TIMISOARA POLITEHNICA UNIVERSITY

MECHANICAL FACULTY

Cavitation erosion behavior of Duplex stainless steels

-SUMMARY-

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Summary

Objectives of the doctor degree thesis:

- profound studies about the cavitation erosion phenomena, especially for establishing new technological modalities to rise the resistance of the Duplex stainless steels by applying volume and superficial heat treatments (quenching at temperatures between 1000 °C and 1100 °C, sensitizing annealing at two critical temperatures, 475 °C and 850 °C, nitration in gaseous environment or with laser beams, covering with composite powders and melting with high energy beams
- generating new testing methods to obtain a more precise control of the cavitation evolution especially in order to reduce the risk of damages
- the morphology and the characterization of microstructures fir the areas exposed to cavitation erosion
- to device of a mathematical model in order to a better description of the cavitation characteristic curves.

<u>Purpose of researches</u>

To put into evidence the importance of different applied treatments to assure an increased resistance to cavitation erosion of the Duplex stainless steel X2CrNiMoN22-5-3.

The novelty of the doctoral thesis consist in founding a **correlation** between the structural state of the material and the degradation velocity through cavitation erosion (expressed through the parameter "$\frac{1}{MDER}$" which represent the inverse of the erosion mean depth penetration), as well as the optimization of the fabrication in order to increase the life of the details which work in such conditions.

1. ACTUAL STAGE OF RESEARCHES REGARDING THE CAVITATION EROSION OF DUPLEX STAINLESS STEELS

In equilibrium state, the microstructure of Duplex stainless steels contains approximate 50% ferrite and 50% austenite, being sometimes named austenite-ferrite steels. This material present a great stability at corrosion under stresses in liquids containing chlorine ions and an excellent stability to pointed corrosion. Simultaneously this material has high values for mechanical resistance (yield limit and breaking limit). The most important alloying elements are Cr, Ni, Mo and N [110], [115]. Chromium and molybdenum determines the formation of ferrite having a cubic crystalline lattice with body centered volume (c.b.c.) while nickel and nitrogen determines the formation of austenite having a cubic crystalline lattice face-centered (c.f.c). The alloying elements Cr, Mo and Ni determine an increased corrosion resistance, especially point corrosion, tenso-cracking in chlorine ions environments. Nitrogen has an important role in the increase of the mechanical characteristics. In the same time nitrogen increases the temperature at which austenite begin to be formed from ferrite and favorites the precipitation of the chromium nitrates at the grain interfaces ferrite-ferrite and ferrite-austenite.

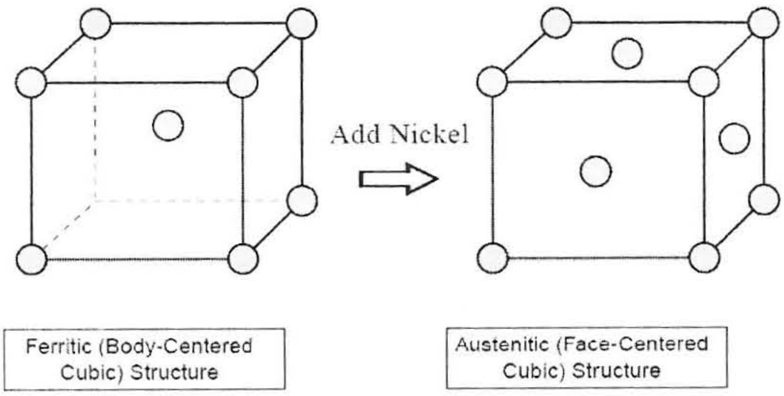


Fig.1 Crystal lattice modification after alloying with Ni

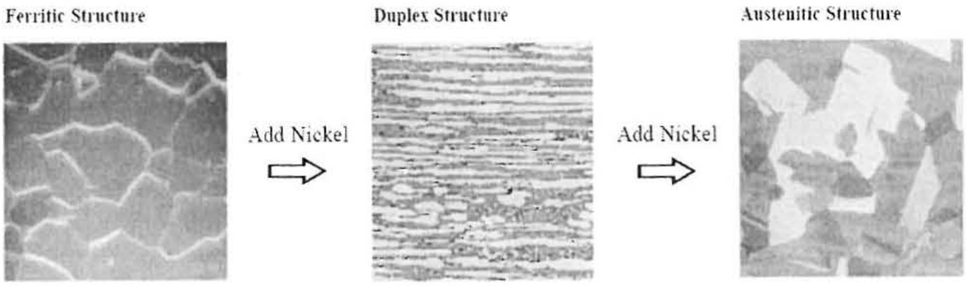


Fig. 2 Microscopic structure modification after alloying with Ni

As a result of the good mechanical properties and excellent corrosion resistance in various environments and running conditions the Duplex stainless steels are employed in numerous industries, such as:

- those in which the cavitation erosion phenomenon is present especially hydro mechanical (turbine runners and blades, pump impellers, marine and river ship propellers);
- offshore drilling equipment;
- seawater desalination plants;

- installations for chemical industry (transport and storage of chemicals);
- food industry equipment.

Fig. 3 Domains in which stainless steels are employed

2. RESEARCHED MATERIAL. EXPERIMENTAL PROCEDURE

The material investigated in the present work is the stainless steel Duplex 2205, having the symbol X2CrNiMoN22-5-3 in conformity with European normative EN 10088 and UNS S31803 after ASTM A276.

Table 1

Mechanical characteristics in the status of delivery

Yield limit $Rp_{0,2}$ [N/mm²]	Tensile strength Rm [N/mm²]	Elongation A5 [%]	Rupture constriction Z [%]	Hardness HB [daN/mm²]
545	736	28	52	275

Chemical composition % of mass								
C	Si	Mn	Cr	Ni	Mo	P	S	N
0,017	0,72	1,8	22,08	5,02	2,9	0,021	0,012	0,16

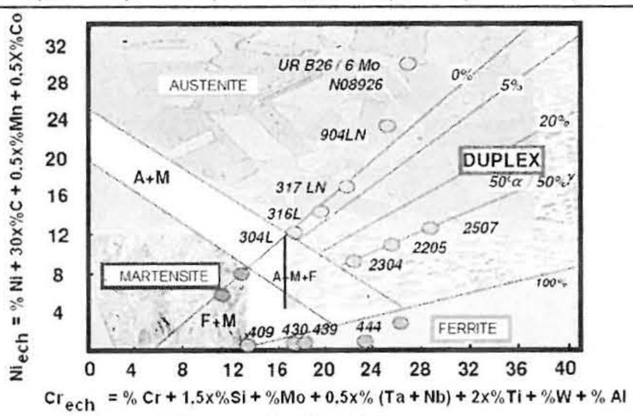


Fig.4 Schäfler Diagram

From this material have been realized cylindrical specimens (fig. 2.3) which were subjected to the following heat treatments:

- Solution treatment;
- **Solution treatment** followed by annealing for sensitize at two critical temperatures;
- nitration in gaseous environment and with laser beam;
- heat spraying with great speed flame, HVOF followed by remelting with laser beam.

