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# Corrosion Resistance of Plasma Nitrided Structural Steels and Modern Methods of Testing

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**Abstract:**

*The corrosion of metals is at first a technical and technological problem of duty and economical point of view. The corrosion degradation is in principle a weight-shortage of metals, which can cause a construction damage by one or several uncontrolled chemical reactions, which are proceeding spontaneously. Many methods of corrosion testing are used to check the corrosion resistance of machines components. Many of these methods are leading to the degradation or destruction of the checked specimens (the atmospheric corrosion testing, laboratory testing by the NSS, AASS, CASS method or solid drops method). The modern corrosion testing methods gives results of corrosion resistance by non-destructive methods testing, e.g. the electrochemical testing. Some of mentioned testing method were used, at first to evaluate the benefit o nitriding and nitrooxidizing to corrosion resistance of some structural steels and at second to judge the agreement of these methods.*

**Keywords:**

*Corrosion, structural steels, plasma nitriding, nitrooxidizing, corrosion testing.*

## 1. Introduction

Corrosion is an undesirable process which is needed to be prevented or to slowed down. For this purpose it is possible to use for example high corrosion resistance structural materials. The primary corrosion protection systems are constructions and technological solutions to protect places with diminished corrosion resistance, as a suitable material combination, construction design and suitable *connection* of the construction parts. As the corrosion resistance increasing technology can be used the surface coating too, which can prevent or slow down the corrosion process. As the next corrosion resistance system can be used the modification of the corroding

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medium by suitable corrosion inhibitors or take the corroding medium corrosion stimulators away. It can be used the combination of these methods, to scale-down of the corrosion mechanisms too [1].

The aim of this article is to resume and compare the results of three corrosion testing methods on plasma nitrided and nitrooxidized (plasox) structural steels.

## 2. Experiment

The different ways of corrosion resistance improvement were studied on following structural steels: ČSN12 050 -DIN 1.1191 (0.49%C; 0.60%Mn; 0.017%P; 0.008%S; 0.24%Si; 0.06%Cr; 0.02%Ni; 0.08%Cu), ČSN 14 340 - DIN 1.8504 (0.35%C; 0.66%Mn; 0.019%P; 0.021%S; 0.24%Si; 1.42%Cr; 0.13%Ni; 0.07%Cu; 0.95%Al; 0.21%V) and ČSN 15 330 - DIN 1.7707 (0.29%C; 0.61%Mn; 0.019%P; 0.015%S; 0.22%Si; 2.48%Cr; 0.07%Ni; 0.12%Cu; 0.24%Mo). The chemical composition of steels was verified by the GDOES method.

*Tab. 1 The plasma nitriding and nitrooxidizing parameters*

| Variation A | Temp. [°C] | Duration [h] | Pressure [mbar] | Voltage [V] | Gas flow [l/h] | | |
|---|---|---|---|---|---|---|---|
| | | | | | $H_2$ | $N_2$ | $CH_4$ |
| Cleaning | 490 | 0.5 | 0.8 | 800 | 20 | 2 | 0 |
| Nitriding | 500 | 8 | 2.8 | 530 | 8 | 24 | x |
| Oxidation | 500 | 1 | 8-9 | - | - | - | - |
| Variation B | Temp. [°C] | Duration [h] | Pressure [mbar] | Voltage [V] | Gas flow [l/h] | | |
| | | | | | $H_2$ | $N_2$ | $CH_4$ |
| Cleaning | 490 | 0.5 | 0.8 | 800 | 20 | 2 | 0 |
| Nitriding | 500 | 20 | 2.8 | 530 | 8 | 24 | x |
| Oxidation | 500 | 1 | 8-9 | - | - | - | - |
| X – flow of $CH_4$ – 0 or 1[l/h], oxidation – water vapour | | | | | | | |

The manufactured structural steel samples (size of 40x5x100 mm) were normalized and heat-treated to obtain optimal mechanical properties. After the heat-treatment were the specimens surfaces rough-grinded to the following average value roughness: 12 050 Ra 0,4 µm, 14 340 Ra 0,9 µm, 15 330 Ra 0,3 µm (measured by Surtronic 3+TAYLOR - HOBSON contact method). The plasma nitriding and plasox parameters are shown in table 1, and the list of the treated specimens marking in table 2. The 12 050 steels samples are marked as 2 01, 2 04 etc., the 14 340 steels samples as 4 01, 4 04 and so on.

