Structural changes of austenitic steel obtained by 532 nm and 1064 nm Nd:YAG laser radiation

M. I. RUSU^{*}, R. ZAMFIR^a, E. RISTICI, D. SAVASTRU, C. TALIANU, S. ZAMFIR^a, A. MOLAGIC^a, C. COTRUT^a National Institute of R&D for Optoelectronics -INOE 2000, Bucharest-Magurele, P. O. Box MG-5, Romania ^aCEMS-UPB, Bucharest, 063199, Romania

Optimum transfer of laser energy to steel surface ensures characteristic changes of austenitic steel to obtain better performance of the work piece whenever it is submitted to wear and/or high temperature or corrosive media. Laser surface hardening through surface melting is done using a focused or near focused beam. Using this procedure one can obtain fine homogeneous structures due to the rapid solidification rates, little thermal penetration, resulting in little distortion, smooth surfaces, reducing work after processing, process flexibility due to possibilities in automation. In the present work, surface hardening with 532 nm and 1064 nm from Nd:YAG pulsed lasers was done to alter surface features of stainless steel samples. Microstructure characterization was carried out by optical and electronic microscopy. Phase transformation during rapid solidification is analyzed and discussed.

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1. Introduction

The objective of the paper is to study the mechanism involved in the transformation of austenite obtained in the superficial layers that are rapidly melted using pulsed laser radiation, in order to increase the wear-corrosion resistance of the hardened layers.

Stainless steels are generally used in aggressive environments due to their passivation capacity.

Stainless steels from austenitic and ferrite classes are characterized by a high resistance against general corrosion, but their usage resistance is low. Also, these steels are sensitive at inter-crystalline and pitting corrosion (corrosion points). Stainless steels from martensitic class behave well as regarding corrosion and usage conditions, but in certain circumstances, like the use of some special tools, the hardening applied to superficial layer must be stronger.

Temporal evolution of temperature distribution in a system depends on the appearance of heat flows from warmer regions to colder ones. In laser thermal processing, the target is solid, and, therefore, the thermal effects are due to the conduction process. Therefore, the thermal effects of laser radiation must be studied on the basis of heat transfer by conduction. Thermal effects depend on power or energy density of laser beam, and are significant at high density values of laser energy.

The correct choice of the laser beam and its parameters is of high importance for the effect of the laser radiation on the sample surface. The role of interference in standard laser calorimetry was analyzed in [1]. The paper gives the solution of classical heat equation when the solid is subjected to two laser beam irradiation. An efficient method for the increasing of resistance to usage-corrosion consists in the thermal laser treatment.

This treatment has been, recently, applied to stainless steels.

In the past years, a special attention was paid to researches involving material super-hardening technologies, based on phase transformations from liquid to solid phase or on alloying with chemical elements in liquid phase [2].

The methods used for the melting of the steel surface are usually heating and ultra fast cooling methods, which tend to keep the amorphous structure of liquid phase (obtained by superficial melting) after solidification, too [3].

The methods of fast heating are based on the capacity of the concentrated energy flows (laser, electron flow) to heat the steel's surface at over 10^6 degrees / second speed and bringing this surface in liquid state; thus the steel's melting temperature is exceeded. A higher cooling speed than the hardening critical speed of the steel leads to a finer modified layer structure.

2. Experimental

The annealing of material was performed by heating at 1150 $^{\circ}$ C, maintaining an hour at this temperature and water cooling. The resulting structure is an austenitic structure with 210 - 220 μ HV_{0.4} micro-hardness. These samples were exposed to local laser heating or melting.

The experimental researches were performed on Cr-Ni stainless steel, monitoring a study of structural transformations, which are produced by irradiating the surface with two Q-switched solid state Nd:YAG lasers working at 1064 nm and 532 nm wavelengths. Chemical

composition of austenitic stainless steel is presented in Table 1.

| Table 1. C | Chemical | composition | of aus | tenitic | steel. |
|------------|----------|-------------|--------|---------|--------|
|------------|----------|-------------|--------|---------|--------|

| Element | С | Si | S | Р | Mn | Cr | Ni | Mo | Ti |
|-------------|---------|--------|--------|--------|--------|--------|--------|--------|--------|
| Percent [%] | 0.377 | 0.4242 | 0.003 | 0.0371 | 1.2105 | 18.386 | 8.1253 | 0.1826 | 0.0081 |
| Element | Co | Cu | Nb | V | Та | W | Pb | Al | Fe |
| Percent [%] | 0.06979 | 0.2608 | 0.0391 | 0.0898 | 0.0027 | 0.0685 | 0.0011 | 0.0087 | 71.046 |

At fast laser heating, in the attacked layer, a modification of chemical composition of austenitic stainless steel due to chrome diffusion and a complete dissolving of possible carbides are produced.

