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Surface Modification of TIMETAL 834 by Excimer Pulse Laser

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ABSTRACT*:*Ti alloys become very popular in various engineering fields due to its light weight, high strength and good corrosion resistance properties. Nevertheless, its application is limited in many areas like: tribology, high temperature oxidation and aggressive corrosion fields due to its poor surface properties. Therefore, the proper surface treatment is required to increase the usageof Ti alloys in such engineering field. In this present study, the surface modification of the Ti alloy is done by the excimer pulse laser. The various parameters used for the laser treatment are: pulse energy 5.4 Jcm^{-2}, pulse repetition frequency 1 Hz, pulse width 20 ns, number of pulses 10 or 30, and atmosphere is ambient. The microstructural characterization is carried out by SEM, compositional analysis is done by the SEM/EDS and the tribological behavior is studied by the fretting wear test. It is observed that, the surface microstructure starts refining with increasing the number of pulse exposure.Initially,after 10 pulses, fine surface ripples prevailed, which became fairly broader later at 30 pulses. It is also found that in between 1 - 10 pulses, surface attained the compositional and microstructural homogeneity. A substantial decrease in average friction coefficient from 0.51 to 0.39 is observed for untreated to 30 pulses treated sample, indicating the significant enhancement in wear properties after laser processing.

KEYWORDS*:* TIMETAL 834, Excimer Laser, SEM, Microstructure, Fretting wear.

I. INTRODUCTION

Titanium and titanium based alloys are one of the most important material in the engineering field. The low density, high strength, and good corrosion resistance properties make it popular in various sectors like aerospace, aeronautical, defence, nuclear power plant, industries and sports applications [1-3].

Although, its mechanical, chemical and physical properties are excellent, it has limited use in many engineering applications like tribology, high temperature oxidation and corrosion in aggressive environment [3-6]. The tribology, corrosion and oxidation are the surface related phenomena which mainly depend on, surface chemistry, surface microstructure (grain size and its shape), morphology and size of different phases. Therefore, by altering the surface chemistry or/and surface microstructure one can achieve the desired surface properties, which is expected to improve its performance and longevity in such engineering fields.

Various attempts had been made by quite a few researcher to modify the tribological, corrosion and oxidation resistance properties of the Ti alloy by thin film coating, nitriding, electro deposition, ion beam, electron beam and continuous wave laser treatment. The treatments like thin film coating, nitriding and electro depositionare time consuming as well as expensive and effective within the equilibrium limit.This may result in the film degradation or pilling effect due to various regions. Likewise, the processes like ion beam, electron beam needs high vacuum and have issues of operational hazards.

In contrast, the material processing by excimer pulse laser has drawn more attention nowadays due to its high absorptivity on the metallic surface, ease of processing, non-equilibrium characteristicsdue to achievable cooling rate of 10^8-10^{10} K/s [7, 8]. The pulse laser processing is expected to give microstructural and compositional homogeneity, metastable phase formation and grain refinement, whichare likely to give better surface performance [7, 9, 10].

The present study examines the surface microstructural changes associated with10 or30 laser pulses irradiation of the sample surface. Also, an assessment of thevariation in tribological properties associated with the microstructural changes are carried out.

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II. MATERIALS AND METHODS

2.1 Material

The nominal composition of the Ti alloy (TIMETAL 834) in (wt. %) is: Ti - 5.72 %Al - 3.97 %Sn - 3.82 %Zr - 0.69 %Nb – 0.57 %Mo - 0.36 %Si. The samples in dimension ~ 10 mm × 10 mm ×2.5 mm were made from the plate. The standard metallography technique was used to prepare the sample.The samples cold mountedbefore mechanical polishing by different grade of emery sheet. Finally, cloth polishing was done with 1 µm alumina (Al_2O_3) slurry ata disc speed of 50 to 90 rpm to obtain a scratch free mirror finished surface. Afterwards, samples were ultrasonicated in acetone to remove the contamination, if any.

