EFFECT OF PLASMA NITRIDING ON THE NOTCH TOUGHNESS OF SPRING STEEL

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Abstract

Specimens made from spring steel 54SiCr6 were nitrided using various parameters of plasma nitriding in order to achieve a layer of variable thickness. The microstructure, phase constitution, hardness and case hardness depth were investigated. Subsequently notch toughness was measured by the instrumented Charpy impact test on 10x10x55 mm test specimens with U-notch 5 mm depth. Instrumented Charpy impact test was carried out at -40 °C, +21 °C and +70 °C. Results of notch toughness were correlated with the structure, chemical composition and the depth of the nitrided layers both on the surface and especially in U-notch of specimens. In the end the fractographic analysis of fracture surfaces were carried out. The notch toughness is negatively influenced as a result of presence of a nitrided layer on the surface of root of U-notch and decrease again as the thickness of nitrided layer increases and with decrease in temperature. It turned out, if the nitrided layer in root of U-notch was “sufficiently” thick, the crack spread had unstable course. The change of the character of the crack spread was also influenced by temperature.

Keywords: Plasma nitriding, notch toughness, U-notch, case depth

1. INTRODUCTION

54SiCr6 steel is low-alloyed noble silicon-chromium spring steel suitable for production of spring components for cars, trucks, as well as railway cars. Fast clips, the components of the railway superstructure, are also interesting example of using spring steel. The advantage of 54SiCr steel is high toughness. The disadvantage is the low corrosion resistance. Spring steel must have good static and dynamic properties and must have a high fatigue life [1]. Flexible components are dynamically loaded machine components which are subjected to high level of operating load at superposition of environment influences (temperature, temperature gradient, aggressiveness environment etc.). It all induces the processes inside the material structure which lead to degradation of the material. Gradual accumulation of degradation processes can then lead to the marginal state of the material [2]. One possible solution to increase utility properties of spring steel is use of plasma nitriding process. Plasma nitriding process is a chemical-heat treatment widely used to increase the surface hardness, fatigue strength, wear and corrosion resistance [3, 4, 5]. For this reason could plasma nitriding increase the utility properties of components made from spring steel. However, the disadvantage of plasma nitrided components is decrease of their notch toughness [6]. It is, in the case of dynamically loaded flexible components, undesirable property. The aim of this work is to determine the effect of various parameters of plasma nitriding on the notch toughness of 54SiCr6 spring steel.

2. EXPERIMENTAL

2.1. Surface treatment

Experimental work was focused on verification of selected mechanical properties of plasma nitrided 54SiCr6 spring steel in wide range of temperatures, through the instrumented Charpy impact test. For the experimental part the standardized test specimens (dimensions 10x10x55 mm) were used. Test specimens, provided with U-notch depth of 5 mm, were heat-treated in accordance with **Table 1** and plasma nitrided according to the parameters specified in **Table 2**. Plasma nitriding was carried out in the PN 60/60 device from RÜBIG

Company. One-step process of plasma nitriding was used for the test specimens. Furthermore, the standard nitriding atmosphere, i.e. the ratio of gas mixture of $H_2 : N_2 = 3 : 1$ and a pressure 280 mbar, was used. When the plasma nitriding was done, part of the test specimens was cut into specified parts on which were measured mechanical and chemical parameters.

Table 1 Parameters of heat-treatment

Procedure	Temperature [°C]
Oil quenching	850
Air tempering	580

Table 2 Parameters of plasma nitriding process

Temperature [°C]	Duration [h]	Gas flow H_2/N_2 [$l \cdot min^{-1}$]	Bias [V]	Pressure [mbar]	Pulse length [μs]
450	10	24/8	530	280	100
	30				
500	8				
	25				
550	6				
	20				

