



UDC 621.762.1

<https://doi.org/10.17073/1997-308X-2025-1-15-22>Research article  
Научная статья

## Effect of quenching and tempering on the structure and properties of hot-deformed powder steels with ultrafine particles

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**Abstract.** This study examines the effect of quenching and tempering on the structure and mechanical properties of hot-deformed powder steels containing ultrafine particles. The research analyzes the structural transformations and mechanical responses during quenching and tempering, focusing on the relationship between heat treatment conditions and the resulting material properties. The experiments involved variations in quenching temperature and tempering time, allowing the identification of optimal conditions for achieving a favorable combination of strength and ductility. The findings highlight the potential to achieve a homogeneous microstructure and high mechanical performance, making these materials suitable for high-load applications. This study underscores the significance of tailoring heat treatment parameters to control both microstructural and mechanical characteristics, thereby broadening the industrial applicability of powder steels.

**Keywords:** heat treatment, powder steels, ultrafine particles, mechanical properties

**For citation:** Egorov M.S., Egorova R.V., Mozgovoy A.V., Gantimurov K.V., Kovtun M.V. Effect of quenching and tempering on the structure and properties of hot-deformed powder steels with ultrafine particles. *Powder Metallurgy and Functional Coatings*. 2025;19(1):15–22. <https://doi.org/10.17073/1997-308X-2025-1-15-22>

## Влияние режимов закалки и отпуска на структуру и свойства горячедеформированных порошковых сталей с ультрадисперсными частицами

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**Аннотация.** Рассматривается влияние режимов закалки и отпуска на структуру и механические свойства горячедеформированных порошковых сталей, содержащих ультрадисперсные частицы. Исследование основано на анализе термических и механических процессов, протекающих при закалке и отпуске, а также их связи с характером структурных изменений, происходящих в материале. Эксперименты включали вариации температуры закалки и времени отпуска, что позволило выявить оптимальные режимы для достижения наилучших механических характеристик – таких, как прочность и пластичность. Полученные результаты указывают на возможность достижения высокой прочности, что делает эти материалы

перспективными для применения в условиях высокой нагрузки. Подчеркивается значимость выбора режимов термообработки для управления микро- и макроструктурой порошковых сталей, что открывает новые возможности для их использования в различных отраслях промышленности.

**Ключевые слова:** термическая обработка, порошковые стали, ультрадисперсные частицы, механические свойства

**Для цитирования:** Егоров М.С., Егорова Р.В., Мозговой А.В., Гантимуров К.В., Ковтун М.В. Влияние режимов закалки и отпуска на структуру и свойства горячедеформированных порошковых сталей с ультрадисперсными частицами. *Известия вузов. Порошковая металлургия и функциональные покрытия*. 2025;19(1):15–22.

<https://doi.org/10.17073/1997-308X-2025-1-15-22>

## Introduction

The properties of powder steels can be improved by complicating their composition and by applying thermal and thermochemical treatments. However, these methods of enhancing the properties of powder steels are characterized by certain challenges, primarily due to residual porosity and chemical and structural heterogeneity [1].

The influence of the structure of powder steels on the thermodynamics of new phase nucleation and transformation kinetics can be controlled through manufacturing technology. The formation of hot-deformed powder steels (HDPS) with minimal residual porosity aligns their critical points more closely with those of compact materials. The quenching temperature for powder steels is primarily determined by the critical points  $A_{c1}$  (the temperature at which austenite begins to transform into pearlite or another phase during cooling and where ferrite starts transforming into austenite during heating) and  $A_{c3}$  (the temperature at which ferrite begins to transform into austenite during heating – a key process for achieving the required steel properties), as well as the carbon content. HDPS are inherently fine-grained. Alloying with non-carbide-forming elements does not affect the tendency of austenite grains to grow at heating temperatures up to 1100 °C. This feature expands the temperature range for quenching; for HDPS with 0.5 % carbon content, this range is 825–845 °C [2–5].

The aim of this study is to investigate the quenching and tempering regimes to determine the optimal mechanical properties of hot-deformed powder steels containing ultrafine particles.

