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3. A. Meisel, G. Leonhardt, and R. Szargan, *Röntgenspektren und Chemische Bindung* [X-Ray Spectra and Chemical Bond] (Leipzig: Akademische Verlagsgesellschaft Geest & Portig K.-G.: 1977) (in German).
4. J. M. Ziman, *Printsipy Teorii Tverdogo Tela* [Principles of the Theory of Solids] (Moscow: Mir: 1974) (Russian translation).
5. M. A. Stucke, D. M. Dimiduk, and D. M. Hazzledine, *High Temperature Ordered Intermetallic Alloys. V* (Eds. I. Baker and R. Darolia) (Pittsburgh, PA, USA: MRS: 1993), p. 471.
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7. B. B. Karpovych and O. B. Borovkoff, *Proc. of Symp. 'Micromaterials Engineering' (Dec. 25–31, 1999)* (Kyiv: RVV IMF: 2000), vol. 2, p. 113 (in Russian).
8. A. E. Krug, *Abstr. Int. Conf. Phys. Phenomena (Dec. 25–31, 1991, Alushta)* (Kharkiv: 1991), p. 12.
9. T. M. Radchenko, *Vplyv Uporyadkuvannya Defektnoyi Struktury na Transportni Vlastyvoli Zmishanykh Krystaliv* [Influence of Ordering of the Defect Structure on Transport Properties of the Mixed Crystals] (Thesis of Disser. for the Degree of Dr. Phys.-Math. Sci.) (Kyiv: G. V. Kurdyumov Institute for Metal Physics, N.A.S.U.: 2015) (in Ukrainian). <https://doi.org/10.13140/RG.2.2.35430.22089>

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10. E. M. Gololobov, V. B. Shipilo, N. I. Sedrenok, and A. I. Dudyak, *Sposob Polucheniya Karbonitridov Metallov* [Production Method of Metal Carbonitrides], Authors' Certificate 722341 SSSR (Publ. November 21, 1979) (in Russian).

11. V. G. Trubachev, K. V. Chuistov, V. N. Gorshkov, and A. E. Perekos, *Sposob Polucheniya Metallicheskih Poroshkov* [The Technology of Metallic Powder Production]: Patent 1639892 SU. MKI, B22 F9/02, 9/14 (Otkrytiya i Izobreteniya, **34**, No. 13: 11) (1991) (in Russian).

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2. V. B. Tarelnyk, O. P. Gaponova, and Ye. V. Konoplianchenko, *Prog. Phys. Met.*, **23**, No. 1: 27 (2022). <https://doi.org/10.15407/ufm.23.01.027>
3. A. Meisel, G. Leonhardt, and R. Szargan, *Röntgenspektren und Chemische Bindung* [X-Ray Spectra and Chemical Bond] (Leipzig: Akademische Verlagsgesellschaft Geest & Portig K.-G.: 1977) (in German).
4. J. M. Ziman, *Printsipy Teorii Tverdogo Tela* [Principles of the Theory of Solids] (Moscow: Mir: 1974) (Russian translation).
5. M. A. Stucke, D. M. Dimiduk, and D. M. Hazzledine, *High Temperature Ordered Intermetallic Alloys*. V (Eds. I. Baker and R. Darolia) (Pittsburgh, PA, USA: MRS: 1993), p. 471.
6. *Handbook of Mathematical Functions with Formulas, Graphs and Mathematical Tables* (Eds. M. Abramowitz and I. A. Stegun), Nat'l Bureau of Standards. Appl. Math. Ser. Vol. 55 (Washington, D.C.: U.S. Govt. Printing Office: 1964).
7. B. B. Karpovych and O. B. Borovkoff, *Proc. of Symp. 'Micromaterials Engineering' (Dec. 25–31, 1999)* (Kyiv: RVV IMF: 2000), vol. 2, p. 113 (in Russian).
8. A. Eh. Krug, *Abstr. Int. Conf. Phys. Phenomena (Dec. 25–31, 1991, Alushta)* (Kharkiv: 1991), p. 12.
9. T. M. Radchenko, *Vplyv Uporyadkuvannya Defektnoyi Struktury na Transportni Vlastyvosti Zmishanykh Krystaliv* [Influence of Ordering of the Defect Structure on Transport Properties of the Mixed Crystals] (Thesis of Diss. for the Degree of Dr. Phys.-Math. Sci.) (Kyiv: G. V. Kurdyumov Institute for Metal Physics, N.A.S.U.: 2015) (in Ukrainian). <https://doi.org/10.13140/RG.2.2.35430.22089>
10. E. M. Gololobov, V. B. Shipilo, N. I. Sedrenok, and A. I. Dudyak, *Sposob Polucheniya Karbonitridov Metallov* [Production Method of Metal Carbonitrides], Authors' Certificate 722341 SSSR (Publ. November 21, 1979) (in Russian).
11. V. G. Trubachev, K. V. Chuistov, V. N. Gorshkov, and A. E. Perekos, *Sposob Polucheniya Metallicheskikh Poroshkov* [The Technology of Metallic Powder Production]: Patent 1639892 SU. MKI, B22 F9/02, 9/14 (Otkrytiya i Izobreteniya, **34**, No. 13: 11) (1991) (in Russian).
12. Yu. M. Koval' and V. V. Nemoshkalenko, *O Prirode Martensitnykh Prevrashcheniy* [On the Nature of Martensitic Transformations] (Kyiv: 1998) (Prepr./N.A.S. of Ukraine. Inst. for Metal Physics. No. 1, 1998) (in Russian).

