Microstructure and Cavitation Erosion Resistance of the X2CrNiMoN22-5-3 Duplex Stainless Steel Subjected to Laser Nitriding

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The cavitation behavior of the Duplex stainless steel X2CrNiMoN22-5-3 was improved by modifying the structure of the surface layer, using the laser nitriding procedure. There have been used different impulse power for the laser beam (from 120W to 240W) for constant impact times. The cavitation erosion tests were effectuated in the Timisoara Poytechnic University Cavitation Erosion Laboratory with the T2 device, which respects all the conditions imposed by the ASTM G32 Standard. There were compared two types of specimens: those laser treated and those subjected only to the conventional solution treatment (heated at 1060 °C and cooled in water). The eroded surfaces were analyzed through hardness measurements, optic microscopy and scanning electronic microscopy (SEM). After a total cavitation exposure of 165 min, the specimens laser nitridet present a reduction of 3.23 till 5.67 times of the mean depth erosion and from 3.03 till 5.26 times of the cavitation erosion rate in comparison with the specimens treated only with solution treatment. This huge improvement is given by the microstructure of the superficial layer enriched in nitrogen.

Keywords: cavitation erosion, laser nitriding, duplex stainless steel, microstructure

As a result of their good mechanical characteristics and the excellent corrosion resistance the Duplex stainless steels are used on a large scale in the chemical and petrochemical industry, for offshore constructions, nuclear industry, food factories etc. [1, 15]. In comparison with austenitic stainless steels, the Duplex ones have an increased concentration in chromium and a reduced concentration of nickel. The most important alloying elements are Cr, Ni, Mo and N. The chromium and molybdenum favorize the ferrite formation while nickel and nitrogen stabilize the austenite. Some Duplex brands contain also other elements such as Mn, Cu or W.

The alloing elements Cr, Mo and N increase the corrosion resistance, especially for spot and cracking corrosion, in environments with Cl ions [14]. The nitrogen has also an important role in the mechanical resistance increase. The Duplex stainless steels have a greater corrosion stability than the austenitic steels X2CrNiMo17-12-2 (AISI 316L) and a 150% greater flow limit [10]. The constant uprising of the nickiel cost, in the last years, motivate the the increase of the use of this kind of stainless steels. Even if numerous systems in the mentionate industries are subjected to cavitation erosion there are few informations upon the cavitation behavior of these steels to this kind of stresses [5]. Some researchers reported an increased cavitation erosion with cracks and pitts initiated at the boundaries between the two phases austenite and ferrite [3, 5, 9, 13].

The present researches propose the use of laser beams for the nitriding thermochemical treatment with the purpose to modify the superficial layer of a duplex stainless steel X2CrNiMoN22-5-3 in order to improve the cavitation erosion resistance. This treatment represent an attractive technological way for the improvement of the tribologic properties, by modifying only the superficial layers which receive an increased hardness without modifications in the material core. For transversal velocities of the laser beam with approximate 100 mm/s [16, 17] there is an ample and rapid dissolution of the nitrogen in the melted superficial layer. In order to compare the cavitation erosion resistance, the tests were carried out both for samples having only normal solution treatments with heating at 1060°C and cooling in water as well as for samples nitrided with laser beams.

Tested material and the Used Laboratory Device

The researched material is the 2205 Duplex stainless steel symblolyzed X2CrNiMoN22-5-3 by European norme EN 10088 and UNS S31803 after ASTM A276. In table 1 are given the chemical composition and in table 2 the mechanical charcteristics.

The material was delivered as hot rolled bars having a diameter of 30 mm. From the bars were realized cylindric specimens (Φ20 x 60 mm) subjected to a solution treatment (heated at 1060°C and cooled in water. Subsequent the specimens were subjected to laser nitriding

Table 1
CHEMICAL COMPOSITION OF THE ANALYZED MATERIAL

Material	Chemical composition, % mass								
	C	Si	Mn	Cr	Ni	Mo	P	S	N
X2CrNiMoN22-5-3	0.017	0.72	1.8	22.08	5.02	2.9	0.021	0.012	0.16

