Investigation of The Laser Nano -Indentation Effects During Irradiation of AISI304 Stainless Steel

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Abstract: In this work, the surface of Austenitic Stainless steel AISI 304 was irradiated by Excimer laser 308nm at constant power 6mJ and repetition rate 200Hz. The selected alloy was irradiated at different number of pulses ranges from 2000 to 50000 Pulses. A significant change in the mechanical properties of AISI 304 is observed based on the microstructure changes. Laser surface irradiation process is a very complicated process and is found to be affected by the microstructure and chemical composition of the alloy. The mechanical properties such as Hardness and reduced modulus of the austenitic stainless steel 304 were improved. Hardness and modulus were decreased with both depth and load. The maximum improvement in nano-hardness and reduced modulus has been investigated in the surface of the sample and rapid decrease in the mechanical properties has been recorded along the depth. Both of quantitative and qualitative techniques were the main tools in results analyses.

[Hebatalrahman A , Rossetto Gilberto and Carta Giovanni. **Investigation of The Laser Nano -Indentation Effects During Irradiation of AISI304 Stainless Steel.** Academia Arena, 2012;4(2):8-15] (ISSN 1553-992X). http://www.sciencepub.net. 3

Keywords: Excimer Lasers, nano-measurements, Quantitative analysis, microstructure

1. Introduction

Stainless steel as alternative to Conventional types of alloys in building applications. In the recent years a need has been developed in several countries for a more comprehensive design specification for cold-formed and hot-rolled stainless steel structural members. Because of the limited life of carbon steel in building and structures that are exposed to moderately to highly aggressive atmospheres, stainless steels, in spite of their higher cost, can be an alternative to carbon steels [(1) :(3)]. The excellent performance of the austenitic stainless steels in fire conditions and during seismic events is a good reason to introduce stainless steels as structural elements in construction. Attention being paid to crash-worthiness, energy absorption is a key property of the material used for structural parts. Stainless steels and among them austenitic stainless steels have the advantage over aluminum alloys and carbon steels of being highly strain rate sensitive. This means that the faster the loading is applied, the more the material resists deformation [(5),(4)]. This is a particularly good feature for crash worthiness as in all earthquakes the loadings are applied very rapidly. Due to the good energy absorption properties of stainless steel a number of alternative ideas are being continuously developed throughout the building materials [(6),(7)].

2. Experimental Work

The alloys used through this work was supplied by Sandvik Co, France in the form of sheets of stainless steel AISI 304 commercial grade. Table (1) show the chemical composition of the alloy used in the current work. The samples were irradiated by laser table (2) show the laser irradiation conditions of the samples.

Table(1) Chemical composition of Austenitic stainless steel 304

Material	C%	Si%	Mn%	P%	S%	Cr%	Mo%	Ni%
Stainless steel 304 Austenitic	**≤0.07**	**1.0 max**	**2.0 max**	**0.045 max**	**0.03 max**	**16.5: 18.5**	**2.5: 3**	**11.0 14.0**

Table (2) Laser Irradiation Conditions

Type	Wavelength nm	No of pulses	Energy per pulse mJ	Repetition rate (Hz)
Excimer	UV 308.6	0,2000,5000,10250, 15000,50000	6	200
duration time=6nano second & total energy =energy per pulse*number of pulses				

Nano- measurements (Indentation data analysis)

Nanoindentation was used to determine film hardness and elastic modulus using a Nanotest 600 instrument from Micro materials Ltd with a Berkovich (three-sided pyramidal) diamond indenter. The peak loads in the rage 1 – 140 mN were used, with loading rate = unloading rate that were varied in proportion to the peak loads starting at a value of 0.05 mN/s for the 1 indentations, while common experimental conditions as initial (contact) load 0.05 mN and holding period at peak load 10 s were used for all the measurements. The indentations were repeated at least five times at each load on different regions of the sample surface apart 100 μm. The hardness and reduced modulus have been determined from these indentation curves using a method originally proposed by Oliver and Pharr, [W. C. Oliver, G. M. Pharr, J. Mater. Res. 7 (1992) p. 1564], which fits a power-law function to the unloading curve. All nano- measurements has been done, the Young's modulus of the samples can be calculated from the reduced modulus via the equation which includes the effects of non-rigid indenters on the load-displacement behavior:

$$1/E_r = (1-\nu_s^2)/E_s + (1-\nu_i^2)/E_i \qquad (1)$$

where Es and νs are, respectively, the Young's modulus and Poisson's ratio for the specimen, Ei (1141 Gpa) and νi (0.07) the corresponding indenter quantities and Er the reduced modulus, Which has been obtained by the initial slope of the unloading curve[(8),(9)].