2.2 Laser surface treatment

The Coherent GmbH make model - Micro LAS COMPex Pro 205excimer pulse laser instrument was used in this study. The laser photon wavelength was 248 nm and pulse width chosen was 20 ns.Pulse repetition frequency 1 Hz andenergy density (fluence) 5.4 $Jcm^{-2}$was maintained for the laser treatment and surface wasexposed to 10 or 30 numbers of pulses. The all laser treatment was executed in ambient condition (T=27 °C) and (P=1atm).

2.3 *Processing method*

The schematic illustration of the experimental set-up of laser processing unit is shown in the Fig.1.Starting from the laser source (1), the laser beam gets reflected from the multiple reflectors (2, 3 and 5) and then it goes through the lens (6) and finally falls on the sample surface (7). The samples are held on the stage (8).The sample stage can move mechanically or electronically in x, y and z direction and the pyroelectric sensors (4) measure the laser pulse energy. The process controlling unit (9) monitoring the process stages. In all laser treatments the beam was at right angle to the sample surface. Also, an attenuator was placed outside the laser for beam energy adjustment.

2.4 Surface characterization

The samplesmicrostructure wascharacterized through SEM (model ZEISS EVO 50). The compositional analysis was done by the EDS attachment with SEM. The imaging mode used was SE and BSE, at accelerating voltage 20 kV. The Tribological behavior (fretting wear) assessment was carried out by a ball on flat configuration, with a 6 mm diameter tungsten carbide (WC) counter body. The fretting wear test was conducted for: 10000cycles,5 Hz frequency with 100 µmslip amplitude.All samples were tested in dry condition.

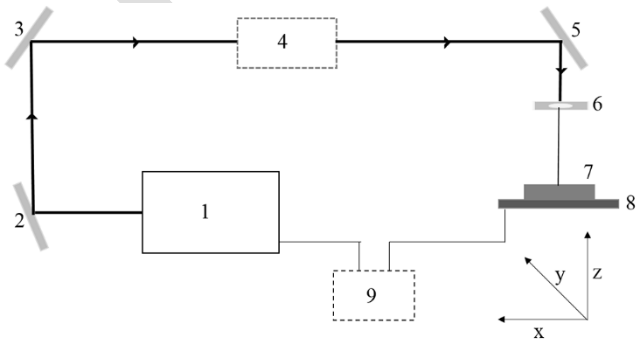

Fig.1. Schematic of excimer laser processing set-up.

III. RESULTS

3.1 Microstructure of Initial sample

Figure 2 illustrates the SEM microstructure of untreated sample. Initial sample shows the presence of dual phase (α and β) structure, the size of α grains is approximately ~ 25 - 45 µm with equiaxed morphology, whereas the β grains are present in lamellar form. The silicide particles can be seen decorating the grain boundaries.

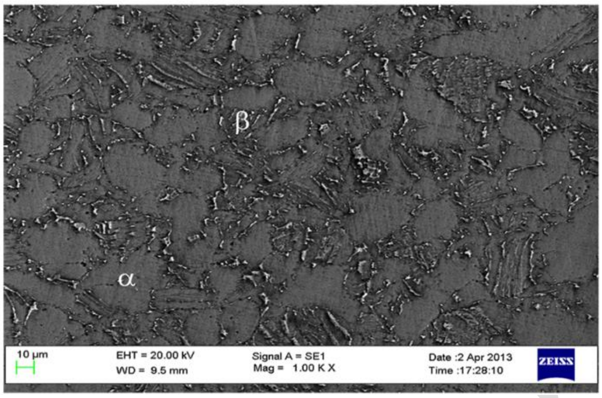

Fig. 2. SEM micrographs of untretaed and laser treated sample; (a) untreated;