Table 2
MECHANICAL CHARACTERISTICS AFTER SOLUTION TREATMENT

Yeld point, $Rp_{0,2}$ [N/mm^2]	Tensile strength, Rm [N/mm^2]	Fracture elongation, A5 [%]	Fracture constriction, Z [%]	Hardness, HB, [daN/mm^2]
545	736	28	52	275

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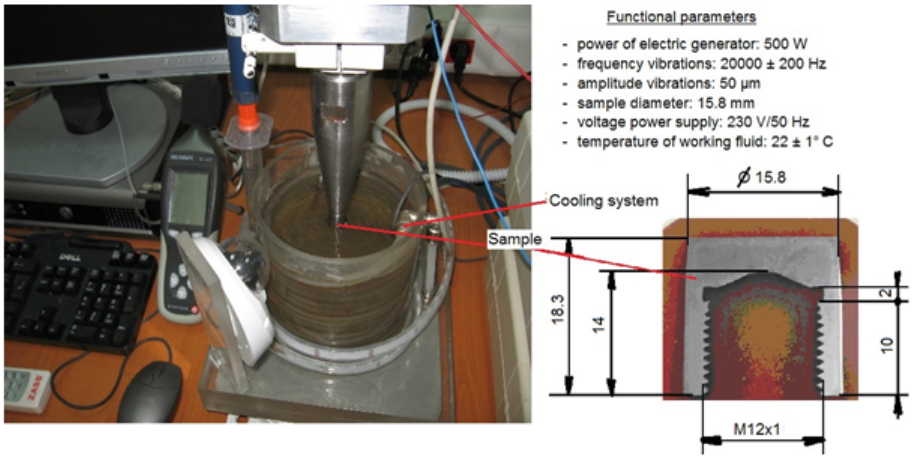


Fig.1 The vibratory cavitation device (left) and a section through the specimen exposed to cavitation (right)

treatment on a station Trumpf HL 124 P LCU. This treatment targeted the formation of a superficial layer with increased hardness and good behavior to cavitation erosion. The laser beam scan the specimen active surface with a velocity of 4.07 mm/s, during 10 ms in an atmosphere of pure nitrogen with a flow capacity of 33 l/min. There were used different impuse powers, as follows:

- the first set was exposed at 240 W;
- the second set was exposed at 180 W;
- the third set was exposed at 120 W.

For the cavitation erosion testings was used a vibratory device with piezoelectric crystals realized respecting the Standard ASTM G32- 2010 [18] (fig.1).

Before introducing the specimen in the cavitation erosion device the activew surface was polished at $R_a = 0.2 \div 0.8$ µm, with the help of an installation Buehler Phoenix Beta. In conformity with the Cavitation Erosion Laboratory customes [2-4, 13-14], the total exposure time was of 165 min divided in two periodes of 5 and 10 min and the following of 15 min each.

On the basis of the recorded mass losses Dm_i, measured after each testing period *i*, was determined the cumulative mass losses m_i, with the relation:

$$m_i = \sum_{i=1}^{12} \Delta m_i \quad (1)$$

The experimental values for the MDE (mean depth erosoion) as well as the MDER (mean depth erosion rate) were determined with the relations:

- for the cumulative mean depth erosion:

$$MDE_i = \sum_{i=1}^{12} \left(\frac{4 \cdot \Delta m_i}{\rho \cdot \pi \cdot d_p^2}\right) \quad (2)$$

- for the mean depth erosion rate:

$$MDER_i = \Delta MDE_i / \Delta t_i \quad (3)$$

where:

i - is the testing periode;

Δm_i - is the mass lost for the periode i, measured in grammes;

ρ - is the material density in g/mm^3;

Δt_i - is the exposure time of the period *i* (5 min, 10 minutes or 15 min);

d_p – is the diameter of the area exposed to cavitation (d_p = 15.9 mm); this convention is recommended by ASTM G 32 because the difference between the eroded area and the total one is wery small and the diameter can be easy measured, with great precision;

ΔMDE_i - Is the value of the erosion mean depth penetration in periode Δt_i.

