THE INFLUENCE OF THE NITRIDED LAYER DEPTH IN THE ROOT OF V-NOTCH TO NOTCH TOUGHNESS OF 30CrMoV9 STEEL

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Abstract

The aim of this work is to describe the influence of plasma nitride layers with different case depths which were created in the root of V-notches to notch toughness of 30CrMoV9 steel. The different case depths of nitride layers were achieved by three nitriding processes at temperatures 450 °C, 500 °C and 550 °C. Duration of nitriding process was chosen with regard to the achievement of uniform depth layer on steel surface because determination the final depth of the root of the V-notch is difficult. Chemical composition of steel was verified by GDOES/Bulk method using the LECO SA 2000 device. The experimental works were realized on V-notch samples of size 10x10x55 mm (according to CSN ISO 148-1 standard). The notch toughness tests of steel were carried out using the instrumental Charpy hammer at temperatures -40 °C, +21 °C and + 70 °C. Thickness and microhardness of plasma nitrided layers was evaluated using the automatic microhardness LECO M-400-H device. The metallography was aslo evaluated using a light microscope OLYMPUS GX51. The results of experiments showed that plasma nitriding process has a direct impact on the change of notch toughness parameters. The notch toughness of plasma nitrided steel was decreased. The primary influence on the notch toughness has the case depth of the nitrided layer at the root of the V-notch. The measurements thereinafter showed that the notch toughness values at low temperature (-40 °C) decreased but at higher temperature (+70 °C) decreased too.

Keywords: Plasma nitriding, Notch toughness, V-notch, Case depth

1. INTRODUCTION

Nowadays, thermo-chemical diffusion processes play an important role in modern manufacturing technologies [1]. Plasma nitriding process assisted by glow discharge plasma deals with surface hardening and advanced surface modification technology which has recently become industrially important [2]. Plasma nitriding is used for increasing of fatigue strength, surface hardness, and corrosion or wear resistance of industrial components for a wide variety of applications [3]. On the other hand, it is necessary to take into account the negative effect of plasma nitriding on the values of impact energy and notch toughness [4, 5]. The submitted paper deals with the influence of selected parameters of plasma nitriding to the fracture behaviour of steel samples made of 30CrMoV9 (CSN 41 5330) steel provided with V-notches (dimensions of V-notch are in accordance with ISO 148-1 standard [6]) compared with not nitrided steel samples. The influence of the nitrided layer depth in the root of V-notch to notch toughness of steel was evaluated by the dynamic notch toughness test using the instrumental Charpy hammer Zwick RKP 450 IWI device according to ISO 14556 standard [7] at test temperatures -40 °C, +21 °C and +70 °C. The results of notch toughness are supplemented by evaluation of surface hardness, metallographic analysis and measurement of case depth of the nitrided layer in V-notches root. Finally, the fractography analysis was performed.

2. EXPERIMENTAL PROCEDURES

The experimental programme of the work was focused in the field of the influence of fracture failure mechanisms of nitrided V-notch roots at test temperatures and the course of notch toughness KCV values. The value of notch toughness KCV was calculated from the values of measured impact energy KV. For the experiment was the nitride 30CrMoV9 (CSN 41 5330) steel used Chemical composition of the selected steel was verified by the GDOES/Bulk method using the LECO SA 2000 spectrometer and the measured values of elements are stated in **Table 1**.

Table 1 Chemical composition of steel 30CrMoV9 (CSN 41 5330)

Chemical composition (%wt)								
Element	C	Mn	Si	P	S	Mo	Cr	V
DIN standard	0.24 - 0.34	0.40 - 0.80	0.17 - 0.35	< 0.035	< 0.035	0.20 - 0.30	2.30 - 2.70	0.15 - 0.30
GDOES/Bulk*	0.32	0.74	0.21	0.014	0.0025	0.16	2.75	0.12

1. *Parameters of GDOES/Bulk analysis: U = 800 V, I = 30 mA, p(Ar) = 314 Pa.