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Electronic and Magnetic Properties of the Rhodonite

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In this study, the electronic and magnetic properties of the mineral rhodonite (MnSiO_3) are analysed, using both experimental and theoretical approaches. The investigation employs the experimental Faraday's method and theoretical DFT + U calculations. The inverse magnetic susceptibility $\chi^{-1}(T)$ of rhodonite is found to follow the Curie–Weiss law. A distinct anomaly is observed in the $\chi^{-1}(T)$ curve at 735 K, indicating a polymorphic phase transition from rhodonite to pyroxmangite, which is further confirmed by differential scanning calorimetry (DSC) analysis. The electron density of states is found to be asymmetric. Based on its electrical characteristics, rhodonite exhibits a wide band gap, indicating that it behaves as a dielectric mineral.

Key words: minerals, Faraday's method, magnetic susceptibility, Curie–Weiss law, magnetic moment, DFT + U calculations.

У даному дослідженні проведено комплексну аналізу електронних і магнетних властивостей мінералу родоніту (MnSiO_3) із застосуванням як експериментальних, так і теоретичних методів. Експериментальна частина виконана методом Фарадея, а теоретичне моделювання — за допомогою розрахунків у рамках функціоналу густини із врахуванням кулонівської кореляції (DFT + U). Обернена магнетна сприйнятливості $\chi^{-1}(T)$ родоніту задовольняє закон Кюрі–Вайса. За 735 К у залежності $\chi^{-1}(T)$ виявлено чітку аномалію, яку інтерпретують як поліморфний фазовий перехід від родоніту до пірохмангіту, що додатково підтверджено методом диферен-

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ційної сканувальної калориметрії (ДСК). Розподіл електронної густини станів виявився асиметричним. За своїми електричними характеристиками родоніт є широкозонним діелектриком.

Ключові слова: мінерали, метод Фарадея, магнетна сприйнятливість, закон Кюрі–Вайса, магнетний момент, DFT + U -обчислення.

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1. INTRODUCTION

Rhodonite, a manganese-rich mineral belonging to the pyroxenoid group, is characterized by a triclinic symmetry, similar to all other pyroxenoids. While the crystal lattice of rhodonite was initially studied in the conventional $P1$ space group, a more comprehensive structural analysis by Ohashi and Finger [1] led to the adoption of the $C1$ space group. This revised symmetry facilitates easier physical and chemical comparisons with pyroxenes and other pyroxenoids.

Rhodonite was first identified by Liebau and his research team [2], and four years later, its crystallographic data were refined and clarified by Peacor and Nisecki [3]. Although the mineral often contains several additional chemical elements, its ideal chemical formula is commonly represented as MnSiO_3 . This stoichiometric composition was synthetically obtained by Ito [4]; however, a pure and well-ordered mineral of this kind has not been found in nature. Liebau *et al.* [2] proposed a more realistic formula, $(\text{Mn,Ca})\text{SiO}_3$, where Fe and Mg typically appear as minor substituents [5].