The curve approximating the experimental values is established with the analytical equations presened in [3], [12]. The used relations are:

- For the mean depth erosion:

$$MDE(t) = A \cdot t \cdot (1 - e^{-B \cdot t}) \quad (4)$$

- For the mean depth erosion rate:

$$MDER(t) = A \cdot (1 - e^{-B \cdot t}) + A \cdot B \cdot t \cdot e^{-B \cdot t} \quad (5)$$

where:

A - is a scale parameter, statistically established from the condition of the minimum scatter;

B - is the curve shape parameter.

For intermediate exposure times, as well as for the final one (165 min), the eroded surfaces were analyzed both with the stereo microscope OPLIMPUS SYX7 and the scanning electron microscope. Subsequent the specimens were longitudinally sectioned (fig.2) and metallographically prepared for the examination of the marginal layer in which appear and are propagated the cavitation erosion cracks and pits.

The examination have been made both with a Leica DM2700M optic microscope and with a scanning electronic

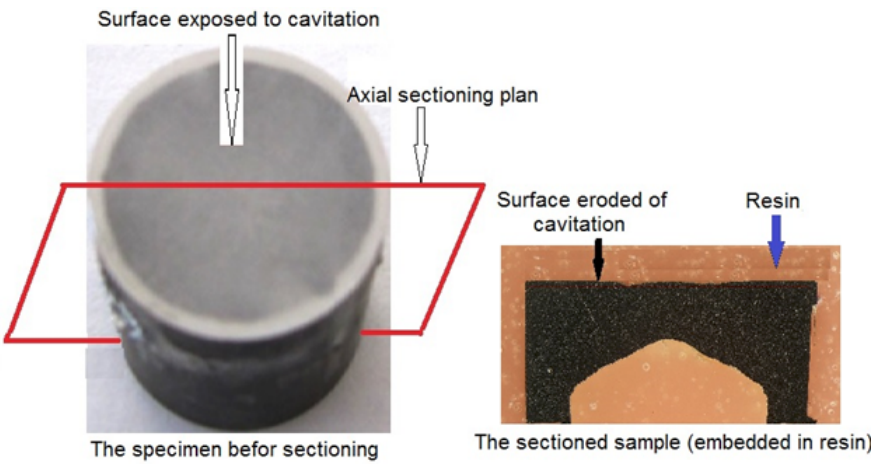


Fig.2 The specimen prepared for microscopy

microscope. It was also measured the variation of the microhardness Vickers (HV0.2) at different distances from the surface.

Results and discussions

Material structure and hardness

Figures 3 and 4 presents the surface appearance of the laser nitrided specimens and the cross section structure through the nitrogen enriched layer.

From these figures it can seen the ondulated and jagged look of the surface, characteristic for laser processing (fig. 3) but also the absence of the metallic continuity defects between the layer and the sublayer (fig. 4a). In the same time we can see a good metallurgical connection between the layer and the sublayer (fig. 4a).

The core microstructure (fig. 4b) put into evidence the presence of austenite islands into a ferrite matrix. The thermochemical treatment with laser beam determine modifcations in the quantitative ratio of the two phases (F/A) enriched in nitrogen as well as the redistribution through diffusion of the alloying elements between them. The austenite proportion of the nitrided layer depends essentially on the carbon and nitrogen content and on the relative quantities of the alphagene respective gammagene elements. The SEM examinations (fig. 5a) show an epitaxial grows of the crystalline grains and a delay by the nitrogen of the preicipitation phenomenon of the secondary phases with a hardening and a embrittlement role. In the same time, the redistribution phenomenon of alphagene or gammagene alloying elements is limited (fig. 5b) because the great cooling velocity eliminate their diffusion, in great measure. The increased solubility of the nitrogene in austenite promote the formation of these phase and delay the formation of the intermetallic phases. Supplementary, the nitrogene substantially increases the mechanical strength as a result of the hardening by solid solutions. The greatest effect upon austenite hardening is given by the interstial dissolved elements (C and N); they are followed by substitionaly dissolved elements, with alphagene character (Mo and Si); the smallest effect is given by the gammagene elements (Mn and Ni).