2.2. Experimental methods

Analysis of the chemical composition was performed by optical emission spectroscopy (GDOES)/Bulk method on LECO SA 2000 spectrometer. Chemical composition of the above-specified steel as per the DIN standard is stated in **Table 3**. Microstructure was evaluated by optical microscope Olympus GX51. The surface hardness was measured by the instrumented hardness tester Zwick ZHU 2.5 (measured at load 9.807 N). Thickness and microhardness of plasma nitrided layers were measured by microhardness testing method in accordance with standard DIN 50190 [7] on automatic microhardness tester LECO LM 47 AT. The fundamental part of experiments was devoted to instrumental Charpy impact test which was performed on instrumented pendulum hammer Zwick Roel RPK 450 with a nominal energy of 300 J and impact velocity of 5.234 $m \cdot s^{-1}$. Charpy impact test were performed on specimens, which were heated and cooled to -40 °C, +21 °C and +70 °C. In case of measuring at -40 °C and +70 °C, synthetic oil was used as cooling and heating medium. Fractographical analysis of fracture surfaces was performed on the environmental scanning microscope Tescan Vega TS 5135 and a scanning electron microscope JEOL JSM-6490LV. The goal of the fractographic analysis was to assess, in terms of quality, the influence of temperature and nitrided layer on the character of fracture surfaces obtained from the notched impact strength test. The morphology of fracture was photographed on individual specimens in the axis of fracture perpendicular to the notch, applying x100 and x1000 magnification.

Table 3 Chemical composition of 54SiCr6 spring steel

C	Mn	Si	P	S	Ni	Cr	Cu
GDOES/Bulk							
0.51	0.73	1.59	0.007	0.011	0.04	0.69	0.04
DIN Standard							
0.50-0.60	0.50-0.80	1.30-1.60	max. 0.035	max. 0.035	max. 0.50	0.50-0.70	max. 0.30

2.3. Experimental results

Metallographic analysis of specimens was performed in the core of specimen, in area under the U-notch and on the specimen surface. A fundamental area of metallographic analysis, of course, was evaluation of microstructure in roots of U-notches. Microstructure of 54SiCr6 steel formed after heat treatment and subsequent plasma nitriding at 450 °C (duration time of 10 h and 30 h), is documented in **Figure 1**. Structure of tempered martensite is evident in the roots of the U-notches. It is also obvious significant horizontal structure. It is also visible very thin white layer of nitrides. Tempered martensite was observed in areas of U-notch roots after plasma nitriding at 500 °C (duration time 8 h and 25 h). In this case of plasma nitriding it is also visible a very thin white layer of nitride (see **Figure 2**). Complete absence of white layer is possible to see on the evaluation of microstructure in areas of U-notches in case of plasma nitriding at 550 °C (both 6 h and 20 h). Structure is composed of tempered martensite and sorbite (**Figure 3**).

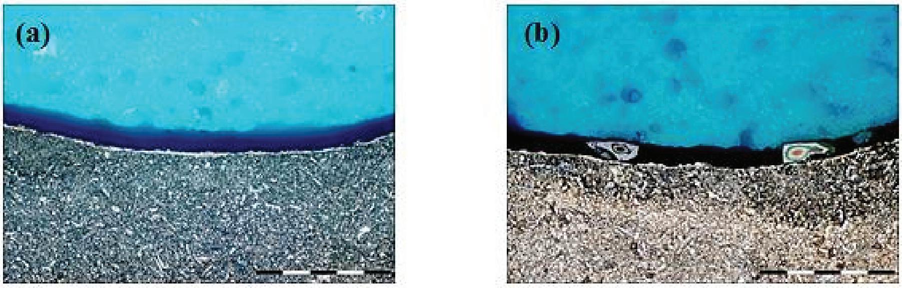


Figure 1 The microstructure of the root area of the U-notch; (a) plasma nitriding at 450 °C/10 h, (b) plasma nitriding at 450 °C/30 h, 2% Nital, 500x