The heat capacity (C_p^0) of rhodonite was measured by Richards and Huebner over the temperature range from 6 K to approximately 350 K, with no evidence of a λ -type anomaly (magnetic phase transition), indicating the mineral paramagnetic nature [6]. The unique magnetic behaviour of manganese-rich minerals, particularly, rhodonite, is of interest for both theoretical and practical research, potentially enabling various applications. The temperature dependence of magnetic susceptibility in minerals can be precisely studied using the Faraday's method, which allows for measurements over a wide temperature range [7, 8].

Density functional theory (DFT), a theoretical approach, whose computational results closely align with experimental observations, is increasingly utilized in the study of mineral properties. Investigating the electronic and magnetic characteristics of rhodonite, a manganese silicate mineral, through DFT calculations not only broadens the research scope, but also enhances the reliability of experimental findings. By employing DFT within the software packages such as Quantum ESPRESSO [9, 10], researchers can accurately model the electronic structure of rhodonite. This enables the elucidation of its band

structure and the density of states, which are critical for understanding its conductivity and other electronic properties. Reinforcing theoretical predictions with experimental validation significantly strengthens the credibility of the research.

2. METHODOLOGY

2.1. Experimental Details

The temperature dependence of the magnetic susceptibility, $\chi(T)$, of the rhodonite mineral was measured using the Faraday's method. The measurements were carried out in an inert helium atmosphere. The experimental values of the magnetic susceptibility at various temperatures were determined using the following expression:

$$\chi = \chi_r \frac{m_r (U_{h+s} - U_h)}{m_s (U_{h+r} - U_h)}. \quad (1)$$

In this context, U_{h+s} represents the compensating voltage measured for the combined sample and holder, while U_{h+r} corresponds to the compensating voltage for the standard reference and holder. U_h denotes the voltage drop of the empty holder (made of alumina, Al_2O_3). The symbols m_r and m_s refer to the masses of the reference and the sample, respectively, and χ_r indicates the magnetic susceptibility of the reference material. Mohr's salt was selected as the reference compound in the experiment.

The relative measurement uncertainty of the magnetic susceptibility in the setup does not exceed 3%.

2.2. Computational Details

For strongly correlated electron systems (particularly, those involving elements with occupied d and f orbitals such as Mn, Fe, Co, Ni and the lanthanides and actinides), the application of the extended density functional theory + Hubbard U (DFT + U) method is considered appropriate. This approach accounts for the on-site Coulomb repulsion between localized electrons, thereby, producing results, which are more representative for the real physical behaviour of the material. The crystallographic data required for the DFT + U calculations were obtained based on the experimental results reported by Nelson and Griffen [5]. The DFT + U calculations were performed for the ferromagnetic (FM) phase, using the Quantum ESPRESSO package [11]. The Hubbard U parameters employed were as follow: $U(\text{Mn}) = 4$ eV, $U(\text{Si}) = 0$ eV, and $U(\text{O}) = 0$ eV. Ultrasoft pseudopotentials were utilized

in all calculations.

3. RESULTS AND DISCUSSION

3.1. Magnetic-Susceptibility Measurements

The temperature dependence of the magnetic susceptibility, $\chi(T)$, for the rhodonite (MnSiO_3) mineral is illustrated in Fig. 1. As the temperature increases, the magnetic susceptibility decreases gradually. This behaviour is attributed to the enhanced thermal agitation, which leads to increasing disorder among the atomic magnetic moments. Figure 2 presents the inverse magnetic susceptibility (χ^{-1}) and the results of differential scanning calorimetry (DSC) analysis. In panel (a), the χ^{-1} curve exhibits a linear increase with temperature, followed by a no-

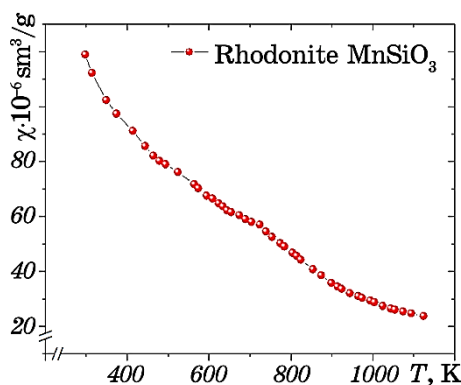


Fig. 1. The $\chi(T)$ dependence graph for rhodonite.

