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Cite as: AIP Conference Proceedings **2053**, 040050 (2018); https://doi.org/10.1063/1.5084488
Published Online: 19 December 2018

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Improving the Scratch Test Properties of Plasma-Nitrided Stainless Austenitic Steel by Preliminary Nanostructuring Frictional Treatment

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Abstract: The paper studies the effect of nanostructuring deformation surface processing – frictional treatment performed prior to the operation of nitriding in electron beam plasma – on the quality of the surface and tribological properties of the nitrided layer of the AISI 321 chromium-nickel austenitic steel. For the surface layers nitrided in the undeformed coarse-crystalline and nanostructured states, considerable differences in the critical loads and the fracture patterns under scratch testing are observed, despite practically the same microhardness of the nitrided surfaces under study. It is demonstrated that preliminary frictional treatment increases the rupture strength of the nitrided layer under scratch testing and stabilizes the tribological properties of the surface on the macroscopic scale.

INTRODUCTION

Nitriding in low-energy electron beam plasma [1, 2] is an advanced method for thermochemical modifying of non-heat-treatable austenitic chromium-nickel steels. A decrease in the nitriding temperature to below 450 °C inhibits the formation of the nitride phase CrN in the near-surface layer of steel and allows a metastable supersaturated γ-solid nitrogen solution to be formed, which is highly hard (12 to 15 GPa) and corrosion resistant [3]. Since the effectiveness of nitriding is controlled by diffusion processes in a material being modified, it is important and urgent to increase the rate of nitrogen diffusion into the material bulk at low nitriding temperatures. Nanostructuring frictional treatment preceding the operation of nitriding in electron beam plasma reduces the temperature of effective nitriding to 350 °C [4]. Besides, in the frictional treatment of austenitic Cr-Ni steel with an artificial diamond indenter in argon, simultaneously with effective hardening, low surface roughness (Ra ~0.1 μm) is achieved [5, 6], this also being an important aspect in the implementation of finishing technologies.

Scratch testing with a sliding indenter enables one to estimate the resistance of the surface layer to contact loads when a material interacts with a moving hard particle and to determine critical loads and the fracture behavior in the surface layer as the normal load on the indenter increases [7].

The aim of this study is to detect the effect of preliminary nanostructuring frictional treatment of Cr-Ni austenitic steel before nitriding in electron beam plasma on the resistance of the nitrided layer to the contact fracture and tribological characteristics of the surface under scratch testing.

Mechanics, Resource and Diagnostics of Materials and Structures (MRDMS-2018)
AIP Conf. Proc. 2053, 040050-1–040050-5; https://doi.org/10.1063/1.5084488
Published by AIP Publishing. 978-0-7354-1781-6/$30.00