3.2 Microstructure of Laser treated sample

Figure 3a-b depicts the SEM micrographs corresponding to 10 and 30 pulse laser treated samples, respectively. After 10 pulses (Fig. 3a) the surface microstructure is completely modified and no distinction is possible between the α and β grains on the surface. These surface waves (ripples) are found periodically from the edge to the central area in the treated region. The width of these ripples is found to be approximately ~ 11 µm. However, these periodic ripples become broader ~ 15 µm after 30 pulses. Also, by comparing the SE and BSE images of 10 pulse (Fig. 4) treated sample, it is observed that, there is no contrast changes thought out the treated surface (Fig.4b), which suggested that the 10 pulses treated samples are compositionally homogenized. Therefore, it is expected that, the higher number of pulse treated samples show similar results as is seen from the Fig.3b (after 30 pulses).

3.3 SEM-EDS analysis of 10 pulse Laser treated sample

To verify the compositional homogeneity, the x-ray EDS analysis is carried out from the hill and valley regions (area 2 and 3) of 10 pulses treated sample (Fig.4 (a)) and the elemental distribution data is presented in the Table 1. The observation shows a marginal difference in composition of the hill and valley regions. The composition difference (in wt. %) of these two regions is found to be ~ 0.92, 0.76, 0.06, 0.28, 0.06 for Ti, Al, Sn, Zr and Si, respectively, indicating the compositional homogeneity. Likewise, the composition is further compared with untreated sample region 1 (α phase) of Fig.4a. The result indicates a marginal increment in the Sn, Zr and a minimal decrement in Ti. In addition, a slight amount of Si is observed in the treated sample.

3.4 Fretting wear behavior

To examine the change in the tribological properties after laser treatment, fretting wear test was performed on untreated and laser treated sample. Figure 5 illustrates the coefficient of friction of untreated and laser treated sample. The observation showed the coefficient of friction (COF) of untreated, 10 and 30 pulses treated samples are ~ 0.51, 0.46 and 0.39, respectively. The comparison indicates that the COF is decreased significantly after the laser treatment. It is well known that, the harder surface offers less friction. Therefore, the observed lower COF for the30 pulse treated sample in the present study can be associatedwith a higher surface hardness achievement, compared to the 10 pulse.

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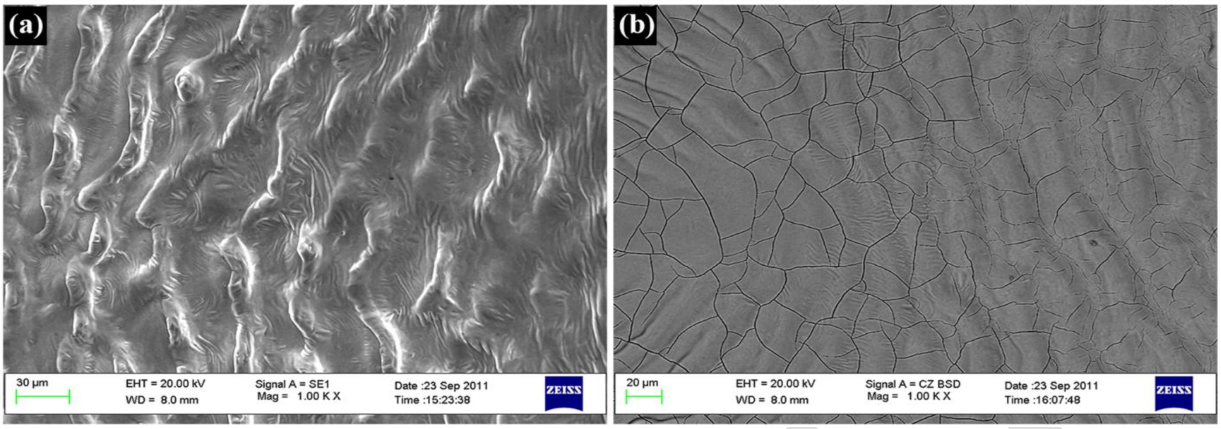

Fig.3 SEM micrographs of laser treated sample: (a) 10 pulse SE image; (b) 30 pulse BSE image.