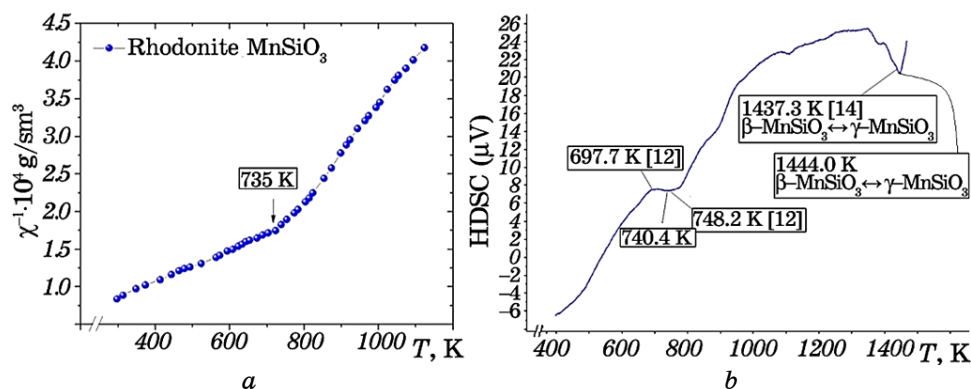


Fig. 2. The $\chi^{-1}(T)$ dependence for rhodonite (a) and its DSC analysis (b).

ticeable inflection point at 735 K. This sharp change in magnetic susceptibility indicates the occurrence of a polymorphic phase transition. To verify the presence of this transition, DSC analysis was conducted and compared with findings from previous studies reported in the literature.

The temperature-dependent variation of the DSC signal shown in panel (b) was obtained using a STA PT1600 TG-DSC/DTA system. An endothermic effect was recorded at 740.4 K, indicating a thermal event. Similar anomalies in this temperature range were also reported by Maresch and Mottana [12], who identified the α -rhodonite (MnSiO_3) $\leftrightarrow$ β -pyroxmangite (MnSiO_3) polymorphic phase transition at closely related temperatures, specifically, at 697.7 K and 748.2 K, under different experimental conditions. This correspondence further supports the occurrence of a phase transition in the studied sample.

The α -rhodonite (MnSiO_3) $\rightarrow$ β -pyroxmangite (MnSiO_3) polymorphic phase transition is illustrated in Fig. 3. The observed endothermic effect within the 697.7–748.2 K range closely corresponds to the magnetic anomaly detected at 735 K, indicating a strong correlation between the magnetic transition (χ^{-1}) and the thermal response (DSC). At higher temperatures, a pronounced endothermic effect was observed at 1444.0 K, suggesting an additional significant thermal transformation.

This represents a second phase transition occurring at a temperature close to the β - $\text{MnSiO}_3 \leftrightarrow \gamma$ - MnSiO_3 transformation reported at 1437.3 K by Proidak *et al.* [14]. The transition temperature observed in our study (at 1444.0 K) shows excellent agreement with the findings of [14], with the minor discrepancy ($\cong 7$ K) attributable to variations in experimental conditions such as scan rate, sample impurities, and instrument sensitivity.

As shown in Table 1, extrapolation of the linear region of the $\chi^{-1}(T)$ dependence within the temperature range of 297–723 K yields a posi-

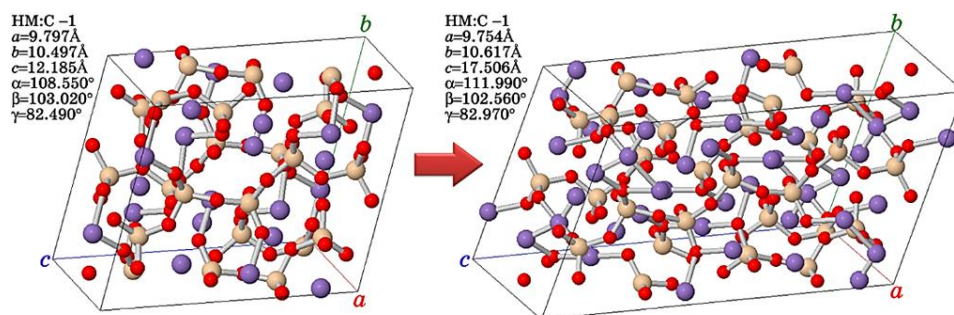


Fig. 3. Polymorphic phase transition from rhodonite (MnSiO_3) to pyroxmangite (MnSiO_3) [13].