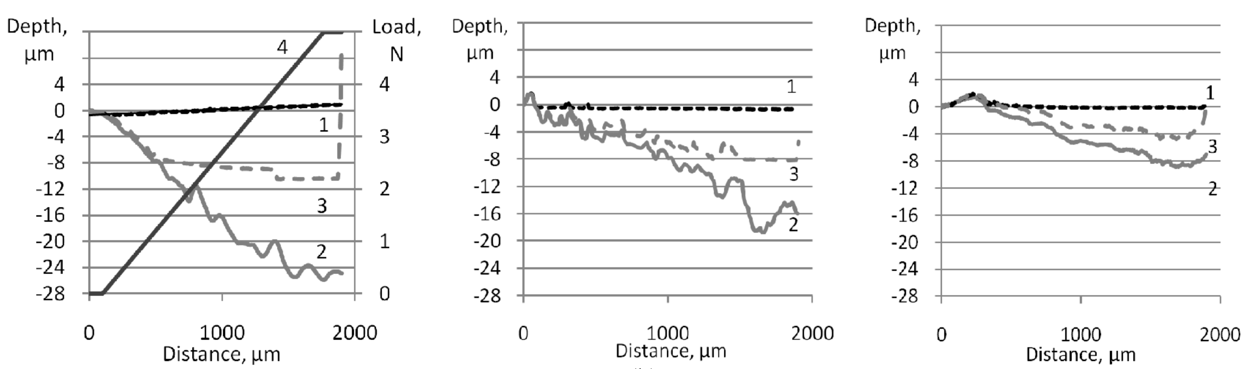
EXPERIMENTAL PROCEDURE

The AISI 321 corrosion resistant austenitic steel was studied, its composition being as follows, wt%: 0.04C, 16.77Cr, 8.44Ni, 1.15Mn, 0.67Si, 0.32Ti, 0.31Cu, 0.26Mo, 0.12Co, 0.12V, 0.04P, 0.03Nb, 0.005S, the rest Fe. Specimens sized 40×20×10 mm were cut out from a steel sheet by spark cutting and quenched from 1100 °C with water cooling then they were mechanically ground, electropolished and treated by friction. Frictional treatment was performed by a sliding artificial diamond indenter with a hemisphere radius R=3 mm in a nonoxidizing argon environment under a load on the indenter P=294 N, with a single scan by an indenter shifting by 0.02 mm per each double stroke of reciprocating motion. Thereafter, the specimens were chemically cleaned by acetone in an ultrasonic bath for 10 min and placed into a vacuum chamber for subsequent ion-plasma treatment. The source of electrons with a plasma cathode based on a glow discharge with a cold hollow cathode, a widened anode part and a single-input wide beam system [1] was used to generate plasma in the volume of the vacuum chamber. Electropolished and additionally friction-treated specimens were nitrided in electron beam plasma for 4 h at 400 °C.

The nitrided surfaces were studied by electron scanning microscopy (SEM) with the use of a Tescan Vega II XMU microscope and on a Wyko NT-1100 optic profilometer. Scratch testing was conducted on a NanoTest 600 testing complex by a Rockwell indenter (cone) with a diamond tip 25 μm in diameter moving at a velocity of 1 μm/s under a normal load linearly increasing from 0.5 to 5000 mN (510 g) and under a constant normal load of 300 mN (30 g). The surface microrelief before and after scratch testing was studied during scanning with the same indenter at a load of 0.5 mN (51 mg). The critical load L_c of the beginning of the nitrided layer fracture was determined by comparing the diagram of load variation in testing with the track microphotographs obtained by optic microscopy (OM) with the use of an Altami 1C microscope. Three scratch tests were performed for each specimen.

RESULTS AND DISCUSSION

The results of scratch testing by a conical indenter with a normal load linearly increasing from 0.5 mN to 5000 mN (5 N) are shown in Fig. 1. The indenter is seen to penetrate to the maximum depth (25 μm) when the surface is loaded after electropolishing (EP) (Fig. 1a). The maximum depth of penetration into the nitrided layer of the initial coarse-crystalline steel (Fig. 1b) is twice (18.2 μm) that for the nitrided layer of the steel preliminarily nanostructured during frictional treatment (FT) – 8.8 μm (Fig. 1c). Herewith, in the case of nitriding (N) with preliminary frictional treatment, curves 2 and 3 have a smoother form, this being indicative of a more uniform deformation of the surface layer under scratch testing.



(a) (b) (c)

FIGURE 1. Scratch test results in the "penetration depth – distance from the track start" coordinates with an increasing normal load for AISI 321 steel specimens after EP (a), EP+N (b) and FT+N (c): 1 – surface profile before testing (measurement under a scanning load of 0.5 mN); 2 – indenter penetration under increasing normal load; 3 – surface profile after testing (measurement under a scanning load of 0.5 mN); 4 – normal load application mode during testing

The metallographic analysis of the tracks has shown that, in the case of nitriding after electropolishing, under loads above 3000 mN large spalls sized at least ~50 μm start to form (Fig. 2a). At the bottom of some tracks, at a load of 5000 mN there appear arc tensile cracks caused by tensile stresses arising behind the moving indenter. After preliminary frictional treatment at loads of 3500 to 5000 mN there appear only small lateral microcracks and recovery spallation along one or both sides of the track (Fig. 2b). Consequently, preliminary frictional treatment increases the critical load L_c of the start of the fracture of the nitrided layer from 3000 to 3500 mN; however, there are no large spalls or stress cracks after frictional treatment and nitriding up to a maximum load of 5000 mN (Fig. 2b).