The specimen was subjected to cavitation erosion in a vibratory device with piezoceramic crystals in the Cavitation Laboratory of Timisoara Polytechnic, following the rules adopted by the Laboratory.

Fig. 2.3 The specimen during the cavitation erosion procedure

3. VOLUMIC HEATH TRETMENTS AND CAVITATION EROSION RESISTANCE

From the investigated steel have been manufactured several lots of three specimens with a diameter of 15,9 mm which were subjected to various heat treatments (heated and maintained 30 min at 1060°C, followed by cooling in furnace or rapid cooling in water). Afterwards, some specimens cooled in water were subjected to sensitizing annealing either at 475°C during 4 hours, either at 850°C during 2 hours with subsequent cooing in air. The purpose was to find the effects of transformation or precipitations, in solid state, upon the cavitation erosion resistance.

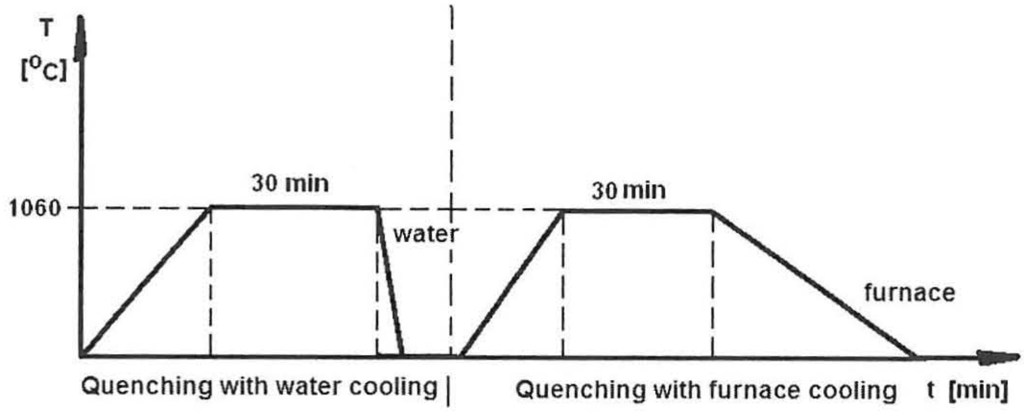


Fig. 5 Heat treating diagram

Table 2 Heating history of tested specimens

	Solution treatment			Awareness annealing		
	T [°C]	t [min]	Cooled	T [°C]	t [min]	Cooling
SC1	1060	30	water	-	-	-
SC2	1060	30	oven	-	-	-
S1	1060	30	water	475	240	air
S2	1060	30	water	850	120	air

History of the specimen heat subjection

After the applied heat treatments on the generating line of the specimen has been a realized hardness measurement (between 8 and 10 measuring points). It was found out that both se sensitive treatments produce an increase of the material hardness in comparison with the structural state obtained after the initial quenching. If after the quenching the hardness values are 270...280 HV1 after the heating at 475°C the hardness became 325...335 HV1 and with heating at la 850°C, the values became 358...365 HV1.

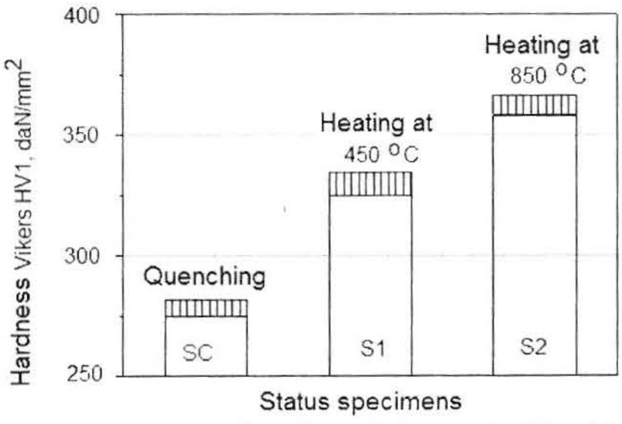
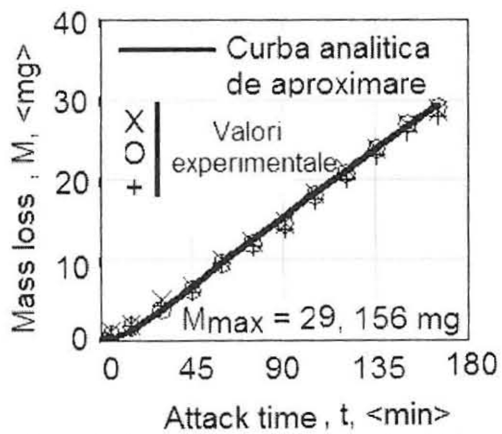


Fig. 6 Hardness histogram function of the applied heat treatment

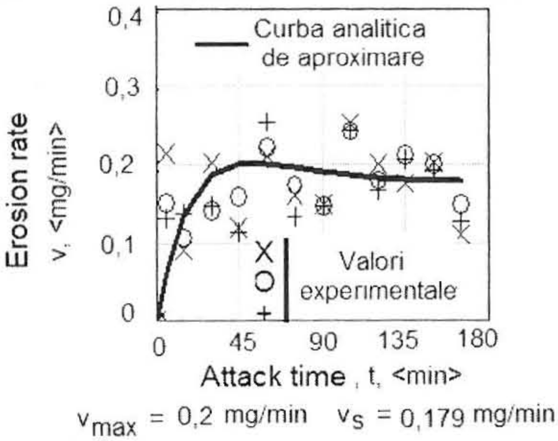
The use of „cavitation characteristic curves" are of extreme importance because their shape and the scatter of the measured points give excellent information about the material behavior during the exposure to cavitation. For the construction of the curve were used the mean values of the mass losses for all three tested specimens for each intermediary testing time. From the presented curves it can be seen that the smallest losses were obtained for the quenched specimens subjected to an annealing at 850°C during two hours and cooled in air.

Comparisons between mean depth erosion curves obtained with furnace cooling or cooled only in water, suggest that in the first exposure interval the cavitation attacked surface has an apparent greater resistance for the first procedure. But the cavitation erosion with significance is the linear zone (after 45 minutes of exposure, together with the tendency in which they reach this stabilization zone) where the cavitation erosion resistance is almost identical regardless of the cooling variant adopted (after an exposure of approximate 120 minutes the stabilizations rates are so close, that they can be considered as being in the same error interval).

