Optica Applicata, Vol. XXXIX, No. 4, 2009

Topography of X39Cr13 steel surface after heat and surface treatment

MONIKA GWOŹDZIK*, ZYGMUNT NITKIEWICZ

Institute of Materials Engineering, Częstochowa University of Technology,
al. Armii Krajowej, 42-200 Częstochowa, Poland

*Corresponding author: gwozdzik@mim.pcz.czest.pl

The paper presents results of surface condition examinations of martensitic X39Cr13 steel subject to heat and surface treatment (plasma nitriding). The heat treatment consisted of compressed nitrogen quenching from 1050 °C (1323 K) and two-hours tempering at 300 °C (573 K) and 620 °C (893 K) for specimens subject to nitriding. The plasma nitriding was carried out in an ion-nitriding installation with cooled anode, at temperature of 460 °C (733 K), at pressure of 150 Pa and during $t = 20$ h (72 ks); $25\%N_2+75\%H_2$ was used as the reactive atmosphere. Specimens surface was examined using a profilographometer designed for surface 2D and 3D examinations using a contact method. Surface topography measurements were carried out on specimens taken from 1 mm thick sheet. 1.5 mm × 1.5 mm surface was the measurement area. The sampling interval in *X* and *Y* axis was 1 μm, while the measuring speed was 0.5 mm/s. The studies carried out allowed evaluating the conditions of examined steel surface through the comparison of stereometric parameters of the surface.

Keywords: topography, martensitic steel, heat treatment, plasma nitriding.

1. Introduction

An intensive increase in the interest in alloys used in medicine, and in particular martensitic steels from the group of corrosion-resisting steels, has been witnessed in recent years [1–3]. The current state of knowledge of metallic materials creates newer and newer possibilities of their modification, and in particular enables manufacturing increasingly complex surface layers, which allow forming practical properties of these materials to much larger extent. The modification of martensitic steels surface layer through nitriding may increase the service life of instruments produced. The plasma nitriding is a surface treatment method, which apart from improving the service life ensures low energy consumption and high cleanliness from ecological point of view [1, 4, 5].

The analysis of surface topography is important for materials used in medicine; most often examinations are carried out using a profilographometer [6] or atomic force microscope [7, 8], which allow determining increasingly wide range of parameters and materials' surface properties.

2. Material and methodology of tests

Martensitic steel, classified as stainless steel, grade X39Cr13 according to standard PN-EN 10088-1:1998 [9], was selected for laboratory tests. Tests were carried out on 1 mm thick heat and surface treated (plasma nitrided) specimens.

The heat treatment consisted of compressed nitrogen quenching from the austenitising temperature of 1050 °C (1323 K). The time of holding at this temperature was 20 minutes (1.2 ks). After quenching the steel was subject to two-hours (7.2 ks) tempering at 300 °C (573 K) with compressed nitrogen cooling. Instead, prior to nitriding, the steel was tempered at 620 °C (893 K).

The plasma nitriding was carried out in a JON-600 ion nitriding installation with cooled anode. During the plasma nitriding the charge was heated as a result of surface bombardment by ions generated in the discharge plasma. After placing in the furnace and obtaining the required vacuum, the specimens during the first stage were rinsed with hydrogen for two hours (7.2 ks). Then, to activate the surface via removal of an oxide layer spontaneously creating as a result of cathode spraying, the specimens were subject to spraying in the mixture of 33% Ar and 67% H_2, at the discharge voltage of 850 V. The plasma nitriding was carried out at $T = 460$ °C (733 K), at the pressure of 150 Pa and during $t = 20$ h (72 ks). The glow discharge intensity was in the range of 8–10 A; 25%N_2 + 75%H_2 was used as the reactive atmosphere. Before nitriding the specimens were grinded and polished.

Stereometric examinations of the surface were carried out using a FormTaly Series 2 profilographometer made by Taylor Hobson, applying a contact method. All operations and computations on the measurement files were performed using the TalyMap Universal software. The obtained file represented the original surface subject to further analysis and stereometric description. The list of denotations and names of parameters analysed (3D) is given in the Table.

The stereometric description of each measured specimen consisted of:
- visualisation (2D) in the form of colour intensities map,
- distribution of "islands/particles" per unit surface,
- isometric image (3D) of surface fragments.

T a b l e. Names of stereometric parameters of the surface.

Denotation	Unit	Description
Sp	μm	Height of the highest surface elevation
Sv	μm	Depth of the deepest surface cavity
Sa	μm	Mean arithmetic deviation of surface roughness height from the reference plane

For specimens examined 1.5 mm × 1.5 mm surface was the measurement area. The sampling interval in *X* and *Y* axis was 1 µm, the measuring speed was 0.5 mm/s.

3. Results of examinations

Results of specimens' examinations are specified in Figs. 1 and 2.