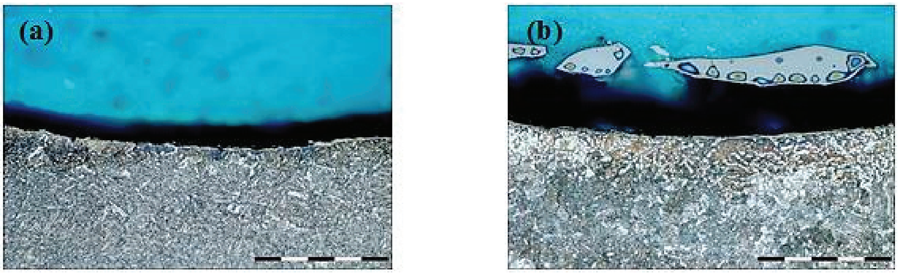


Figure 2 The microstructure of the root area of the U-notch; (a) plasma nitriding at 500 °C/8 h, (b) plasma nitriding at 500 °C/25 h, 2% Nital, 500x

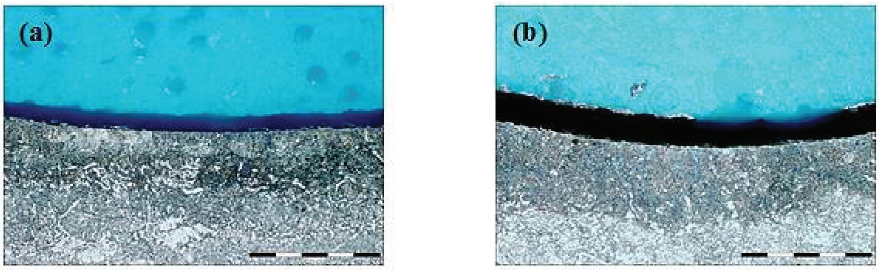


Figure 3 The microstructure of the root area of the U-notch; (a) plasma nitriding at 550 °C/6 h, (b) plasma nitriding at 550 °C/20 h, 2% Nital, 500x

Based on the results of measurement of microhardness of the individual specimens, the case depths of formed nitrided layer were graphically evaluated. The microhardness and thickness of nitrided layer was measured by Vickers microhardness method at 50 g load (HV 0.05) and 10 s dwell time. The primary measurement was performed from the surface of the notch root towards the core material, the secondary measurement from the “free” surface of specimens towards the core (specimen surface outside the notch). Comparing the values of the case depth on the surface of the test specimens and in the roots of U-notches, for all regimes of plasma nitriding, is shown in **Table 4**. We cannot say that the increase of duration time of the plasma nitriding leads to an increase in the case depth of the nitrided layer in the notch roots.

Table 4 The measured case depths of the nitrided layers on the surface and in the roots of the U-notches

Parameters of plasma nitriding		**Measured case depth of nitrided layer [mm]**	
Temperature [°C]	**Duration [h]**	**Surface**	**U-notch**
450	10	0.15	0.09
	30	0.32	0.11
500	8	0.16	0.12
	25	0.26	0.12
550	6	0.12	0.13
	20	0.23	0.07

Surface hardness of the specimens was measured by the Vickers method in accordance with standard EN ISO 6507-1 [8]. The loading of indenter was 9.807 N (HV_1). The results of measuring the surface hardness of heat-treated test and plasma nitrided specimens are shown in **Table 5**.

Table 5 Results of surface hardness measurements

Parameters of plasma nitriding		**Hardness**
Heat-treated specimens		426.20 ± 12.15
450 °C	10 h	749.00 ± 8.71
	30 h	865.70 ± 8.01
500 °C	8 h	910.70 ± 3.04
	25 h	945.73 ± 3.19
550 °C	6 h	853.50 ± 16.06
	20 h	800.53 ± 0.39

The basic characteristic, i.e. the energy consumed on the deformation and fracture of a specimen, the instrumented pendulum enabled to determine, during impact loading, the relation of power *F* and the time *t*. The values of notched impact strength were calculated from the measured impact energy values according the Equation 1:

$$KCU = \frac{KU}{S_0} \; [J/cm^2] \tag{1}$$

where *KU* stands for impact energy and S_0 stands for the cross section of a rod at the point of the notch (*h x b* in cm^2).