*Tab. 2 Marking of treated steel samples*

| Sample | Treatment | Sample | Treatment |
|---|---|---|---|
| 2 04 ÷ 2 07 | A – nitrided 8 h | 2 16 ÷ 2 19 | B – nitrided 20 h |
| 4 04 ÷ 4 07 | A – nitrided 8 h | 4 16 ÷ 4 19 | B – nitrided 20 h |
| 5 04 ÷ 5 07 | A – nitrided 8 h | 5 16 ÷ 5 19 | B – nitrided 20 h |
| 2 08 ÷ 2 11 | A – plasox | 2 20 ÷ 2 23 | B – plasox |
| 4 08 ÷ 4 11 | A – plasox | 4 20 ÷ 4 23 | B – plasox |
| 5 08 ÷ 5 11 | A – plasox | 5 20 ÷ 5 23 | B – plasox |
| 2 12 ÷ 2 15 | A – plasox + $CH_4$ | 2 24 ÷ 2 27 | B –plasox + $CH_4$ |
| 4 12 ÷ 4 15 | A – plasox + $CH_4$ | 4 24 ÷ 4 27 | B –plasox + $CH_4$ |
| 5 12 ÷ 5 15 | A – plasox + $CH_4$ | 5 24 ÷ 5 27 | B –plasox + $CH_4$ |

Samples 2 01, 2 30, 4 01, 4 41, 5 01 and 5 40 were not nitrided.

## *2.1. The NSS Corrosion Test*

Methodology of corrosion testing in the condensation chamber offers the following methods: testing in the mist of neutral sodium chloride (NSS method), acidified sodium chloride by acetic acid or by sodium hydroxide (AASS method), acidified sodium chloride and cupric dichloride (CASS method), aqueous vapour condensation with sulphur dioxide and salt drops test [2]. For the corrosion resistivity evaluation of the nitrided and nitrooxidized structural steels the neutral sodium chloride (NSS) and sulphur dioxide ($SO_2$) testing methods were chosen.

By using of the NSS method the uniform type of corrosion attack and point or pitting corrosion were supposed as a reason of the nitride layer incompactness. The uniform type of corrosion means a corrosion attack of an area of the surface and represents not only an aesthetic degradation, but mainly the loss in the lifetime and functionality. This type of corrosion can also cause a markedly high decrease of the wall thickness. The point and pitting corrosion show on the imperfections and incoherency of the plasma nitrided layer. This type of corrosion attack is really dangerous because can induce the fatigue of materials, the pitting borders work as stress raisers and can cause the cracking of layers and materials.

The corrosion tests were carried out in the Liebisch GmbH & Co (S 400 M-TR) corrosion cell in accordance with the ČSN ISO 9227 standard. The samples were degreased before the corrosion testing by the technical benzoline and ethylalcohol and the edgings and the suspension apertures were conserved by resistant sticky tape. The necessity of protecting of the edgings and suspension apertures by resistant sticky tape was proved by previous experiments [3], [4].

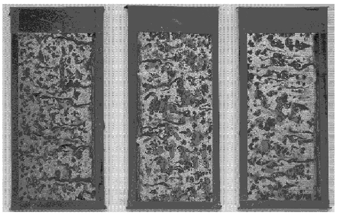
The NSS corrosion test conditions: the temperature of 35 ± 2 ºC, 5 % neutral sodium chloride dilution, the amount of vapour condensation 1-2 $ml.h^{-1}$ on square 80 $cm^2$, pH 6.5 – 7.2, the suspension angle 20º from the vertical line, the period of evaluation 2, 6, 24, 48, 96, 144 and 240 hours.

On the above mentioned time exposed samples were evaluated visually (photographic documentation) and by means of gravimetry (corrosion increments) (see table 3).

