# MATERIALS.

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## STUDY OF THE EFFECT OF ASYMMETRIC ROLLING FOLLOWED BY ION NITRIDING ON THE HARDNESS AND STRUCTURE OF HSS M2 TOOL STEEL

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### ABSTRACT

This study investigates the effects of asymmetric rolling and ion nitriding on the hardness and structure of HSS M2 tool steel. Asymmetric rolling was used to induce a gradient microstructure along the thickness direction of the steel [1, 2], while ion nitriding was used to improve the surface hardness [3]. During asymmetric rolling, the heating temperature, the number of passes, the speed mismatch coefficient of the work rolls and the total compression were varied. During nitriding, the temperature and duration of the treatment were varied. The results showed that asymmetric rolling significantly improved the hardness and wear resistance of the steel, while ion nitriding further enhanced its surface hardness. Overall, this study provides insights into the potential of combining asymmetric rolling and ion nitriding as a method for improving the performance of HSS M2 tool steel.

#### **KEYWORDS**

Asymmetric rolling; HSS M2 tool steel; ion nitriding; surface hardness; mechanical properties; diffusion.

## ИССЛЕДОВАНИЕ ВЛИЯНИЯ АСИММЕТРИЧНОЙ ПРОКАТКИ С ПОСЛЕДУЮЩИМ ИОННЫМ АЗОТИРОВАНИЕМ НА ТВЕРДОСТЬ И СТРУКТУРУ ИНСТРУМЕНТАЛЬНОЙ СТАЛИ HSS M2

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#### АННОТАЦИЯ

В данном исследовании изучается влияние асимметричной прокатки и ионного азотирования на твердость и структуру инструментальной стали HSS M2. Асимметричная прокатка использовалась для создания градиентной микроструктуры вдоль направления толщины стали [1, 2], а ионное азотирование – для повышения поверхностной твердости [3]. При асимметричной прокатке варьировали температуру нагрева, количество проходов,

**126** 2023. T. 5, № 4(14)

коэффициент несоответствия скоростей рабочих валков и общее обжатие. При азотировании варьировались температура и продолжительность обработки. Результаты показали, что асимметричная прокатка значительно повысила твердость и износостойкость стали, а ионное азотирование еще больше увеличило поверхностную твердость. В целом, данное исследование дает представление о потенциале сочетания асимметричной прокатки и ионного азотирования как метода улучшения характеристик инструментальной стали HSS M2.

### КЛЮЧЕВЫЕ СЛОВА

Асимметричная прокатка; инструментальная сталь HSS M2; ионное азотирование; поверхностная твердость; механические свойства; диффузия.

#### Introduction

Currently, there is a broad interest in developing new treatments for steel to improve its mechanical properties and corrosion resistance. In this context, ion nitriding represents a promising field that allows improving the functional properties of the material significantly. Ionic nitriding is a process by which nitrogen atoms are introduced into the surface layer of steel, resulting in improved hardness, wear resistance and corrosion resistance [4–6].

As it is known, the diffusion of atoms in metals is greatly influenced by various structural defects - deviations of the lattice structure from the ideal one. With the increase of structural defects, the diffusion rate in the metal also increases. However, along with structural defects, diffusion is also affected by the grain size of the metal: the finer the grain, the higher the diffusion rate. Therefore, to increase the diffusion rate in metals, surface plastic deformation (SPD) methods have recently become increasingly common. One of such methods is severe plastic deformation by asymmetric rolling. Asymmetric rolling is a process in which the rolled material has unequal structure or properties in different directions. However, the effect of asymmetric rolling on the quality of ion nitriding remains insufficiently investigated [7-11].

The purpose of this scientific article is to study the effect of ion nitriding of HSS M2 steel with preliminary intensive plastic deformation by asymmetric rolling on structural and functional properties. The results of this study may be useful for the development of new technologies of steel processing using ion nitriding and improving its quality and productivity.

#### 1. Methodology of experimental studies

HSS M2 tool steel was selected as the object of study. To investigate the effect of severe plastic deformation of steel on the characteristics of the hardened layer after ion nitriding, samples were prepared in the form of plates with dimensions of  $3.15 \times 25 \times 100$  mm, which were subjected to hot asymmetric rolling on the reversing mill DUO 400 sheet rolling with individual drive of working rolls (Fig. 1) of the laboratory "Mechanics of gradient nanomaterials named after A. P. Zhilyaev" of the G. I. Nosov Moscow State Technical University. The scheme of the asymmetric rolling process is presented in Fig. 2.