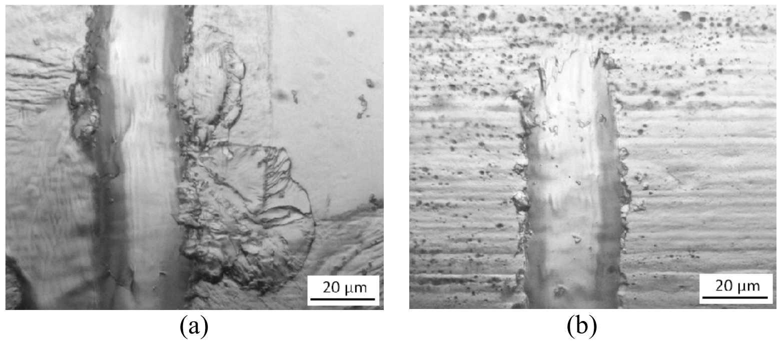


FIGURE 2. The form of the tracks (OM) on the AISI 321 steel specimen surface after scratch tests: a – EP+N (the load on the indenter 2980 mN); b – FT+N (the load on the indenter 5000 mN)

Microscratch testing under constant normal load of 300 mN (30 g) has shown that the mean value of the friction coefficient for the quenched steel before nitriding is 0.25 (Fig. 3, curve 1), and for the nitrided specimens it is at close lower levels – 0.17 and 0.14 for nitriding after electropolishing and after frictional treatment, respectively (Fig. 3, curves 2 and 3). Herewith, in the case of plasma nitriding of the steel in the coarse-crystalline state (electropolishing + nitriding) there is a wide scatter of the values of the friction coefficient, namely, from 0.07 to 0.36 (Fig. 3, curve 2). However, after nitriding with preliminary friction treatment, the friction coefficient only slightly fluctuates near the mean value during testing (Fig. 3, curve 3), similarly to the friction coefficient in the scratch testing of the initial steel after electropolishing (Fig. 3, curve 1).

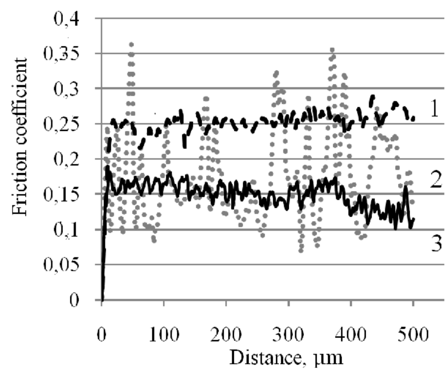
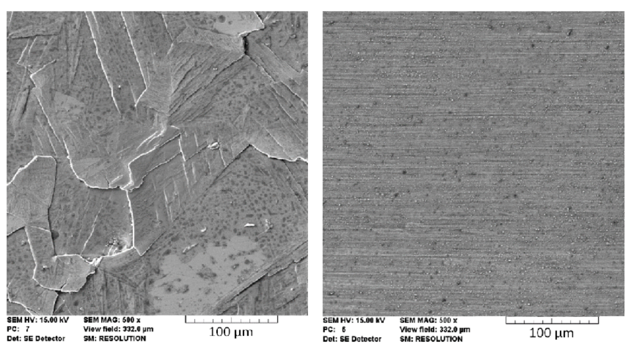


FIGURE 3. The friction coefficient in scratch testing under constant loading P=300 mN of the AISI 321 steel after EP (1), EP+N (2) and FT+N (3)

The experimental results testify to essential differences in the deformation and fracture patterns obtained in scratch testing with a conical indenter for the nitrided layers formed on the surface of the undeformed coarse-crystalline austenitic steel and that nanostructured by frictional treatment. The steel nitrided after frictional treatment has more stable tribological properties at the microscopic level and higher resistance to elastic-plastic deformation and fracture (Figs. 1 to 3). At the same time thin (several micrometers) layers on both nitrided surfaces under study (EP+N and FT+N) have approximately equal level of microhardness (1430-1450 $HV_{0.025}$ and 1310-1350 $HV_{0.3}$ [4]. This is due to a relatively small penetration depth of a Vickers indenter compared to the thickness of the analyzed nitrided layers. It has been found by the nuclear reaction method [4] that, after nitriding of the AISI 321 steel in electron beam plasma at 400 °C, both non-deformed and frictional treated specimens showed nitrogen concentration of 31-32 at.% at a depth of 2.25 μ from the surface. In what follows, we discuss possible reasons for the differences detected in the scratch tests.