### *2.2. The NSS Corrosion Test Evaluation*

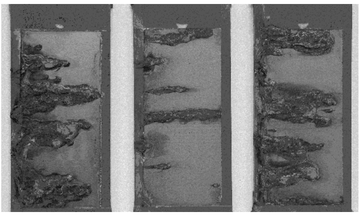
During the NSS corrosion test of plasma nitrided a nitrooxidized steels different corrosion resistance results were observed, as the effect of the chemical composition of the basic material and the type of the surface treatment. After 2 hours corrosion conditioning the not-nitrided steel samples surfaces were visible affected (numbers 2 01, 4 01, 5 01) and after 6 hours of corrosion conditioning were fully degradated (see fig. 1, 2). For a longer conditioning time only the gravimetric method of evaluation was applied (see table 3). After 96 hours the most of specimens was strongly rusted and had to be excluded from the consequential corrosion conditioning. The best corrosion resistance results have displayed the nitrooxidized steels, namely the 4 20 (plasox) – after 240 h of corrosion conditioning without corrosion attack. The 4 04 (8h nitridation), 4 08 (plasox), 4 12 (plasox), 4 24 (plasox) specimens could be considered as the following best results.

Generally it can be said, that the time of 8 hours (and even shorter) of plasma nitriding process is sufficient for the appreciable increase of corrosion resistance. The effective improvement of the corrosion resistance more than the long time nitriding implement of oxidation process. Using of the $CH_4$ in the plasma atmosphere doesn´t lead to the improvement of the nitrooxidized structural steels, according to results of the visual evaluation. Exception from this results can be found for the 14 340 steel (see table 3 and compare marking of steels specimens with table 2).



2 01 4 01 5 01

*Fig. 1 Not-nitrided specimens*
*2 hours NSS test*



2 08 4 08 5 08

*Fig. 2 8 h plasox specimens*
*96 hours NSS test*

### *2.3. Sulfur Dioxide ($SO_2$) Corrosion Test*

The sulfur dioxide corrosion test, in accordance with the ČSN ISO 6988 (ČSN 03 8130) standard, simulates an increased effect of the industrial environment. This accelerated corrosion test is very aggressive and it is suitable way of the corrosion testing of various types of protection coatings, because the humid air with the sulfur dioxide produce the visible signs of corrosion very easily. This testing method is useful for the detection of pits, poruses or other sources of coating failure.

The $SO_2$ corrosion test conditions: the temperature of 40 ºC, the period of classification 8 hours and next 16 hours on the air at the room temperature (22ºC ± 2ºC), the completed testing period was 24 hours [5], [6].

*Tab. 3 The gravimetric results of the NSS corrosion test*

| Spec. | Weight [g] | Weight increase [g] | | Weight increase $[g/cm^2]$ | |
|---|---|---|---|---|---|
| | beginning | after 48h | after 144h, **240h** | after 48h | after 144h, **240h** |
| Not-nitrided | | | | | |
| 2 01 | 133.8757 | 0.2755 | 0.4499 | 0.0088 | 0.0144 |
| 4 01 | 113.6923 | 0.2628 | *0.2164 | 0.0085 | 0.0070 |
| 5 01 | 126.6744 | 0.2803 | 0.3496 | 0.0091 | 0.0114 |
| Plasma nitridation (8 h) | | | | | |
| 2 04 | 136.7562 | 0.2593 | *0.2531 | 0.0091 | 0.0088 |
| 4 04 | 110.0046 | 0.1311 | **0.3246** | 0.0044 | **0.0110** |
| 5 04 | 129.4788 | 0.2545 | *0.2467 | 0.0083 | 0.0080 |
| Plasox | | | | | |
| 2 08 | 136.5783 | 0.2146 | 0.3301 | 0.0072 | 0.0111 |
| 4 08 | 11.9417 | 0.1745 | **0.3314** | 0.0059 | **0.0112** |
| 5 08 | 129.3753 | 0.1361 | **0.5148** | 0.0046 | **0.0173** |
| 2 12 | 135.3693 | 0.2510 | 0.2645 | 0.0086 | 0.0091 |
| 4 12 | 113.3371 | 0.1371 | **0.2771** | 0.0044 | **0.0089** |
| 5 12 | 127.547 | 0.2215 | 0.2277 | 0.0070 | 0.0072 |
| Plasma nitridation (20 h) | | | | | |
| 2 16 | 137.2151 | 0.2346 | 0.2840 | 0.0082 | 0.0099 |
| 4 16 | 116.153 | 0.1628 | *0.1221 | 0.0057 | 0.0043 |
| 5 16 | 126.7867 | 0.2717 | 0.2628 | 0.0091 | 0.0088 |
| Plasox | | | | | |
| 2 20 | 138.1071 | 0.2217 | 0.3663 | 0.0075 | 0.0125 |
| 4 20 | 111.6612 | 0.1192 | **0.1926** | 0.0041 | **0.0066** |
| 5 20 | 126.4934 | 0.1535 | **0.6065** | 0.0053 | **0.0209** |
| 2 24 | 135.7227 | 0.2251 | *0.2170 | 0.0077 | 0.0074 |
| 4 24 | 112.6644 | 0.0974 | **0.1399** | 0.0038 | **0.0054** |
| 5 24 | 127.1561 | 0.2600 | *0.1942 | 0.0085 | 0.0063 |