## Materials and methods

The study utilized domestic powders of grades PZhRV 2.200.26 (TU 14-1-5365-98, water-atomized and reduced iron powder) and N4D2M (TU 14-5402-2002, alloyed powder) produced by Severstal PJSC (Cherepovets, Russia) [4; 5]. Ultrafine additives of silicon nitride ( $\text{Si}_3\text{N}_4$ ) and nickel oxide (NiO) produced

by Plazmoterm (Moscow, Russia) [6] were added to the charge.

Before use, the powders were analyzed using the Analysette 22 MicroTecplus universal laser particle size analyzer (Fritsch, Germany) and the Beckman Coulter AU480 submicron particle analyzer (USA). The charge was prepared using an RT-NM05S twin-cone mixer (Taiwan) and an Assonic SPC ultrasonic station (China) for sieving and mixing powders with ultrafine particles. Static cold pressing was performed on a TS0500-6 hydraulic press (China) with a maximum load capacity of 50 tons using laboratory dies. Homogenizing sintering was carried out in the heat treatment laboratory of the “Materials Science and Metal Technology” department of DSTU in an SNOL 6.7/1300 muffle electric furnace (AB UMEGA, Lithuania) at temperatures ranging from 900 °C to 1150 °C in a protective gas atmosphere of dissociated ammonia. The sintering time ranged from 15 to 180 min. Subsequent heat treatment of the hot-deformed powder steels was conducted in the same furnaces.

Dynamic hot pressing (DHP) of the billets was performed on a K2232 crank press (Russia) with single-action operation. Before the DHP operation, powder billets were heated in a muffle electric resistance furnace (950–1150 °C) in a dissociated ammonia atmosphere. The furnace temperature was monitored using a platinum-palladium thermocouple [7].

Tensile tests were conducted in accordance with GOST 18227-85 using an MGS-V15 servo-hydraulic floor testing machine in automatic mode with a personal computer. Fig. 1 shows the diagram of the sample subjected to testing.

The hardness of the samples was measured using a Rockwell hardness tester (TK-2M, Tochmashpribor, Ivanovo, Russia) with diamond cone indentation under a total load of 1471 N.

The samples of PZhRV 2.200.26 + 0.5 % C and N4D2M + 0.5 % C were subjected to quenching followed by tempering after hot re-pressing at  $t = 1150$  °C, with the addition of ultrafine particles (2 % NiO, 0.1 %  $\text{Si}_3\text{N}_4$ ) to each material. Cooling was performed

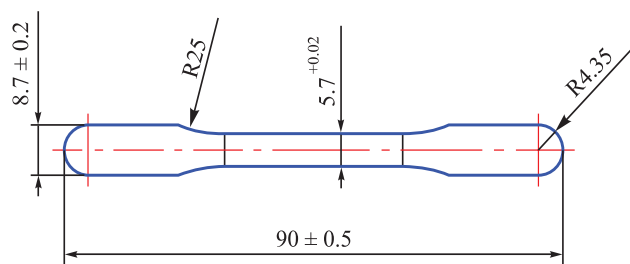


Fig. 1. Technical drawing of the sample for tensile testing

Рис. 1. Чертеж образца для испытания на растяжение

in water and oil, with cooling rates at the temperature of minimum austenite stability being 600–500 °C/s (in water) and 150–100 °C/s (in oil), respectively. The chemical composition of the studied powders, the characteristics of the ultrafine particles, and the technology for producing sintered samples are described in detail in [2].

## Results and discussion

Quenching of hot-deformed powder steels (HDPS) makes it possible to obtain a homogeneous martensitic structure with high hardness ( $HV = 7.5$  GPa). This is due to the low porosity and favorable structure formed during hot pressing.

Fig. 2 shows the microstructure of HDPS based on PZhrV 2.200.26 powder containing 0.5 % C + 2 % NiO. The martensitic structure is clearly defined, with a small number of pores up to 3 μm in size. This quenched steel structure does not contain ferrite or retained austenite, confirming that the quenching process was conducted correctly [8; 9]. The hardness of the quenched HDPS at a quenching temperature of 835 °C is presented in Table 1.

Modification of steels with silicon nitride increases hardness after quenching. The final formation of the structure and properties of HDPS occurs during tempering. The effect of tempering temperature on the mechanical properties of HDPS is presented in Table 2.