In areas of higher test temperatures are values of notch toughness shifted towards higher values. Decrease of test temperature led to decrease of notch toughness. Reference (heat-treated) specimens reached higher values of notch toughness than plasma nitrided specimens. With the increase of nitriding duration, which in most cases led to achieve to greater case depths in the roots of U-notches, was associated decrease of notch toughness values, but this dependence was not always be true. The highest notch toughness values were obtained after nitriding at 550 °C and it can be argued that this regime of plasma nitriding process was most suitable for test specimens provided with U-notches. Minor differences between the individual values of notch toughness at selected test temperatures were achieved even at a nitriding temperature of 450 °C. The achieved results of notch toughness, for all regimes of plasma nitriding, are shown in **Table 5** up to **Table 7**.

Table 5 Results of notch toughness of 54SiCr6 steel (plasma nitriding at 450 °C, duration 10 h and 30 h)

Test temperature +21 °C

Parameters of plasma nitriding	$\overline{KU}$ [J]	$\overline{KCU}$ [J/cm^2]	Case depth [mm]
Heat-treated specimens	16.40 ± 0.33	34.38 ± 2.46	-
450 °C; 10 h	10.41 ± 0.78	21.12 ± 1.62	0.09
450 °C; 30 h	10.00 ± 2.63	20.28 ± 5.35	0.11

Test temperature -40 °C

Heat-treated specimens	12.29 ± 0.54	24.99 ± 1.22	-
450 °C; 10 h	2.47 ± 0.68	4.99 ± 1.36	0.09
450 °C; 30 h	1.47 ± 0.19	2.97 ± 0.40	0.11

Test temperature +70 °C

Heat-treated specimens	19.26 ± 0.85	39.14 ± 1.81	-
450 °C; 10 h	14.90 ± 1.80	30.19 ± 3.71	0.09
450 °C; 30 h	12.93 ± 1.61	26.16 ± 3.24	0.11

Table 6 Results of notch toughness of 54SiCr6 steel (plasma nitriding at 500 °C, duration 8 h and 25 h)

Test temperature +21 °C

Parameters of plasma nitriding	$\overline{KU}$ [J]	$\overline{KCU}$ [J/cm^2]	Case depth [mm]
Heat-treated specimens	16.40 ± 0.33	34.38 ± 2.46	-
500 °C; 8 h	5.79 ± 0.44	11.75 ± 0.92	0.08
500 °C; 25 h	7.03 ± 1.10	14.22 ± 2.20	0.12

Test temperature -40 °C

Heat-treated specimens	12.29 ± 0.54	24.99 ± 1.22	-
500 °C; 8 h	4.42 ± 0.46	8.96 ± 0.95	0.08
500 °C; 25 h	2.74 ± 0.74	5.56 ± 1.49	0.12