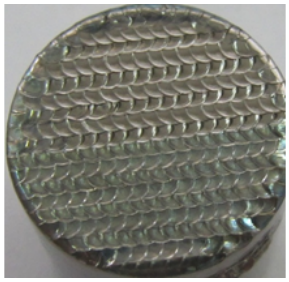
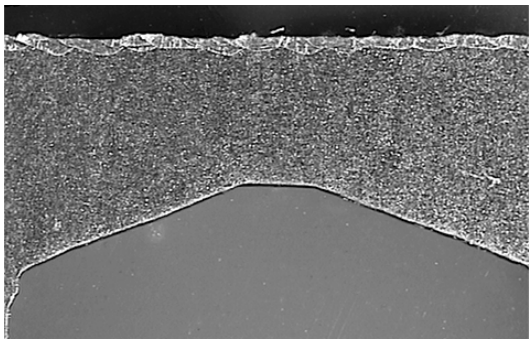
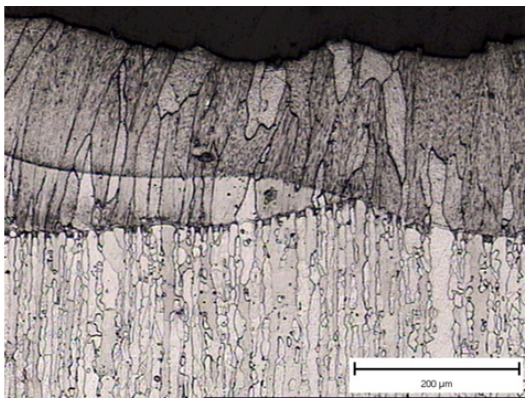


Fig. 3 View of the laser nitrated surface with 240W

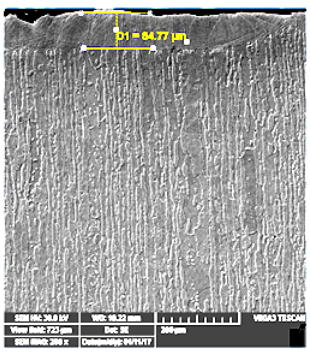


a

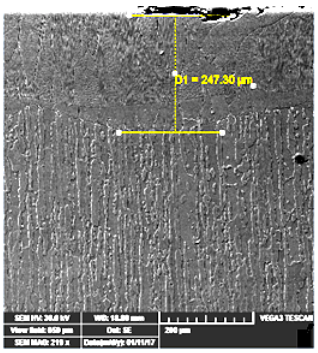


b

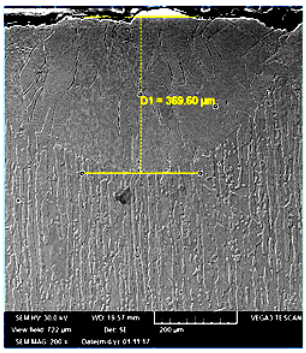
Fig. 4 Structure of the longitudinal section through a laser nitrided samples: a – x 40; b – x 200

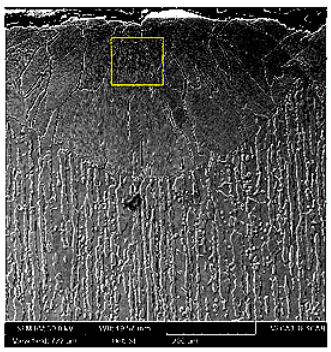


P = 120 W



P = 180 W





P = 240 W

a

Fig.5 Microstructure of the system layer-sublayer (a) and the chemical composition of some crystalline grains (b)

	Series	unn. C [wt.%]	nor. C [wt.%]	Atom C [at.%]	Error (1 Sigma) [wt.%]
Silicon	K series	0.52	0.53	0.92	0.07
Chromium	K series	21.41	21.71	20.35	0.60
Manganese	K series	0.78	0.80	0.71	0.06
Iron	K series	63.12	64.00	55.85	1.65
Nickel	K series	4.49	4.55	3.78	0.17
Molybdenum	K series	3.60	3.65	1.86	0.42
Nitrogen	K series	4.69	4.76	16.55	1.44
	Total	**98.63**	**100.00**	**100.00**	

b

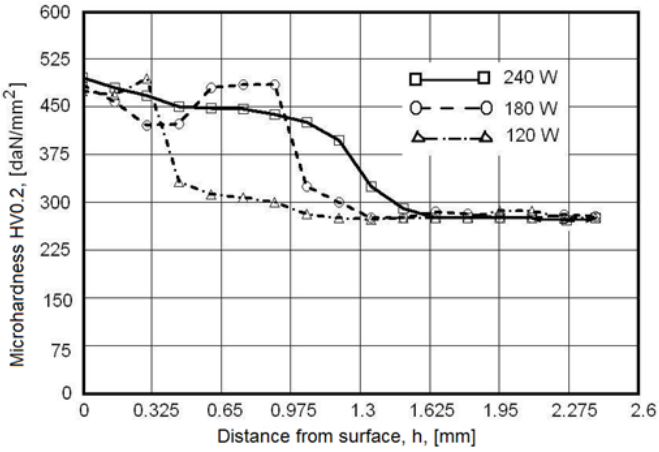


Fig.6 The microhardness variation in the nitrated layer, for three values of the laser beam power

By changing the power of the laser beam from 120 W to 240 W increase the depth of the nitrided layer from approximate 84 μm at approximate 369 μm (fig. 5 a).

In figure 6 are presented the hardness variation in the crossection of the nitrided layer, for all three power values of the laser beam. It can be seen that the hardness near the surface (0…0.65 mm) has values of HV0.2 = 480 – 500 daN/mm^2 while the core has similar values with those obtained through quenching HV0.2 = 280 – 290 daN/mm^2.

Cavitation erosion resistance

In figure 7 is presented the MDE (mean depth erosion) evolution with the increase of the exposure time and in figure 8 the evoulution of MDER (t), for both the nitrided and not nitrided specimens. Regardless of the power used (120 W, 180 W or 240 W), it can be seen that the laser nitriding give remarkable cavitation erosion resistance in comparison with solution treatment.

The main observations that come out from these figures are:

-the mediation curves of the experimental results have shapes and aspects obtainable only for materials with very good or excellent cavitation eosion resistance;

-regarless of laser beam regime, the dispersion of the experimental points is very reduced which means that the surface exposed to cavitation is homogeneous from the point of wiev of the qualities responsible for cavitation erosion resistance;

-the maximum erosion rate appear in the interval 60…70 min of exposure after which the MDER remain at the same

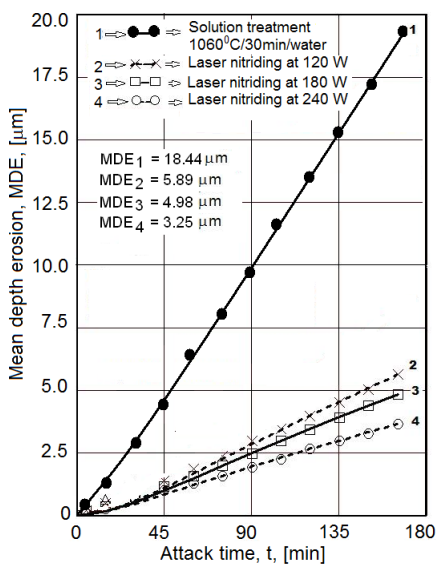


Fig.7 The evolution of erosion penetration depth against the cavitation time exposure

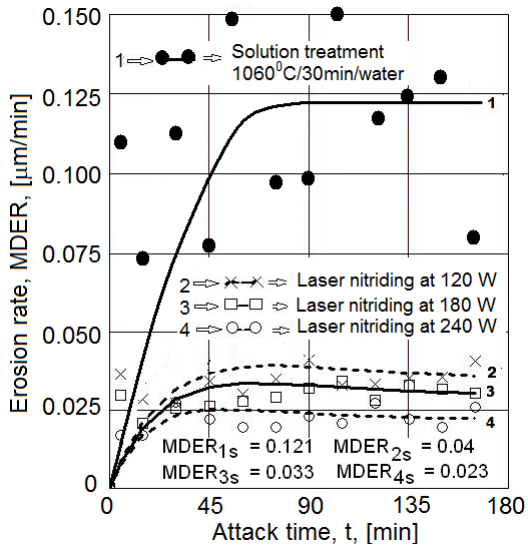


Fig.8 Mean depth erosion rate against time exposure to cavitation

value; it means that the length of the exposure time was correctly chosen as well as the laser treatment (impulse extension and frequency);

