Hardening Roll Surface by Plasma Nitriding with Subsequent Hardfacing

A Pesin¹, D Pustovoytov¹, R Vafin², I Yagafarov², E Vardanyan²

¹Nosov Magnitogorsk State Technical University, Magnitogorsk - 455000, Russia
²Ufa State Aviation Technical University, Ufa - 450000, Russia

Email: vafinrk@mail.ru

Abstract. The wear of the surface layer of rolls after ion nitriding in glow discharge, followed by a coating of TiN - TiAlN plasma arc are studied and simulated. The stress-strain state of the material rolls under asymmetric rolling with ultra-high shear deformations is simulated. The effect of thermal fields, formed upon contact of the tool and a deformable sheet, the structure of aluminum alloys, are considered.

1. Introduction

The purposeful asymmetry in the process of plastic deformation is gained due to the roll velocity misalignment by proportions from 1:2 to 1:4, when the cold rolling is provided under the high contact friction with severe single deformations (no less than 50%) [1]. The asymmetry results in the significant increase of the friction force which provides the high shear deformations of the material, and in the surface wear of rolls.

Currently, the development of the new ion-plasma techniques for hardening the roll surface which provide the purposeful change of the structure and phase composition of the surface layer and the physical and mechanical characteristics of the tool [2,3] is an urgent issue. The stimulating techniques of ion – nitriding with the crossed electric and magnetic fields [4-8] due to the charged particles densification followed by the TiN-TiAlN coating are considered to be perspective. The material modification is achieved through the increase in the number of the ionizing events, the ion mixing, sputtering, flash heat, atom and molecule deposition, defect formation, chemical interaction, and the radioactive diffusion stimulation.

2. Materials and Research Methods

The numerical simulation of the asymmetric thin sheet rolling was performed as a two-dimensional problem using the DEFORM 2D software. Also, the metal heating was considered, so the thermal and mechanical problems were solved as well. To solve the thermophysical problem, the thermophysical properties of the Al 5083 alloy were predefined as follows: the heat conductivity coefficient was 180.2 N/s/°C, heat capacity coefficient was 2.433 N/mm²/°C, emissivity was 0.7. Also, the thermophysical properties of the roll material were predefined – AISI D2 from the DEFORM 2D library; the heat conductivity coefficient was 50.71 N/s/°C; the heat capacity was...
3.81 N/mm²/°C. The curve of the Al 5083 yield was predefined from the DEFORM 2D library within the temperature band 20 — 500 °C.

The experimental setup to research the glow discharge characteristics in crossed electrical and magnetic fields was developed using the commercial ELU-5 equipment. The setup uses the pulsed power supply with the duty cycle S=80 % and the frequency of 50kHz.

The vacuum chamber was equipped with a standard magnetron with a length of 450 mm, a height of 50 mm, and a width of 100 mm and the magnetic field induction of 0.03 T, which was attached to the regular place of the vacuum chamber. As the working gas, argon as well as the mixture of nitrogen, argon and acetylene in the proportion of N₂ 50% - 80%, Ar 25% - 10%, C₂H₂ 25% - 10% were used. After the pressure got below 2 Pa, the chamber was blown with argon and filled with the working gas. The working gas pressure was altered within 5 - 200 Pa. The experimental circuit of the experimental ion nitriding is displayed in figure 1.

![Diagram](image.png)

Figure 1. Scheme of the experiment: 1 – vacuum chamber, 2 – magnet system, 3 – samples, 4 - lines of magnetic induction, 5 – high-density nitrogen plasma, 6 – arc plasma source

The research was conducted with the cylindrical-shaped samples of the shear steel P6M5 and X12, each was 12 mm in diameter and 4 mm high. The samples were mechanically polished before nitriding. After nitriding, the samples were coated by TiN and TiAlN compositions using the NNV-6.6-I1 machine. The process sequence was as follows. Firstly, the sample surface was cleaned and activated with argon plasma for 20 min when the bias voltage of -700 V was applied to the specimens. When cleaning, the samples were heated up to 440 °C. Then, they were coated with the TiN and TiAlN compositions. The following coating modes were applied: the nitrogen pressure was 0.11 – 0.13 Pa, the discharge current was 15A supported by altering the heating current of the emission cathode, the arc current of the titan cathode was 90A, of the aluminum one 60A. The stoichiometric compound of the TiN and TiAlN coating was obtained checking the arc current of the titan and aluminum cathodes. Simultaneously, the bias voltage of -200 V was applied to the materials. The temperature of the specimens under coating did not exceed 500 °C.