| | |
|---|---|
| * | The corrosion products loss |
| **XXX** | Gravimetric evaluation after 240h |

### 2.4. *Evaluation of The Sulfur Dioxide Corrosion Test*

The $SO_2$ corrosion tests have shown, that this corroding medium is for the plasma nitrided steels exceedingly aggressive, practically all the specimens were destroyed during the first corrosion test period. The degradation of the plasma nitrided steel surfaces were comparable to the not-nitrided one. Generally can be said, that the best results were won on the oxinitrided steels, like for the NSS test. The best corrosion resistance results were found for the 14 340 steel specimens (plasma nitrooxidized and nitrided) and short-time plasma nitrided structural steels, like in the NSS test. The corrosion resistance of the long-time plasma nitrided structural steels (20 hours) can be effective improved by oxidation after the plasma nitriding process (but without the $CH_4$ in the plasma atmosphere). Results of the $SO_2$ corrosion tests validated the NSS corrosion tests results.

### 2.5. *Electrochemical Corrosion Testing*

The electrochemical corrosion testing of materials is considered as the most modern corrosion research. These testing methods are intensively developed and step by step standardized. In our research the potentiokinetic polarization method was used. By this method the electrode potential is continuously changed and is recorded as the current/potential characteristic (see fig. 3) to win the full potentiokinetic polarization curves.

For the potentiokinetic, PC controlled measurements the VoltaLab$^{TM}$21 electrochemical laboratory system was used, together with the PGP 201 Potentiostat/galvanostat type, VoltaMaster I software. The electrochemical tests conditions: the electrochemical testing was carried out in the 0,1 mol/l of NaCl aqueous solution at the room temperature with the access of air in the standard cell - the 50 ml plastic vessel with Pt spiral auxiliary electrode.

The potentiokinetic polarmeasurement was realised at the starting potential -600 mV with following shift of 1 mV/s until to the backpotential ($E_v$) was reached and return to the starting potential or to the current density under -10 μA/cm$^2$. The obtained values are in table 4. Based on the potentiokinetical polarization curves I = F (E) (see fig. 3) the values of the polarization resistance ($R_p$) and the corresponding current density ($I_{corr}$) were obtained and the average values of the corrosion rate ($v_{corr}$) could be quantified. As the next, the values of the puncture potential ($E_d$ – the potential of depassivation) for $I_d$ = 4 μA/cm$^2$, the repassivation potential ($E_r$) for $I_r$ = 5 μA/cm$^2$ were deducted and the backpotential ($E_v$) on the repassivation potential according to curve intersection ($E_{ri}$ - intersection) were determined. The results are shown in the table 4.