Metallographic Examinations

The specimens were prepared for examination first by grinding on different grades of silicon carbide "SiC" papers coarse grinding followed by fine grinding, finally polishing was conducted with Alumina powder (3μm) size. The details of the microstructure were revealed after etching by standard etching solution of the alloy selected. All specimens had to be etched and polished several times to obtain best results and to produce a uniform level of sample examination[(10),(11)]. The surfaces of the samples before and after laser irradiation were examined using an Olympus optical microscope Model BHM at selected magnification.

Quantitative Metallography Measurements

A quantitative analysis of the microstructures was produced carried out over 4 fields across the surface to indicate:

1. Average grain size number by linear intercept technique.
2. Average grain diameters D_A using number of modules per unit area
3. Average intercept distance μm
4. Area of average grain section mm^2 x $10^{\wedge 6}$
5. Average number of grains per mm^3 x$10^{\wedge 6}$
6. Nominal grains per mm^2
7. Nominal grains per mm^2 at 100x

calculations were carried out in several areas in each specimen investigated at 100X projected on a screen measuring 500 mm^2. The measurement is average of over five readings at least for each condition. The scattering value for each specimen was $\pm$1%. All calculations were measured around a circle to consider all directions 360°

3. Results and discussions

Nano-measurements of stainless steel 304 irradiation by Excimer Laser at 308nm

The nano-indentation measurements were performed on the surface of the stainless steel 304 before laser irradiation in order to determine its hardness and elastic properties. Figures (1) shows the variation of hardness with maximum load for the austenitic stainless steel 304 irradiated by Excimer laser 308nm at 200Hz, 2.2mJ, at different number of pulses. The hardness decreased as the load increased, maximum hardness is at the surface, the higher number of pulses, the higher the hardness at maximum load of 20mN. Figure (2) shows the variation of hardness with Maximum depth for the austenitic stainless steel 304 irradiated by Excimer laser 308nm at 200Hz, 2.2mJ at different number of pulses. The higher number of pulses the higher the hardness at an indentation depth of about 350nm. Figure (3) shows the variation of reduced modulus with load for the austenitic stainless steel 304 irradiated by Excimer laser 308nm, 200Hz, 2.2mJ, at different number of pulses. The change in reduced modulus is less pronounced than variation in hardness at the same range of number of pulses. The gradual decrease in modulus is at maximum load 10mN. Figure (4) shows the variation of reduced modulus with maximum depth for the austenitic stainless steel 304 irradiated at the same conditions. The variation of reduced modulus is significant only at an indentation depth of 400nm. The variation in hardness and reduced modulus with maximum loads and depth respectively, covering the loading range 1 – 200 mN. The sample shows a decrease in the hardness and in the reduced modulus as the indentation load increases; probably the higher hardness near to the surface represents a change in the mechanical properties of the near surface region due to previous spontaneous oxidation of the sample or work hardening due to polishing. Samples with similar preparation conditions have been irradiated by Excimer laser 308nm, 200Hz and 6mJ at 2000, 5000 and 50000 pulses respectively. The hardness value decreased on the surface due to stress relief, which occurs due to laser irradiation. Laser irradiation effect has been limited to the surface of the sample. This was followed by the value dropped within the range of 0 to 500 nm to the hardness value of the base metal. 2 Gpa. Laser effect was not enough to produce any significant effect on yielding of the material so the reduced modulus almost unchanged with both depth and load.