For all the studied materials, a similar trend in property changes is observed: as the tempering temperature increases, the ultimate strength ( $\sigma_v$ ) and hardness (HRC) of the steels decrease, while the ductility parameter ( $\psi$ ) increases, reaching its maximum  $t = 550$  °C. At this temperature, the overall set of mechanical properties is superior to those of the initial and annealed steels [7–9].

The microstructures of quenched and tempered HDPS N4D2M + 0.5 % C + 2 % NiO are shown in Fig. 3.

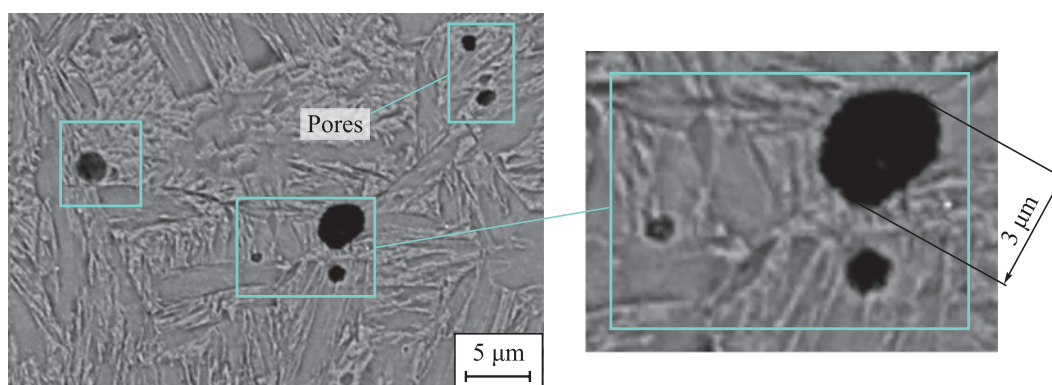


Fig. 2. Martensite of hot-deformed powder steel of PZhrV grade 2.200.26 + 0.5 % C + 2 % NiO

Pore size: 1–3 μm

Рис. 2. Мартенсит горячедеформированной порошковой стали марки ПЖРВ 2.200.26 + 0,5 % C + 2 % NiO

Размер пор: 1–3 мкм

Table 1. Hardness (HRC) of quenched HDPS

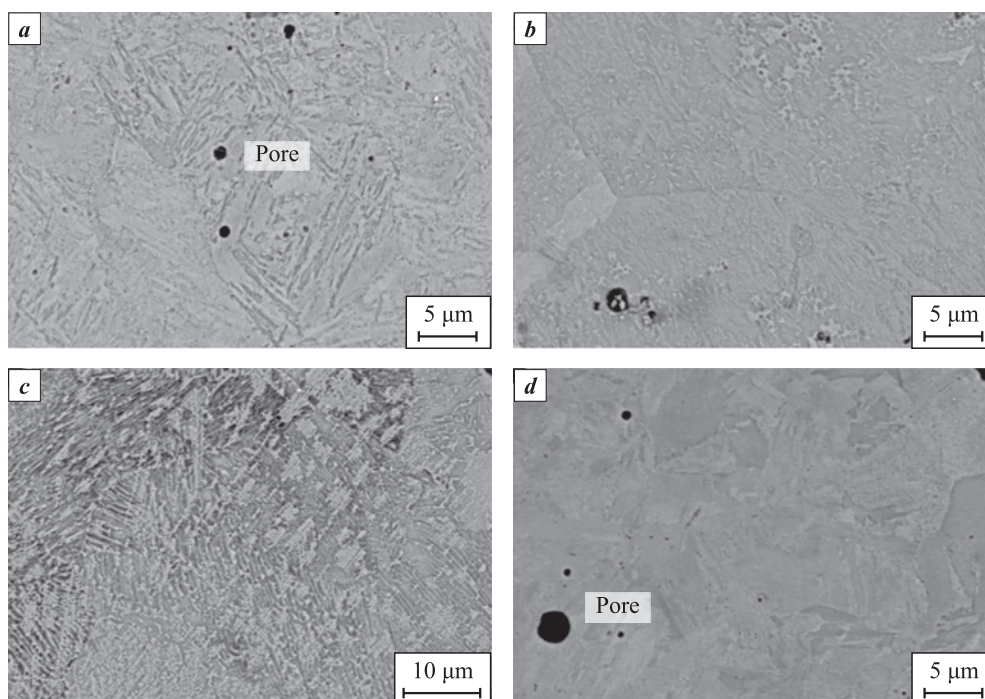
Таблица 1. Твердость (HRC) закаленных ГДПС