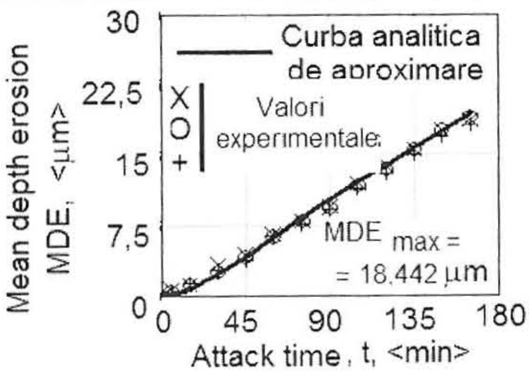
General example



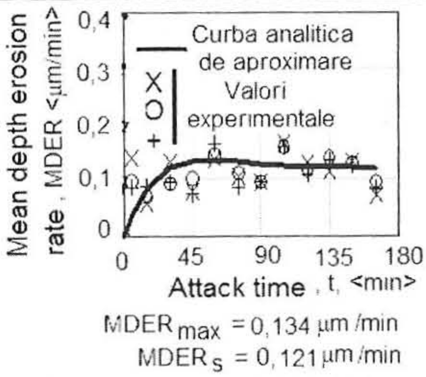
Cumulative mass losses against time



Velocity of mass losses against time



Mean depth erosion against time



Mean depth erosion rate against time

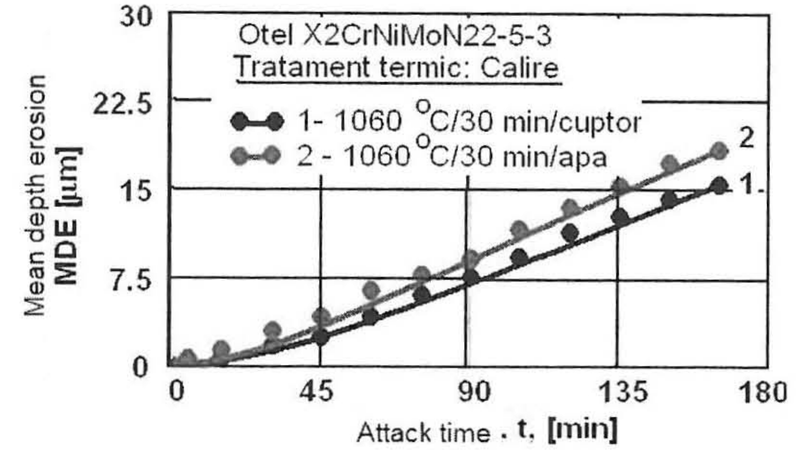


Fig. 11 Mean depth erosion against time exposure

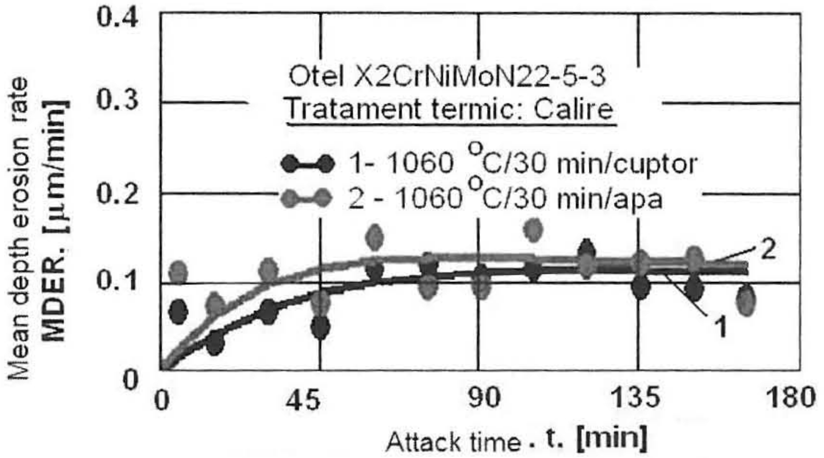
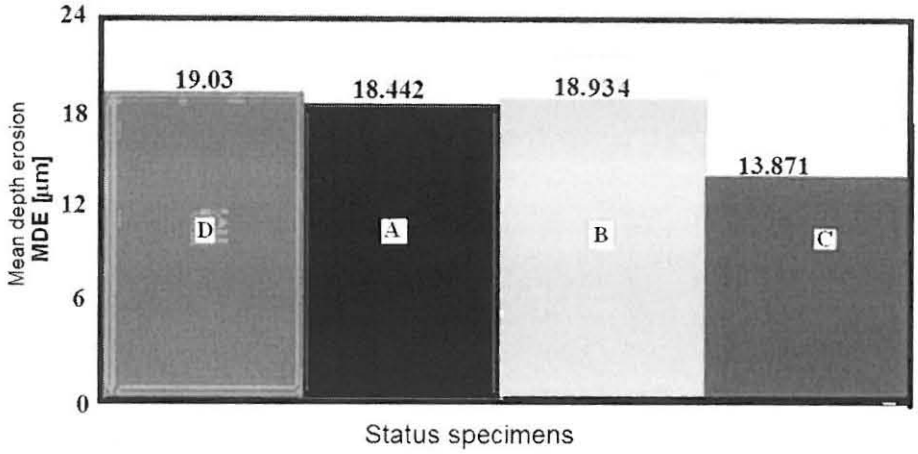


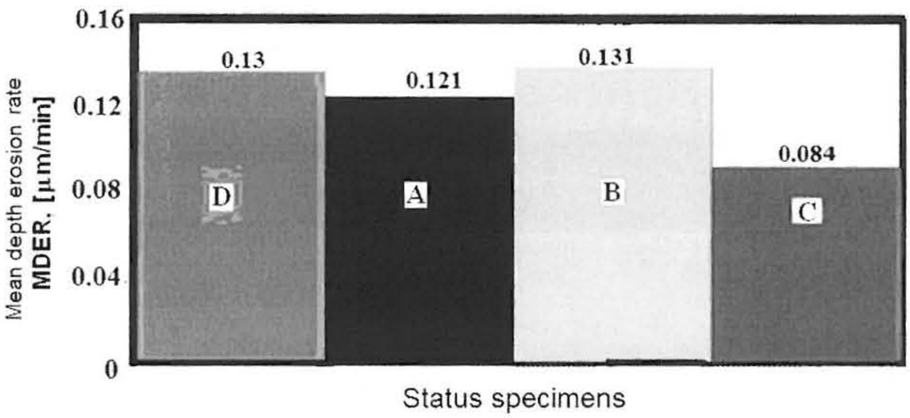
Fig. 12 Mean depth erosion rate against time

The scatter of the experimental values with regard to the characteristic curves is given by the influence of the uneven microstructure upon the cavitation erosion attack.