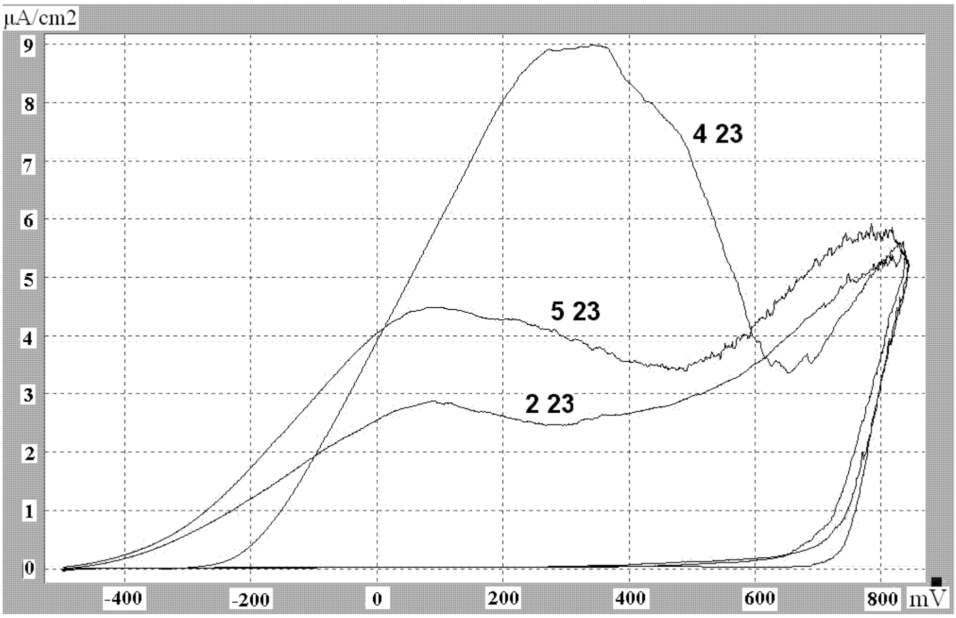


*Fig. 3 Potentiokinetic polarization curves of the specimens 2 23, 4 23 and 5 23*

### 2.6. Evaluation of Electrochemical Corrosion Testing

The puncture potential (depassivation) describes the pitting corrosion resistance of steels, it depends on the surface chemical composition and on the surface quality. The repassivation potential describes the resistivity against the pitting corrosion spreading, it depends on the volumetric chemical composition and is practically independent on the surface quality. The pitting corrosion resistance is generally higher, if the values of the named potentials are much higher, it means higher difference between the $E_d - E_{corr}$ and simultaneously $E_r - E_{corr}$ potentials. The range of the surface and pitting corrosion is represented by calculated $V_{corr}$ values.

The results of the electrochemical testing have shown, that for the pitting corrosion resistance increase of the 12 050 and 15 330 steel the 8 hours plasma nitriding is sufficient. Very good results offer the 20 hours plasma nitriding and post oxidizing of steel, too. The corrosion resistance differences between the 8 hours and 20 hours plasma nitrided steels and nitrooxidized steels in $CH_4$ free nitriding atmosphere and nitrooxidized steels in the nitriding atmosphere with $CH_4$ are thanks the electrochemical corrosion way of testing much more obvious (see table 4).

At the end, one can say, that this kind of testing is really fast, non-destructive and fast burgeoning testing method.

*Tab 4. The potentiokinetic measurements results*

| | Ecor (mV) | Ed (mV) | Er (mV) | Ev (mV) | Icorr ($\mu A/cm^2$) | Vcorr (mm/year) | Ed-Ecor | Er-Ecor |
|---|---|---|---|---|---|---|---|---|
| 2 30 | -543 | -480 | -628 | -245 | 0.0233 | 0.278 | 63 | 85 |
| 4 41 | -567 | -503 | -562 | -268 | 0.023 | 0.274 | 64 | 5 |
| 5 40 | -588 | -500 | -577 | -231 | 0.014 | 0.167 | 88 | 11 |
| 2 07 | -241 | 657 | 372 | 772 | 0.4624 | 0.006 | 898 | 613 |
| 4 07 | -220 | 293 | -172 | 541 | 0.5728 | 0.007 | 513 | 48 |
| 5 07 | -231 | 632 | 280 | 791 | 0.8109 | 0.01 | 863 | 511 |
| 2 11 | -157 | 672 | 283 | 836 | - | - | 829 | 440 |
| 4 11 | -201 | 525 | 143 | 837 | 0.1199 | 0.001 | 726 | 344 |
| 5 11 | -170 | -387 | 245 | 839 | 0.098 | 0.001 | 217 | 415 |
| 2 15 | -273 | 705 | -195 | 1073 | 0.7308 | 0.009 | 978 | 78 |
| 4 15 | -345 | 129 | -309 | 426 | 5.3795 | 0.064 | 474 | 36 |
| 5 15 | -359 | -9 | -321 | 1082 | 4.0761 | 0.049 | 350 | 38 |
| 2 19 | -144 | 650 | 145 | 803 | 0.2667 | 0.003 | 794 | 289 |
| 4 19 | -269 | 473 | 93 | 758 | 1.4778 | 0.018 | 742 | 362 |
| 5 19 | -474 | 598 | -449 | 779 | 5.2322 | 0.063 | 1072 | 25 |
| 2 23 | -207 | 706 | 289 | 841 | 0.1335 | 0.002 | 913 | 496 |
| 4 23 | -230 | 573 | 301 | 837 | 0.6452 | 0.008 | 803 | 531 |
| 5 23 | -234 | 487 | 239 | 842 | 0.3163 | 0.004 | 721 | 473 |
| 2 27 | -332 | -102 | -305 | 340 | 3.1727 | 0.038 | 230 | 27 |
| 4 27 | -206 | 617 | 172 | 1153 | 1.2941 | 0.015 | 823 | 378 |
| 5 27 | -387 | -124 | -354 | 306 | 3.7491 | 0.045 | 263 | 33 |