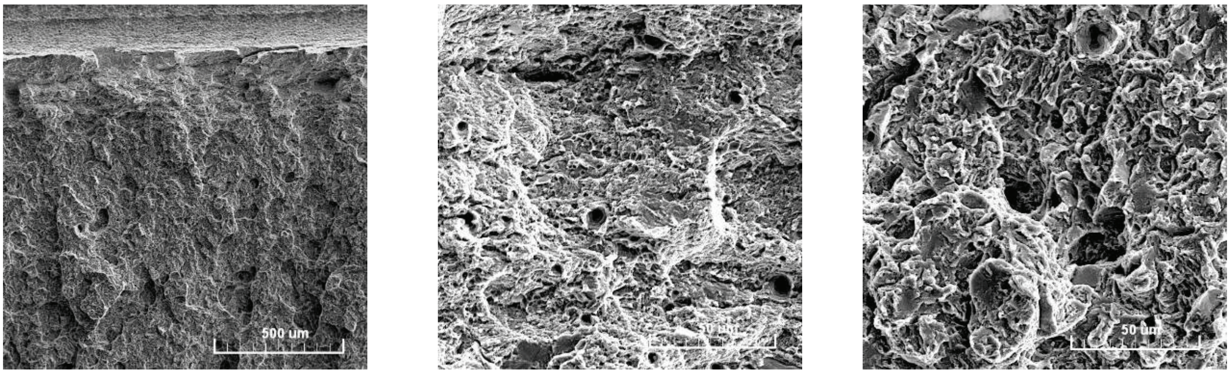
Test temperature +70 °C

Heat-treated specimens	19.26 ± 0.85	39.14 ± 1.81	-
500 °C; 8 h	13.21 ± 0.26	26.77 ± 0.59	0.08
500 °C; 25 h	12.35 ± 1.32	25.04 ± 2.74	0.12

Fractographical analysis determined the type of fracture and deformation characteristics of the fracture surface in areas under roots of U-notches and in cores of specimens. The failure mechanism in the lower temperature range of test temperatures was transcrystalline quasi-cleavage. A different failure mechanism was identified at higher temperatures (transcrystalline ductile), but no influence on the notch toughness values has been proved. The results of the fractographic analysis show that in the zone under the notch and in the sphere along the edges of the test specimens only ductile dimple fracture mostly of shear character was identified. It follows the below-provided fractographis analysis photographs (**Figure 4**) that the degree of directing of fracture micromorphology grows (rolling direction applies more) with growing test temperature in specimens of steel. As shown in **Figure 5** and **Figure 6**, in the zone under the notch is highly visible sharply demarcated region of the nitrided layer, which is characterized by a different mechanism of failure. The failure mechanism is transcrystalline quasi-cleavage; there is smaller portion of ductile failure compared with heat-treated specimens.

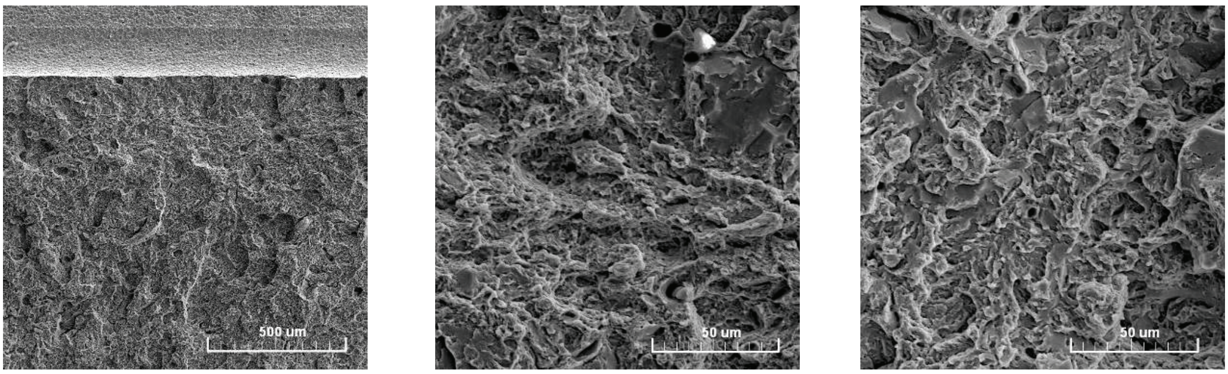
Table 7 Results of notch toughness of 54SiCr6 steel (plasma nitriding at 550 °C, duration 6 h and 20 h)

Parameters of plasma nitriding	$\overline{\mathrm{KU}}$ [J]	$\overline{\mathrm{KCU}}$ [J/cm²]	Case depth [mm]
Test temperature +21 °C			
Heat-treated specimens	16.40 ± 0.33	34.38 ± 2.46	-
550 °C; 6 h	11.70 ± 1.57	23.70 ± 3.15	0.13
550 °C; 20 h	12.23 ± 2.10	24.68 ± 4.21	0.07
Test temperature -40 °C			
Heat-treated specimens	12.29 ± 0.54	24.99 ± 1.22	-
550 °C; 6 h	3.99 ± 0.17	8.10 ± 0.37	0.13
550 °C; 20 h	4.00 ± 0.81	8.06 ± 1.57	0.07
Test temperature +70 °C			
Heat-treated specimens	19.26 ± 0.85	39.14 ± 1.81	-
550 °C; 6 h	15.08 ± 0.50	30.62 ± 0.94	0.13
550 °C; 20 h	16.91 ± 2.56	34.29 ± 5.22	0.07