The SEM examination of the surfaces of the nitrided specimens has shown that, nitriding of the coarse-crystalline steel causes a relief (Fig. 4a), resulting from inhomogeneous deformation and reorientation (“bulging”) of coarse grains and subgrains due to the formation of nitride phases predominantly along the boundaries [8] and an increase in the lattice parameter of large austenite grains with nitrogen saturation. The arithmetic mean deviation from the surface roughness profile after electropolishing followed by nitriding has a significant value (Ra=0.87 μm). Besides, as the coarse-crystalline specimens are nitrided, in a very thin (tenths of μm) layer austenite may transform into the α (bcc) phase by the shearing mechanism [9], and this enhances deformation and increases stresses in the layer and their inhomogeneity. Obviously, the coarse relief of the nitrided surface and the increased inhomogeneous stresses in the layer cause an uneven development of elastic and plastic deformation (Fig. 1b, curves 2 and 3), as well as significant fluctuations in the friction coefficients (Fig. 3, curve 2) during indenter sliding under conditions of scratch testing after electropolishing followed by nitriding.

Preliminary frictional treatment decreases the roughness parameter Ra of the nitrided surface to 0.19 μm. After frictional treatment and nitriding, the surface is smooth (Fig. 4b), without rough relief typical of nitriding of the coarse-crystalline state (Fig. 4a). The absence of a pronounced relief results from the high dispersion of the structure under frictional treatment [5, 6] and, possibly, the features of the formation of the stress state under subsequent nitriding of a deeper layer [4]. Nanostructuring frictional treatment not only favors nitrogen diffusion to a greater depth owing to a great length of the nanograin boundaries and their high diffusion activity, but also supplies the smoother transition from the high-strength nitrided layer to the soft bulk metal due to strain-hardening, which can reach ~450 μm in depth for metastable austenitic steel [10]. This seems to contribute to the formation of a more favorable stress state in the surface layer after frictional treatment and nitriding as compared to nitriding of the coarse-crystalline specimen. After electropolishing and nitriding, nitrogen is concentrated at a smaller depth near the surface, with a sharper transition from the high-strength layer to the softer matrix, thus creating a high level of residual stresses. Besides, when the preliminarily deformed state is nitrided, 95 vol% of strain-induced α′-martensite is formed on the surface of the AISI 321 steel as early as under frictional treatment [4, 8]. This prevents the development of the γ→α transformation under nitriding (as distinct from nitriding of the undeformed steel [9]) and, accordingly, the appearance of additional stresses induced by the bcc-phase with a greater unit volume than that of the fcc-phase.



(a) (b)

FIGURE 4. SEM images of the AISI 321 steel specimen surfaces after EP+N (a) and FT+N (b)

The absence of the coarse relief on the nitrided surface after frictional treatment and nitriding (Fig. 4b) and the formation of an apparently favorable stress state provide a stable development of elastic-plastic deformation (Fig. 1c, curves 2, 3) and small fluctuations in the friction coefficient (Fig. 2, curve 3) under scratch testing. The low tendency to cracking and spalling observed for the FT+N treatment (as compared to EP+N) and a smaller depth of penetration with a sliding conical indenter (Figs. 1 and 2) seem to testify to a deeper nitrided layer and less sharp gradients of hardening and stresses in the transition to a soft matrix in the case of preliminary frictional treatment.

CONCLUSIONS

It has been found that preliminary nanostructuring frictional treatment with a synthetic diamond indenter in an argon environment increases the effectiveness of the application of nitriding in electron beam plasma at 400 °C aimed at increasing the tribological properties of the AISI 321 Cr-Ni austenitic steel under scratch testing. Under conditions of scratch testing, preliminary frictional treatment increases the resistance of the nitrided layer to elastic-plastic deformation and fracture and stabilizes the process of deformation and tribological properties at the microlevel as compared to nitriding of quenched coarse-crystalline steel. This manifests itself in a double decrease in the depth of penetration with a sliding conical indenter, its smoother penetration without large spalls or cracks, increased critical load of the start of fracture in the nitrided layer and smaller oscillations of the friction coefficient in the scratch testing of a nitrided specimen with a preliminary frictional treatment. This improvement of the tribological behavior is due to the prevention of the appearance of a coarse relief, typical of the surface of the nitrided coarse-crystalline steel, on the nanostructured surface during nitration, as well as, presumably, to a smoother transition from the high-strength nitrided layer to the soft matrix and the specific features of the stress state of the friction-hardened steel.

ACKNOWLEDGMENTS

The work was done in the framework of the Complex Program of UB RAS (project No. 18-10-2-39) and the government assignments on themes No. AAAA-A18-118020190116-6 and No. AAAA-A18-118020790148-1. The investigation were performed in the Testing Center of Nanotechnologies and Promising Materials affiliated to the Institute of Metal Physics UB RAS and in the “Plastometriya” collective use center of the Institute of Engineering Science UB RAS.

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