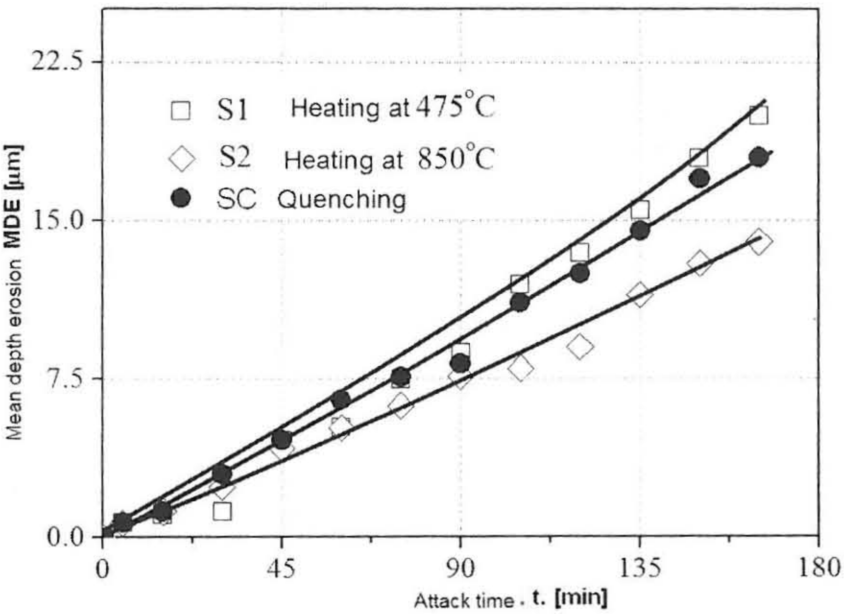
Comparisons between the final depth of cavitation erosion for the steel X2CrNiMoN22-5-3 subjected to different heat treatments and the steel 41Cr4 (material used with success in different technical applications) considered as standard in the Cavitation Laboratory of Timisoara Polytechnic University show that all the new tested steels have better behaviors. The most favorable results were obtained with the steel X2CrNiMoN22-5-3 quenched and annealed at 850°C with furnace cooling.



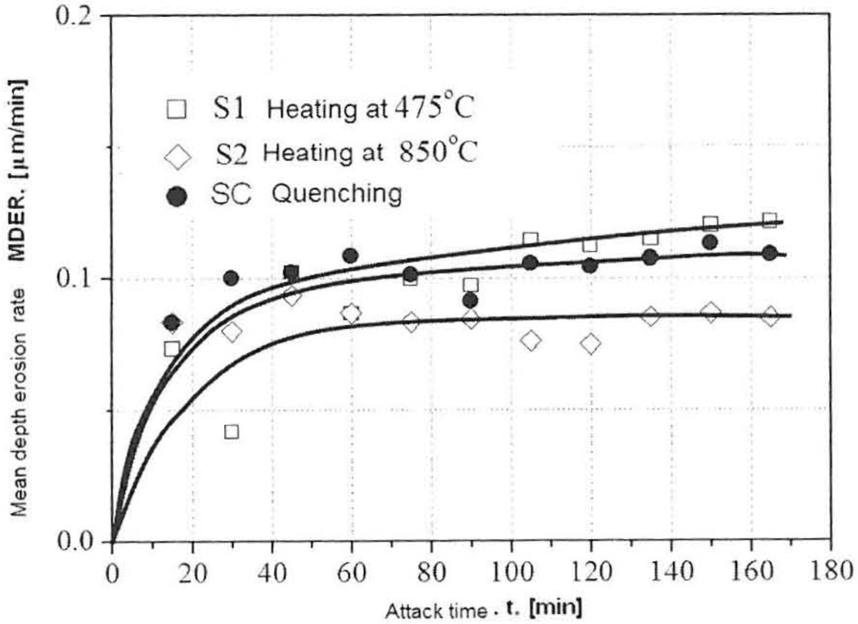
A - Oțel inoxidabil Duplex X2CrNiMoN22-5-3 călit 1060°C /30 min/apă;
B -Oțel inoxidabil Duplex X2CrNiMoN22-5-3 călit 1060°C /30 min/apă + sensibilizare 475°C/4 h/ aer;
C -Oțel inoxidabil Duplex X2CrNiMoN22-5-3 călit 1060°C /30 min/apă + sensibilizare 850°C/2 h/ aer;
D - Oțel aliat pentru imbunatatire, 41Cr4, considerat ca etalon.



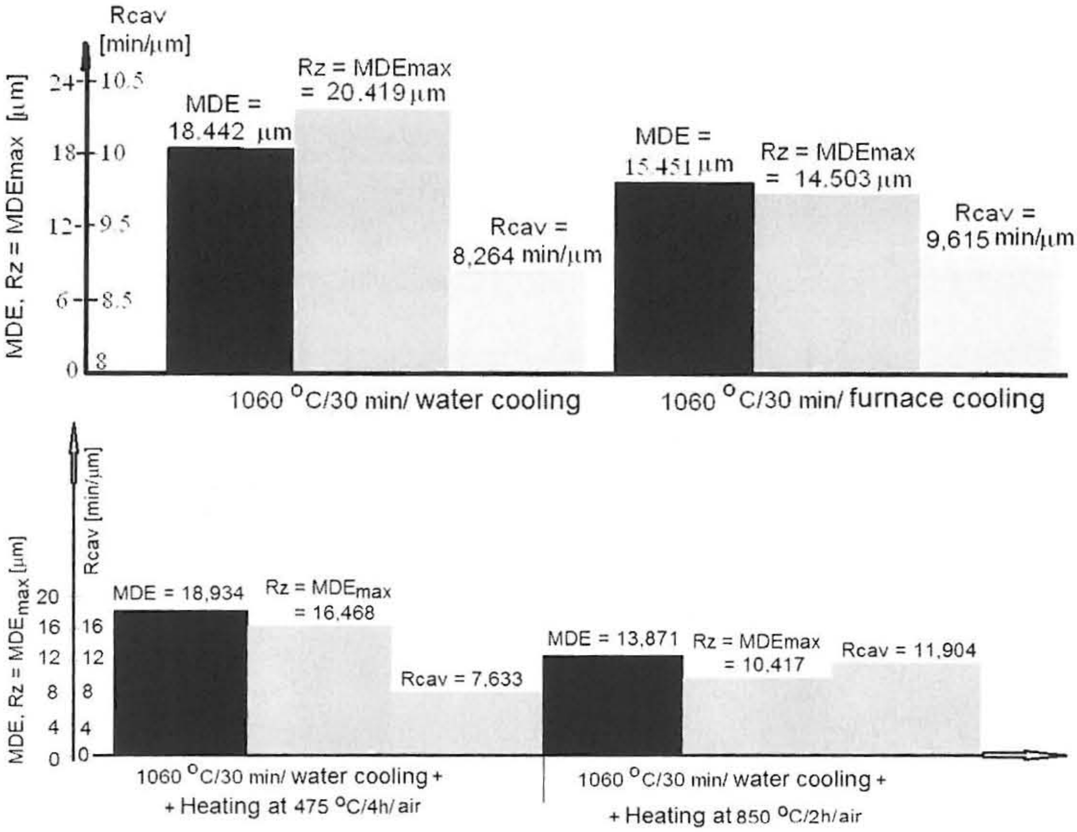
It can be seen that for the final attack (165 minutes), for the annealing at 850°C it was obtained a reduction with approximate 11% of the maximum value of the erosion depth and with approximate 28% of the maximal erosion velocity. On the other hand using only 475°C, even if the hardness is increased as a result of the spinoidal decomposition of ferrite in domains with poor in α, but rich in α'(with increased fragility) deteriorate the resistance to cavitation erosion. Our data show that appear an increase with approximate 22% of the maximum depth of the erosion and with approximate 21% of the maximum erosion rate with regard to the solution treatment.