| Powder steel             |                                    |                                                                 |                 |                           |                                                        |
|--------------------------|------------------------------------|-----------------------------------------------------------------|-----------------|---------------------------|--------------------------------------------------------|
| PZhrV 2.200.26 + 0.5 % C | PZhrV 2.200.26 + 0.5 % C + 2 % NiO | PZhrV 2.200.26 + 0.5 % C + 0.1 % Si <sub>3</sub> N <sub>4</sub> | N4D2M + 0.5 % C | N4D2M + 0.5 % C + 2 % NiO | N4D2M + 0.5 % C + 0.1 % Si <sub>3</sub> N <sub>4</sub> |
| Cooling medium           |                                    |                                                                 |                 |                           |                                                        |
| Water                    |                                    |                                                                 | Oil             |                           |                                                        |
| 50–52                    | 50–52                              | 54                                                              | 49–51           | 49–51                     | 55                                                     |

**Table 2. Dependence of mechanical properties of HDPS on tempering temperature**

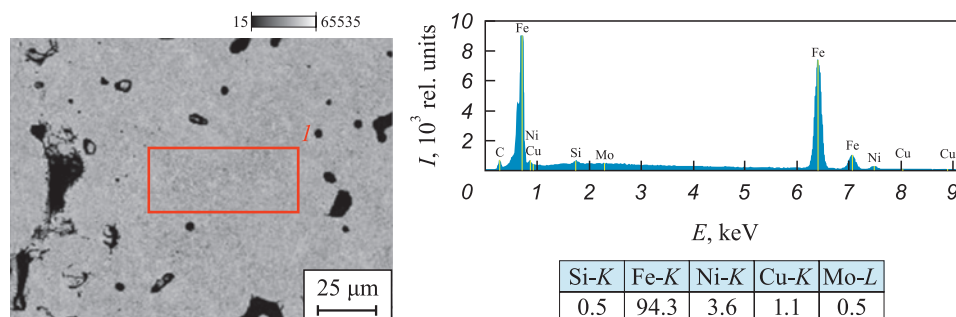
**Таблица 2. Зависимость механических свойств ГДПС от температуры отпуска**

| HDPS composition                                         | Tempering temperature, °C | $\sigma_v$ , МПа | $\psi$ , % | HRC |
|----------------------------------------------------------|---------------------------|------------------|------------|-----|
| PZhRV 2.200.26 + 0.5 % C                                 | 250                       | 1180             | 18         | 45  |
|                                                          | 350                       | 920              | 22         | 42  |
|                                                          | 450                       | 825              | 30         | 38  |
|                                                          | 550                       | 760              | 35         | 33  |
| PZhRV 2.200.26 + 0.5 % C + 0.1 % $\text{Si}_3\text{N}_4$ | 250                       | 1230             | 18         | 47  |
|                                                          | 350                       | 950              | 22         | 45  |
|                                                          | 450                       | 860              | 30         | 40  |
|                                                          | 550                       | 780              | 35         | 35  |
| PZhRV 2.200.26 + 0.5 % C + 2 % NiO                       | 250                       | 1190             | 19         | 45  |
|                                                          | 350                       | 935              | 23         | 42  |
|                                                          | 450                       | 840              | 29         | 38  |
|                                                          | 550                       | 765              | 35         | 33  |
| N4D2M + 0.5 % C                                          | 250                       | 1420             | 16         | 46  |
|                                                          | 350                       | 1260             | 20         | 43  |
|                                                          | 450                       | 1090             | 28         | 39  |
|                                                          | 550                       | 1070             | 32         | 34  |
| N4D2M + 0.5 % C + 0.1 % $\text{Si}_3\text{N}_4$          | 250                       | 1450             | 17         | 48  |
|                                                          | 350                       | 1290             | 24         | 46  |
|                                                          | 450                       | 1130             | 30         | 41  |
|                                                          | 550                       | 1090             | 34         | 35  |
| N4D2M + 0.5 % C + 2 % NiO                                | 250                       | 1430             | 16         | 46  |
|                                                          | 350                       | 1270             | 20         | 43  |
|                                                          | 450                       | 1100             | 28         | 39  |
|                                                          | 550                       | 1080             | 32         | 32  |