IV. DISCUSSION

The transformation of the initial α/β dual phase microstructure to the surface waverippled character is related to the fact that when the laser exposed onthe surface, many processesoccur, viz., surface melting, evaporation, heating, plasma creation, shock wave generation.All these process fairly depends on the laser pulse energy and surface absorptivity. The surface absorptivity of the excimer laser is significantly higher compared to the other continuous wave laser (infrared laser) for metallic surface [10, 11]. Also, it is well known, the surface absorptivity rises with increase inthe surface temperature [10]. Based this, it can be inferred that the first few pulses exhibitto enhanced thesurface absorptivity, which results the intense deposition of energy at the surface and initiated the surface melting, evaporation and surface ionization.

As the pulse width of the excimer laser is 20 ns, if the solidification time is expected to be the same of the pulse width (~ 20 ns), then, the moultedsurface cools rapidly. Such rapid solidification leaves the imprint of the liquid structure in the form of surface wave/ripples. The hydrodynamic and capillary action also plays the major role, for the formation of periodic ripples.

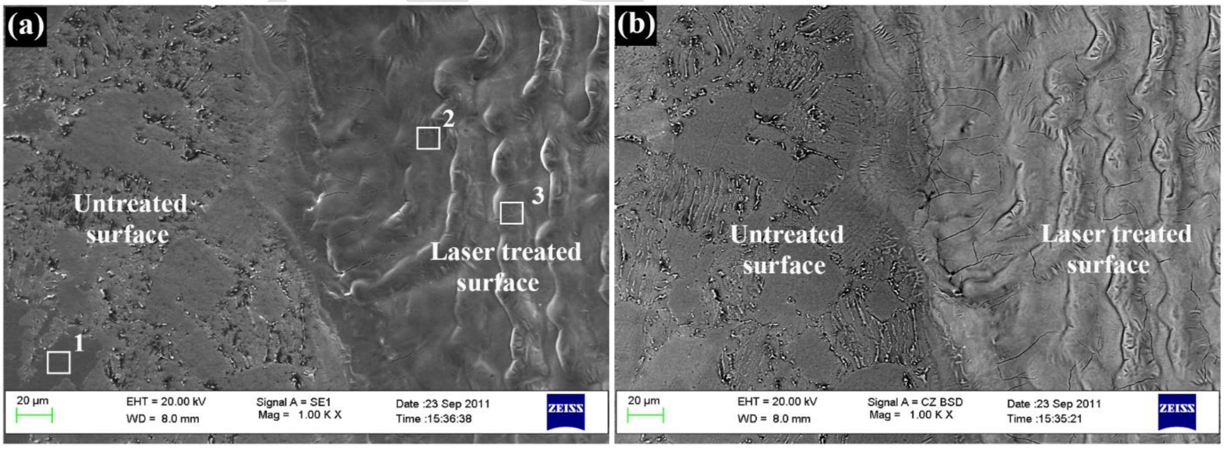

Fig.4. SEM micrographs of 10 pulse laser treated sample; (a) SE image; (b) BSE image; corresponding elemental composition taken from box region 1 and 2 and illustrated in Table 1.

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Table 1 SEM-EDS data of untreated and 10 pulse laser treated sample.

Sample	Region/ Elements	Composition (wt. %)				
		Ti	Al	Sn	Zr	Si
Untreated	Spec 1	86.85	5.86	3.92	3.37	-
Laser Treated	Spec 2	85.15	5.85	4.59	3.91	0.5
	Spec 3	84.23	6.61	4.53	4.19	0.44

However, the calculation of relaxation time for the periodic wave is given as [12]:

$$\tau = \rho\lambda / 8\pi^2\eta \qquad (1)$$

Where, τ is the relaxation time, ρ density, λ wavelength and η viscosity.