TABLE 1. Calculation results.

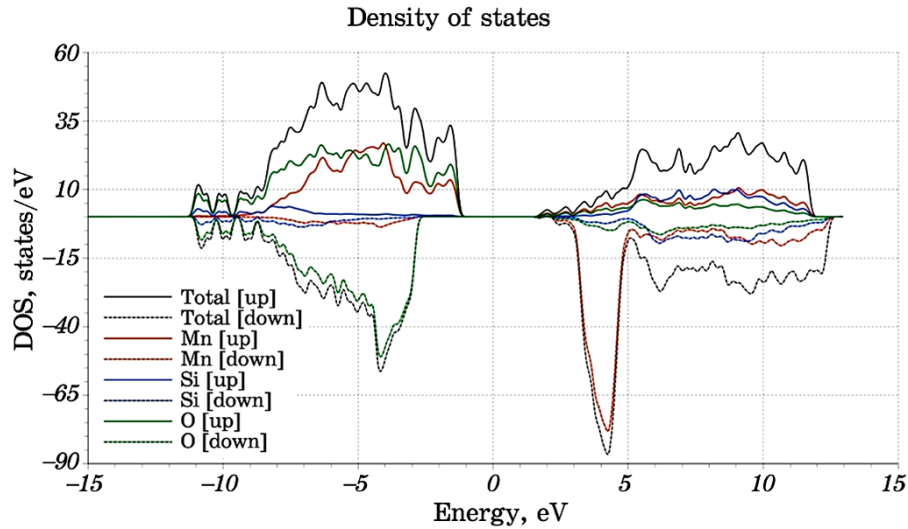
Mineral	Temperature interval, K	Θ	$C, 10^{-4} \text{ sm}^3 \cdot \text{g}^{-1} \cdot \text{K}$	μ_{eff}, μ_B	μ_{eff}, μ_B
rhodonite	297–723	115	18	4.53	6.21
(MnSiO ₃)	738–1123	545	485	2.68	3.67

tive Curie–Weiss temperature of $\Theta = 115$ K, indicating the presence of ferromagnetic interactions between the magnetoactive atoms in the rhodonite crystal structure. At higher temperatures (738–1123 K), Θ increases to 545 K. Concurrently, the effective magnetic moment (μ_{eff}) decreases from $4.53\mu_B$ to $2.68\mu_B$, suggesting the influence of thermal effects and a possible phase transition within the crystal lattice.

3.2. DFT + U Calculation

The DFT + U calculations based on the MnSiO₃ crystal structure of pure rhodonite (free of inclusions) were carried out for the ferromagnetic (FM) phase, using the Quantum ESPRESSO software package.

Figure 4 presents the calculated density of states (DOS), in which the asymmetrical distribution of the spin-up and spin-down states indicates spin polarization within the system. In particular, a pronounced difference is observed between the spin-up and spin-down densities of states for manganese and oxygen near the Fermi energy, indicating the presence of magnetic moments associated with the rho-

**Fig. 4.** Total and partial electron density of states of the rhodonite mineral.

donite atoms. Regarding the electronic properties, a significant band gap appears around the Fermi level, confirming the dielectric nature of rhodonite. As well known, DFT + U calculations tend to underestimate the band-gap energy (E_g); therefore, to obtain values in closer agreement with experimental data, the HSE06 (Heyd–Scuseria–Ernzerhof 2006 hybrid functional) method is recommended.

Table 2 presents the magnetic moments (in Bohr magnetons, μ_B) of Mn, Si, and O atoms as calculated using the DFT + U method. The magnetic moments of all manganese (Mn) atoms range from $4.0628\mu_B$ to $4.0633\mu_B$, indicating their dominant role in the magnetic behaviour of the system. In contrast, silicon (Si) atoms exhibit very small magnetic moments, between $0.0043\mu_B$ and $0.0050\mu_B$, suggesting that they do not

TABLE 2. DFT + U calculated results.