-in comparison with the specimens subjected only to solution treatment the laser beam treatment favorize an important reduction of the MDE (with about approximately 213% for 120 W power regime, about 270 % for 180 W and about 467 % for 240 W) and also MDER (with about 203 % for 120 W, about 267% at 180 W and about 426 % for 240 W);

-the increase of the impuse power from 120 W to 240 W diminishes MDE with about 81% and MDER with about 74%.

The analyze of the eroded surfaces during cavitation (fig. 9) justifies the elements responsible for the performance increase of the cavitation erosion resistance for the nitrided specimens.

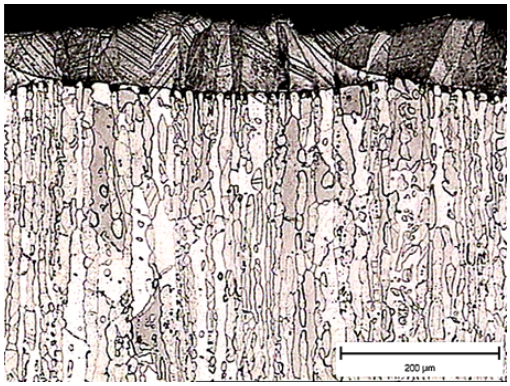


Fig.9 Micrographic images of the longitudinal section through the nitrided samples after the maximum time exposure (165 min)

The increase of cavitation erosion resistance can be attributed to the hardness increase and to the enrichment in nitrogen of the marginal layer. The cavitation erosion of metallic material is the result of the huge pressures during the bubble implosions as well as to a fatigue determined by the multitude of implosions [4, 19]. The cavitation erosion resistance depends on the manner in which the material takes the impacts with either the microjets or the huge pressures of the numerous bubble implosions, in condition of triaxial and repetitive loadings. The take over of those energies generate the increase and propagation of craks, localizate fracture and finally mass losses [3, 6, 8]. Because the researched Duplex stainless steel has an austenite-ferrite microstructure the resistance to cavitation erosion depends both on the mechanical resistance and

the differet ways in which the two phases and their interfaces are plastic deformed during this process [5]. The results presented in the papers [1, 14, 15] show that in the first stages of erosion both phases are deformed till the ferrite receive the maximum capacity of hardening while the austenite phase is hardened and increase the deformation while receiving the energy. As a consequence the sliding strips are activated (fig.9) and cracks are triggered at the interface austenite-ferrite. As a consequence, if the process continues, the cracks appear initially in ferrite and after that also in austenite [14]. As a result in the ferrite grains the losses are produced more rapid that in in the austenite ones [5, 11]. Even if in the present work this behavior was not evaluated, it is nevertheless present, so the increase of the austenitic fraction justify the improvement at cavitation erosion. Some researchers belive that the resistance to cavitation erosion increases continuously by diminishing of the dimensions of the crystalline grains [3, 7, 9, 10]. This reduction of the crystalline grains dimensions has an impact upon the mechanical resistance, through a Hall-Petch effect, and is in concordance with the hardness measurements see figure 6. This increase of the mechanical properties also contributed to the cavitation erosion performance.

All the mentioned characteristics cooperate at the improvement of cavitation erosion resistance of Duplex stainless steels because both the plastic deformation and the fracture is conducted by the microstructure.

Conclusions

The obtained results show that nitriding thermochemical treatment with laser beams is a promising alternative to improve the performances of the pieces working in cavitation erosion conditions.

In comparison with solution treatment, the laser treatment has a great influence upon the reduction of the final erosion depth (with 213% for the 120W regime, with 270% for 180W and 467% for 240W) as well as for the erosion rate (with 203% for the 120W regime, with 267% for 180W and 426% for 240W).

This great behavior improvement is the result of the modification of the austenite fraction enriched in nitrogen which has a greater hardness.

The results of the hardness measurements are in agreement with the cavitation erosion improved behavior for the two states of the specimen treatments.

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