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BEHAVIOUR AT NITRIDING IN FLUIDIZED LAYER OF AUSTENITIC STAINLESS STEELS AT THE TEMPERATURE OF 450°C

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ABSTRACT

*Stainless austenitic steels have a good corrosion resistance but reduced abrasion strength due to low strength below 200*HV_5*. By nitriding one may ensure the obtaining of a superficial layer with increased strength that should maintain unaltered the corrosion resistance. The nitriding in fluidized layer applied at 450 ºC allowed the obtaining of diffusion layers formed of nitrogen enriched austenite with certain nitride separations. For the CrNi, CrNiMo usual steels, the obtained strengths have been higher than 1000* HV_{005}*. In the case of steel X1CrNiMoCu20.18.7 with more than 50% of alloying elements, the strengths did not exceed 500* HV_{005} *due to the low nitrogen concentration, much below the saturation limit determined by the reduced diffusion speed. The corrosion test in saline mist indicates the maintaining of corrosion resistance, the materials ranging in the group of Perfectly resistant. Also, the abrasion strength increased 2-3 times due to the limited diffusion speed at low temperature. The favourable property assembly allows the usage of the treatment for some practical applications.*

KEYWORDS: austenite, nitriding, corrosion, abrasion

1. Introduction

By nitriding we want to improve the strength properties, mainly the superficial strength in order to ensure an abrasion resistance of the austenitic stainless steels. Such steels are characterized by a good corrosion resistance but the strength during solution tempering, the usual delivery and usage condition is low, below 200 HV_5. Nitriding must ensure the formation of an improved strength superficial layer but also of a structure that should ensure the conservation of the corrosion resistance properties. For the performance of the treatment we used the method of nitriding in fluidized layer in quartz sand, washed and dried, and as fluidizing agent and treatment atmosphere a mix of nitrogen and 30% ammoniac. For the research we used samples of 6 austenitic stainless steel CrNi and CrNiMo whose chemistry is presented in Table 1.

Table 1

Steel code	**Related mark SR:EN 10283-98**	**C**	**Mn**	**Si**	**Cu**	**Cr**	**Ni**	**Mo**	**Ti**	**V**
		[%]								
1	X2CrNi18.8	0,12	1,24	1,64	0,06	23,2	9,8	0,11	0,01	0,02
2	X6CrNi18.10	0,06	1,55	0,65	0,08	17,1	9,3	0,05	0,60	
3	X2CrNi18.9	0,03	1,27	0,42	0,19	18,9	8,95	0,15	0,01	0,02
4	X2CrNiMo17.11.2	0,02	2,06	0,78	0,27	20,0	8,8	2,7	0,03	0,03
5	X6CrNiMoTi17.12.2	0,045	0,96	0,54	0,16	18,1	11,6	2,04	0,32	
6	X1CrNiMoCu20.18.7	0,02	1,12	0,41	0,70	20,1	18,1	6,1		0,2

The parameters of the nitrading process have been established on the basis of the experience obtained in the previous research. The 450 ºC nitrading temperature has been chosen, inferior to the interval of rendering sensitive the austenitic stainless steels, for the reduction of the risk of separating the chromium nitrides. The duration of the nitrading treatment has been established at 3 hours.

2. Analysis of the samples nitrided at 450 ºC

For the measuring of strength and its variation in the nitrided layer Vickers trial with small HV_{005} load trial at pressure load of 0.50N has been used. The nitrided samples have been transversally cut, perpendicularly on the nitrided layer, with abundant cooling abrasive disk, grinded on metallographic paper and polished with diamond paste. The results of the strength trials are presented in fig. 1. Based on the strength trials and its variation curves in the superficial layer we could precisely estimate the depth of the nitrided layer. The estimated values are presented in table 2.