S1 Heating at 475°C
S2 Heating at 850°C
SC Quenching
Mean depth erosion MDE [μm]
Attack time, t. [min]
22.5
15.0
7.5
0.0
0
45
90
135
180

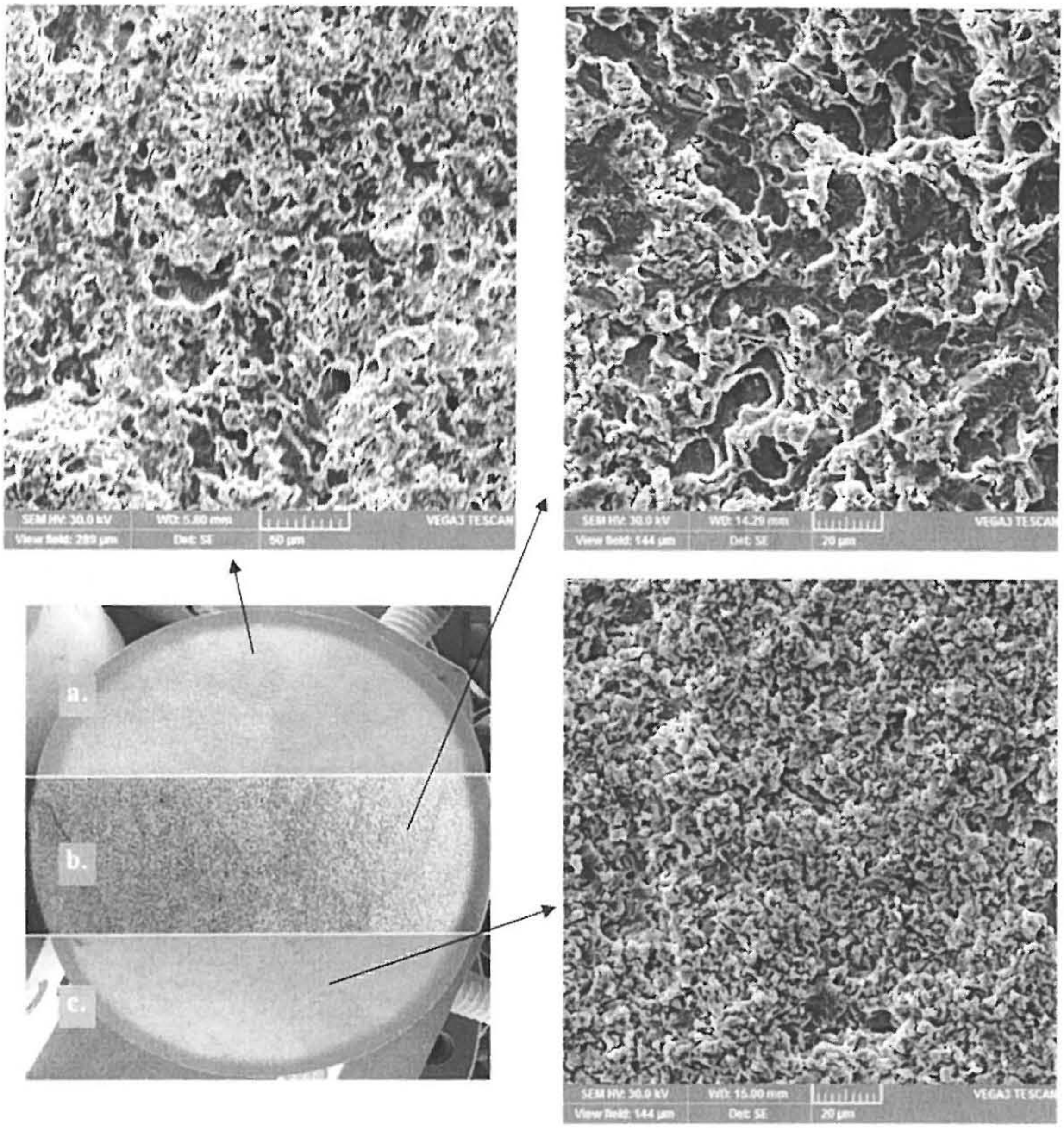


S1 Heating at 475°C
S2 Heating at 850°C
SC Quenching
Mean depth erosion rate MDER. [μm/min]
Attack time, t. [min]
0.2
0.1
0.0
0
20
40
60
80
100
120
140
160
180

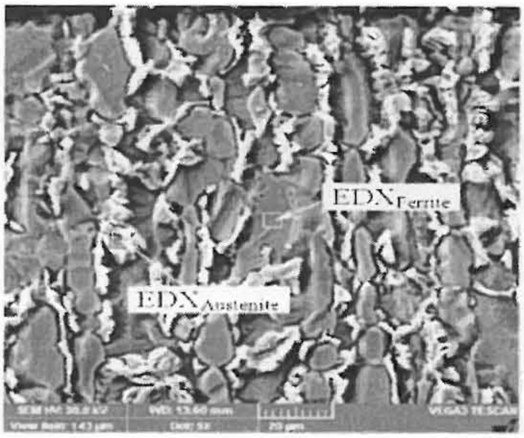


In Fig...... is analyzed the influence of the cooling procedure through three indicators: mean deep erosion, the roughness Rz, and the resistance to cavitation Rcav. From all those indicators, it resulted that by cooling in furnace the cavitation erosion resistance increases. The cause of the increase is the great proportion of austenite by furnace cooling, approximately 75%, towards only 52% obtained with cooling in water, even if the hardness became a little smaller.

In is also obvious from Fig.... an increase of the cavitation erosion resistance using the sensitizing procedure by heating at 850°C, maintaining 2 hours at this temperature, and afterwards cooling in air. The increase of resistance at cavitation erosion estimated in Rcav is greater with 56% toward that obtained by sensitizing at 450°C with maintenance at that temperature till 4 hours and with 44% greater that for the specimens subjected to volume quenching in water.