It is necessary to note, that this corrosion testing methodology of the nitride and nitrooxidized layers is quite new. This is why the obtained results have to be compared and interpreted together with the results of other methods of analyses, which are shortly described in the following parts of this study. This could be also the aim of next development of corrosion testing methods.

## 3. 3D Corrosion Pits Measurement

It would be appropriate to evaluate the state of surface integrity after the corrosion attack for the overall information about the corrosion resistance of steel and about the extend of surface damage in the corrosion medium. The best way to evaluate the surface state after corrosion attack is to compare the surfaces before and after the corrosion attack. The first expectation that the not-nitrided specimens will show an extensive general corrosion while nitrided and nitrooxidized specimens only surfaces full of a lot of corrosion pits was right. According to the ISO 8407:1991 standard the corroded surfaces were by the chemical (500 ml of HCl, 3.5 ml of urotropin and 496.5 ml of $H_2O$ solution) and mechanical way (brush) derusted.

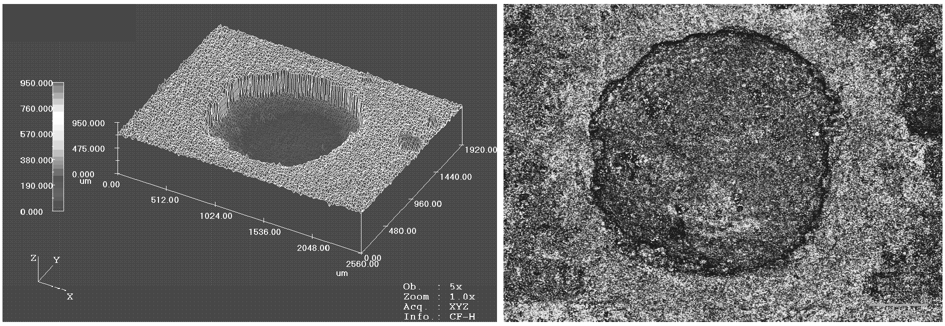
There are many convetional methods used the pitting evaluation. The commonest measuring principles are: measurement using the depth recorder, depth measurement on the cross section of the pits or the method using the successive grinding to the aground of the pit. Application of a new method of the pit depth measurement allows the new laser confocal scanning microscope LEXT OLS 3000 using the wavelength of 408 μm. Thanks the possibility of scanning and focusing of the light beam on the defined pitch from the bottom to the top of the pit the whole 3D picture of the pit is obtained and can be measured (see picture 4 and 5). Using of laser confocal scanning microscopy for the pit depth measurement is the evaluated derusted steel surface undamaged as it is at the aforesaid measurements steps.

### 3.1. Evaluation of 3D Corrosion Pits Measurement

Generally it can be said that all of the not-nitrided steel surfaces (12 050, 14 340, 15 330) were attacked by extensive general type of corrosion. The 8 hours plasma nitrided surfaces were attacked by the general corrosion of about 50% less than the not-nitrided steels and weakly by pitting corrosion. Only the nitrided 14 340 steel surface was attacked simultaneously by the general corrosion (30%) and stronger pitting corrosion (see fig. 4).