**Fig. 3. Microstructure of N4D2M + 0.5 % C + 2 % NiO after quenching and tempering at different temperatures  $t$ , °C: 250 (a); 350 (b); 450 (c); 550 (d)**

**Рис. 3. Микроструктура N4Д2М + 0,5 % С + 2 % NiO после закалки и отпуска при различной температуре  $t$ , °C: 250 (a); 350 (b); 450 (c); 550 (d)**



**Fig. 4.** Results of micro-X-ray spectral analysis of powder steel H4D2M + 0.5 % C + 2 % NiO after heat treatment (quenching and tempering)

**Рис. 4.** Результаты микрорентгеноспектрального анализа порошковой стали H4D2M + 0,5 % C + 2 % NiO после проведения термической обработки (закалка и отпуск)

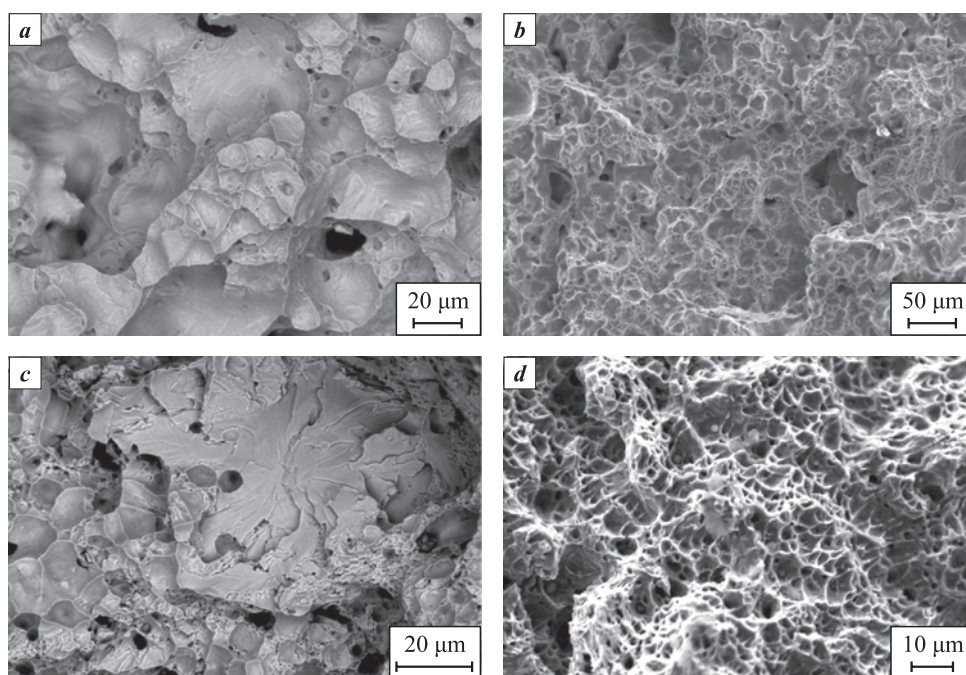
Thus, quenching and tempering allow for achieving the desired structure of HDPS [9–11]. The level of mechanical properties of HDPS depends on the quality of interparticle bonding formed during the sintering and hot re-pressing stages. If this bonding is incomplete, it is not possible to improve mechanical properties through strengthening heat treatment [11; 12].

To monitor the chemical composition of the powder steels obtained after heat treatment (quenching and tempering), a micro X-ray spectral analysis was performed using a scanning electron microscope

(S-3400N, Hitachi, Japan) [12; 13]. The results are presented in Fig. 4.

The presence of all alloying elements in the powder steel after heat treatment was verified through micro-X-ray spectral analysis [7; 10; 12].

Fractographic analysis using the S-3400N scanning electron microscope highlighted the characteristic features of HDPS fractures following heat treatment (quenching and tempering). The fracture surfaces of the quenched and tempered HDPS samples are presented in Fig. 5.