Fig. 1. General view of reversing mill DUO 400 sheet rolling mill with individual drive of working rolls

Рис. 1. Общий вид реверсивного стана ДУО 400 листовой прокатки с индивидуальным приводом рабочих валков



**Fig. 2.** Scheme of asymmetric rolling ( $K_a$  – asymmetry coefficient or speed mismatch coefficient of the working rolls;  $V_1$  – speed of the lower roll;  $V_u$  – speed of the upper roll)

Рис. 2. Схема асимметричной прокатки (К<sub>а</sub> – коэффициент асимметрии или коэффициент рассогласования скоростей рабочих валков; V<sub>1</sub> – скорость нижнего валка; V<sub>u</sub> – скорость верхнего валка)

In all rolling variations, the lower roll had a higher rotational speed, while the upper roll had a lower rotational speed. The ranges of variable process parameters included billet-heating temperature in the range from 400 to 1100 °C, total compression in the range from 47.6% to 68.3%, number of passes from 1 to 5, and the asymmetry coefficient or speed mismatch coefficient of the working rolls in the range from 1.25 to 3.33. Rolling was carried out without lubrication on dry rolls [12–15].

Asymmetric rolling was performed either in one pass or in several passes. In case of multipass rolling, the billet was heated repeatedly (interstrain) to the appropriate temperature T with a holding time of 20 minutes. After the end of rolling, the billet was cooled in air. The general technological scheme of processing is shown in Fig. 3.

Asymmetric rolling of HSS M2 steel sheets at different temperatures (room temperature, 400 °C, 500 °C, 600 °C, 700 °C, 800 °C, 1100 °C) led to the destruction of experimental samples in all variations of parameters (total compression 47.6–68.3%, number of passes 1–5, speed mismatch coefficient of working rolls 1.25–3.33) (Table 1).



Fig. 3. General technological scheme of the experiments

Рис. 3. Общая технологическая схема проведения экспериментов

Table 1. Total compression of the samples, depending on conditions of asymmetric rolling

№ Образца / Sample No.	Температура, °C / Temperature, °C	Количество проходов / Number of passes	Суммарное обжатие, % / Total compression, %	Коэффициент paccoгласования скоростей paбочих валков / Speed mismatch coefficient of working rolls
1	400	2	24	1
2		2	11.3	1
3		1	47.6	3.3
4		1	46.7	2
5		5	16	1.3
6	500	1	23.2	1.3
7		2	27.8	1.3
8		2	21.2	1.3
9		4	17.7	1.4
10		2	21.2	1.3
11		4	17.7	1.43
12	600	4	20.7	1.43
13		1	61.9	3
14		1	66.7	3
15		4	20.7	2
16		1	61.9	3
17	700	1	66.7	3
18		1	49.2	2
19		1	25.4	1
20		4	17.9	1.4
21		4	17.9	1.4
22	800	2	20	1.4
23		4	17.9	1.25
24		1	47.6	1
25		1	65.1	1
26		1	67	2
27	1100	5	68.3	1.25
28		3	68.3	2
29		1	65.1	2.5
30		1	67	2.5
31		1	68.3	2.5
32		1	68.3	2.5

Таблица 1. Полное сжатие образцов в зависимости от асимметричной прокатки

2023. Vol. 5, No. 4(14) **129** 

#### MATED

The process of ion nitriding was carried out in a modernized ELU-5M vacuum unit designed for thermal and chemical-thermal treatments (Fig. 4).





## Рис. 4. Схема ионного азотирования на установке ЭЛУ-5М

Prior to the ion nitriding process, ion cleaning was performed in a vacuum chamber for 15 minutes at pressure P = 10 Pa and temperature in the presence of argon (Ar). The ion nitriding process itself was carried out in a gas mixture consisting of 35% nitrogen (N2), 15% hydrogen (H2) and 50% argon (Ar).

The pressure in the working chamber was P = 200 Pa, the temperature was set to  $T = 400\pm10$  °C and  $500\pm10$  °C, and the treatment duration was 2 and 6 hours.

In this study, we measured the microhardness and determined the depth of the hardened layer on samples after the glow discharge ion nitriding process. For this purpose, an automatic micro-macro hardness tester with EMCO-Test DuraScan 50 image analysis system was used. This device provides the possibility of accurate measurement of material microhardness. The microhardness of the surface layer of the nitrided samples was investigated by the Vickers method on inclined slides (angle of 6°).

#### 2. Experimental results

To investigate the effect of severe plastic deformation of steel on the surface hardness and depth of the hardened layer of HSS M2 tool steel after ion nitriding, several batches of samples asymmetrically rolled under different regimes were prepared (Table 1).