All the nitrooxidized steel surfaces were attacked by pitting corrosion and very slightly by the general corrosion. The pit proportions measurement (see figure 5) has shown, that dimensions of the corrosion pits of the nitrooxidized surfaces are larger than of pits on the plasma nitrided surfaces. Surfaces of the nitrooxidized steels were attacked slightly by the general corrosion but the pits proportions were much larger.

This quite new method of pits proportions measurements is really timeless-consuming, effective and non-destuctive. Probably the only disadvantage of this measurement is that it is in principle unable to see the shape of the pits or corrosion points. That can be clear thanks the cross sections evaluation of pits.



3D – mag. 5x 2D – mag. 5x

*Fig. 4 The 8 h pl. nitrided 14 340 steel surface topography after corrosion attack*

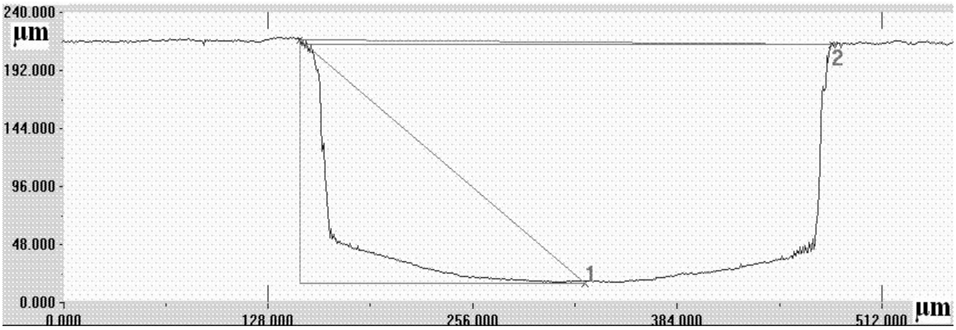


*Fig. 5 The pits proportion measurement (8 h plasma nitrided 12 050 steel)*

## 4. Conclusions

The corrosion tests have shown clear differences between the corrosion resistance of the tested steels and created layers. The different corrosion resistance was dependent on the different duration of the plasma nitriding process and on the chemical composition of the basic material too. As the next - the differences in corrosion resistance arouse out of the differences in the nitriding atmosphere compound.

The best corrosion resistance was obtained by the plasma nitriding and nitrooxidizing of 14 340 steel which contains aluminium and chromium (in contrast to the 12 050 and 15 330 steels).

The results of the corrosion testing methods (NSS, $SO_2$, electrochemical testing) have made possible to compare the fittingness of these methods and to check the corrosion resistance of the plasma nitrided and nitrooxidized steels. The results have shown that to the corrosion resistance increase is the plasma nitriding time of 8 hours (and shorter), but as the best corrosion resistance counts the 8 and 20 hours plasma nitrooxidizing without the $CH_4$. Differences between the resistance to corrosion for the 8 hours and 20 hours plasma nitrided steels and nitrooxidized steels in $CH_4$ free-nitriding atmosphere and nitrooxidized steels in the nitriding atmosphere with $CH_4$ are much more obvious thanks the electrochemical corrosion testing (see table 4).

The results of the several corrosion testing methods proved that the corrosion resistance of the long-time plasma nitrided structural steels (20 hours) can be effective improved by oxidation after the plasma nitriding process (but in the $CH_4$ free-atmosphere).

Our experiments have shown, to a certain extent, a good agreement of results and merits of compared testing methods. It is necessary to state that the electrochemical testing does not lead to destruction of the steel specimens, is fast and allows an additional measurements and testings of these specimens.

Application of the laser scanning confocal microscopy for the measuring of the pits depths was verified. All the nitrooxidized steel surfaces were attacked by pitting corrosion and very slightly by the general corrosion. The pit proportions measurement (see figure 5) has shown, that dimensions of the corrosion pits of the nitrooxidized surfaces are larger than of pits on the plasma nitrided surfaces. Surfaces of the nitrooxidized steels were attacked slightly by the general corrosion but the pits proportions were much larger.

It can be said, that this method is a less time-consuming and allows depth measuring without destroying the surface profile.

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