The used energy densities were compared with those from literature [4] to verify if it was operated in structural modifications field, which indicate hardening induced by laser pulses.

The collected samples were metallographic prepared by their including in bakelite with a special device Buehler type. Afterwards, the samples from this state were exposed to manual polishing.

On these samples, experimental researches of structural modifications, which resulted from superficial layer laser irradiation was developed. For this purpose, some determinations were performed by micro-hardness and optical microscopy.

Laser irradiation equipment consists of two different laser systems:

1. For laser irradiation a laser system (Fig. 1) was used which has an articulated arm, equipped with reflection mirrors so that the output emission energy of laser radiation on 1064 nm is 450 mJ. The power supply and control system of Nd:YAG laser give a laser radiation with repetition frequency in the range of 10 - 20 Hz and 6 - 8 ns pulse width. The mobile arm can be moved over the sample manually.



Fig. 1. Diagram of experimental laser device with mobile arm.

The focusing of laser radiation on 532 nm wavelength (Fig. 2) was achieved by an optical lens or directly on the sample. The samples were placed in front of the laser in a mounting stand in order to ensure the perpendicularity of laser radiation on the sample.



Fig. 2. Block diagram of the experimental set-up.

The laser beam diameter is 6 mm. In this case, the sample is moved in front of laser beam after each exposure, while the laser beam is in a definite position. From 100 to 500 laser pulses were applied on the same area. Laser emission parameters were: 170 mJ energy, 10 Hz repetition frequency and 4-6 ns laser pulse width.

Laser beam monitoring was achieved by preestablished pulses number above each irradiated area. Laser emission energy was measured with a Melles-Griot energymeter for each of 532 nm and 1064 nm wavelengths.

Microscopic analysis and metallographic images were performed by an optical microscope Reichert-Univar with a Polaroid digital camera.



Fig. 3. Image analysis computerized line.

The computerized line for the image analysis consists in (Fig. 3):

- Optical microscope;
- \blacksquare Video camera + adaptor;
- Image acquisition system;
- PC, I.B.M. compatible;
- Printer.

3. Results

3.1. Micro-hardness

The samples were subjected to micro-hardness determinations, on a HANEMANN micro-hardmeter attached to a ZEISS-JENA optical microscope, using a 40 grams load. Using the microscope, the trace diameter imprint in sample by hardmeter prism is determined. Depending on this diameter (d), the micro-hardness is calculated with the formula:

$$H = \frac{1854 \cdot P(g)}{d^2 \cdot n^2} \quad (\mu HV)$$

Where P is the applied load (40 g), and $d = 0.2865 \,\mu m$ is the distance between opposite corners of pyramid base with point angle, between two opposite edges of 136°.

The micro-hardness values are tabulated and represent micro-hardness variation on layer depth.

| Structure | H(µHV), | H(µHV), | Average |
|--------------|---------|---------|---------|
| type | zone 1 | zone 2 | - |
| Dendrite | 250.967 | 231.291 | 241.129 |
| structure | | | |
| Annealing | 259.547 | 273.265 | 266.406 |
| twin | | | |
| structure | | | |
| Unknown | 264.002 | 298.064 | 281.033 |
| structure | | | |
| Non attacked | | 213.842 | |
| zone | | | |

Table 2. Mirror sample, $\lambda = 532$ nm.