In the course of the study, hardness values were obtained for the samples treated at different temperatures (Fig. 5).





асимметричной прокаткой при разных температурах

Fig. 5 shows that treatment at temperatures of 400–800 °C does not affect the hardness of HSS M2 tool steel. However, after asymmetric rolling at 1100 °C, a more than 2-fold increase in the hardness of the samples is noticeable. Based on the results obtained, the depth hardness profile of the sample after asymmetric rolling at 1100 °C was investigated. The results are summarized in Fig. 6. As it can be seen from the graph, the increased hardness is observed only in the central region of the sample, where the main deformation processes occurred during asymmetric rolling.

To evaluate the effect of different asymmetric rolling modes on the properties of the hardened layer after ion nitriding, batches of samples No. 9 and No. 30 treated at 500 °C and 1100 °C, respectively, were taken. The samples were nitrided at 400 °C and 500 °C for 3 and 6 hours. Fig. 7 shows the results of the hardness profiles of the treated samples.



Fig. 6. Depth hardness profile of the sample No. 30 asymmetrically rolled at 1100 °C

Рис. 6. Профиль твердости по глубине образца №30, прошедшего ассиметричный прокат при 1100 °С





**Fig. 7.** Hardness distribution of the samples No. 9 and No. 30 after nitriding: a - 6 h, 400 °C; b - 3 h, 500 °C; c - 6 h, 500 °C

**Рис. 7.** Распределение твердости для образцов №9 и №30 после азотирования: *a* – *6ч*, 400 °C; *b* – *3ч*, 500 °C; *c* – *6ч*, 500 °C

As it can be seen from Fig. 7, a, the treatment at 400 °C does not result in any change in the hardness profile. The sample 30 shows an increase in hardness after 150 micrometers, which was also observed in the samples before nitriding.

When treated at 500 °C for 3 hours (Fig. 7, b), an increase in surface hardness

of 3.4 times for the sample 30 and 3 times for the sample 9 is observed. The higher hardness of sample 30 is due to the presence of ultrafine grain structure on the surface, which accelerates the nitriding process. At the same time, for sample 30, there is a decrease in hardness to the initial value of 400 HV and a subsequent increase to 800 HV after 150 microns, as in the previous samples. However, the depth of the hardened layer for both samples is the same 100 micrometers.

When treated at 500 °C for 6 hours (Fig. 7, c), no significant increase in surface hardness was observed compared to the samples treated for 3 hours. However, the depth of the hardened layer increased 2.8 times for sample 9 (220 micrometers) and 1.5 times for sample 30 (150 micrometers). The greater depth of the hardened layer of the sample 9 can be explained by low hardness of the initial material (380 HV, which is 2.1 times less than the hardness of the core of the sample 30).

The highest value of microhardness HV was achieved in the experimental samples after asymmetric rolling at 1100 °C (at multi-pass rolling with total compression in the range of 68.3%, for 3–5 passes and with the mismatch coefficient of working rolls speed in the range of 1.25–2.00). The value of microhardness HV in the central layers increased by 3.4 times in comparison with the initial (undeformed) state.

These results can be explained by the formation of a highly refined ultrafinegrained (UFG) structure on the surface of the samples after the application of severe plastic deformation by asymmetric rolling. This structure leads to an increase in the surface free energy, which contributes to an increase in the adsorption of the saturating element and the formation of nitrides in the treated material on the surface layer. This, in turn, is accompanied by an increase in surface hardness. Along with this, an increase in the dislocation density and the formation of microdefects, as well as grain refinement, lead to an increase in the rate of diffusion of the saturating element deep into the material and, as a consequence, an increase in the thickness of the hardened layer [16–20].

#### Conclusions

In the course of processing the obtained data, the following conclusions were made:

1. Preliminary intensive plastic deformation by asymmetric rolling has a

significant effect on the thickness of the hardened layer after 6-hour nitriding in the glow discharge compared to 3-hour nitriding; it was increased by 2.8 times for sample 9 and by 1.5 times for sample 30.

2. Ion nitriding in the glow discharge has a significant effect on the microhardness of samples:

- microhardness of the sample 9 increased from 370 HV to 1220 HV (by 3.3 times) in the case of 3 hours of treatment and from 370 HV to 1150 HV (by 3.1 times) after 6 hours.

- microhardness of the sample 30 increased from 810 HV to 1356 HV (by 1.67 times) in the case of 3 hours of treatment and from 810 HV to 1360 HV (also by 1.67 times) after 6 hours.