For the impact test, samples sized 10 x 10 x 55 mm were manufactured and fitted with a V-notch 2 mm deep, with angle 45° and radius of the notch root ρ = 0.25 mm. These samples were subsequently heat-treated through quenching and tempering as recommended in the steel standard. The surfaces of specimens were grinded to the value of Ra = 0.4 μm prior to the plasma nitriding process, thereafter specimens were degreased in acetone and dried. Thus prepared experimental samples were subsequently plasma nitrided in the PN 60/60 RÜBIG device according to the parameters marked in **Table 2**. Before the actual process of plasma nitriding was the procedure of plasma cleaning performed under following conditions: T = 480 °C for 30 min, p = 80 Pa in a gas mixture 20H2: 2N2 (l/h).

Table 2 Parameters of plasma nitriding process

Parameter	Plasma cleaning	Plasma nitriding					
		I		II		III	
Temperature [°C]	480	450	450	500	500	550	550
Time/Duration [h]	0.5	10	30	8	25	6	20
Flow H_2 [l/h]	20	24	24	24	24	24	24
Flow N_2 [l/h]	2	8	8	8	8	8	8
Pressure [Pa]	80	280	280	280	280	280	280

Twenty pieces of samples were prepared for each type of plasma nitriding process. Five pieces of samples were selected for the metallographic evaluation and evaluation of case depth and fifteen pieces of samples for verification of the dynamic parameters through the notch toughness test. The group of fifteen samples, plasma nitrided under same condition, was divided into three subgroups of five samples. Each of subgroups was tested using the instrumented Charpy hammer under different test temperatures (i. e. - 40 °C, +21 °C and +70 °C). The metallographic evaluation of the plasma nitrided steel specimens with V-notches was performed on the metallographically prepared cross-sections by the optical microscope OLYMPUS GX51. Next, the surface hardness (HV1) and the nitrided layer depth measuring were carried out. The depth profiles of the nitride layers in roots of V-notches were evaluated by the measuring of microhardness profiles using the automatic microhardness tester LECO M-400-H in accordance to the DIN 50190-3 standard [8]. The microhardness was measured by Vickers microhardness method under 50 g loading (HV 0.05) and 10 s dwell time in the two selected areas of V-notches labelled as "I" and "II" (see **Fig. 1**). The actual evaluation of notch toughness of plasma nitrided samples with V-notches was verified using the instrumented Charpy hammer RKP 450 ZWICK IWI with nominal energy 300 J in accordance to the ISO 148-1 [6] and ISO 14556 standards [7]. The impact velocity of the pendulum was 5.234 m/s. in case of measuring under lower and higher temperatures, the silicon oil was used as cooling/heating medium. The values of notch toughness were calculated from the measured impact energy values [9, 10]. The fractographic analysis of fracture surfaces was carried out by means of an electron scanning microscope (SEM) TESCAN Vega TS 5135. The morphology of fracture was documented on individual samples in the axis of fracture perpendicular to the notch, in the distance approx. 1/3 from the notch, applying the x100 and x1000 zoom.

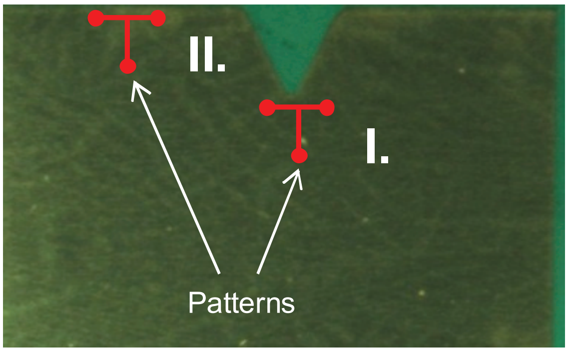


Fig. 1 Image of measured sample; location (position) of measurement pattern on the bottom of V-notch root and pattern of surface of sample

3. RESULTS AND DISCUSSION

3.1. Metallographic analysis, microhardness and surface hardness measurements

The metallographic analysis of plasma nitrided samples from each series was evaluated using the light microscope OLYMPUS GX 51. The evaluation was focused on the analysis of formed nitrided layers with focusing on presence of brittle compound layer in the V-notch roots and on the microstructure of steel. The formed layers were analyzed on the areas of V-notches roots, marked as "I" in **Fig. 2**.