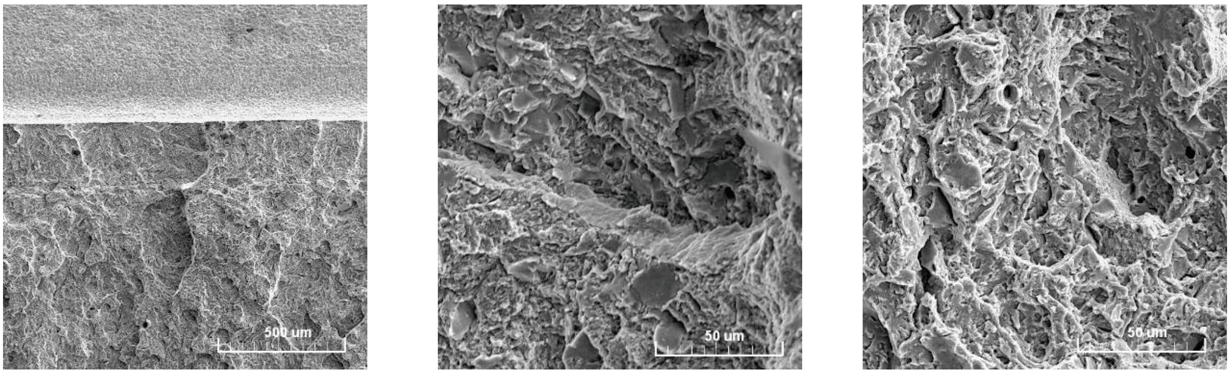
The zone under U-notch; 100x — The zone under U-notch; 1000x — The core of specimen; 1000x

Figure 4 Morphology of fracture of heat-treated specimen (test temperature +21 °C)



The zone under U-notch; 100x — The zone under U-notch; 1000x — The core of specimen; 1000x

Figure 5 Morphology of fracture of plasma nitrided specimen (nitrided at 450 °C, duration 10 h, test temperature +21 °C)



The zone under U-notch; 100x The zone under U-notch; 1000x The core of specimen; 1000x

Figure 6 Morphology of fracture of plasma nitrided specimen (nitrided at 450 °C, duration 30 h, test temperature +21 °C)

3. CONCLUSION

The aim of the experimental programme was to obtain new knowledge about changing notch toughness of plasma nitrided specimens from spring steel 54SiCr6 and assess the influence of the test temperature on final notch toughness. The results of experimental measurements have shown that even a small depth of the nitrided layer formed in the roots of U-notches, negatively affecting notch toughness by reducing their values, which corresponds with the measurements in [6]. Part of the experimental programme was the determination of impact energy and notch toughness values for evaluation of the share of the above-stated quantities in the occurrence of degradation processes that directly influence a material failure mechanism. It follows from experimentally determined KCU values that the values decrease significantly during experiments carried out under lower temperatures. The evaluation of 54SiCr6 steel fracture surfaces after the impact test proved the failure mechanism in heat-treated steel to be transcrystalline ductile and in plasma nitrided steel to be transcrystalline quasi-cleavage. With the increase of nitriding duration and increase the case depth in the notch root, there was a decline in the proportion of ductile fracture in the fracture surfaces of specimens. Heat-treated samples reached higher values of notch toughness than plasma nitrided specimens. With the increase of nitriding duration, which in most cases led to achieve greater depth of the nitrided layer in the notch root, was associated decline in the values of notch toughness, but this dependence not always be true.

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