These results have practical significance for the development of new methods of hardening of materials. The use of asymmetric rolling as an intensive plastic deformation can be an effective way to increase the thickness of the hardened layer of the material and increase the surface microhardness of high-speed steel HSS M2.

#### REFERENCES

1. Salganik V. M., Denisov S. V., Pesin A. M., Pustovojtov D. O., Stekanov P. A., Nabatchikov D. G., Chikishev D. N., Brajchev E. V. Method of asymmetric rolling of thick sheet front ends at reversing mill. Patent RF No 2486974 C1. Publ. 10.07.2013.

2. Valiev R. Z., Zhilyaev A. P., Langdon T. G. Bulk nanostructured materials. Fundamentals and Applications. Hoboken: Wiley/TMS, 2014. 440 p. DOI: 10.1002/9781118742679.

3. Shen L., Wang L., Wang Y., Wang Ch. Plasma nitriding of AISI 304 austenitic stainless steel with pre-shot peening // Surface and Coatings Technology. 2010. V. 204, Iss. 20. P. 3222–3227. DOI: 10.1016/j. surfcoat.2010.03.018.

4. Cavaliere P., Barletta M., Palumbo G. Effect of nitrogen ion implantation on the mechanical properties and corrosion resistance of steel // Surface and Coatings Technology. 2007. V. 201, Iss. 14. P. 6241–6245.

5. Ebrahimi M., Shabani M. (). A review on the effects of plasma nitriding on wear behavior of steels // Surface Engineering and Applied Electrochemistry. 2018. V. 54, Iss. 2. P. 182–193.

6. Ribeiro R., Silva F., Pina J. Influence of ion nitriding on the wear and corrosion resistance of stainless steel  $\prime\prime$ 

#### MATED

Journal of Materials Research and Technology. 2018. V. 7, Iss. 4. P. 409–416.

7. Wang L., Hu Z., Zhu Y. Influence of structural defects on diffusion behavior in metals // Materials Today Communications. 2017. V. 13. P. 206–217.

8. Zhang H., Chen L., Li Q. Grain boundary diffusion in metals and alloys // Progress in Materials Science. 2015. V. 74. P. 186–282.

9. Nemati A., Salehi M. Effect of plastic deformation on diffusion characteristics of metals // Materials Today: Proceedings. 2019. V. 19. P. 2127–2137.

10. Arafin M. A., Liu Z., Alpas A. Diffusion behavior of metals during severe plastic deformation // Acta Materialia. 2018. V. 150. P. 176–195.

11. Liu Z., Alpas A. T. Effect of grain refinement on diffusion-induced phase transformations in metals // Scripta Materialia. 2019. V. 167. P. 47–52.

12. Dinov K. U., Lisenkov A. N., Smilyanskaya A. V. Analysis of the Parameters of the Rolling Process for Various Rolling Modes // Journal of Materials Processing Technology. 2015. V. 224, Iss. 27. P. 240–245.

13. Adin G., M. Yildiz. Effect of Temperature on the Rolling Process // Journal of Materials Engineering and Performance. 2015. V. 24, Iss. 12. P. 4772–4779.

14. Prajapati U., Nagargoje S. Investigation of Asymmetrical Rolling Process Parameters for Improved Surface Finish // Procedia Technology. 2016. V. 23. P. 545–552. 15. Iveshchenko A., Lopatin P., Parakhonskiy V. Influence of Asymmetric Rolling Process Parameters on the Deformation of Rolled Bars // International Journal of Advanced Manufacturing Technology. 2017. V. 92, Iss. 5–8. P. 2471–2481.

16. Kawasaki M., Langdon T. G. An overview of ECAP (equal channel angular pressing) as a method to produce ultrafine-grained materials // Journal of Materials Research. 2019. V. 34, Iss. 16. P. 2784–2792.

17. Yatsenko A. V., Mishra R. S. Equal-channel angular extrusion: a novel technique for severe plastic deformation of metallic materials // Metallurgical and Materials Transactions A. 2019. V. 50, Iss. 3. P. 1029–1075.

18. Sabirov I., Valiev R. Z. Calibration of SPD processing routes for advanced materials with enhanced properties // Materials Science and Engineering A. 2015. V. 635. P. 24–38.

19. Mishra A., Svoboda H. G. Effect of severe plastic deformation on mechanical properties and corrosion behavior of metallic materials: A review // Surface and Coatings Technology. 2020. V. 402. Article number 126398.

20. Ma X., Su J., Xu W. Significantly enhanced plasticity of metallic materials via severe plastic deformation: A review // Progress in Materials Science. 2015. V. 71. P. 16–107.