The repetitive implosions of cavitation bubbles produce in the material important plastic deformation and the subsequent formation of a crack lattice (indicated in the figures by arrows) as in the case of fatigue, by the same phenomenon of cycling stressing. The cracks appear preferentially in the ferrite phase or at the boundaries between ferrite and austenite grains. Because after the heat treatments at 475°C there appear ferrite domains rich in chromium α', these regions are subjected to a greater degradation. The annealing at 475°C during 4 hours does not generate structural modification microscopic visible. In exchange the annealing at 850°C determines the phenomena of secondary phase precipitations. There appeared also small cavities in the ferrite matrix and at the boundaries between ferrite and austenite. As the cavitation progresses the material losses became greater in the ferrite phase.



(Pr. S1 - investigated area EDX)

Specimen	Element, % masă	$EDX_{Ferrite}$	$EDX_{Austenite}$
S1 (in ageement with tab.3.1)	Cr	22.4	21.2
	Ni	5.3	5.9
	Mo	3.1	2.8
	N	≤ 0.04	0.32
S2 (in ageement with tab.3.1)	Cr	21.2	21.8
	Ni	5.4	5.5
	Mo	3.09	2.9
	N	≤ 0.04	0.34

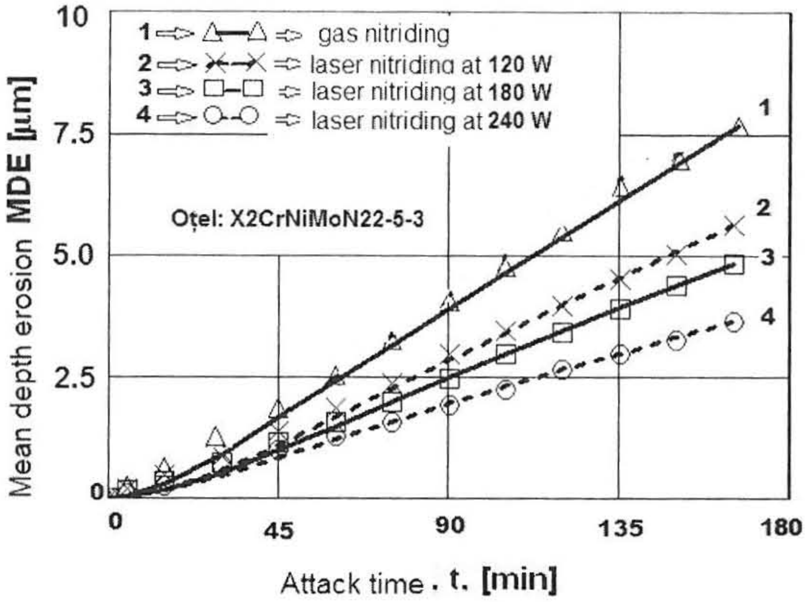
4. HARDNESS INCREASES THROUGH NITRATION TREATMENTS AS WELL AS INCREASES OF CAVITATION EROSION RESISTANCE

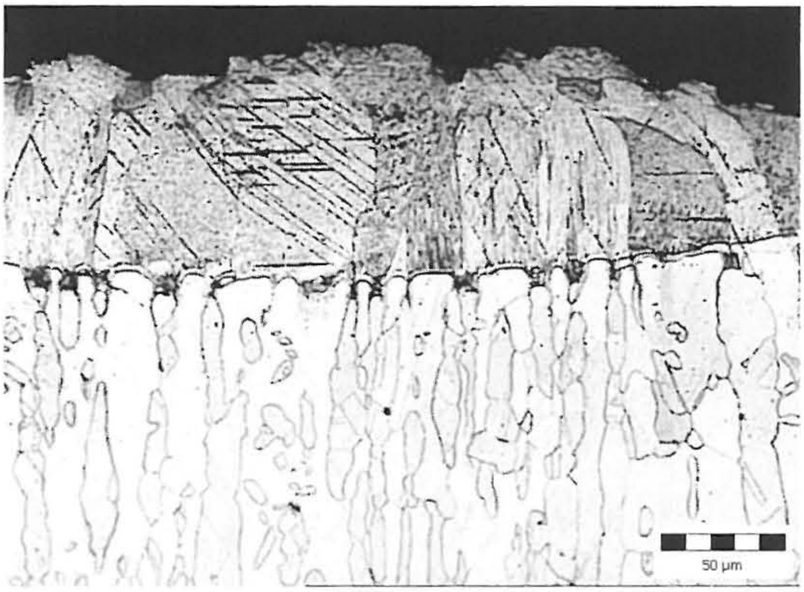
Nitration with laser beam is an attractive technologic modality in improving the tribology qualities of different details by adding superficial layers with good properties, without affecting the base material. For transversal velocity of approximate 100 mm/s appear a rapid dissolution of nitrogen in the marginal melted layer. The specimen frontal surfaces, subjected to cavitation erosion subjected a nitration thermochemical treatment with the help of a pulsating laser Nd-YAG introduced in the HL 124LCU device.

This treatment had in view the formation of superficial layers with increase hardness able to improve the behavior at cavitation erosion.

The laser beam scanned the specimen surfaces 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. The impulse power was modified as follows:

- the first set 240 W
- the second one 180 W
- the third one 120 W





There were obtained the following results:

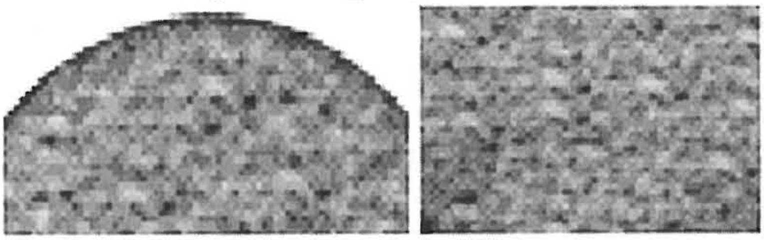
- the characteristic curves show evolutions proper to the materials with very good or even excellent resistance to cavitation erosion;
- the scatter of the experimental points toward the mean curve , regardless of the laser power is very small; this show that the the treated surface is homogeny from the point of view of mechanical properties as well as from the point of view of cavitation erosion.
- the maxim values of the erosion rates appear in the interval 60-70 minute of exposure; this denote a correct treatment procedure and a steady state of the characteristic parameters: impulse duration, frequency and power;

- in comparison with the gas nitration the laser beam treatment determines an important reduction of the erosion depth (about 24% for the power regime 120 W, about 55% for 180 W and about 125% for 240 W) as well as the erosion rate (about 13% for 120 W, about 36% for 120 W and about 96% for 240W);
- The power increase from 120 W to 240 W determine a reduction of the erosion depth with about 81% ant erosion rate with 74%.