Stereometric examinations carried out on specimens for two treatment variants show that in the case of nitriding a characteristic 3D island structure may be observed. On the other hand, for heat-treated specimens there are no island structures whatsoever, although from 3D image observations it results that this surface is more developed. Roughness measurements show that the height of the highest elevation for nitriding is more than twice higher than for material quenched and low-temperature tempered. Instead, the depth of the deepest surface cavity is larger in the case of heat treatment as compared with nitriding, however, this difference is not that significant as in the case of the highest elevation. The mean roughness values obtained for

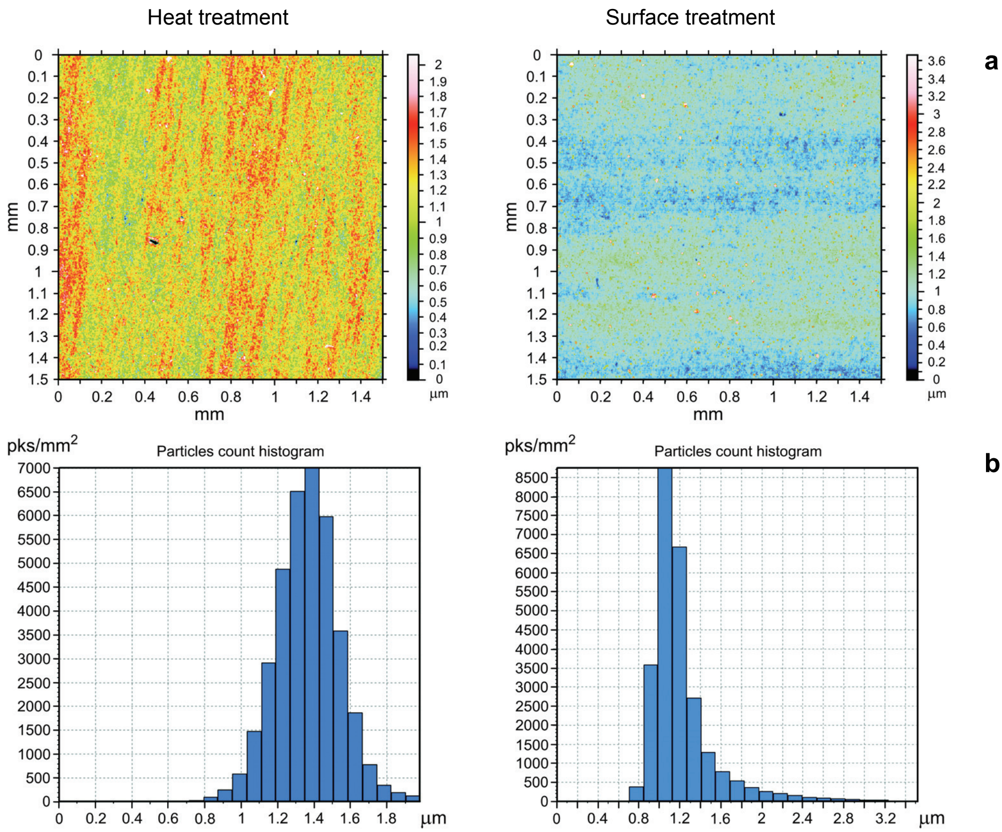

Fig. 1. To be continued on next page.

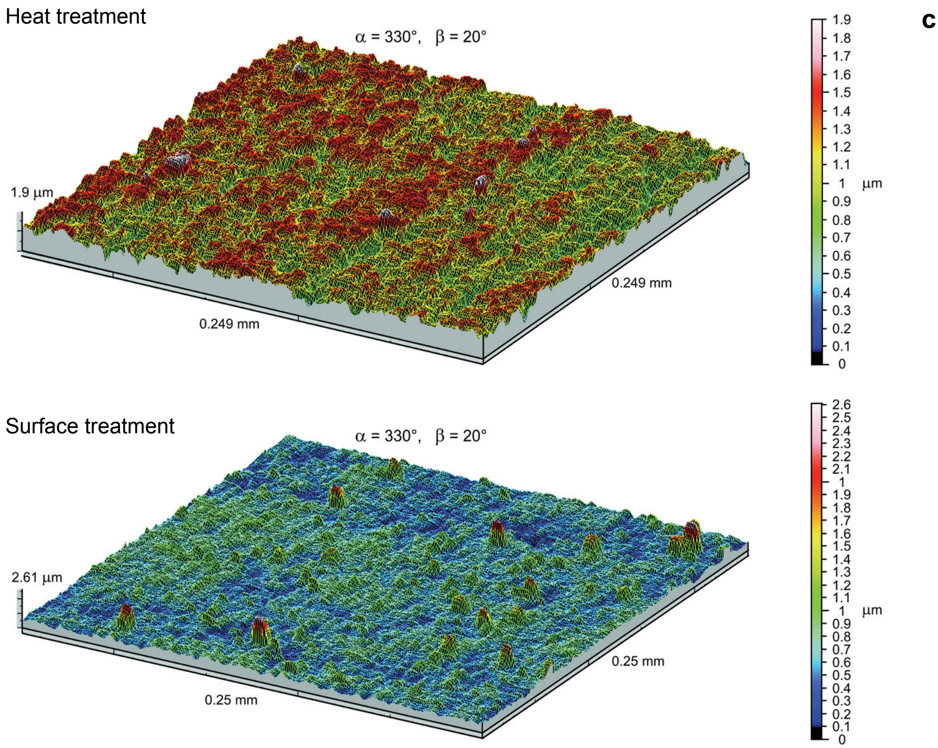

Fig. 1. Results of specimens' surface examinations: surface image as a colour intensities map (**a**); distribution of "islands/particles" per unit surface (**b**); isometric image (3D) of examined surface (**c**).