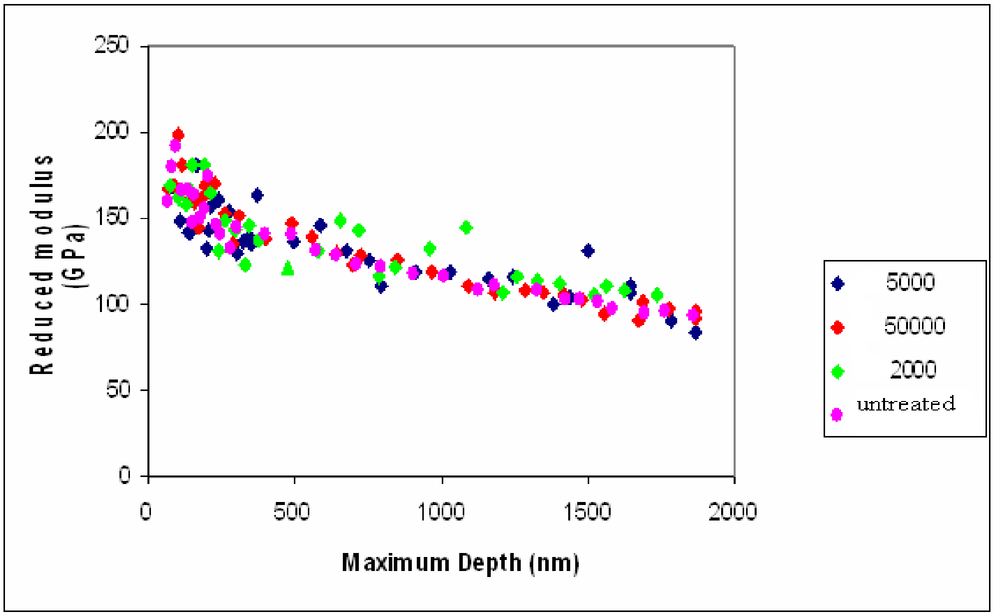


Fig (1) The variation of hardness with maximum load for the austenitic stainless steel 304 irradiated by Excimer laser 308nm, 200Hz, 2.2mJ, at different number of pulses.

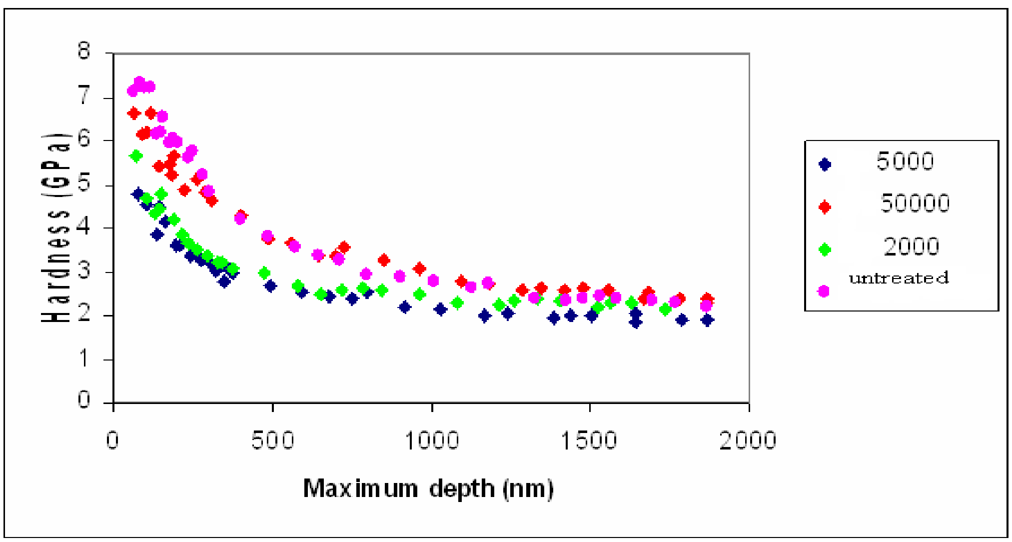


Fig (2) The variation of hardness with Maximum depth for the austenitic stainless steel 304 irradiated by Excimer laser 308nm,200Hz, 2.2mJ, at different number of pulses