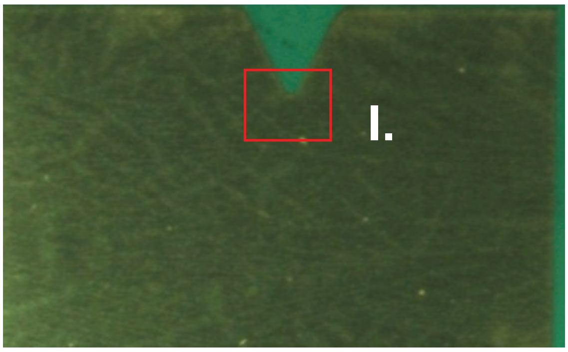


Fig. 2 Marking the measured areas of nitrided samples

The evaluation of the microstructure in areas of V-notches showed that in any case of plasma nitriding the compound layer in V-notch root were not created. The created nitrided layers were formed only by diffusion layers and the microstructure of nitrided samples was determined by tempered martensite and sorbite. Metallographic images of V-notch roots after selected plasma nitriding processes are shown in **Fig. 3** up to **Fig. 5**. The next step was the measurement of created nitrided layers in V-notch roots. Microhardness profile measurements were performed in roots of notches, marked as "I" and on the surface of samples marked as "II", in **Fig. 1** and these values was converted to the average values which were summarized for all modes of plasma nitriding in **Table 3**. The differences between shorter and longer nitriding duration were approximately of 0.01 mm in V-notch roots, although the differences between shorter and longer nitriding duration on surfaces were higher than twice. Increased case depths in V-notch roots were achieved after shorter nitriding duration and the lowest values were obtained at nitriding temperature under 550 °C (see **Table 3**). The decreased values of case depth in all V-notch roots are probably caused by a small diameter of V-notch root and therefore behave as a blind cavity with limited access of plasma [11].

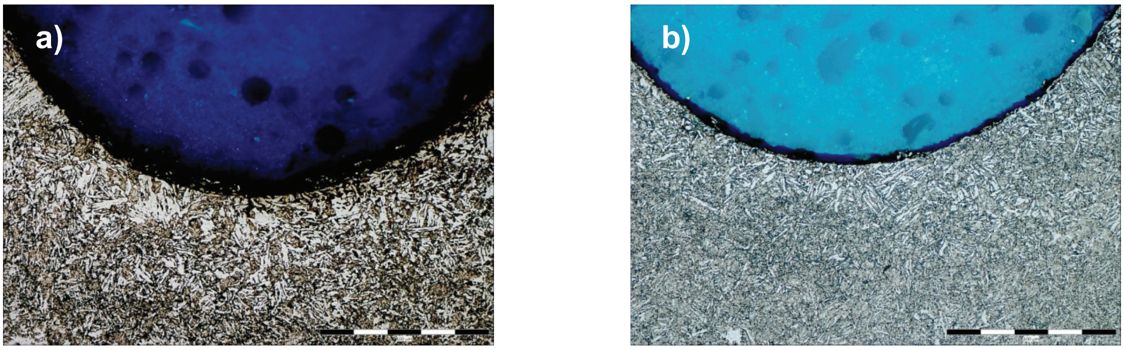


Fig. 3 Microstructure of V-notch roots after 10 h (a) and after 30 h (b) of nitriding at 450 °C

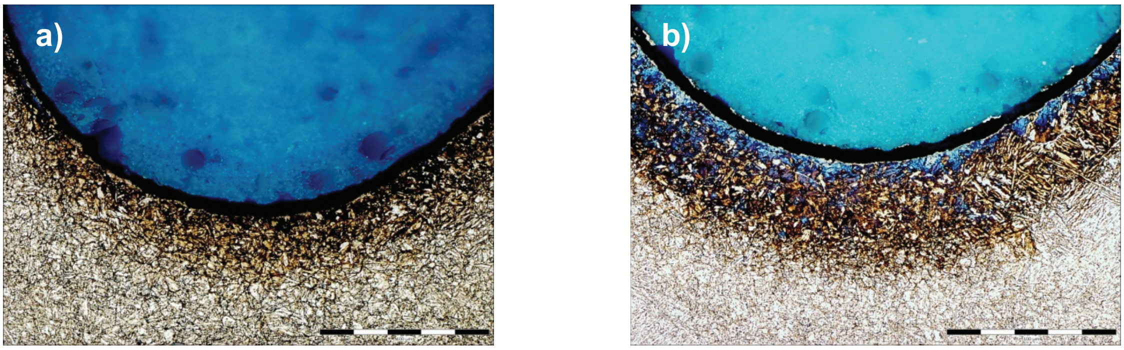


Fig. 4 Microstructure of V-notch roots after 8 h (c) and after 25 h (d) of nitriding at 500 °C

