

# Nanosecond Laser Surface Patterning of Bio Grade 316L Stainless Steel for Controlling its Wettability Characteristics

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**ABSTRACT-** In this work, potential of the nanosecond laser processing technique on manipulating the surface wettability of 316L bio grade stainless steel is investigated. Results show that the steel wettability toward water, improves significantly after the laser treatment. Different analyses are assessed in correlation with wettability using Scanning Electron Microscope (SEM), Scanning Tunneling Microscope (STM) and Energy Dispersive X-ray spectroscopy (EDX). It is found that the improvement in the wettability relates to the combined effects of the increase in the surface roughness, oxygen content and the form of the created surface morphologies. Laser fluence is found as the most dominant processing parameter and the higher the incident fluence results in the higher surface roughness and improvement of the wettability. However, measurements indicate that all the treated surfaces become hydrophobic after air exposure for a few days. It is shown that the time dependency of the surface wettability relates to the chemical activity and the reduction of the Oxygen/Carbon (O/C) ratio on the treated surfaces. The behaviors are further studied with investigating the effects of the keeping environment. The long-term wettability alteration differs for the samples that are kept in different mediums. Results indicate that the nanosecond pulsed laser treatment is a versatile approach to create either hydrophobic or hydrophilic steel surfaces for industrial and medical applications.

**KEYWORDS:** Laser matter interaction, 316L stainless steel, surface morphology, wettability, hydrophilic surface

## I. INTRODUCTION

Stainless Steel (SS) grade 316L has found numerous and diverse applications in different industries such as automotive, aerospace or many other manufacturing technologies. More over according to its unique properties of good corrosion resistance and biocompatibility, AISI 316L SS is used as a reliable biomaterial in manufacturing medical implants. It does not react with body fluids and tissues and has not harmful effects in the body. It can also bind with human bones for fulfillment of an appropriate osseo integration [1-3].

Although the mechanical performances of metals are governed by their bulk properties, its interactions with surroundings are determined by the characteristics of its surface. Surface topography, morphology, roughness, hardness, chemical composition and wettability are among the most important features determining the success or failure of a surface in a specific application. Thus the surface features upgrading is an undeniable requirement.

Ion implantation [3], electrochemical and chemical processing [4-5], plasma surface treatment [6], and thermo mechanical

processing [7] are the most prevalent methods for surface treatments. These methods have some disadvantages including long processing time, high energy and material consumption, poor precision and flexibility, lack in scope of automation, requirement of complex heat treatment schedules, and easy deformation of work-piece being treated [8-10]. Coating as another popular technique, has drawbacks mainly due to the limited bond strength between the coating and substrate and the subsequent lifetime of coating. These issues arise mainly due to low coating density, non-uniform coating thickness and mismatch of coefficient of thermal expansion [11]. The chemical method, the most widely used, is the cleanest method but may lead to incorporation of harmful elements into the material, requiring additional decontamination stages, prolonging and increasing the cost of this process, in addition to causing serious environmental damage from waste disposal [12]. Surface engineering methods based on application of electron, ion and laser beams are free from such limitations. But laser treatment is more advantageous compared to electron and ion beam based processing. Electron and ion beams: i. causes the rapid deceleration of high energy electrons and generates X-rays which is a possible health hazard, and ii. Need an expensive ionization chamber and ultra-high vacuum level [12]. Laser treatment can be free of the aforementioned short-comings and provides precise control over the morphology of processed area and has the ability to process complex parts and specific areas of the component without affecting the bulk material. Moreover it is direct, single step, repeatable, applicable for 3-D surface structuring with negligible risk of contamination [2, 13-15].

Here we report the potential of the laser surface processing technique in controlling the wettability of 316L SS without the assistance of any additional coatings. Despite the most of the recent publications which have used femtosecond lasers or relatively higher laser fluences [15-18], a nanosecond

Nd:YAG laser delivering low fluences is utilized in this investigation. Effects of the laser fluence and pulse number are studied and the mechanisms of the changes are discussed extensively. The time dependency of the wettability characteristics is investigated and a simple and successful method for turning from hydrophilic to hydrophobic condition is proposed. Potentials of the water and ethanol in long-term keepings of the treated samples are also evaluated.

## II. EXPERIMENTAL DETAILS

316L SS samples with dimensions of 10mm×10mm×1mm are used for the investigation. Compositions of the samples are tabulated in Table 1. In order to obtain more uniform surfaces, and having experiments with specified initial conditions, the samples were gradually wet-grounded with SiC papers to an average roughness value (Ra) of about 350 nm. To get rid of unwanted destructions, water was used as the lubricant and cooler. It could furthermore wash away the contaminations of the polishing. Subsequently samples were cleaned for 20 min in acetone ultrasonically and to ensure a clean surface, they were rinsed in the DI water at neutral pH for several times. Tests were performed in air atmosphere with a Q-switched pulsed Nd:YAG laser. Experimental parameters that were considered for the processing are summarized in Table2. Lateral displacements without any pulse overlaps were realized for obtaining an entire machined surface.

To investigate the effects of the laser radiation on the wettability characteristics of the samples, contact angle measurement using DI water are carried out. The droplets are released in a controlled manner onto the surface of a sample from the tip of a micropipette, with the resultant volume of the drops being 2  $\mu$ l. After about one minute which is an appropriate time for a droplet to be stabilized, its image on the surface is taken using a high resolution CCD camera. The contact angle of the water droplet is

determined by analyzing its image using the image processing techniques.

Scanning Electron Microscope (SEM) and Energy Dispersive X-ray spectroscopy (EDX) (VEGA\TESCAN) are used to study the surface morphology alterations and the

elemental composition changes of the samples. Scanning Tunneling Microscope (STM) (NAMA-STM model SS3) is also used for the surface roughness measurements of the laser treated samples. The reported roughness values are the mean values of the 4 different samplings on a surface.

Table 1. Elemental composition of the 316L SS sample

316L SS											
Element	Cr	Ni	Mo	Si	Co	S	P	C	N	Mn	Fe
Wt%	16.82	10.14	2.03	0.55	0.08	0.03	0.03	0.02	0.061	1.51	68.72

Table 2. Laser parameters employed during surface processing

Parameter	Value
Wavelength (nm)	532, 1064
Operation mode	TEM00
Pulse width (ns)	12
Pulse repetition rate (HZ)	5
Spot diameter (mm)	3.5
Laser fluence ( $J/cm^2$ )	0.1 - 1.1
Pulses per spot	100 ~2500
Laser energy (mJ)	9.6-105.7

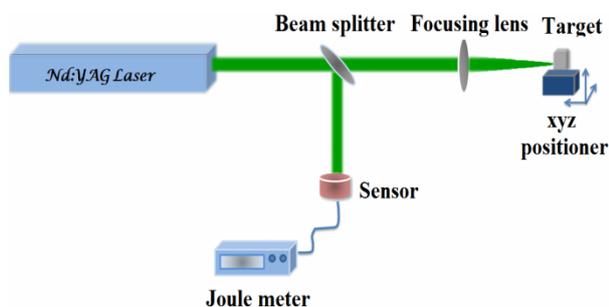


Fig. 1. Schematics of experimental setup

### III. RESULTS AND DISCUSSION

#### A. Surface Morphology

Structural features of the laser treated targets with different incident laser fluences and 2500 subsequent laser shots per spot are shown in Figs. 1 and 2.

2-Dimensional Fast Fourier Transform (2D-FFT) analyses (the inserts in the lower left corners of the images) and three dimensional gray scale images of the SEM figures are also

shown in Figs. 2 and 3. They confirm the high regularity and uniformity of the structures distribution in different directions. According to the SEM results, the imprints of the treatments are detectable on the samples irradiated at  $0.1 J/cm^2$  ( $\lambda=532nm$ ). Increasing the laser fluence so the incident energy, enhances the melting at surface discontinuities like scratches and impurities and causes the onset of the microstructures formation. Creation of such patterns might be related to rapid heating, melting and cooling of the surface layer. As the incident fluences are not high, so no material ejections to the periphery are realized.

The created morphologies can directly affect the surface properties of the samples such as the wettability.

Further increasing the laser fluence, especially at  $\lambda=1064nm$  laser wavelength, results in the intensified hydrodynamic effects so the larger micro structures creation, Fig. 2 D, C. These results might be related to the created turbulent melt pool and the hydrodynamics instabilities such as Kelvin–Helmholtz or Rayleigh–Taylor [19] and the subsequent rapid quenching and re-solidification of the melt. According to the results, there isn't any crack or defects on the treated samples.

#### B. Surface Wettability

Based on the nature of the attractive forces existing across a liquid-solid interface, wetting can be classified into the two broad

categories of physical wetting and chemical wetting [23]. In physical wetting the attractive energy required to wet a surface is provided by the reversible physical forces (van der Waals) and in chemical wetting adhesion is achieved as a result of reactions occurring

between the mating surfaces, giving rise to chemical bonds. In practice, according to the types of a material used, complex combinations of various bonding mechanisms actually occur [23].

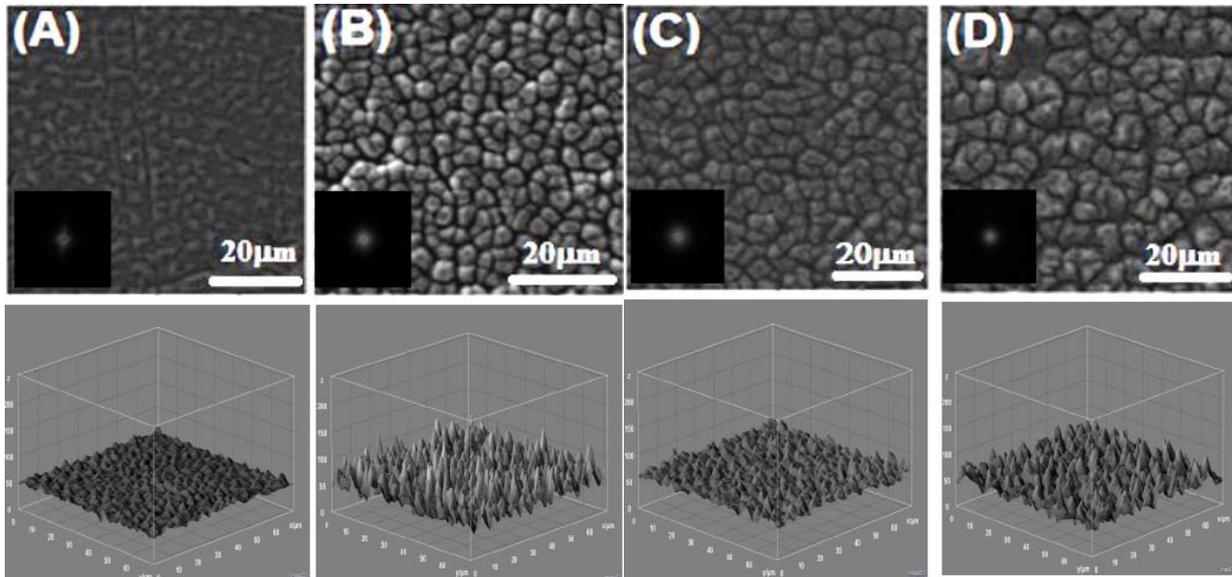


Fig. 2. SEM images illustrating structural features of the laser treated samples at 0.1, 0.2, 0.4 and 0.6 J/cm<sup>2</sup> laser fluences with N=2500 laser shots ( $\lambda = 532$  nm). Lower row shows the three dimensional gray scale images of the SEM figures.

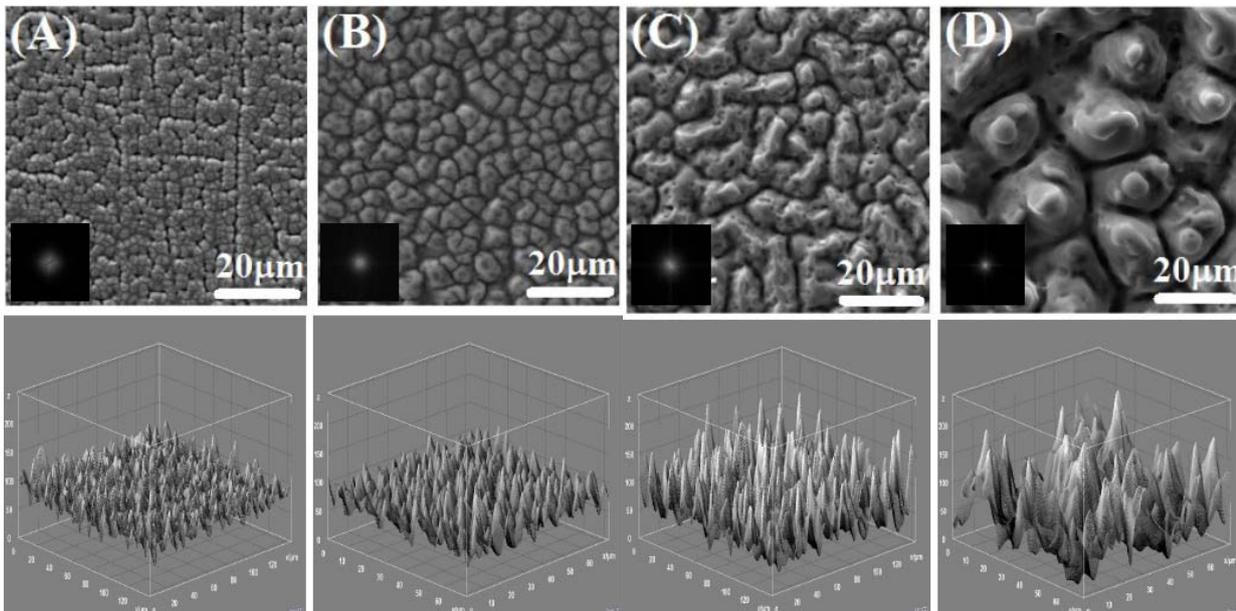


Fig. 3. SEM Images illustrating the structural features of the laser treated samples at 0.2, 0.5, 0.8 and 1.1 J/cm<sup>2</sup> laser fluences with N=2500 laser shots ( $\lambda = 1064$  nm). Lower row shows the three dimensional gray scale images of the SEM figures.

Contact angle measurement is in fact a simple method for evaluating the wettability of a

surface. Water contact angles on the samples that were treated mechanically are tabulated in

Table 3. It is obvious that more roughening of the samples with 80grit SiC paper increases the surface energy and lowers the contact angle of the water. This behavior reveals the important relation between the surface roughness and its wettability. But despite the remarkable differences in the surface roughness of the samples processed with 1000 and 80 grit SiC papers, the difference in the wettability is only about 4 degrees.

Table 3. Water contact Angles (CA) on the 316LSS surface, after mechanically roughening and after exposure to air for two months.

Sample	CA after cleaning	CA after two month
316LSS treated with SiC paper (1000)	74.1	79.1
316LSS treated with SiC paper (80)	70.7	85.1

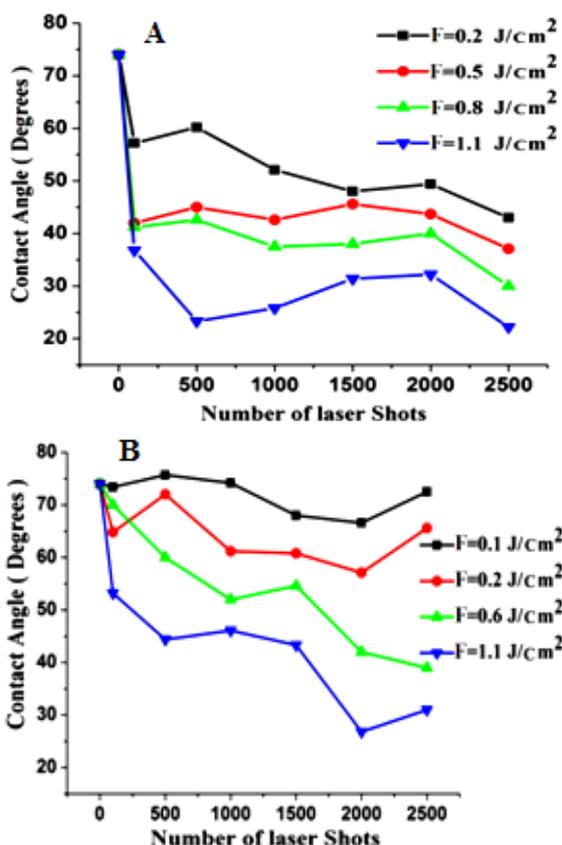


Fig. 4. Water contact angle variation versus the number of incident laser shots on the samples treated with different laser fluences at (A)  $\lambda=1064$  nm and (B)  $\lambda=532$ nm.

Variation of the water contact angle as a function of the number of incident laser shots

for different laser fluences are depicted in Fig. 4. It is obvious that the laser treatment has a remarkable effect on the surface wettability of the samples. Water contact angle of as low as 30 degrees is also achievable with controlling the processing parameters.

The water contact angle on the samples irradiated at  $\lambda=1064$  nm laser wavelength, decreases with increasing the laser fluence but the effect of the incident laser pulse number is not as significant as the laser fluence, Fig 4A. It is obvious that at least  $1.1\text{J}/\text{cm}^2$  laser fluence is required to reach the angles below 30 degrees. Water contact angle of  $\sim 25^\circ$  is the minimum angle that is obtained with the treatment at  $\lambda=1064$  nm and  $1.1\text{J}/\text{cm}^2$  laser fluence. On the other hand, processing at low laser fluences such as  $0.1\text{ J}/\text{cm}^2$  or even  $0.2\text{ J}/\text{cm}^2$  at  $\lambda=532$  nm have no remarkable consequence on the surface wettability of the steel, Fig. 4B. Increasing the incident fluence up to  $0.6$  or  $1.1\text{ J}/\text{cm}^2$  improves the wettability of the samples. Experiments using  $1064$  nm laser wavelength result in more hydrophilic states compared to the ones utilizing  $\lambda=532$  nm.

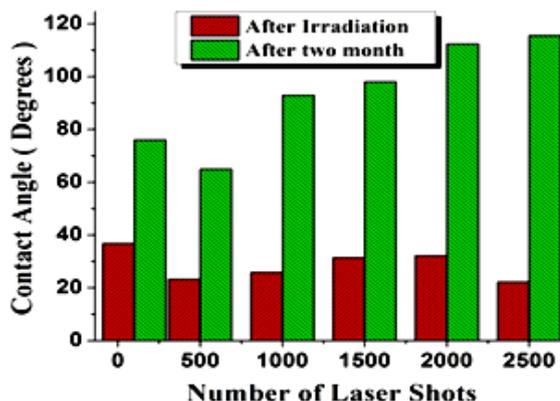


Fig. 5. Variation of the water contact angle on the samples irradiated with  $1.1\text{ J}/\text{cm}^2$  laser fluence at  $\lambda=1064$  nm versus the number of incident laser shots, immediately after the irradiation and after two month exposure to air.

However, although the samples become hydrophilic right after the laser processing, a decrease in surface hydrophilicity of almost all of the treated samples are observed with time

and exposure to air. Fig. 5 and Table 3 clearly illustrate these changes. The maximum water contact angle of  $115.5^\circ$  is observed on the sample irradiated with  $1.1\text{J}/\text{cm}^2$  laser fluence and 2500 pulses at  $\lambda = 1064\text{ nm}$ .

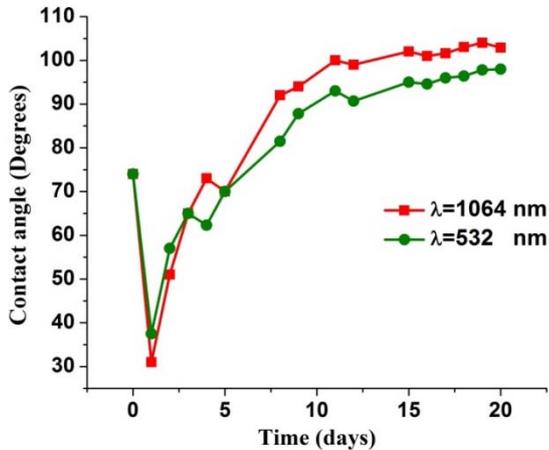


Fig. 6. Daily water contact angle alteration on the samples treated with  $1.3\text{ J}/\text{cm}^2$  laser fluence and 1500 shots at  $\lambda = 1064\text{ nm}$ .

These alterations occur at both the investigated laser wavelengths. Daily inspections of the water contact angle alterations indicate that a limited time after the treatment is required for reaching a hydrophobic condition. Fig. 5, depicts that the contact angle starts to increase exponentially in the first days after the irradiation but after about two weeks a saturation state is achieved.

### C. EDX Analysis

Indeed, since wettability is governed by the first atomic layers of a surface, the elemental analysis of the surfaces before and after treatment could be useful in understanding the changes. Fig. 6, shows that the surface oxygen content increases remarkably after the laser treatment. It is found from the EDX analysis that the oxidation happens for both of the investigated wavelengths following the laser treatment and the atomic percentage of the oxygen on the treated surface increases significantly.

Color modifications after the laser treatment can also point out the possible chemical changes like oxidization of the surface. The

oxidation could be related to the melting of the surface by the laser beam during the treatment. This in turn leads to the oxygen diffusion through the molten material and the subsequent oxidation of the surface. It is well known that the changes in the surface oxygen content on a metal surface are responsible for the overall increase in the wettability of them [16, 20]. Oxides are believed to can chemically alter the hydrophilicity of the surfaces due to their strong affinity for hydroxylation [21].

In addition to wettability, presence of the oxide(s) on a surface is considered very desirable from different fields of view: Oxidation can resist the corrosion of the sample, cause local hardening and improvement of the wear resistance, and can also influence the implant surface adhesion properties [13, 22].

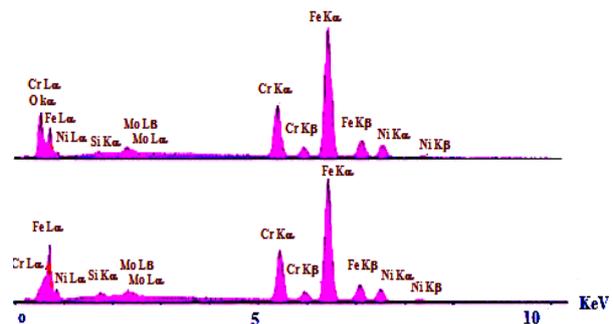


Fig. 7. EDX spectrum of the 316LSS surface before (lower graph) and after (upper graph) treatment with  $1.1\text{J}/\text{cm}^2$  laser fluence and  $N=1000$  laser pulses ( $\lambda=1064\text{nm}$ ).

### D. Surface Roughness

Two main models that have been proposed to describe the effects of the surface roughness on its hydrophobicity are the Wenzel and the Cassie-Baxter models. In the Wenzel model the liquid is assumed to be in contact with every part of the rough surface. The Cassie state, however, is referred to when air is trapped between the rough surface and the liquid droplet [18].

In the Wenzel model, the contact angle on the roughened surface,  $\theta_f$ , is given by Eq. 1

where  $r$  is the ratio between the actual ( $A$ ) and projected ( $A_0$ ) surface areas and  $\theta_s$  is the contact angle measured on the equivalent smooth surface.

$$\cos \theta_f = (A/A_0) \cos \theta_s = r \cos \theta_s \quad (1)$$

Because  $r$  is always greater than 1, the contact angle will increase with raising the roughness of a hydrophobic surface, whereas the contact angle will decrease when the roughness of a hydrophilic surface increases.

In the Cassie- Baxter model, the measured contact angle on a roughened surface is given by Eq. 2, where  $\phi_s$  represents the fraction of the solid in contact with liquid and  $\theta_s$  is the contact angle measured on the equivalent flat surface. Smaller the value of  $\phi_s$ , means the smaller the contact area between the solid and the liquid. Because  $\phi_s$  is less than 1, this model always predicts an increase in  $\theta_f$ , independent of the value of  $\theta_s$ . Only the Cassie state allows hydrophobicity of a roughened material which is inherently hydrophilic.

$$\cos \theta_f = -1 + \phi_s (1 + \cos \theta_s) \quad (2)$$

Table 4 illustrates the mean roughness values of the laser treated surfaces at different fluences. With increasing the incident laser fluence, surface roughness increases. Samples that are treated at 1064nm wavelength have rougher surfaces than the samples that are processed in the same conditions but using  $\lambda=532\text{nm}$ . This is in agreement with the SEM results and could be related to the fact that the infrared radiation is coupled to the metallic samples less efficiently than the visible radiation [24], then the melting in 1064nm irradiation is shallow so rougher surface is resulted. But in the 532 nm experiments the absorption of the laser beam is additional. Thus deeper melt pools or ablation could be realized which results in more uniform and smoother surfaces.

The maximum roughness change of approximately 145% and 98% are observed on

the samples that are laser processed with respectively 1064 nm and 532nm wavelengths. The reduction of the contact angle after laser processing can be linked to the increase in the surface roughness.

Table 4. Mean roughness of the laser irradiated surfaces with 1500 laser shots at different laser fluences.

Laser fluence (J/cm <sup>2</sup> )	$\lambda=1064\text{nm}$	$\lambda=532\text{nm}$
0.1	401±18	371±14
0.2	657±14	413±18
0.4	698±24	483±9
0.5	715±12	501±11
0.6	748±16	585±18
0.8	814±14	609±20
1.1	847±17	591±27

The higher the incident laser fluence is resulted in the higher the surface roughness and the enhanced wettability. So it seems that the Wenzel's state, is more likely and the roughness is an important parameter controlling the wettability of the laser treated samples.

In addition to the surface roughness, the surface chemistry (via the surface oxidization) and the form of the surface morphology have an important role in making the surfaces more hydrophilic

### E. The effect of the keeping environment

As the surface pattern and morphology does not change with time therefore, they cannot explain the wettability alteration by time. It seems that chemical reactions on the sample surface might be realized that could be responsible for the observed hydrophobicity after some time. For better understanding, the samples irradiated with 1500 pulses at 1.3 J/cm<sup>2</sup> laser fluence were kept for a month in three different ambient, in air exposure at laboratory, Ethanol (Merck, 99.99%) and De-ionized water. Fig. 8 illustrates the values of the water contact angle on the treated samples after a month. It is obvious that the changes for the samples exposed to air are very remarkable and the sample experiences ~232% alterations.

Samples were kept for a month exposed to air, DI water and ethanol. The water contact angle increases  $18^\circ$  and  $21^\circ$  on the samples that were kept in DI water and ethanol confinement respectively. This increase is compared to the initial angle immediately after the treatment. The differences of these environments is as low as  $3^\circ$  which is negligible compared to the changes of the sample that was kept in air exposure. This indicates the importance of the keeping environment. Keeping in liquid confinement, samples are far from any contaminations. But in the case of the sample that was kept in air exposure, the high surface tension of the oxides causes the surfaces to be very active and capable for different chemical reactions with contaminants such as the available carbonaceous  $\text{CO}_2$  and  $\text{CO}$  ones in air. Carbonaceous layers are non-polar and can cause hydrophobicity [18].

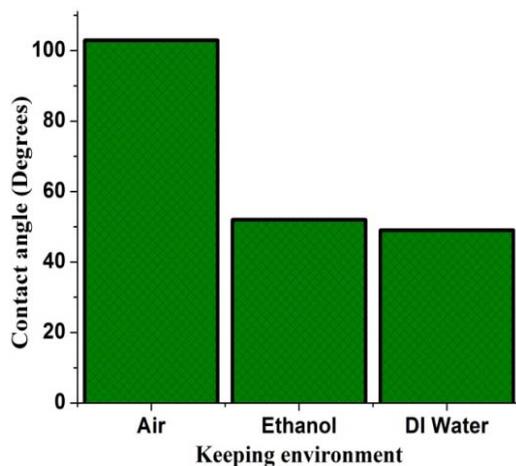


Fig. 8. Contact angle of a water droplet on the samples treated with  $1.3\text{J}/\text{cm}^2$  laser fluence and 1500 pulse trains at  $\lambda = 1064\text{ nm}$ .

EDX analysis proves the supposition and indicates a reduction from 2.14 (immediately after irradiation) to 0.71 (after a month being in air exposure) for the O/C ratio on the 316L SS samples surfaces. This in turn can cause the material to be a hydrophobic product. In this case the inherent hydrophobicity of the screening layer gets amplified by the surface morphology.

#### IV. CONCLUSION

Nanosecond laser irradiation experiments were carried out on 316L SS samples to change their surface properties such as morphology and wettability toward DI water. In conclusion, Outcomes can be summarized as following: a) Regular micro structures without any cracks or defects are created on the processed surfaces using low laser fluences, b) wettability changes of the laser treated samples are more remarkable than the ones that are treated with rough Sic papers. c) With increasing the laser fluence, water contact angle on the treated samples decreases at both of the studied wavelengths, d) the higher the incident laser fluence results in the higher the surface roughness. e) As the wettability also increases with laser fluence, the Wenzel's state is more likely. f) Processing at 1064 nm laser wavelength results in rougher surfaces compared to  $\lambda=532\text{ nm}$  so the better wettability. g) surface oxygen content increases remarkably after laser processing. The enhanced wettability could be related to an increase in the surface roughness; surface oxygen content and the form of the created surface morphologies. h) a remarkable alteration from hydrophilic to hydrophobic condition is detected in about 10 days for the samples that are exposed to ambient air. As the surface structures, does not change with time, through the EDX analysis and the differences of the wettability alterations for the samples that were kept in different environments for a month, it is found that the behavior may be related to the chemical reactions and the absorbance of the hydrophobic contaminants and therefore the reduction of the O/C ratio on the treated surfaces. i) DI water was found as the ambient that has a good potential for long term keepings of the laser treated hydrophilic metals.

Overall, patterning of 316LSS with low nanosecond laser fluences and exposing them to air is a one-step, convenient and successful method, for controlling the surface wettability of steel.

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