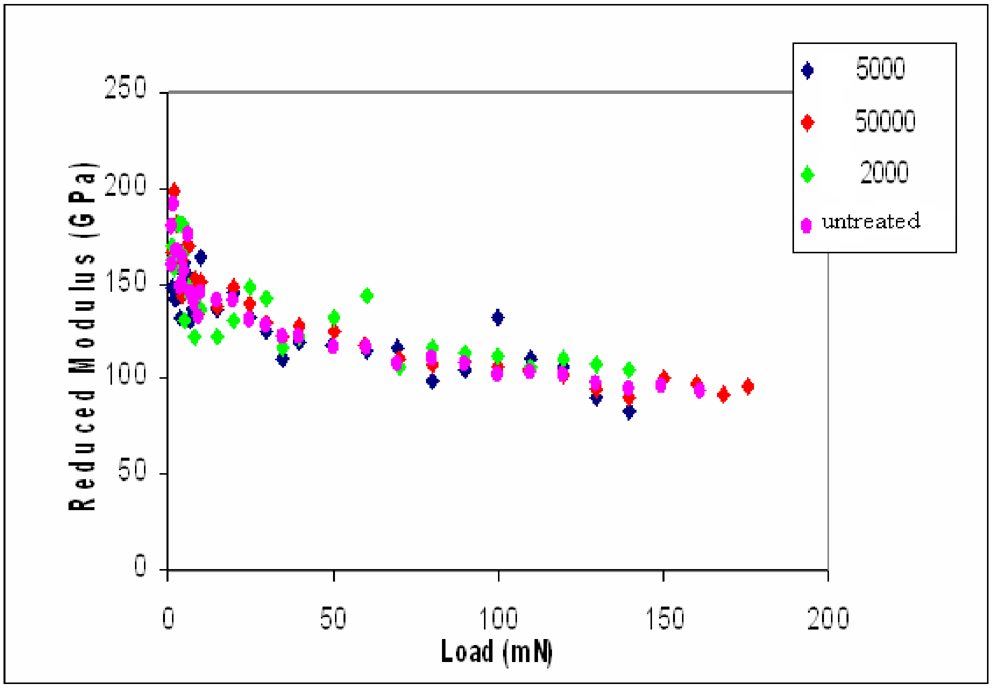


Fig (3) The variation of reduced modulus with load for the austenitic stainless steel304 irradiated by Excimer laser 308nm, 200Hz, 2.2mJ, at different number of pulses.