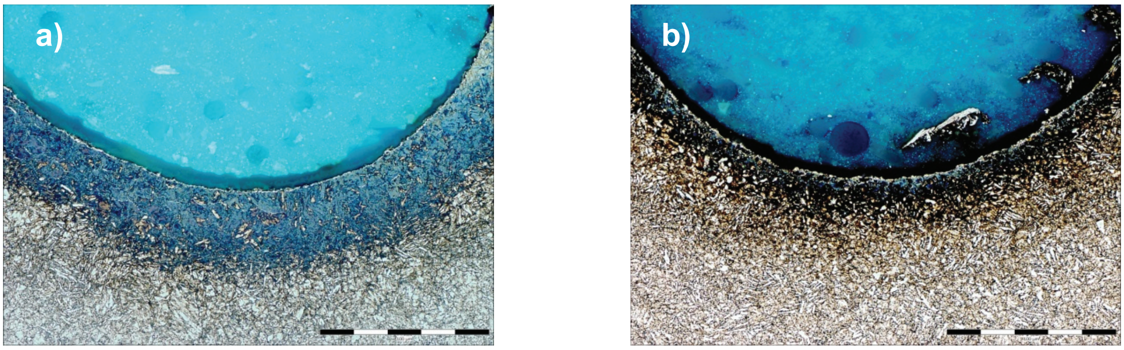


Fig. 5 Microstructure of V-notch roots after 6 h (a) and after 20 h (b) of nitriding at 550 °C

It is known that the case depth of nitride layer affects a number of mechanical properties of nitrided steel such as surface hardness, fatigue limit and toughness [4, 5, 12]. The values of surface hardness of the material may serve as one of the important indicators of the assumed resulting toughness. For this reason, the surface hardness was evaluated. The surface hardness of plasma nitrided steel specimens was measured using the instrumented hardness tester ZWICK Roell ZHU 2.5. Five measurements were carried out on each sample and the surface hardness was set as the average value of these five values. The determined surface hardness values reached higher values, because of presence of the alloying elements in the case of used 30CrMoV6 (CSN 41 5330) steel. These alloying elements with high affinity for nitrogen like Al, Mo, V, Cr and W [13, 14]. The monitored steel contains especially Mo, V and Cr. The results of the surface hardness of plasma nitrided samples are summarized in **Table 3**. The results shows that the surface hardness after plasma nitriding was increased almost three times, from the original value of 400 HV1 (quenched and tempered before plasma nitriding) up to 1167 HV1 (after plasma nitriding process at 500 °C and nitriding duration 8 hours).

Table 3 The results of measurements

Plasma nitriding parameters		Measured case depth [mm]		Surface hardness [HV 1]
Temperature [°C]	Time [h]	Surface	V-notch	
Ref.*	0	/	/	400.10 ± 0.40
450	10	0.15	0.07	1044.60 ± 8.46
	30	0.31	0.08	1150.53 ± 4.87
500	8	0.19	0.09	1167.73 ± 21.44
	25	0.37	0.08	1009.27 ± 0.77
550	6	0.16	0.06	985.10 ± 23.98
	20	0.31	0.05	1037.26 ± 12.11

3.2. Charpy impact test

A fundamental part of the experimental work was to perform the instrumented Charpy test in accordance to the ISO 148-1 [6] and ISO 14556 standards [7]. The results of the absorbed energy (impact energy) and the results of achieved notch toughness values of reference samples compared with nitrided samples for all test temperatures are shown in **Table 4**.