3. Results and Discussion

The analysis of the numerical simulation in DEFORM 2D revealed that, comparing to the symmetric distortion, the rolling force decreased by 2.4 – 3.2 times under 50 – 75% speelerizing and the misalignment of roll velocities Vi/V₂ = 2 — 4. However, there was a significant torque increase on the rolls: by 1.5 – 3.5 times on the bottom roll, by 1.1 – 2.6 times on the top roll. The deformation heating of Al 5083 alloy in the deformation zone which reached 345 °C when £ is 75 %, Vi is 100 mm/s, Vi/V₂ is
4, and p is 0.4, strongly impacts the force parameters of the asymmetric rolling. When decreasing the rolling velocity down to 1 mm/s, the metal heating does not exceed 50 °C, the rolling force, however, increases from 4 to 7 kN by 1.75 times (per 1 mm of the sheet width), the rolling torque on the bottom roll increases by 1.8 times, from 137 kN to 245 kN (per 1 mm of the sheet width), and by 1.96 times on the top roll: from 85 kN to 167 kN (per 1 mm of the sheet width).

The results of the electron microscopical study of samples demonstrate that the nitriding in crossed electrical and magnetic fields of P6M5 and X12 steel results in the formation of nitride layer under which a diffusion sublayer with microtexture is formed.

When nitriding the P6M5 steel in a glow discharge plasma for 4 hours, a hardened layer of 200 µm is formed (figure 2). It is known [2] that the hardened layer height reaches 25 µm after 3 hours of gas nitriding.

The analysis of X12 microtexture revealed the thin nitride zone of 10 – 15 µm (figure 3) with minimal grain boundary nitride precipitate in the diffusion zone. The nitride layer in the images of microtexture looks to be completely texture-free, it includes the iron nitride and the alloying element nitride. Both the nitride and diffusion layers observed microscopically are considered as the common thickness of the nitrogenized layer. The research of the microtexture and the phase composition of the diffusion sublayer (figure 7) revealed the nitrogenous solid solution of the parent metal, its nitrides and the nitrides of alloying elements [7]. During the alpha-phase diffusion saturation, the CrN phases in the shape of fine substances of 1 – 2 µm are separated (figure 3).

There is no clear boundary between the nitride layer to sublayers is smooth, that is one of the key requirements to the nitrogenized layer [3].

The thickness of the nitride layer in X12 steel was 10 µm after the 4 hour nitriding in crossed electrical and magnetic fields (figure 3) and in P6M5 it was 80 µm (figure 2). The high percentage of Cr in X12 steel provides the thinning of the hardened layer, as may be supposed, due to the chrome nitride layer which prevents the nitrogen diffusion as well as the diffusion layer formation.

There is no parent phase on the sample surface after nitriding in the crossed electrical and magnetic fields. Instead of that, a range of phases is formed as a result of interaction of the glow discharge plasma with the matrix material.

**Figure 2.** The section microtexture of P6M5 steel after 4 hour nitriding in crossed electrical and magnetic fields, where p is 80Pa

**Figure 3.** The section microtexture of X12 steel after 4 hour nitriding in crossed electrical and magnetic fields, where p is 80Pa
Figure 4. X-Ray diffraction patterns of the P6M5 surface layers before (a) and after the 4 hour nitriding in crossed electrical and magnetic fields, where p is 80Pa (b)

Figure 5. X-ray diffraction patterns of the X12 surface layers before (a) and after the 4 hour nitriding in crossed electrical and magnetic fields, where p is 80Pa (b)

The X-ray pattern (figure 4) of the P6M5 surface after the ion nitriding revealed that around the 40º—50º angles, there is a broadening at the Fe₃(N,C) and CrN peak bases which can be caused by several phases with the similar values of interplanar distance and the retained compression stress.

The analysis of the X12 surface after the nitriding in crossed electrical and magnetic fields demonstrated the reflexes of the Fe₃N ε-phase as well as the phases which was composed of CrN. The significant fall in intensity of the α-iron peaks (figure 5) after nitriding indicates the decreasing α-phase fraction in the surface layer as a result of forming the Fe₃N ε-phase. After the treatment in the nitrogen – argon – acetylene mixture with proportions of N₂ 50% - 80%, Ar 25% - 10%, C₂H₂ 25% - 10%, the sample
surfaces revealed the reaction to iron carbide \((\text{Fe}_5\text{C}_2)\) as well. Using the nitrogen – argon – acetylene mixture with proportions of \(\text{N}_2 50\% - 80\%\), \(\text{Ar} 25\% - 10\%\), \(\text{C}_2\text{H}_2 25\% - 10\%\) in nitriding provides the deactivation of the oxygen remained and the diffusion saturation.