Table 2. *Values of the layer depth nitrided at 450 ºC.*

Steel code	**Layer depth** [μm]
1-X2CrNi18.8	14
2-X6CrNi18.10	20
3-X2CrNi18.9	20
4-X2CrNiMo17.11.2	17
5-X6CrNiMoTi17.12.2	16
6-X1CrNiMoCu20.18.7	19*

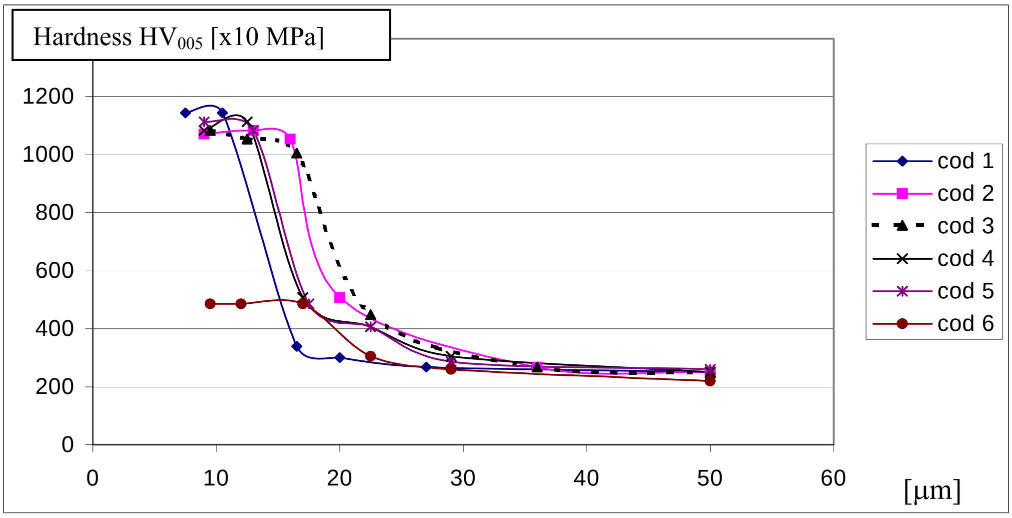

Fig. 1. *Strength variation in the nitrided layer at 450 ºC.*

The analysis of the strength trials results show the presence of the nitrided layer and important increase of strength at the steels code 1, 2, 3, 4, 5 of about 1000-1100 HV_{005} and lower increase at the steel code 6 of maximum 486 HV_{005}. The values of the lower strengths on the case of nitriding code 6 steel X1CrNiMoCu20.18.7 shows that in the layer the nitrogen concentration is lower, which is justified by the high concentration of the alloying elements over 50% and especially of the 18% Ni and cca 1% Cu concentration which amplify the austenizing effect of Ni and reduces the speed of the diffusion processes. The diffusion speed is also limited by the reduced temperature of the process which leads to the reduced concentration of the nitrogen and also to the reduced depth of 20 μm.

3. Analysis of the samples nitrided at 450 ºC

The analysis of the samples nitrided at 450 ºC does not show the presence of any distinct presence superficial layer. This proves that the layer is obtained by diffusion, being formed of austenite alloyed with nitrogen, the phase noted by most authors with. The large strength difference between the first five usual steels and high alloyed steel code 6 is caused by the different nitrogen concentration in the layer. In figure 2 is presented the microstructure of the steel code 4 X2CrNiMo17.11.2 nitrided at 450ºC. X-ray analysis of the layer nitrided at 450 ºC confirms the results of the microstructure analysis.

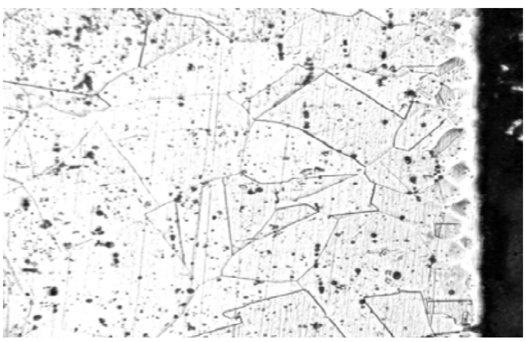

Fig. 2 *Microstructure of the steel code 4 X2CrNiMo17.11.2 nitrided at 450ºC, x400*

In figure 3, there are presented comparatively the diffractograms for the samples of the steel code 4 X2CrNiMo17.11.2 in the initial state and nitrided at 450 ºC and for the high alloyed nitrided steel code 6 X1CrNiMoCu20.18.7. In the diffractograms of sample having code 4, initial steel, there are droplets corresponding to the diffraction angles of the γ_{Fe} phase, initial austenite. In the diffractogram of sample having the code 4 nitrided at 450 ºC there are droplets of high amplitude corresponding to the diffraction angles of the γ_N phase, nitrogen enriched austenite and droplets of lower amplitude for the diffraction angles corresponding to the CrN phase.