Table 4 The results of absorbed energy a notch toughness at selected test temperatures

Plasma nitriding parameters		Test temperature [°C]	Absorbed energy KV [J]	Notch toughness KCV [J·cm^{-2}]
Temperature [°C]	Duration [h]			
0 (Ref.*)	0 (Ref.*)	-40	73.29 ± 2.79	91.33 ± 3.72
450	10		25.16 ± 4.85	31.39 ± 6.05
	30		13.25 ± 0.50	16.51 ± 0.63
500	8		19.79 ± 4.85	24.63 ± 6.02
	25		15.08 ± 1.70	18.80 ± 2.12
550	6		23.83 ± 1.20	29.90 ± 1.40
	20		26.88 ± 5.26	33.66 ± 6.54
0 (Ref.*)	0 (Ref.*)	+21	109.87 ± 14.13	136.87 ± 17.00
450	10		100.30 ± 2.89	125.37 ± 3.39
	30		75.09 ± 4.86	93.92 ± 6.17
500	8		95.06 ± 5.85	118.75 ± 7.41
	25		76.29 ± 0.79	95.36 ± 0.82
550	6		90.44 ± 4.24	113.19 ± 5.11
	20		70.67 ± 5.97	88.40 ± 7.54
0 (Ref.*)	0 (Ref.*)	+70	94.52 ± 5.08	117.26 ± 6.31
450	10		89.38 ± 1.63	111.72 ± 2.44
	30		71.00 ± 3.63	88.38 ± 4.57
500	8		82.08 ± 4.09	102.80 ± 5.39
	25		54.55 ± 5.12	68.15 ± 6.46
550	6		72.26 ± 1.89	90.95 ± 2.62
	20		80.54 ± 2.09	100.74 ± 2.89

The values of absorbed energy and notch toughness achieved the lowest results at test temperature -40 °C (the decrease was approx. of 70 % compared to the values obtained at the temperature +21 °C). Though the highest values of absorbed energy and notch toughness was not achieved at test temperature +70 °C but at test temperature +21 °C (by approx. 15 %). Values of absorbed energy and notch toughness were increased under increased test temperatures. Though the highest values of absorbed energy and notch toughness was not achieved at test temperature +70 °C but at test temperature +21 °C (by approx. 15 %). The results also showed that longer nitriding duration at the same nitriding temperature tended to lower values of absorbed energy and notch toughness except plasma nitriding at 550 °C and test temperature -40 °C and +70 °C. It is also evident a tendency of slight decrease of absorbed energy values and notch toughness with increasing of nitriding temperature in case of shorter nitriding duration. Longer nitriding duration led to higher values of absorbed energy and notch toughness only at nitriding temperature 450 °C. Higher nitriding temperatures achieved the maximum values of notch toughness after 20 hours at nitriding temperature 550 °C and test temperature -40 °C, after 25 hours at nitriding temperature 500 °C and test temperature +21 °C and after 20 hours at nitriding temperature 550 °C and test temperature +70 °C. It is evident that all selected modes of plasma nitriding processes caused a decrease of values of absorbed energy and notch toughness compared with reference not nitride samples. These findings are consistent with previous studies [4, 5, 14, 15, 16]. The notch toughness values dependence on achieved nitrided layer depths in the V-notch roots after plasma nitriding at 450 °C, 500 °C and 550 °C is displayed in **Fig. 6**. It is evident that values of notch toughness decreases with increasing of case depth in V-notch roots. In this case of plasma nitriding process at 450 °C, it was achieved higher value of case depth after longer nitriding duration. In case of nitriding temperature 500 °C, higher value of case depth after shorter nitriding duration was achieved. This trend was also the same in the case of notch toughness values. The highest nitriding temperature also led to creation of higher value of case depth after shorter nitriding duration. The values of notch toughness actually with increasing nitriding duration were increased slightly except the value of notch toughness achieved at test temperature +21 °C. The graphical dependencies of notch toughness on the case depths in V-notch roots is displayed in **Fig. 6**, it is evident that the highest values of notch toughness were really achieved at test temperature +21 °C. The explanation of this phenomenon has not been done in this work.