No.	Atom	Bohr magne- ton (μ_B)	No.	Atom	Bohr magne- ton (μ_B)	No.	Atom	Bohr magne- ton (μ_B)	No.	Atom	Bohr magne- ton (μ_B)
1	Mn	4.0628	26	Si	0.0045	51	O	0.0354	76	O	0.0233
2	Mn	4.0628	27	Si	0.0045	52	O	0.0354	77	O	0.0267
3	Mn	4.0628	28	Si	0.0045	53	O	0.0418	78	O	0.0267
4	Mn	4.0628	29	Si	0.0043	54	O	0.0418	79	O	0.0267
5	Mn	4.0633	30	Si	0.0043	55	O	0.0418	80	O	0.0267
6	Mn	4.0633	31	Si	0.0043	56	O	0.0418	81	O	0.0127
7	Mn	4.0633	32	Si	0.0043	57	O	0.0331	82	O	0.0127
8	Mn	4.0633	33	Si	0.0050	58	O	0.0331	83	O	0.0127
9	Mn	4.0599	34	Si	0.0050	59	O	0.0331	84	O	0.0127
10	Mn	4.0599	35	Si	0.0050	60	O	0.0331	85	O	0.0123
11	Mn	4.0599	36	Si	0.0050	61	O	0.0427	86	O	0.0123
12	Mn	4.0599	37	Si	0.0045	62	O	0.0427	87	O	0.0123
13	Mn	4.0299	38	Si	0.0045	63	O	0.0427	88	O	0.0123
14	Mn	4.0299	39	Si	0.0045	64	O	0.0427	89	O	0.0101
15	Mn	4.0299	40	Si	0.0045	65	O	0.0362	90	O	0.0101
16	Mn	4.0299	41	O	0.0454	66	O	0.0362	91	O	0.0101
17	Mn	4.0864	42	O	0.0454	67	O	0.0362	92	O	0.0101
18	Mn	4.0864	43	O	0.0454	68	O	0.0362	93	O	0.0127
19	Mn	4.0864	44	O	0.0454	69	O	0.0303	94	O	0.0127
20	Mn	4.0864	45	O	0.0378	70	O	0.0303	95	O	0.0127
21	Si	0.0053	46	O	0.0378	71	O	0.0303	96	O	0.0127
22	Si	0.0053	47	O	0.0378	72	O	0.0303	97	O	0.0168
23	Si	0.0053	48	O	0.0378	73	O	0.0233	98	O	0.0168
24	Si	0.0053	49	O	0.0354	74	O	0.0233	99	O	0.0168
25	Si	0.0045	50	O	0.0354	75	O	0.0233	100	O	0.0168

significantly contribute to the magnetization and primarily participate in chemical bonding within the crystal lattice. Oxygen (O) atoms develop minor magnetic moments ranging from $0.0127\mu_B$ to $0.0418\mu_B$. These results confirm that Mn atoms are the principal contributors to magnetism in the rhodonite mineral.

3.3. ELF Analysis

The electron localization function (ELF) map is a visual tool that illustrates the spatial localization of electrons and plays a crucial role in identifying interatomic electron densities and the nature of chemical bonding [15, 16]. It also reflects the electrical-conductivity characteristics within the molecular or crystal structures. Figure 5 shows the ELF map of the rhodonite (MnSiO_3) mineral. A strong ionic character is observed between MnI^+ and OI^- ions, although the valence electrons of oxygen also indicate partial covalence. Electrons are clearly local-