After the 180 minutes of coating deposition, the coating of the TiN composition of 9 \(\mu\)m (figure 6) as well as the TiAlN coating with thickness of 6 \(\mu\)m (figure 7). Then, the materials were cooled to 100 °C under vacuum. The micro-hardness dependence on depth is presented in Figure 8.

The high wearing is provided by the TiN and TiAlN coating as well as by hard flexible nitrogenized layers under coating. Moreover, smoothing the sharp transition between the “soft” material and the hard coating contributed to the wearing quality and decreased the hardness gradient between two different textures as well.

Compared to the conventional nitriding, the advantage of the ion nitriding in magnetic field in the microhardness distribution through the obtained layer is obvious. The thickness of the hardened layer was increased from 54 \(\mu\)m to 84 \(\mu\)m, i.e. by 1.5 times. The samples placed in magnetic field demonstrated smoother microhardness depth distribution (figure 8a).

![Figure 6. The P6M5 microtexture after the ion nitriding in glow discharge with magnetic field and TiN coating](image)

![Figure 7. The P6M5 microtexture after the ion nitriding in glow discharge with magnetic field and TiAlN coating](image)

![Figure 8. The micro-hardness dependence on depth in the P6M5 samples after the ion nitriding under 500°C: a – in magnetic field \((P=44 \text{ Pa})\); b – in the absence of magnetic field \((P=120 \text{ Pa})\)](image)
4. Conclusion

- The analysis of the numerical simulation in DEFORM 2D revealed that comparing to the symmetric distortion the rolling force decreased by 2.4 – 3.2 times when 50 – 75% spellerizing and the misalignment of roll velocities \( \frac{V_1}{V_2} = 2 — 4 \). However, there was a significant torque increase on the rolls: by 1.5 – 3.5 times on the bottom roll, by 1.1 – 2.6 times on the top roll.
- The crossed electrical and magnetic fields provide the energy and density increase of the charged particles current to the treated surface.
- It was experimentally proved that the 4 hour ion nitriding in the crossed electrical and magnetic fields of the P6M5 and X12 steel under pressure of 80 Pa resulted in the surface modified layer which was compounded of \( \text{Fe}_3\text{N} \), \( \text{CrN} \) nitride phases of increased hardness. The \( \text{CrN} \) phase appeared as fine particles of 1-2 nm.
- The crossed electrical and magnetic fields increase the thickness of the hardfaced layer of the P6M5 sample up to 200 \( \mu \)m for 4 hours in comparison with 25 \( \mu \)m for 3 hours under gas nitriding.
- The ion nitriding in glow discharge with magnetic field is an efficient technique to prevent the TiN and TiAlN coating from excessive plastic deformation of the support.

Acknowledgments
The study was supported by the grant from the Russian Science Foundation (#15-19-10030)

References
[1] Ji Y.H., Park J.J. 2009 Materials Science and Engineering 499 14
[2] Goncharenko I.M. 2000 Evolution of the structure and phase composition of hardened 4140 steel in the process of plasma nitriding 5th International Conference on Modification of Materials with Particle Beams and Plasma Flows (Tomsk: Institute of Physics and Technology TPU) pp 330–333
[3] Edenhofer B. 1976 Metal and Material Technological 8(8) 421
[4] Arnell R.D., Kelly P.J. 1999 Surface and Coatings Technology 112 170
[5] Brading H.J., Morton P.H., Earweaker G. 1992 Surface engineering 8(3) 206
[6] Bradley J.W., Arnell R.D., Armour D.G. 1997 Surf. and Coat. Technol. 197 538
[7] Vafin R.K., Ramazanov K.N. 2010 Surface Modification of Tool Steel during Ion Nitriding in Magnetic Field 10th International Conference on Modification of Materials with Particle Beams and Plasma Flows (Tomsk: Publishing House of the IOA SB RAS) pp 458–461
[8] Koval N.N. 2000 Elion nitriding of steels 5th International Conference on Modification of Materials with Particle Beams and Plasma Flows (Tomsk: Institute of Physics and Technology TPU) pp 327–329