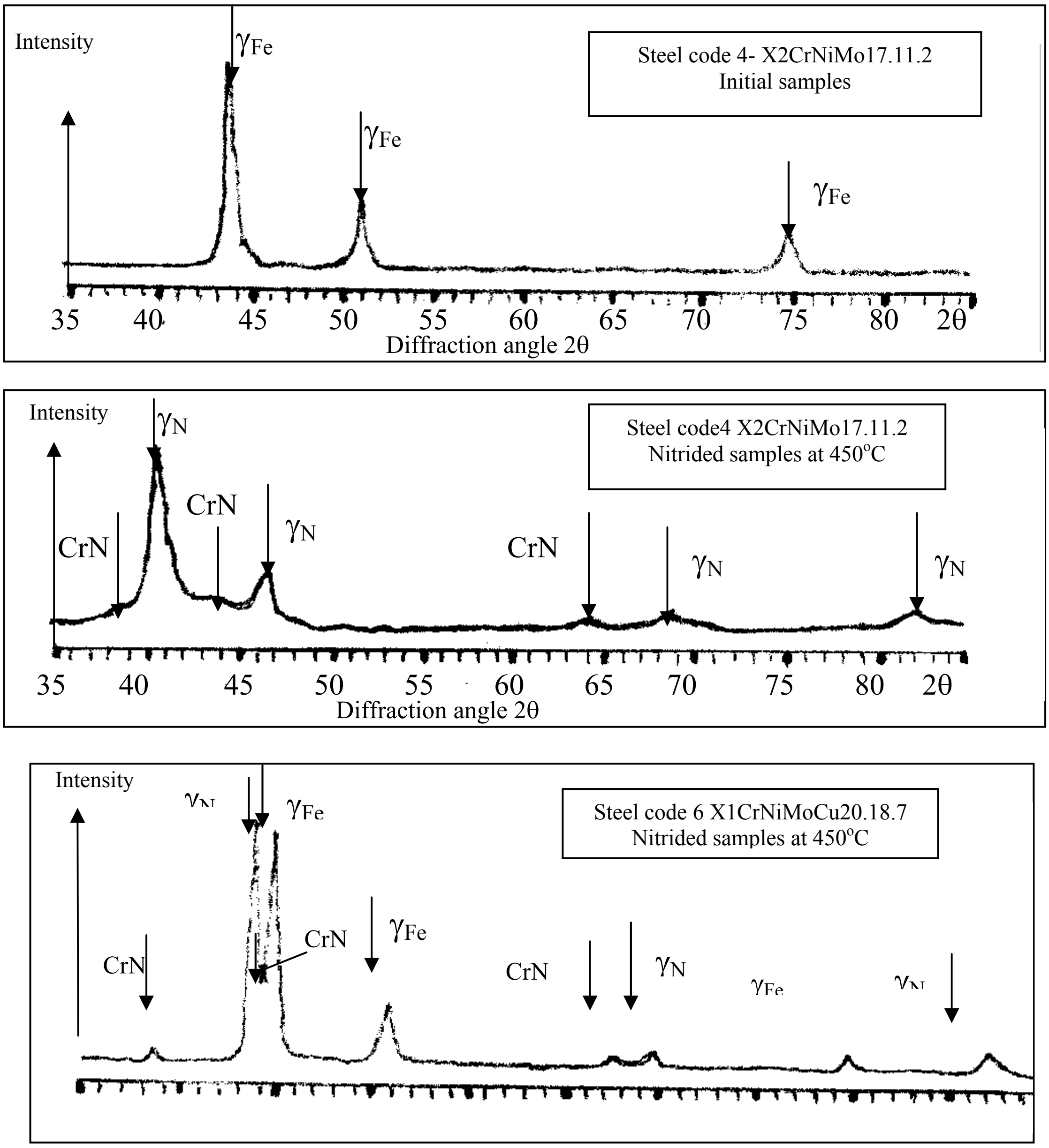

Fig. 3*. Diffractograms of the initial samples and those nitrided at 450ºC steel code 4 X2CrNiMo17.11.2 and code 6 X1CrNiMoCu20.18.7.*