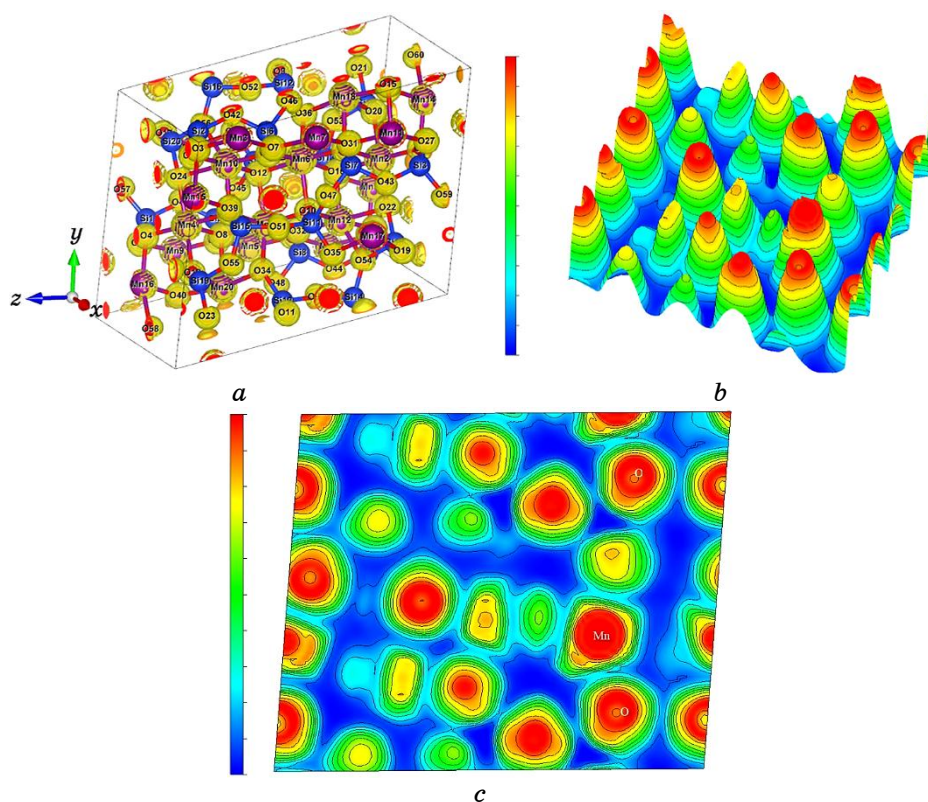


Fig. 5. ELF map of the rhodonite mineral, including 3D views (a), (b), and a 2D projection (c).

ized around Mn–O bonds, leading to the formation of electric dipoles, when an external electric field is applied. Such local dipole formations give rise to dielectric behaviour. The Si–O bonds exhibit polar and strongly covalent characteristics, with electron density skewed toward the oxygen atoms.

4. CONCLUSION

Based on the combined experimental and theoretical (DFT + U) investigations of the rhodonite mineral, the following conclusions were drawn. The $\chi^{-1}(T)$ curve exhibits linear behaviour, confirming that rhodonite follows the Curie–Weiss law. A kink in the $\chi^{-1}(T)$ plot at approximately 735 K corresponds to a polymorphic phase transition from rhodonite to pyroxmangite, consistent with the findings reported in Ref. [12]. At higher temperatures, the DSC analysis reveals a phase transition (β -MnSiO₃ $\leftrightarrow$ γ -MnSiO₃) at 1444.0 K that agrees with the results in Ref. [14]. From the perspective of electronic properties, the presence of a wide band gap near the Fermi level indicates that rhodonite is a dielectric mineral. Magnetic-moment calculations for Mn, Si, and O atoms confirm that Mn atoms are the primary contributors to the magnetic behaviour of the mineral. The ELF (electron localization function) map further supports the dielectric nature of rhodonite, revealing localized electron regions consistent with strong Mn–O bonding. Although the heat capacity (C_p^0) of rhodonite has been experimentally measured by Richard and Huebner over the temperature range from 6 K to $\approx$ 350 K, no λ -type peak indicative of a magnetic phase transition was observed [6], supporting the interpretation that rhodonite exhibits paramagnetic behaviour. However, to classify definitively rhodonite as an ideal paramagnet, additional experimental measurements below 6 K are required.

AUTHORS' CONTRIBUTIONS

O. K. Kuvandikov conceived the research idea and supervised the study. Z. M. Shodiev contributed to the interpretation of the results and manuscript editing. J. Sh. Akhtamov performed the theoretical calculations, conducted the experiments, analysed the results, and wrote the manuscript with input from all authors. All authors have read and approved the final version of the manuscript.

REFERENCES

1. Y. Ohashi and L. W. Finger, *Carnegie Inst. Washington Year Book*, **74**: 564 (1975).

2. F. Liebau, *Acta Crystallogr.*, **12**: 177 (1959).
3. D. R. Peacor and N. Niizeki, *Z. Kristallogr.*, **119**: 98 (1963).
4. J. Ito, *Am. Mineral.*, **57**: 865 (1972).
5. W. R. Nelson, *Am. Mineral.*, **90**, Nos. 5–6: 969 (2005).
6. R. A. Robie, J. S. Huebner, and B. S. Hemingway, *Am. Mineral.*, **80**, Nos. 5–6: 560 (1995).
7. O. K. Kuvandikov, Z. M. Shodiev, Kh. B. Khasanov, B. A. Khairullaev, and J. Sh. Akhtamov, *Metallofiz. Noveishie Tekhnol.*, **47**, No. 6: 595 (2025).
8. O. