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Improvement The Corrosion Behavior And Wear Characteristics Of Aisi 304 Stainless Steel By Using Nd -Yag Laser Surface Treatment

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ABSTRACT: This article aimed to study the effect of pulsed laser surface melting (LSM) on the corrosion behavior and wear characteristic of AISI 304 stainless steel which are used widely in medical and food equipment. Laser beam of Nd-YAG (neodymium – doped yttrium aluminum garnet Nd: Y3Al5O12) was done with parameters of 0.8 mm spot size, 10 mm/sec. travel speed, 2 ms pulse duration, 1-3 Hz pulse repetition rates (frequencies), 1064 nm wavelengths and 4J fixed energy. Surface treatment process include surface melting by rapid heating of the materials surface and followed by fast cooling to modify the microstructure and phase change of surface metal. The microstructure of samples were performed by using the optical microscope and confirmed by X-ray diffraction analysis to identify the phase's transformation after laser treatment. Scan electro microscopy was considered in investigated the surface morphology before and after laser treatment. Surface hardening values was obtained by MicroVicker test. Corrosion test was carried out using the electrochemical corrosion cell and measured by Tafel extrapolation method of 304 stainless steel in a chloride acid (HCl) solution at 37 0C. Results shows that the treated samples by laser surface melting have a higher hardness and wear resistance than untreated sample. Also corrosion results indicated that the treated samples were a higher pitting corrosion resistance than untreated samples.

KEYWORDS: Nd-YAG Laser, 304 stainless steel, pitting corrosion, microhardness, chloride acid.

INTRODUCTION

The avails of the laser processes applied of surface engineering are high iteration, high speed, and worked area depend of the laser focus, high pliability and automation. Industry laser applications in the surface treatment of materials are focused on polymers, aluminum and titanium and many applications in food industry equipment of stainless steels [1]. In the case of metal surface treatment was treated with parameters as high energy laser, one part of laser energy is reflex and the other is absorbed by metal bulk [2, 3]. In study of laser treatment of 304 stainless steel, it can be found at some problems due to the probability of corrosion behavior which is inherently increased by the fashioning of delta - ferrite (δ) due to a higher dissolvability for chlorid acide in ferrite than austenite [4, 5]. A studied the improvement of surface properties of AISI304 stainless steel by laser melting [6]. They found dissolution of inclusions such as MnS, FeS, CrxCy, and other carbides. The dissolution of CrxCy phases which excess Cr available for the chromium formation of a thin from oxide layer. The capability of susceptibility of sensualize of 304 stainless steels to pitting corrosion is minimized by laser surface melting through the degradation of inclusions precipitates and otherwise the homogenization of chromium distribution. A study the effect of laser parameters on the mechanical property's microstructure, and corrosion resistance of stainless steel AISI316L by laser surface melting [7].

They found that the technique by laser surface melting was refine the grain size and improved the microhardness of 316 L orthodontic bracket at. Some researcher was studied the impact of laser radiation on metal structure and corrosion resistance of stainless steel 413 L, which is used as biomedical material in the artificial saliva solution

[8]. They found that the corrosion behavior was improvement of corrosion after laser surface melting due to enhancement of microstructure by refine grain size. The aim of this work is to study the effect of novel technique using Nd- YAG laser surface melting to improve the corrosion behavior of AISI 304 stainless steel. A chloride acid (HCl) concentration up to 18% at 37 ^{0}C was used to cleaning and sterilization of medical equipment which manufacturing from AISI 304 stainless steel, because of kills a bacterium and virus in widely percentage. In other hand a chloride acid solution caused corrosion in surface of this equipment. In other application of 304 stainless steel was used in food mixer tool which exposure damage of metal surface due to mechanical wears abrasive. Therefore laser surface treatment help to improvement the wear characteristic of metal surface of AISI 304 stainless steel. The novel of these studies was help to solve the problems due to offering applications.

MATERIALS AND METHODS

Material

Stainless steel grad AISI 304 is used in this study due to as wide applications in food industry, architectural paneling, dairy equipment and pharmaceutical production equipment field. The chemical composition was done by spectrometer analysis device which obtainable in the university of technology – Iraq, as shown in Table 1. The specimens were prepared from stainless steel plates with 5 mm thick and dimensions of area as 1.5 cm × 1.5 cm, which are prepared by using machine cutting by a wire process. Specimen's surfaces were prepared by variance of paper degree of 320, 500 and 1000 of SiC emery. The last operation were polishing by cloth with diamond past of 0.3 μm to get a flat surface. The mechanical properties of stainless steel AISI 304 are shown in Table 2.

Table 1: Chemical analysis of AISI 304 stainless steel.

Element (Wt%)	Analytical	Standard
C	0.07	0.08
Mn	0.05	2.00
Si	1.08	1.00
P	0.35	0.045
Ni	0.001	8.00-10.50
Cr	18.65	17.50-19.50
Ni	0.001	0.002
Fe	-	Balance

Table 2: Mechanical properties of AISI 304 stainless steel [9].

Tensile (Mpa)	Yield strength (Mpa)	Elongation (%)	Hardness (HRB)
515	205	40	750

Laser Surface Instrument

Figure 1, show the Laser irradiated beam instrument was used in this work. Laser type was Nd-YAG (neodymium – doped yttrium aluminum garnet Nd: Y3Al5O12) with parameters were Area of sample was 1 cm^2 and the laser beam at 8 cm distance from the lens with 0.8mm spot size, 10 mm/sec. speed, pulse duration 2 ms, repetition pulse rates (frequencies) 1,3 Hz, wavelengths of 1064 nm and fixed energy of 4J. Table 3, was explain the specifications of Nd:YAG laser instrument in this work.

Figure 1: The laser device in this study.

Table 3: The specifications of Nd:YAG laser device.

Laser type	Nd-YAG Q switch laser
Wavelength	Dounle wavelength (1054,323) nm
Controller	7.4 color touch LCD display
Pulse energy	1600mj
Power	1000w
Width of pulse	6ns
Frequency	1-6Hz
Spot diameter	1-7mm adjustable

Metallographic, Microhardness, SEM and XRD Tests.

The microstructure examination was carried out the Stainless steel grade 304 samples were prepared by grinding, polishing, etching process and examination by using optical microscope type (MBL3300) model (kruss optronic -Germany) connected with computer. The grinding process was done with water by using emery paper of SiC with the various grainier of (220,320,500, &1000). Polishing process was done to the samples by using Al_2O_3 with size 0.2µm and followed by cloth. All samples must be dry with hot air after cleaned with water and alcohol. Etching process was done to the samples by using etching with solution consists (49 $CuSO_4$ + 20 ml HCl + 20 ml distilled water). Final the samples were washed with worm water and alcohol and dried in oven. The hardness test was carried out micro hardness on cross section of specimen. The micro hardness test of samples using Digital MicroVicker Hardness Tester type (TH714). The 3-5 reading of hardness values and taken average of distance between any two adjacent readings was 1 mm. Examination the microstructure and topography of sample surface by using (SEM) scanning electron microscope type (vega3lm). X-Ray diffraction type (Shimadzu- 6000) with Cu-tube and wavelength 1.540 Aº, to determine the phases before and after laser surface of 304 stainless steel samples.

Electrochemical corrosion Cell

Electrochemical corrosion test done on specimens were made according the standard ASTM G71-31 with dimensions (1×1 cm^2) which immersed in the 13% chloride acid (HCl) solution at 37ºC. All specimens were machined, cut and polished mechanically to a mirror finish, washed in distilled water and stored in desiccators. Polarization experiments were carried out in “WINKING M Lab 200” Potentiostat from Bank-Elektronik with electrochemical standard cell as shown in Figure 2, with provision for working electrode, auxiliary electrode (Pt electrode). Electrochemical measurements were executed with a potentiostat at a scan rate 3 mV/sec. Tafel extrapolate method used to determine the corrosion potential (E corr.) and corrosion current (I corr.) [9].

Figure 2: Electrochemical corrosion cell

Wear test

The wear device was used to measure the wear rate is shown in Fig. 3. The instrument was consists of a rotary electric motor with a speed of (500 r. p. m) attached with gearbox and shaft, pin -on-disk principles to measure the wear rate. Light load (1000gm) was used and a sensitive electronic balance was used for measuring the weight. The distance of sliding wear was equal to (82mm) and the speed of sliding was equal to (4.2 mm/min). The samples were weighed before and after the test, every 20 min.

The wear rate was calculated from the following equation:

W.R = W2-W1/ S.D (1) [10]

S.D = S × t (mm) (2) [10]

Where

W1: is the sample mass before wear test (gm), W2: is the sample mass after wear test (gm), S.D: is the distance of sliding wear (mm), S: is the Speed of sliding (mm/ min), t: is the time of sliding (min) and W.R: is the wear rate (gm/ mm).

Figure 3: Instrument of wear test.

RESULTS AND DISCUSSION

Microstructure and phase transformations results

The microstructure of sample AISI304SS before LSM (untreated) as shown in Figure 4a, which shows the formation of a fully γ- austenite phase and twin bands with some inclusions and the columnar grain due to manufacturing process of hot roll forged of steel. While Figure 4b explains the analysis results of XRD are affirmation of the γ- austenite phase in stainless steel.

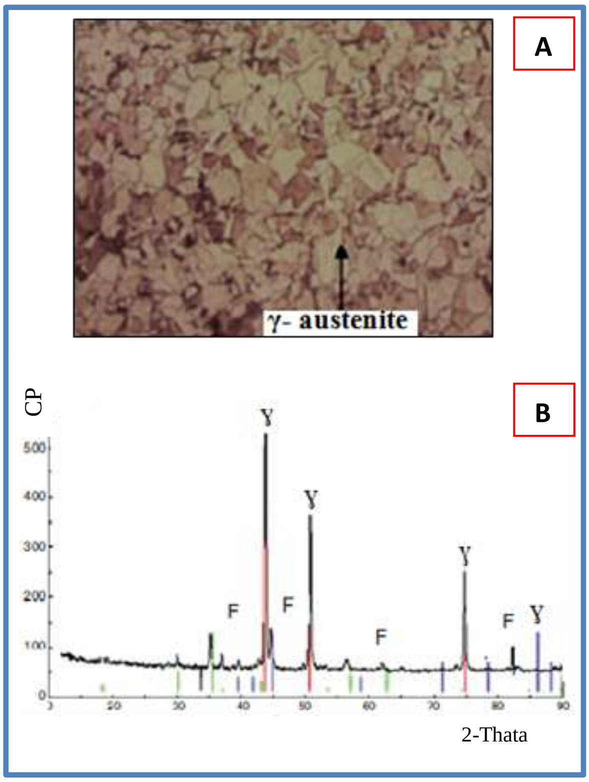

Figure 4: A - Microstructure of 304stainless steel before laser at 200x and, B - XRD analyses after laser treatment.

Figure 5 A Show the microstructures modification after laser surface melting with parameters at 4J at pulse repetition rates 1,3 and wavelengths of 1064 nm. It was noticed a three zones in sample after laser melted as base metal, molten zone and heat affect zone with different grain size and a fine dendritic structure which distributed in the melted pool. Figure 5 B, explain the SEM topography of sample after laser treated, we noted the molten pool and base metal. After the laser surface melting, the metal surfaces were smooth and fine grain comparison with untreated sample as shown in Figure 4a. It was found that the microstructure consists of delta -ferrite in the dendritic solidification of austenite matrix phase. During the solidification of austenitic phase, the first phase formation is delta ferrite as the at drops the temperatures below the protected transformation it led to transform into gamma austenite [10]. X-ray diffraction result show the different phases which consist of chromium carbides after laser surface treatment as shown in Table 6.

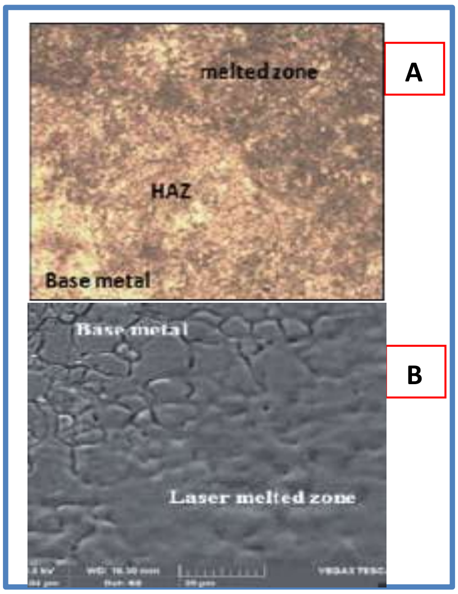

Figure 5: Optical micrograph (A) and; SEM images (B) for the laser treated sample, 100x.

Table 3: The phases by X-Ray diffraction of laser treated sample

2theta	d measured (nm)	I/I_O	Phase
33.7	0.266	55	Cr_7C_3
39.5	0.228	61	Cr_7C_3
44.9	0.210	68	Fe_3C
61.8	0.152	67	x-Fe_2C
73.7	0.128	69	Cr_7C_3
76.1	0.125	85	$Cr_{23}C_6$
73.7	0.128	75	Cr_7C_3
76.1	0.125	77	Cr23C6
81.9	0.11	0.11	Fe7C3

Microhardness test results

The results of microhardness values for untreated specimens of 304 stainless steel and treated specimens by irradiate with laser beam parameters. It has been found that the treated specimens have hardness values 1002 HRB and value of untreated specimen was 396 HRB. This is because of the irradiate laser interaction with metal of stainless steel followed by the rapid heating and cooling cycles. Increase in the hardness value after laser treated due to the refinement structure of grains γ-austenite phase after laser treated and formation of chromium carbide that's effect to an increase the hardness values.

Corrosion test results

Corrosion examination was employ to evaluate of pitting corrosion of 304 SS samples before and after laser surface melting by using electrochemical corrosion cell. The specimens were cleaned and put in solution from 13% chloride acid (HCl) solution at 37 ºC. Figure 6 shows the behavior curves of cyclic polarization for as received (untreated) sample. It was noticed that anodic polarization path was includes of two regions active- passive and passivation zones until reach breakdown the passivity on metal surface film, also the curves paths are detect of an increase in (Icorr.) with behavior of cycle pitting. Figure 7, show the polarization curves paths for treated sample after corrosion test in chloride acid HCl solution at 37 ºC. That treated and untreated samples of 304 stainless steel have the oneself path in polarization behavior as in similar corrosion mechanism. But the differentiability was notice that untreated sample have a larger cycle of hysteresis loop than treated sample and the pitting corrosion of sample after laser treated due to the presence of chloride ions which combine with dissolved hydrogen to formed HCl acid which effect to decrease the pitting corrosion rate. The results showed that the pitting corrosion resistance of stainless steel was increases significantly after laser treatment and noted the laser beam gives a smoother surface (average roughness is 0.78 and 0.07 before and after laser respectively). Improvement the pitting corrosion resistance after laser surface melting because of formation layer from oxide chromium and chromium carbide that's effect to increase pitting corrosion. These results are agreements with research [11, 12].

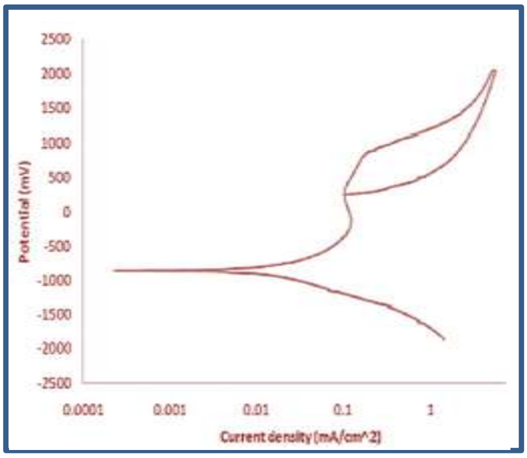

Figure 6: Polarization curves path for untreated sample AISI 304 stainless steel.

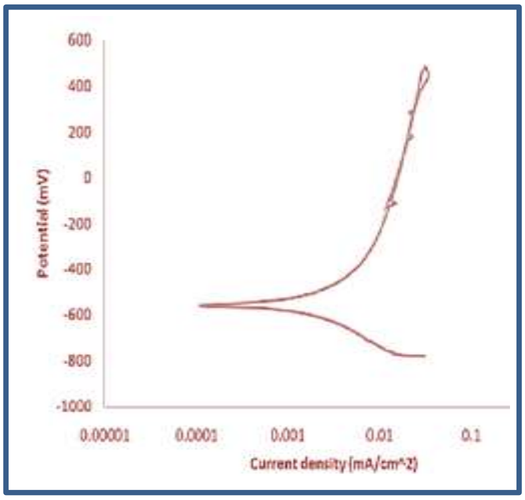

Figure 7: Polarization curves path for laser treated sample AISI 304 stainless steel.

Topography damage after Corrosion

Figure 8; shows the topography images of damage surface examination by optical morphology of AISI 304 stainless steel after corrosion test in 13% chloride acid solution(HCl) at 37 ^{0}C. It's clearly shown large damages in surface by corrosion before laser surface melting and it's large and deep pits corrosion as shown in Fig.8A, matching with [13]. While the topography surface of sample after laser surface melting which has a less damage of pitting corrosion? While it was noted the shallow pits and very small pits dispensed in metal surface of specimen after laser treatment s as shown in Fig. 8B. That, because of formation of protective film by chromium oxide Cr2O3 on the surface after laser surface melting process that led to enhancement the resistance of pitting corrosion, these result are agreements with research [14, 15].

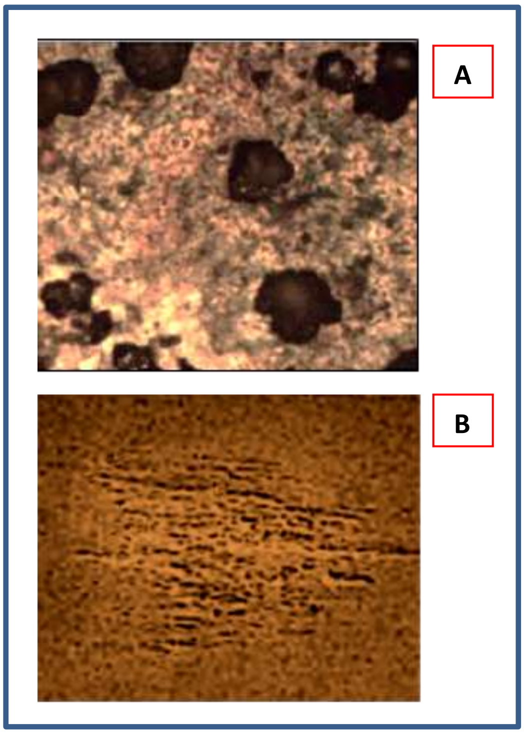

Figure 8: Surface metallography damage for; (a) untreated and (b) laser treated samples after corrosion in the 13% HCl solution.

Wear test results

Figure 9; Shows the wear test results of AISI 304 stainless steel samples before and after laser surface melting. It was found a decrease in wear resistance (increase in weight loss) in sample before treatment, because decrease in hardness of surface. Also increase damage surface as noted in Fig.10A. In other across the laser surface treatment was effect to increase in wear resistance (decrease in weight loss) because increase in hardness of surface metal due to refine the microstructure, that's matching with behavior of sample surface as less damage in topography surface in Fig.10B, these results are agreements with research [16, 17].

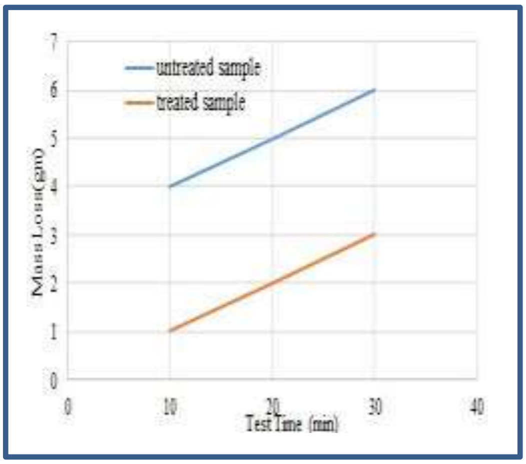

Figure 9: Wear test results before and after laser treatment of sample 304 stainless steel.

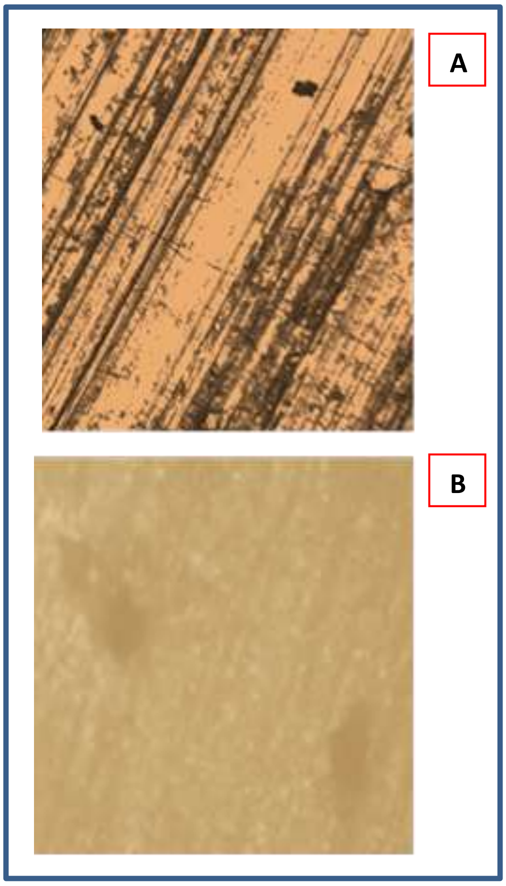

Figure 10: Micrographs of surface damages after wear test; A, before laser and B, after laser, 400x.

CONCLUSION

Nd-YAG laser surface treatment of 304 stainless steel with laser conditions as fixed energy 4J at pulse repetition rates of 1-3 Hz and wavelength 1064 nm, which allows obtaining:

1. Improved the microstructure of 304 stainless steel to a fine grain of γ- austenite phase in fusion zone after laser melting.
2. Increased the metal surface hardness after surface laser melting by formation carbide phase.
3. To obtained a pitting corrosion type with better corrosion resistances by minimizes (I corr.)
4. Enhances the pitting corrosion resistance by minimize the pitting corrosion rate by formation a protective film layer from chromium oxide on the metal surface.
5. Laser treated sample of 304 stainless steel have a smaller hysteresis loop than untreated sample.
6. The topography surface damage of AISI 304 stainless steel after corrosion test in chloride acid solution have a big and deep pits corrosion in the metal surface of untreated sample compare with treated sample.
7. Increased wear resistance (decrease in weight losses) of 304 stainless steel after laser treated comparison with untreated sample.

Recommendation for future studies was to study the effect of continuous Nd-YAG laser with different laser parameters applied on corrosion of medical surface metal (316 L stainless steel) was immersed in artificial solution.

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