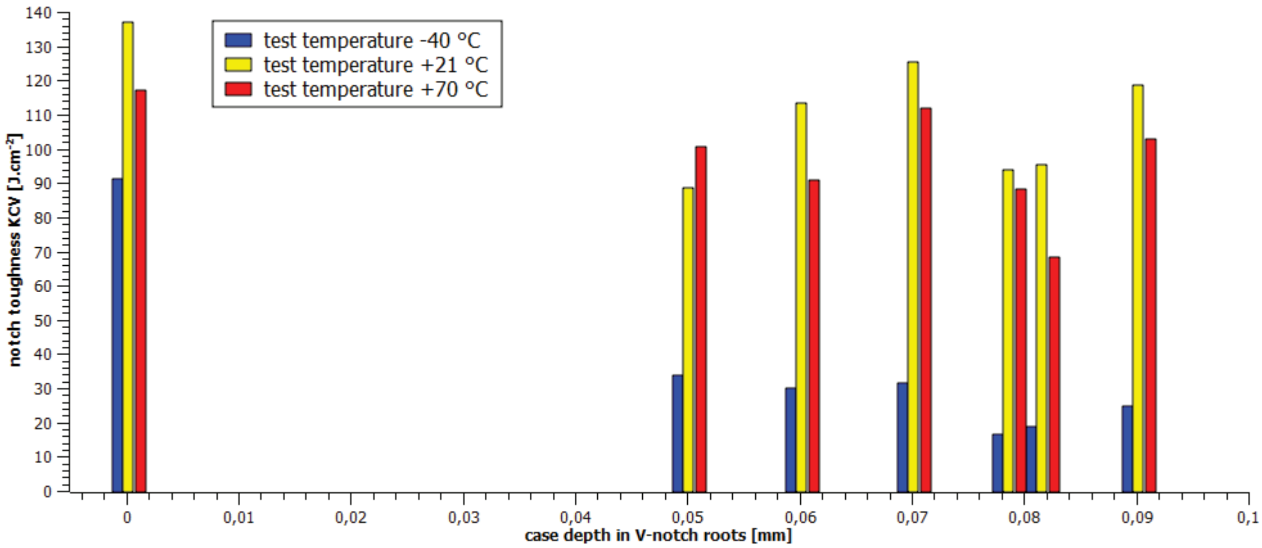
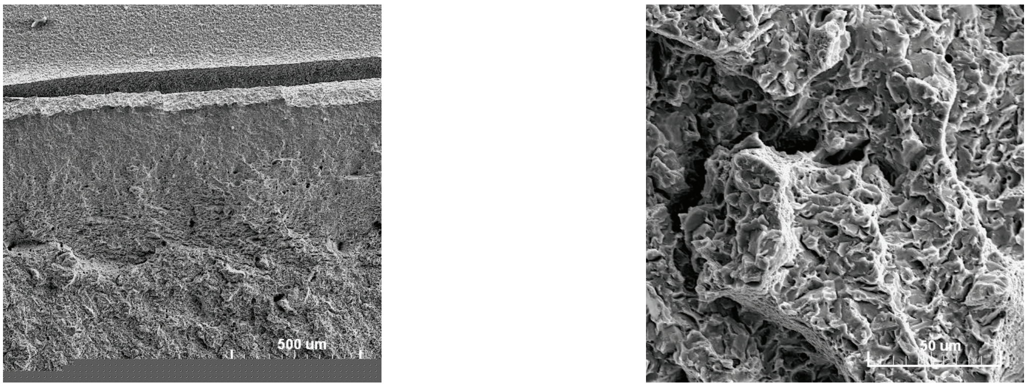