Although, the data for η is not available for the present alloy, the η value has been reported for a similar alpha alloyTi6424 alloy to be 4.84 mPa.s[13]. Assuming this value, the calculation indicates that the estimated relaxation time ispossibly~ 30.05 µs in the present case. Hence, the surface solidification time > the pulse width. Thus, the molten surface get enough time for re-solidified after eachlaser pulse exposure. This explains the unique microstructuralfeature as seen in the Fig. 3a and b. The periodicity of these surface ripples is found to be ~ 20 and 15 µm for 10 and 30 pulses treated sample, whereas the ripple width is estimated to be ~ 11 and 15 µm. The similar wave pattern has been reported by the Yu et al [14] on the Ni-P substrate by excimer laser treatment.

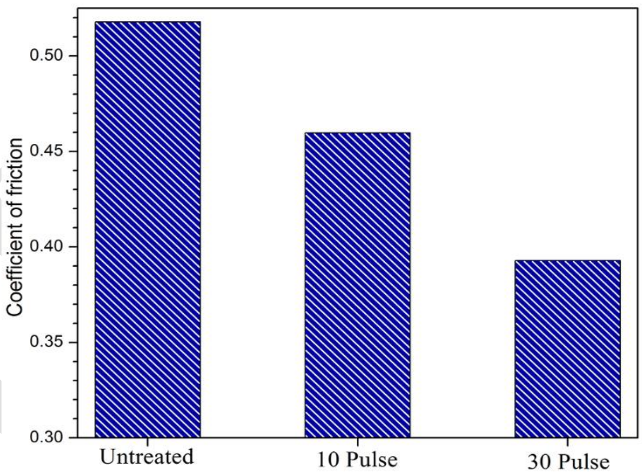

Fig.5. Vartiation in coefficient of friction with number of laser pulses.

The authors found that the wave periodicity increasedwith increasein the pulse number.

Milovanovic et al [15] also reported the similar ripple structure of excimer laser processed Ti6Al4V sample and estimated the surface periodicity ~ 20 µm for samples treated at 7.2 Jcm^{-2} fluence. The surface roughness was found toincrease with the progress in wavelength and pulse number. However, in the presentcase,it isexpected that the surface roughness becomes lower with progress in number of pulses becauseof the ripple broadening is higher (~ 15 µm) for 30 pulses treated sample.

The comparison of compositional changes (BSE images) indicates the expected compositional homogeneity after the laser treatment as seen in the Fig. 3 and 4. It is very obvious as the whole surface is in the molten liquid form after laser exposure, rapidly cools off. Thus, there is no time for nucleation and growth due to the termination of diffusion by rapid cooling. Likewise, the EDS analysis of the hill and valley regions of the ripple indicates marginal changes in

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atomic fraction of Ti, Al, Zr,Sn and Si. The composition is almost similar to the untreated α-phase, indicating no phase transformation after 10 pulses. It is possible that, after the laser exposure to the sample, it remains in the α-phase. A similar result had been reported by Kleinsschmidt et al on titanium film at ultrafast cooling rate [16].

The COF is significantly decreased from 10 to 30 pulse treated samples which is associated with the microstructural changes. This can be related to the surface hardness and surface roughness. It is expected, the hardness of the laser treated sample is significantly improved from 10 to 30 pulse treated samples.This could decrease the coefficient of friction [17]. Also, it is expected the surface roughness is higher in the case of 10 pulses compared to the 30 pulses treated sample which is responsible for the higher coefficient of friction for 10 pulse laser treated sample.

V. CONCLUSION

The conclusions emerging from the present study are listed below:

1. The surfacemicrostructural refinement takes place after the 10 and 30 pulse treatment. The distinction between α and β grains disappears after the laser processing.
2. The ripples are observed at the surface after the laser treatment.These ripples are finer and broader for 10 and 30 pulse treated sample, respectively.
3. The sample attained compositional homogeneity in between 1 to 10 pulse treatment.
4. The laser treated samples showed less friction coefficient compared to the untreated sample. The decrement is significant for 30 pulses treated sample.

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