during the resolidification, it will not get enough time for nucleation and growth for crystallization. Consequently, it leads to the formation of amorphous structure at the surface.



Figure 2: Optical micrograph of laser treated sample; (a) in ambient condition, (b) in argon atmosphere.



Figure 3: Variation of crater size and density with the process atmosphere.

#### Tribological behaviour

Figure 4 shows the average friction coefficient of untreated and laser treated sample. The observation indicates that the average friction coefficient for untreated ( $\sim 0.51$ ) and argon atmosphere treated sample ( $\sim 0.50$ ) is almost similar, whereas, sample treated in ambient condition showed slightly lower coefficient of friction

(COF) (~ 0.45). The lattice disorder created by the laser treatment is expected to increase the hardness and hence contribute to lowering of the coefficient of friction <sup>11, 12</sup>. In contrast, the possible reason for the similarity in COF of untreated and argon atmosphere treated sample can be attributed to the formation of higher number of craters and surface undulations during laser treatment.



Figure 4: Variation of coefficient of friction with the process atmosphere.

## Conclusions

The conclusion from the present study are summarized below:

1. The initial  $\alpha$  and  $\beta$  coarse grains at the surface gets completely modified and form single phase structure. The surface ripple character and surface craters are observed.

- The average surface crater size is higher (~ 55 µm) for the air treated sample, whereas the crater density (25/cm<sup>2</sup>) is more for the argon treated sample.
- 3. The coefficient of friction for argon treated sample remains same as the untreated (0.5) whereas it decreases (0.45) for sample treated in ambient condition.

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