It results that in the structure there are small quantities of chromium nitrates along with the high alloyed gamma solution enriched with nitrogen in high concentration, close to the saturation limit. The presence of the two phases justifies the increase in strength recorded in the layer nitrided at 450 ºC. On the diffractogram of code 6 sample nitrided at 450 ºC there are droplets of high amplitude corresponding to the diffraction angles of the γ_{Fe} phase, initial austenite and γ_N nitrogen enriched austenite. The distinct prominence of the two austenite types is caused by the fact that the interstitial penetration of the nitrogen in the austenite determines a dilation of the network and thus, a modification of the network parameter. There are also much lower amplitude droplets for the diffraction angles corresponding to the CrN phase. The presence of these phases lead to the increase of strength, its lower value being determined by the lower concentration of nitrogen in the austenite and small quantities of punctiform nitrates separated in the austenite.

4. The analysis of corrosion behaviour in saline mist of the steels nitrided at 450 ºC

For the estimation of the corrosion resistance of the samples the accelerated corrosion test in saline mist STAS 9229-95 has been used. The estimation of the corrosion behaviour has been done comparatively between the original samples with austenitic structure for the tempering for dipping in solution and nitrided samples. The composition of the solution for the obtaining of the saline mist has had a salt concentration of 35 g/litter as follows: 27 g/l NaCl, 6 g/l $MgCl_2$, 1 g/l $CaCl_2$, 1 g/l KCl with addition of boron acid for the adjustment of the pH in solution. The corrosion in saline mist is accelerated and this is explained by the high contribution of oxygen and the temperature of 35 ºC in the enclosure. In figure 4 there are presented comparatively the average corrosion speeds in saline mist STAS 9229-95 for the initial trials and those nitrided at 450 ºC. The test took place over 300 hours with weighing every 100 hours.