Fig. 6 The notch toughness dependence on case depths in V-notch roots

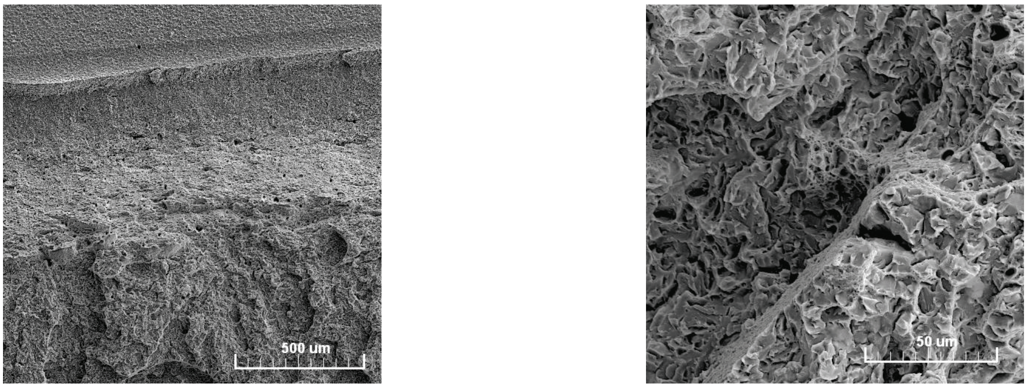
3.3. Fractography

The last part of the experimental work was a fractography analysis of fracture surfaces of broken samples. It is evident, from preceding measurements, that more significant decrease of the notch toughness values was identified at the lowest test temperature -40 °C which was approx. of 70 % compared to the value determined at the temperature +21 °C. The failure mechanism in the lowest temperature range of test temperatures was evaluated as transcrystallic brittle fracture (**Fig. 7**).



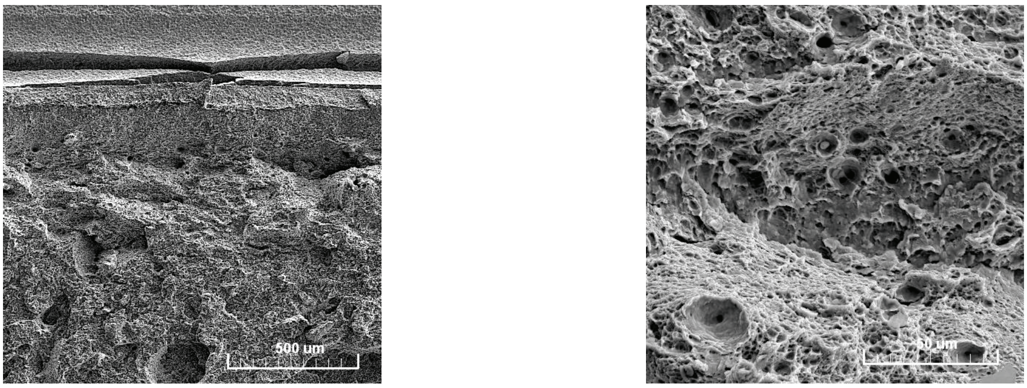
A B

Fig. 7 Micromorphology of fracture surface of plasma nitrided sample. Plasma nitriding 450 °C, 10 hours, test temperature -40 °C. Magnification 100x - A, 1000x - B



A B

Fig. 8 Micromorphology of fracture surface of plasma nitrided sample. Plasma nitriding 450 °C, 10 hours, test temperature +21 °C. Magnification 100x - A, 1000x right - B



A B

Fig. 9 Micromorphology of fracture surface of plasma nitrided sample. Plasma nitriding 450 °C, 10 hours, test temperature +70 °C. Magnification 100x - A, 1000x - B

It has been proved that the type of failure had an influence on the notch toughness values. The results of the fractographic analysis show that in the zone under the notch root and in the sphere along the edges of the test samples only the ductile dimple fracture mostly of shear character was identified. Micromorphology of fracture surface of the sample at a test temperature of +21 ° C has corresponded to brittle transcrystallic failure with areas of transcrystallic ductile failure, i.e. it is a mixed fracture (see **Fig. 8**). A different failure mechanism of 30CrMoV9 (CSN 41 5330) steel was identified at the highest testing temperatures. For this case the transcrystallic ductile fracture mechanism predominate was identified (**Fig. 9**). As shown in **Figs. 7 up to 9** with the change of test temperature the morphology of fracture surface was changed too. This phenomenon occurred in all temperature of plasma nitriding processes. The micromorphology of fracture surfaces clearly shows a change of nature of fracture surface of V-notch. On the micromorphology of fracture surfaces is the change of the nature of fracture surface of V-notch visible. Brittle fracture characteristic for nitrided layer passes into ductile fracture towards the core of material, which is evident from **Fig. 8** and **Fig. 9**. An exception was found in the case of the fracture surface at a test temperature of -40 ° C, consisting of only brittle failure (see **Fig. 9**).