**Fig. 5.** Fractographs of powder steels with ultrafine particles after tempering  
 $t, ^\circ\text{C}$ : 250 (a, c); 550 (b, d)

a, b – H4D2M + 0.5 % C + 2 % NiO; c, d – PZHVR 2.200.26 + 0.5 % C + 2 % NiO

**Рис. 5.** Фрактограммы изломов порошковых сталей с ультрадисперсными частицами после отпуска  
 $t, ^\circ\text{C}$ : 250 (a, в); 550 (б, г)

a, b – H4D2M + 0,5 % C + 2 % NiO; c, d – ПЖРВ 2.200.26 + 0,5 % C + 2 % NiO

An analysis of the fractographs revealed that the dominant features on the fracture surfaces of HDPS tempered at  $t = 250\text{ }^{\circ}\text{C}$  are intergranular and transgranular cleavages, appearing at different levels and distinguished by varying sizes of the crack propagation zones [14–16]. In Fig. 5, *a* and *c* steps on large cleavage elements are clearly visible, giving the structure a river-like pattern – a characteristic feature of intergranular fracture. On smaller facets, smooth surfaces formed by crack propagation along crystallographic planes are observed, which are typical of transgranular cleavage [17–20]. Discontinuities in both intergranular and transgranular cleavage zones make it difficult to identify the preferred site of crack initiation. This observation indirectly suggests a balance of interatomic bonding forces within grains and along grain boundaries, indicating the successful formation of intragranular bonding during HDPS production [2; 12].

## Conclusion

The study examined the effects of quenching and tempering on the structure and properties of HDPS with ultrafine particles. Maximum hardness at a quenching temperature of  $835\text{ }^{\circ}\text{C}$  was observed in steels with compositions PZhRV 2.200.26 + 0.5 % C + 0.1 %  $\text{Si}_3\text{N}_4$  ( $\text{HRC} = 54$ ) and N4D2M + 0.5 % C + 0.1 %  $\text{Si}_3\text{N}_4$  ( $\text{HRC} = 55$ ). Modifying steels with silicon nitride improved hardness after quenching. For these steels, maximum ultimate strength values were recorded at a tempering temperature of  $250\text{ }^{\circ}\text{C}$ :  $\sigma_v = 1230\text{ MPa}$  (PZhRV 2.200.26 + 0.5 % C + 0.1 %  $\text{Si}_3\text{N}_4$ ) and  $\sigma_v = 1450\text{ MPa}$  (N4D2M + 0.5 % C + 0.1 %  $\text{Si}_3\text{N}_4$ ). At  $550\text{ }^{\circ}\text{C}$ , these steels exhibited maximum ductility indicators:  $\psi = 35\text{ }%$  (PZhRV 2.200.26 + 0.5 % C + 0.1 %  $\text{Si}_3\text{N}_4$ ) and  $\psi = 34\text{ }%$  (N4D2M + 0.5 % C + 0.1 %  $\text{Si}_3\text{N}_4$ ). The addition of 0.1 %  $\text{Si}_3\text{N}_4$  increased ultimate strength at  $250\text{ }^{\circ}\text{C}$  by 50 MPa for PZhRV 2.200.26 + 0.5 % C and by 30 MPa for N4D2M + 0.5 % C. Adding 2 % NiO to both materials slightly improved strength properties (by 10–15 MPa).

For HDPS tempered at  $550\text{ }^{\circ}\text{C}$ , the fracture surfaces predominantly displayed a dimpled morphology, with individual dimples ranging in diameter from 8 to  $20\text{ }\mu\text{m}$ . The clear resolution of dimple depths and ridge heights indicates the material's high capacity for microplastic deformation at the crack propagation site [19; 20].

This study demonstrates that strengthening heat treatment is a key tool for enhancing the mechanical properties of hot-deformed powder steels. By carefully adjusting quenching and tempering conditions, it is possible to improve the material's strength, ductility, and hardness. Managing mechanical properties depends

on the effective formation of intragranular bonding during production, which optimizes the microstructure and significantly enhances the performance of the final product. The combination of heat treatment and bonding control offers a promising pathway for advancing the quality and functionality of powder steels. These findings open new opportunities for developing materials with tailored properties, which are essential for modern mechanical engineering and other high-tech industries [10; 12].

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Received 13.11.2024

Revised 03.12.2024

Accepted 05.12.2024

Статья поступила 13.11.2024 г.

Доработана 03.12.2024 г.

Принята к публикации 05.12.2024 г.