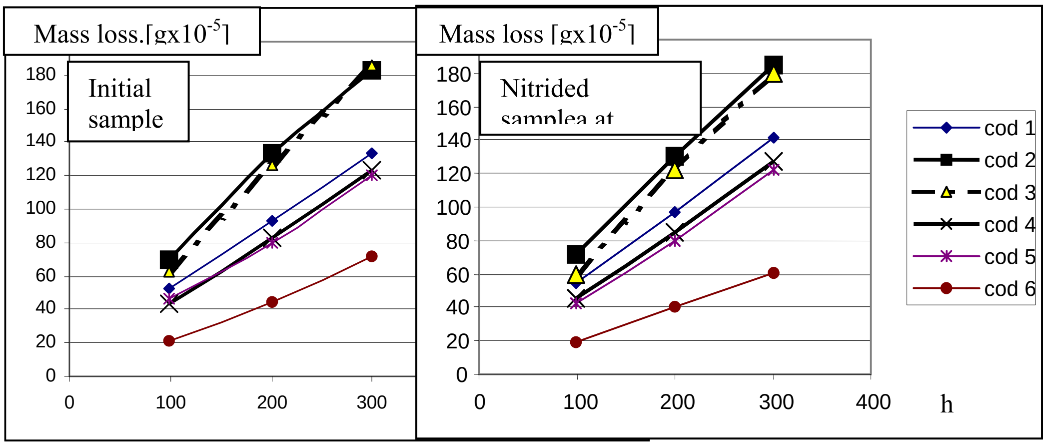

Fig. 4 *Time variation of mass losses for initial and nitrided materials.*

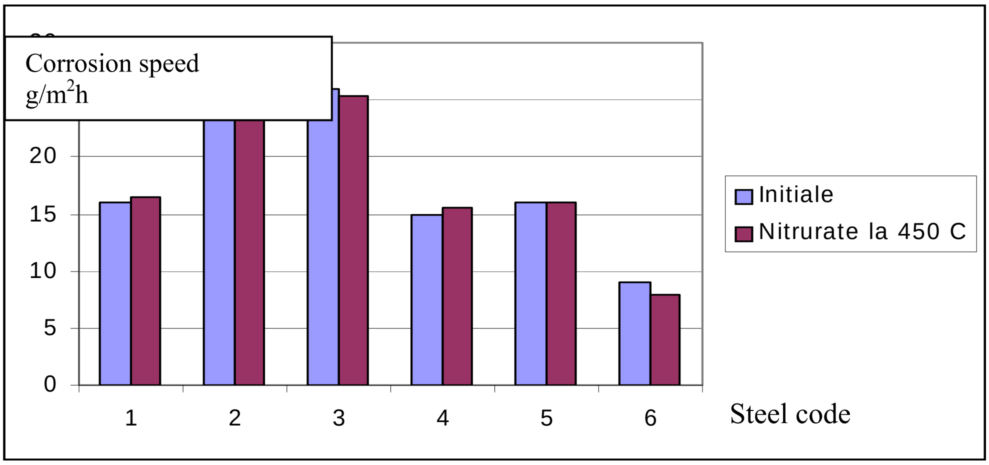

Fig. 5. *Average corrosion speeds in saline mist for the initial and nitrided materials at 450^{o}C*

The analysis of the results show a similar behaviour of the original samples and of those nitrided at 450 ºC at the corrosion test in saline mist, the mass lasses and determined corrosion speeds are vary close values, figure 5. According to the determined values, the steels nitrided at 450 ºC range within the same corrosion resistance classes as the initial steels, the measured values indicating slight increases or decreases determined by other factors.

Thus, steels code 1, 2, 3, 4, 5, both original and nitrided at 450 ºC range within the *Perfectly resistant* material group with merit note 1.2 and steel code 6, both initial and nitrided at 450 ºC ranges within the *Perfectly resistant* material group with merit note 1.1. These results are also confirmed by the traced potential curves. In figure 6 ther are presented comparatively, the potential dynamic curves traced for the initial materials and those nitrided at 450ºC of steel code 4 –X2CrNiMo17.11.2.

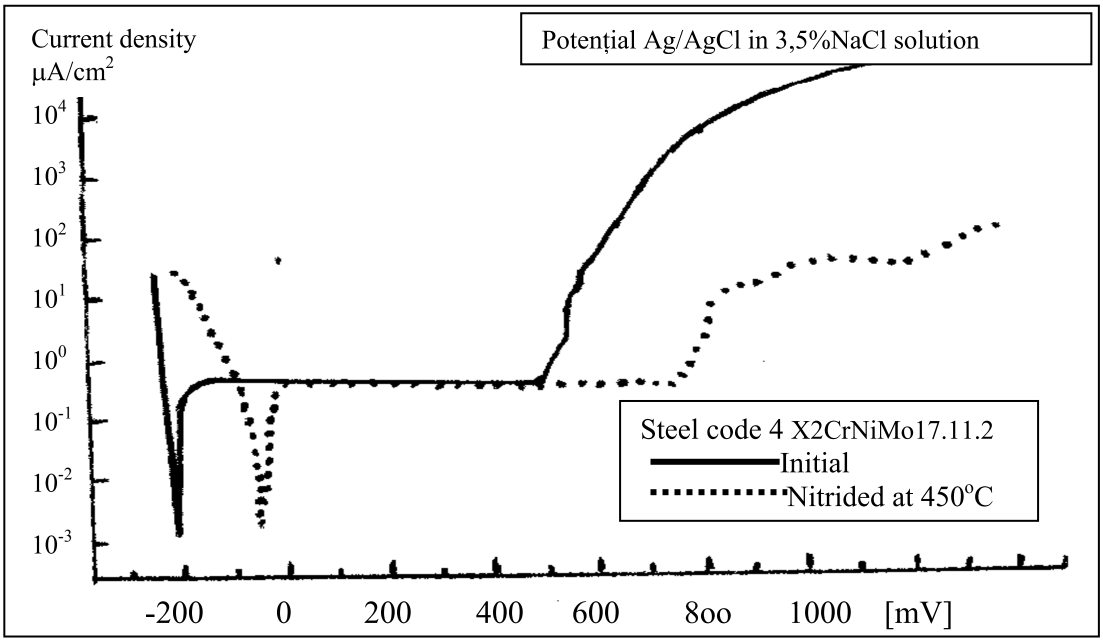

Fig. 6. *The potential dynamic curves traced for the original materials and those*

From the analysis of the potential curves results that in the case of the samples nitrided at 450 ºC in comparison with the initial samples, the passive domain is enlarged and shifts to more electropositive values and the current density in the passive domain is maintained even manifesting a downward tendency.