individual treatment variants are similar. According to SINGH *et al*. [10] the initial surface roughness had a great effect on the surface roughness stainless steel after plasma nitriding. Mirror polished samples exhibited high surface roughness increment after plasma nitriding compared to the rough polished, machined and ground samples. The increase in surface roughness also depends on the processing gas compositions in addition to the treatment time and temperature [11].

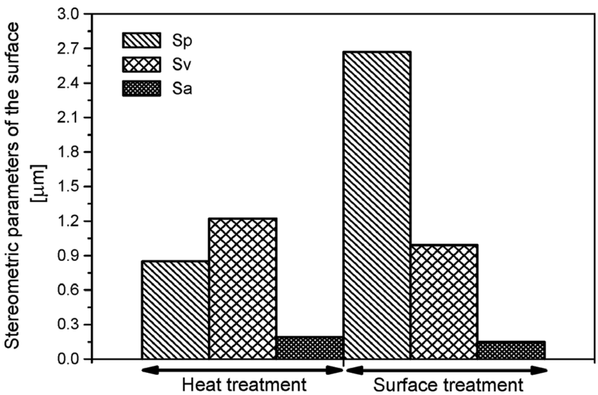

Fig. 2. Stereometric parameters of the surface.

4. Summary

The analysis was performed for specimens subject to two treatment variants: heat and surface. The surface morphology was diagnosed using a profilographometer. Basic examinations of heat and surface treated X39Cr13 steel surface topography allowed stating that the plasma nitriding has no significant impact on Sa parameter. From 3D images observations it results that the surface of heat treated specimens is definitely more developed as compared with nitrided specimens. Characteristic islands occur in the case of nitriding, which have not been observed for heat-treated specimens.

References

[1] GWOŹDZIK M., NITKIEWICZ Z., I*nfluence of tempering temperature on the kind of carbides in X39Cr13 steel*, 35th School of Materials Engineering, Crakow–Krynica, 25–28 September 2007, pp. 10–14 (in Polish).

[2] TUCKART W., FORLERER E., IURMAN L., *Delayed cracking in plasma nitriding of AISI 420 stainless steel*, Surface and Coatings Technology **202**(1), 2007, pp. 199–202.

[3] RODRÍGUEZ-BARACALDO R., BENITO J.A., PUCHI-CABRERA E.S., STAIA M.H., *High temperature wear resistance of (TiAl)N PVD coating on untreated and gas nitrided AISI H13 steel with different heat treatments*, Wear **262**(3–4), 2007, pp. 380–389.

[4] FRĄCZEK T., PASZENDA Z., NITKIEWICZ Z., GWOŹDZIK M., BASIAGA M., *Areology of unconventional plasma nitriding of austenitic steels (304 and 316L)*, Engineering of Biomaterials **10**(69–72), 2007, pp. 30–32.

[5] GWOŹDZIK M., NITKIEWICZ Z., DYJA D., *Wear resistance of martensitic steel after plasma nitriding*, Acta Metallurgica Slovaca **13**, 5/2007, pp. 439–443.

[6] CYBO J., JURA S., *Functional Description of Isometric Structures in Quantitative Metallography*, Silesian University of Technology Publishers, Gliwice 1995 (in Polish).

[7] HOSEMANN P., HAWLEY M., MORI G., LI N., MALOY S.A., *AFM and MFM characterization of oxide layers grown on stainless steels in lead bismuth eutectic*, Journal of Nuclear Materials **376**(3), 2008, pp. 289–292.

[8] SÁNCHEZ J., FULLEA J., ANDRADE C., GAITERO J.J., PORRO A., *AFM study of the early corrosion of a high strength steel in a diluted sodium chloride solution*, Corrosion Science **50**(7), 2008, pp. 1820–1824.

[9] PN-EN 10088-1:1998: *Stainless steels. Grades*.

[10] SINGH G.P., ALPHONSA J., BARHAI P.K., RAYJADA P.A., RAOLE P.M., MUKHERJEE S., *Effect of surface roughness on the properties of the layer formed on AISI 304 stainless steel after plasma nitriding*, Surface and Coatings Technology **200**(20–21), 2006, pp. 5807–5811.

[11] LARISCH B., BRUSKY U., SPIES H.J., *Plasma nitriding of stainless steels at low temperatures*, Surface and Coatings Technology **116–119**, 1999, pp. 205–211.

Received June 19, 2009
in revised form September 1, 2009