5. THE CAVITATION EROSION INCREASES OF THE COMPOUND HARDENED WITH WC-9CO-5CR-1NI PARTICLES DEPOSITED BY, HVOF TECHNIQUE UPON - DUPLEX STAINLESS STEEL -

At present, in numerous industrial branches ((aerospace industry, vehicle industry, shipbuilding industry etc.) the protection of the working areas with composite powders against the harmful environment (such as chemical corrosion, abrasive or cavitation erosion) became indispensable [34, 35, 81,127,128].

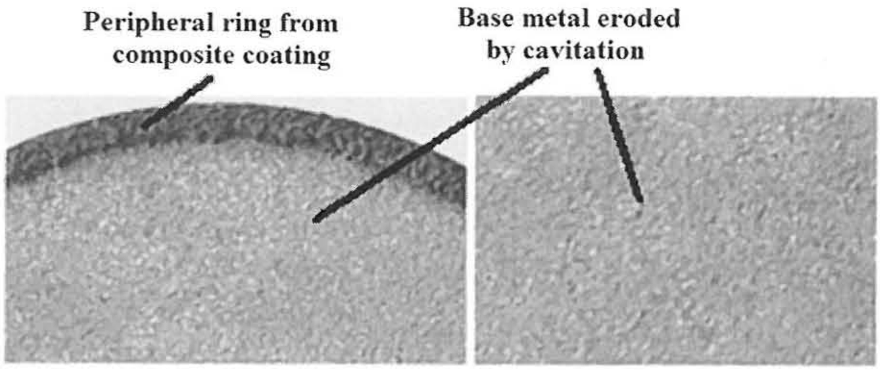
Composite sprayed coating



a) Peripheral zone b) Central zone

Minute 0

The initiation of pitting and cracking phenomena take place at the boundaries of the grains as well as inner the ferrite grains. In the same time as a result of the cold strain effect under the implosion of cavitation bubbles take place a mackling of the austenite grains and even a martensitic transformation.



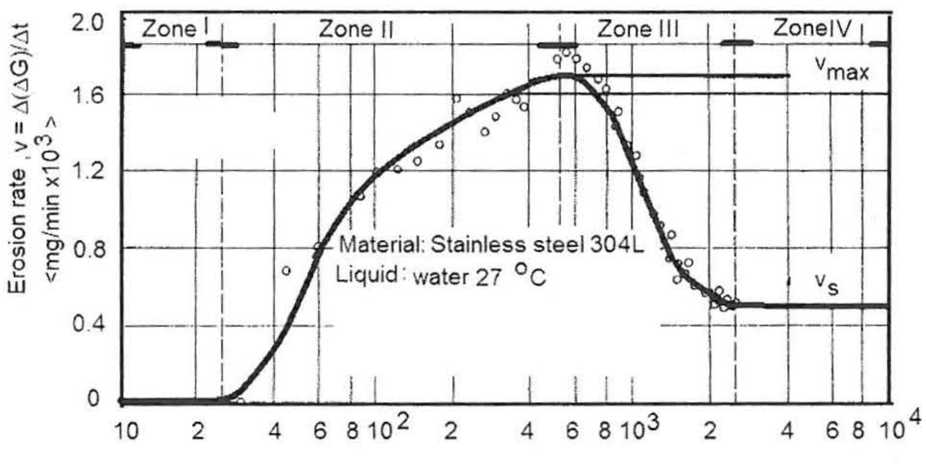
a) Peripheral zone b) Central zone
Minute165

The layer obtained by recasting with laser beams with 6 ms pulsing duration consisted from strips with pronounced points of carbides and numerous goals between them. After a cavitation exposure of 120 minutes the entire layer is removed and the erosion take place in the basic surface.

This procedure does not assure an improvement to cavitation erosion.

6. CONTRIBUTIONS WITH REGARD TO THE ANALYTICAL RELATIONS DEFINING THE CAVITATION EROSION CHARACTERISTIC CURVES

This contribution consist in establishing a new form of the equation for the erosion rate taking as beginning point the model established in 1953 by Thiruvengadam and accepted as reference by the ASTM standards an by all the researchers in the field.



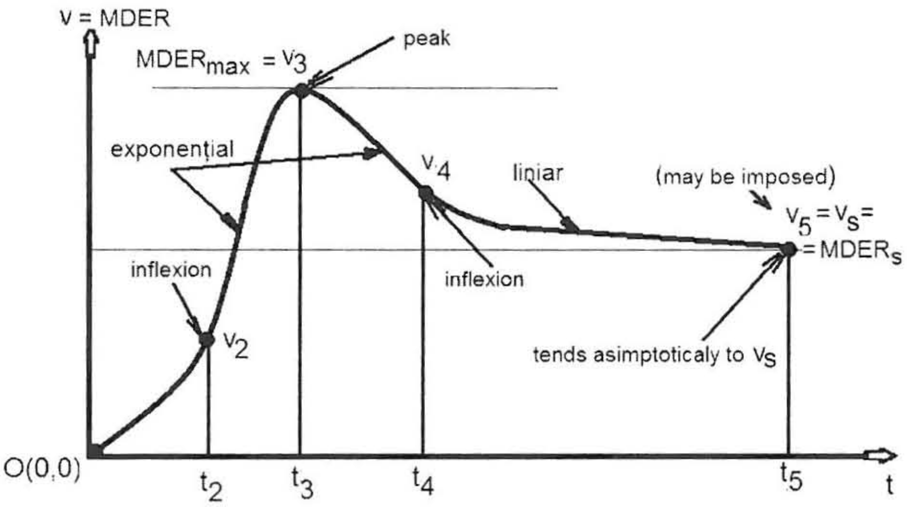
Taking as departure point the relation established in 2004 by Bordeasu and coworkers for the erosion rate MDER(t):

$$MDER(t) = \frac{dMDE(t)}{dt} = A(1 - e^{-Bt}) + ABte^{-Bt} \qquad (6.9)$$

Admitting a variation similarly with those in the figure below (alike as shape with that established by Thiruvengadam) with the mentioned conditions we added in the Bordeasu relation (6.9) a supplementary term (-Ct) and we have obtained the relation (6.10) in which is taken into consideration a linear decreasing of the erosion rate in the zone of the stabilized erosion. By integrating the relation (6.10) we have obtained the equation (6.16) which give the variation of the mean depth erosion.

$$MDER(t) = v(t) = A(1-e^{-Bt}) + ABte^{-Bt} - Ct \qquad (6.10)$$

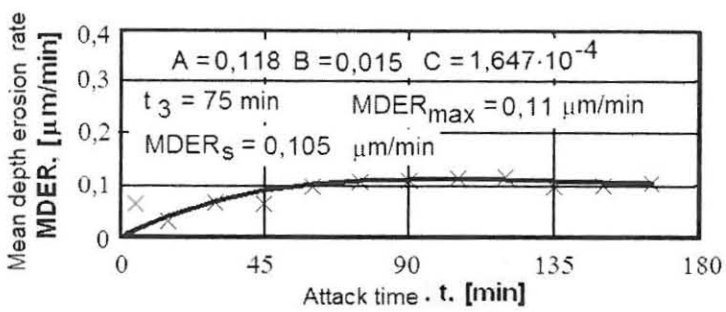
$$MDE(t) = At(1-e^{-Bt}) - Ct^2/2 \qquad (6.16)$$



✖ For the shape parameter B it must be chosen a value between the interval (0,012...0,03), established on the data resulted from the statistical processing of 40 types of stainless steels tested in LMHT.