The shape of the polarization curves indicates a good corrosion behaviour of the steel nitrided at 450 ºC, justified by the fact that the structure of the steel is mono phase, formed of solid γ_N austenite solution enriched with nitrogen.

Nitrogen proves to have a beneficial effect over the corrosion resistance and corrosive behaviour in general.

5. Analysis of the abrasive wearing strength of the steels nitrided at 450 ºC

The abrasive wearing test was performed according to STAS 9639-81, using a friction coupling abrasive disk-pin. The conditions of the trial were: sample section 50 mm^2, granulation pointage of the abrasive disk-800, disk speed 25 rpm, radial sample advance 0.5 mm/rotation, pressure on sample 10 MPa, average sliding speed 10 m/min, length of the travel space 25 m. The results of the abrasion test, namely the mass losses by wearing for the samples nitrided at 450 ºC as compared to the original samples are presented in table 3 namely figure 7. The analysis of the results indicate an increase of the abrasive wearing strength of the samples nitrided at 450 ºC, 2,5-3 times comparatively with initial steel code 1,2,3,4,5 and 2 times for the steel code 6. The increses to wearing strength depends also on the thickness of the layer and hardness variation in the nitrided layer.

***Table 3.** Abrasion test results*

Steel code	Initial materials		Nitrided materials at 450°C		
	Hardnes HV_{005}	**Abrasive wear**	**Layer depth**	**Hardnes HV_{005}**	**Abrasive wear**
	[x10 MPa]	[g]	[µm]	[x10 MPa]	[g]
1-X2CrNi18.8	198	0,0066	14	1144-500	0,0024
2-X6CrNi18.10	202	0,0065	20	1084-500	0,0026
3-X2CrNi18.9	188	0,0065	20	1084-500	0,0026
4-X2CrNiMo17.11.2	180	0,0069	17	1114-500	0,0024
5-X6CrNiMoTi17.12.2	204	0,0064	16	1114-500	0,0022
6 X1CrNiMoCu20.18.7	182	0,0071	19*	486	0,0037

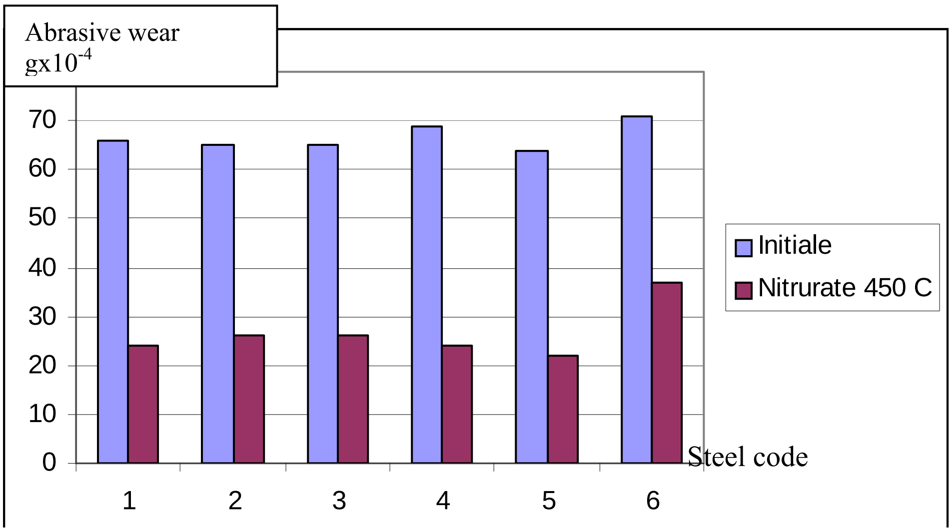

***Fig.7** Behavior of initial and nitrided samples at 450^{o}C to abrasive wear*

6. Conclusions

Anssambly of corrosion resistance properties for nitrided samples show for 450 °C especially favourable situation. The corrosion resistance of the nitrided materials or even better wearing strength has increased 2.5-3 times.

High alloyed code 6 steel with Cr, Ni, Mo with over 50% alloying elements has a more special behaviour due to the lower nitrogen diffusion speed caused by the high concentration of the alloying elements, especially Nichel.

The diffusion speed is also reduced due to the low temperature which makes the depth of the nitrided layer not to exceed 20 µm, yet sufficient for some practical applications.

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