Table 3. Sample covered with absorbent carbon, $\lambda = 532$ nm.

| Structure | H(µHV), | H(μHV), | Average |
|-----------|---------|---------|---------|
| type | zone 1 | zone 2 | |
| Dendrite | 334.128 | 321.639 | 327.883 |
| structure | | | |
| Annealing | 326.064 | 330.252 | 328.158 |
| twin | | | |
| structure | | | |
| Unknown | 400.436 | 409.302 | 404.869 |
| structure | | | |
| Non | | 214.312 | |
| attacked | | | |
| zone | | | |

Table 4. Sample covered with absorbent carbon, $\lambda = 1064$ nm.

| Structure | H(µHV), zone 1 (more | | H(µHV), zone 2 | Average |
|-----------|-------------------------|------|-------------------|----------|
| | penetrate | ed) | | |
| Centre | 2 | 255. | 259.547 | 257.375 |
| | 203 | | | |
| Edges | 2 | 242. | 255.203 | 249.0025 |
| | 802 | | | |

In laser annealing, it is well known that the cooling is achieved by thermal conduction. In these terms, the irradiated marginal zone will be cooled much faster than the central zone. This is why the micro-hardenings values are much higher in marginal zone, in which the structure is not obvious at optical microscope, at analyzed size. In the circumstances in which the sample was covered with absorbent carbon, it exhibits higher values of microhardenings. This proves a strong radiation absorption and, therefore, a high temperature of the sample. Increasing the radiation wavelength does not cause a significant growth of micro-hardeness.

3.2. Optical microscopy

Figs. 4a, 4b, 4c, 4d show the images of the structures obtained in the heated area on polished surface using 100 laser pulses (532 nm) (Fig. 4a); 200 pulses (Fig. 4 b); 500 pulses (Fig. 4c) and 100 pulses for sample covered with absorbent carbon (magnification: $\times 100, \times 200$). Fig. 5 shows the structure of a sample with the surface covered with absorbent C and using a Nd:YAG laser emitting on 1064 nm wavelength.



Fig. 4. a (λ=532 nm, 100 pulses, ×100).



Fig. 4. b (λ =532 nm, 200 pulses, ×200).



Fig. 4. (c) (λ =532 nm, 500 pulses, ×200).



Fig. 4. (d) (Sample covered with absorbent C, 100 pulses, \times 200).



Fig. 5. Structures in heated area on covered surface with absorbent C at $\lambda = 1064$ nm with 200 magnification ($\lambda = 1064$ nm, ×200).

4. Discussion

Due to heating and ultra fast cooling speeds, the transformation mechanism occurs so that nanocrystalline and/or amorphous structures are obtained. These structures confer to stainless steels high mechanical and chemical properties.

The critical parameter is the laser energy absorbed by the irradiated system. For comparing the irradiation efficiency of different wavelengths of laser radiations, we must consider the optical properties of irradiated system, too.

Every thermal treatment leads to certain physical and mechanical properties of treated samples, related to the modified material structure.

Regarding to laser radiation power densities range and the number of pulses, another observation is important. At a specified point, the material reaches the melting temperature and the melted material front becomes deeper. Another power density threshold follows when at the surface, the temperature of melting zone increase and closes up to the vaporization one.

5. Conclusions

Laser radiation induces in material a temperature that determines the heating and/or the melting of irradiated zone function on number of laser pulses and energy density. So, for 532 nm laser radiation, with a number of 100 laser pulses a heating is obtained.

When the number of laser pulses increases from 200 up to 500, the melting zone grows without reaching vaporization. In this zone one observes a dendrite structure as a result of solidification. At the edges of this zone appears another zone in which the dendrite aspect of solidification is not evident. In order to paint out this feature an electronic microscope was used. In heated zone, the crystallized austenite structure appears.

If the material surface is covered with a layer of absorbent carbon before the irradiation in order to increase the absorbance of irradiation, we observe that even at a small number of pulses (100), a heating leading to melting occur.

For the longer wavelength of laser radiation (1064 nm) the heating process causes the melting more rapidly and the resulted structure has a dendrite aspect with the ferrite separation δ .

For determination of hardness, a Hanemann hardener attached at an optical microscope type Zeiss-Jena using a charge of 40 grams was used.

The micro-hardening values show an obvious increase from the base mass structure. This modification determined by the heating and cooling processes depends on the heating temperature speed reached by laser radiation and the cooling speed.

More laser irradiations in different conditions are needed to find the optimum laser regime in order to obtain a good hardness for specific steel samples.

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* Corresponding author: madalin@inoe.inoe.ro