4. CONCLUSION

In this article was the main aim to determine the influence of plasma nitriding processes on to changes of selected mechanical parameters of nitrided 30CrMoV9 (CSN 41 5330) steel. It was experimentally found and confirmed that the plasma nitriding process has an effect on the reduction of the notch toughness values in case of 30CrMoV9 (CSN 41 5330) steel. Although the case depth in the V-notch roots achieved only hundredths of a millimeter, the notch toughness values were significantly reduced especially at test temperature of -40 °C. It was found that experimentally obtained KCV values at test temperature -40 °C were decreased by approx. of 70 % compared to the value determined at the test temperature +21 °C. Based on the comparison of the fractographic analysis results carried out for all test temperatures and determined KCV values, it can be concluded that the limit state occurrence was detected at temperature -40 °C, because the micromorphology of failure surface determined at this test temperature had quite brittle fractures. The experimental work confirmed the fact, that plasma nitriding increases the surface hardness but simultaneously decreases the notch toughness values. The most noticeable difference was recorded at low temperature test.

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REFERENCES

[1] GRÄFEN, W., EDENHOFER, B. New developments in thermos-chemical diffusion processes. In Surface and Coatings Technology, Vol. 200, 2005, pp. 1830-1836.

[2] ALVES, C., Da SILVA, E. F., MARTINELLI, A. E. Effect of workpiece geometry on the uniformity of nitrided layers, In Surface and Coatings Technology, Vol. 139, 2001, pp. 1-5.

[3] KARAKAN, M., ALSARAN, A., CELIK, A. Effect of various gas mixture on plasma nitriding behavior of AISI 5140 steel, In Mater. Character., Vol. 49, 2003, pp. 241-246.

[4] DOBROCKÝ, D., DOAN, T., KUSMIČ, D., HRUBÝ, V. (2014): The Change of Notch Toughness Parameters of Steel After Plasma Nitriding. In ICMT´- 2014, pp. 66-74.

[5] DOBROCKÝ, D., KUSMIČ, D. (2015): The Effect of Plasma Nitriding Process on the Change of Dynamic Parameters of Steel DIN 1654/4. In Manufacturing Technology, Vol. 15, No. 1, pp. 14 - 20.

[6] ISO 148-1:2009 Metallic materials - Charpy pendulum impact test - Part 1: Test method

[7] ISO 14556:2000 Steel - Charpy V-notch pendulum impact test - Instrumented test method

[8] DIN 50190-3 - Hardness depth of heat-treated parts; determination of the effective depth of hardening after nitriding. Deutsches Institut für Normung E. V., 1979.

[9] YU, H. L., JEONG, D. Y. Application of stress-triaxiality dependent fracture criteria for unnotched Charpy specimens. In G. C. SIH et al. (Eds.), Transferability and Applicability of Current Mechanics Approaches. ECUST: Shanghai, 2009, pp. 41-51.

[10] YU, H. L., JEONG, D. Y. Application of stress-triaxiality dependent fracture criterion in the finite element analysis of unnotched Charpy specimens, In Theor. Appl. Mech., Vol. 54, 2010, pp. 54-62.

[11] POKORNÝ, Z., KADLEC, J., HRUBÝ, V., JOSKA, Z., TRAN, D. Q. Mechanical Properties of Steels after Plasma Nitriding Process. In Journal of Materials Science and Engineering, Vol. A1, 2011, pp. 42-45.

[12] POKORNÝ, Z., KADLEC, J., HRUBÝ, V., JOSKA, Z., TRAN, D. Q., BERAN, D. Plasma nitriding of bored barrels. In Advances in Military Technology, Vol. 20, No. 20, 2011, pp. 69-76.

[13] HOLEMÁŘ, A., HRUBÝ, V. Iontová nitridace v praxi. SNTL, Praha, 1989, pp. 178-180.

[14] PYE, D. Practical nitriding and ferritic nitrocarburizing. 2nd edition, Ohio: ASM International Materials Park, 2003, pp. 127-129.

[15] LATTNER, M., HOLESOVSKY, F. Effect of Machining the Load Capacity Notched Components. In Manufacturing Technology, vol. 14, 2014, pp. 47-50.

[16] MADL, J., RAZEK, V., KOUTNY, V., KAFKA, J. Surface Integrity in Notches Machining. In: Manufacturing Technology, vol. 13, 2013, pp. 188-193.