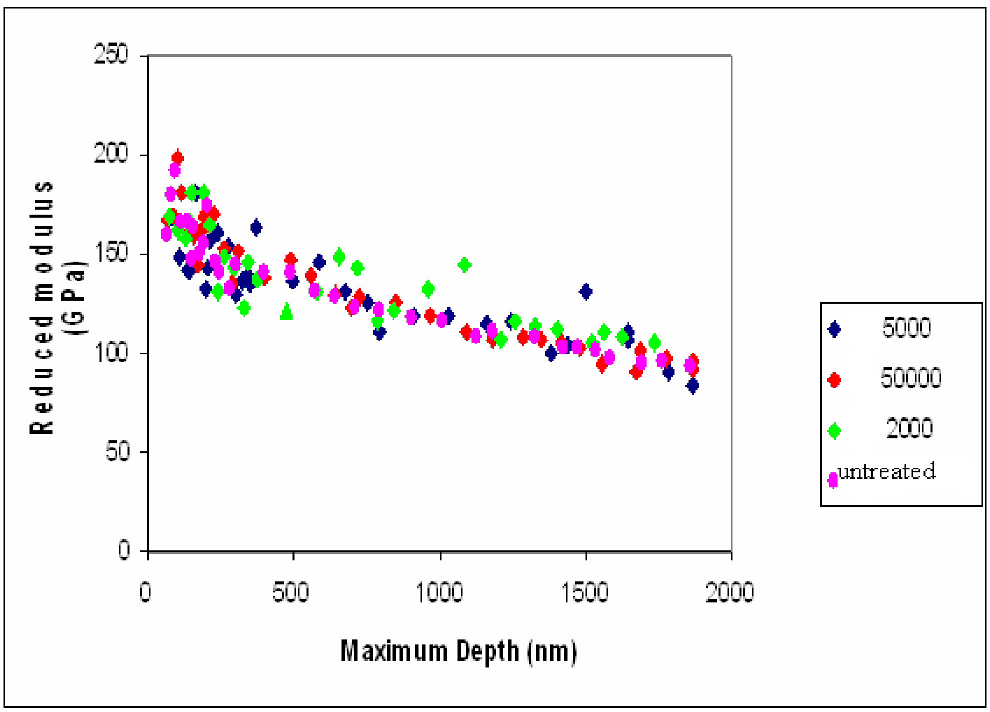


Fig (4) The variation of reduced modulus with maximum depth for the austenitic stainless steel 304 irradiated by Excimer laser 308nm,200Hz, 2.2mJ, at different number of pulses.

Quantitative analysis of stainless steel 304

The variation in mechanical properties such as hardness and modulus was related to the variation in microstructure changes as a function of laser irradiation conditions. Table (4) shows the variation of Grain size, average diameter of the grain, average intercept distance and average number of grains per unit area and unit volume. Quantitative analysis gives us the size, distribution and shape of the microstructure. Fig (5) and Fig (6) show the variation in grain size number and average diameter of the grain with number of pulses respectively. By comparing these figures with hardness-number of pulses curve and Modulus-number of pulses curve the following features appeared:-

* There was good agreement between variations that occurred in the microstructure and the variation in mechanical properties
* Microstructure changed due to laser irradiation.
* All transformations in our study range in solid state

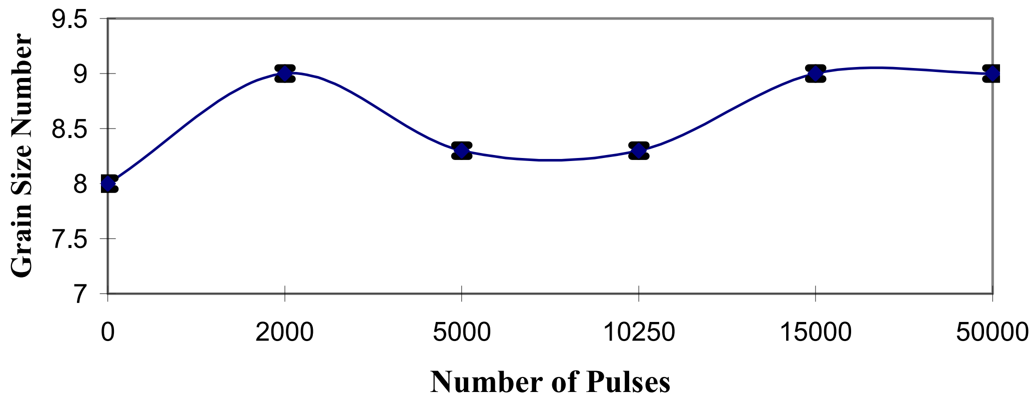
Pulse laser treatment in normal atmosphere is an attractive technique that differs from usual coating methods. A new very thin layer with different microstructure and different mechanical characteristics will be formed on the alloy surface. The laser interaction is the basis for an effective treatment. To induce the chemical-physical reaction with the atmospheric environment high power, short pulse lasers are used.

When comparing these techniques with common surface treatment methods, for instance in the laser nitriding process [1],[2], the depth of nitriding is about 400nm for the iron and stainless steel after 256 pulses. Many aspects of Excimer laser nitriding in iron and steels like the influence of laser fluence and gas pressure**,** the phase formation and thermal stability [4],[3] and the mass transport mechanism[5],[6] make the nitriding process very complicated and less economic.

Other irradiation techniques have many disadvantages over the Gamma radiation doses extended up to more than I Mrad which the samples started to change its color to the brown-black indicated the carbonization or burning [11],[8].