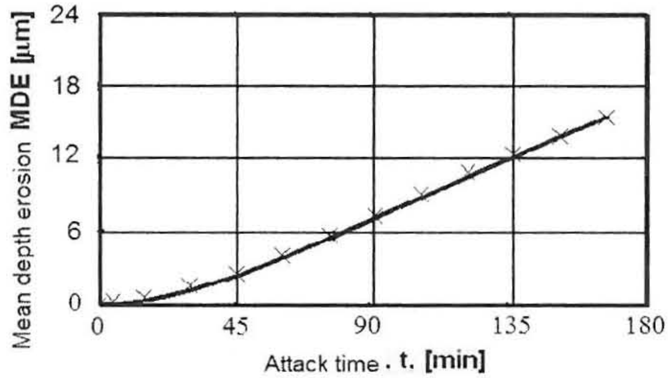
✖ The constant values A and C are obtained from the conditions imposed to the shape of the curve, taking into account the scatter of the experimental points for the time values t3 and t5.

✖ The approximation with the new relation is better than that obtained with the Bordeasu relation in the stabilization period, the parameter value being better (see the example for the following curves, for which are given the the values of the statical parameters a_s, a_i and σ).



$a_s = 6,49 \cdot 10^{-3}$ μm/min $a_i = -4,619 \cdot 10^{-3}$ μm/min

$\sigma = 8,522 \cdot 10^{-3}$ μm/min



Starea oțelului	a_s [μm/min]	a_i [μm/min]	σ [μm/min]
Quenching 1060°C /30 min/furnace-rel.(5.9)	$6,65 \cdot 10^{-3}$	- 0,011	$8,579 \cdot 10^{-3}$
Quenching 1060°C /30 min/furnace-rel.(5.10)	$6,49 \cdot 10^{-3}$	$-4,619 \cdot 10^{-3}$	$8,522 \cdot 10^{-3}$

In this way, the introduction of a supplementary term increase de approximation degree of the experimental points).

7. Final conclusions and original contributions. New research directions

The doctoral work **"Cavitation erosion behavior of Duplex stainless steels"** is a interdisciplinary one (Materials engineering-Mechanical engineering) comprised in the actual research tendency to find new solutions for improving the performance of the equipments stressed with cavitation in general and cavitation erosion in special.

The main conclusions and the original contributions of the work can be synthesized as follows:

1. The implosion of cavitation bubbles, filled in principal with steam but also in a smaller measure with gases, is accompanied either by a rapid increase of pressure either by high velocity jets, both phenomena produce a violent erosion of the solid surface which guides the flow. Mainly de erosion can reach 1 till 10 mm yearly but in some peculiar cases the erosion was of the order of centimeters in a few minutes. For diminishing this tremendous erosion there are taken simultaneously two measures: an adequate hydrodynamic design and using materials with high cavitation erosion resistance (such a material can be the Duplex stainless steel).

2. The interaction between the principal alloying elements (Cr, Ni, Mo, N) of the Duplex steels aim on the one hand to obtain a structural equilibrium between the two phases (ferrite and austenite) and by the other hand to avoid the formation of fragile inter-metallic compounds (Sigma σ, Chi χ, nitrates Cr_2N, carbides $M_{23}C_6$, etc.)

3. The annealing heat treatment for sensitiveness at 475 °C applied after the quenching from 1060 °C with cooling in water increase the hardness from 275 HV (quenched state) to 330 HV (sensitive state) but increase with approximate 21% the cavitation erosion rate. Because the steel microstructure has approximate 50% austenite the brittleness of the ferrite phase α' reach in chromium (which appear during the annealing at 475°C) is not so detrimental at cavitation erosion as to a ferrite stainless steel.

4. By ageing through hardening at 850°C (361 HV) it resulted a reduction of approximate 11% of the maxim depth erosion and approximate 28% of the maximum erosion rate, in comparison with the quenching at 1600°C with cooling in water. The explanation is given by unleash of precipitation phenomena for some secondary phases and also by the reformation of ferrite from austenite.

5. For gas nitration is obtained a maximum hardness of approximate 630 - 640 HV and a depth of the hardened layer of approximate 0.12 mm. The cavitation erosion resistance increases with 9%, and the mean depth erosion is reduced with 152%.

6. For laser beam nitration by increasing the impulse power from 120 W to 240 W the depth of the diffusion layer increases from 0,14 at 0,20 mm, and simultaneously the cavitation erosion resistance increases with approximate 81%, when we consider the erosion depth or 74 %, if the erosion rate is taken into consideration.

7. The surfaces covered with the composite powder WC-9Co-5Cr-1Ni through the HVOF procedure receive a relative small resistance to cavitation erosion as a result of the weak adherence of the deposited layer to the base metal.

8. The judicious selection of basic parameters for the laser beam jet determine, after re-melting, the formation of a surface with fine strips, without failings (cracks, uncovered portions, prominent picks, burned material) with great hardness which offer o high cavitation erosion resistance.

9. The introduction of a supplementary term in the mathematical transcription of the cavitation erosion rate curve, under the hypothesis the erosion rate in the attenuation period has a linear decreasing slope towards the stabilization period, improve de degree of the approximation of the experimental points.

The combination between the investigations concerning the applicative side (regarding especially the improvement of the outfits working in cavitation conditions) and those regarding the phenomenological side (with the specific purpose to elucidate the material reaction subjected to cavitation erosion) inscribe this work in the modern methodology and tendencies used for scientific researches.

New research directions

Using the results of the investigations undertaken in the doctoral degree thesis can be formulated the following directions for the future researches: The cavitation erosion resistance of the welded layers during the repair work periods of the hydro-mechanical equipments.

The influence of different mechanical and heat treatments upon the cavitation erosion resistance.

Researches regarding the structural degradation of the Duplex stainless steels in the incipient period of cavitation erosion.

Selective references

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