Table (4) Quantitative analysis for stainless steel 304 irradiated by Excimer Laser 308nm, 200Hz, 6m

Micro-grain size measurements	Before irradiation	2000 pulse & 5000	15000pulse & 10250	50000 Pulse
ASTM grain size number	8	9	8.3	8
Average diameter of grain µm	22	16	20	22
Average intercept distance µm	20	14.1	17.7	20
Area of average grain section mm^2 x 10^6	504	225	400	504
Average number of grains per mm^3 x10^6	0.0707	0.2	0.1000	0.0707
Nominal grain per mm^2	1980	4440	2500	1980
Nominal grain per mm^2 at 100x	128	256-287	161.3	128



Fig(5)The relation between Number of Pulses and Grain size Number for stainless steel 304 irradiated by 308nm,6mJ,200Hz

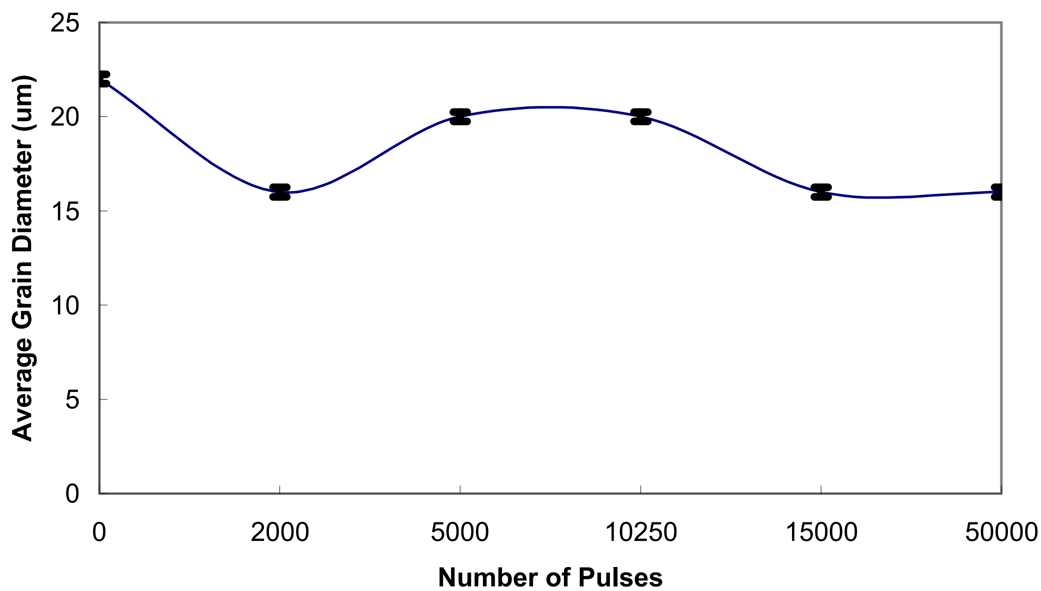


Fig (6)The relation between Number of Pulses and Average Diameter of The Grain(um) for Stainless steel 304 irradiated by 308nm,6mJ,200Hz

The qualitative analysis of stainless steel 304

A wide range of microstructure variations can be obtained as a result of variation in number of laser pulses. Fig (7) shows the variation in microstructure of stainless steel 304 irradiated by Excimer laser 308nm at different number of pulses 2000, 5000, 10250, 15000 and 50000 respectively. The microstructure was compatible with the quantitative analysis mentioned in the above section; laser irradiation leads to change in grain size. At lower number of pulses, grain size number increases and average diameter of the grains decreases (grain refinement). When the number of pulses increased over 10250 pulses, grain growth happened, which is the main reason for the decrease in mechanical properties at higher number of pulses over 10250. Fig (7) shows the variation in microstructure of stainless steel 304 irradiated by Excimer laser 193nm

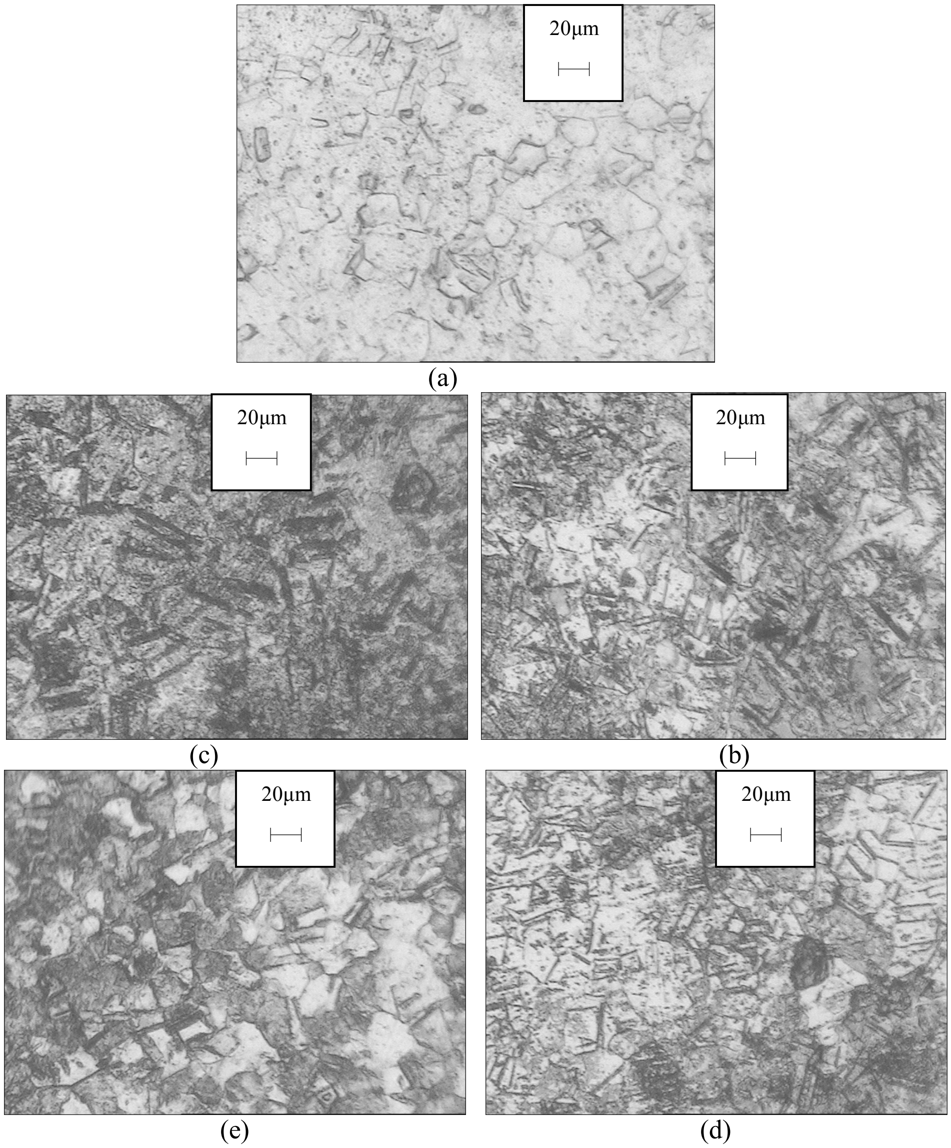


Fig (7) The Effect of Excimer Laser irradiation at 308nm, 6mJ, 200Hz at 300 x Different Number of Pulses on the microstructure of austenitic stainless steel 304. (a) Untreated (b)2000pulses (c)5000pulses (D)15000pulses (e)50000pulses

4. Conclusions

Based on the experimental results observed in this work, the following conclusions may be drawn:

1) The optimized experimental conditions to obtain improvement in the mechanical properties for austenitic stainless steel AISI 304 during laser irradiation in our study range can be summarized as Excimer laser at 308nm, 200Hz, 6mj and 5000pulses.
2) Laser irradiation of austenitic stainless steel is recommended for building elements to improve mechanical properties and enhance surface finish.
3) A significant change in the mechanical properties of the selected alloy is based on the microstructure changes.
4) Laser surface irradiation process is a very complicated process and is found to be affected by the microstructure and chemical composition of the alloys.
5) The mechanical properties such as hardness, modulus of the austenitic stainless steel were improved at the surface of the samples. Hardness and modulus were decreased with both depth and load

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12/12/2011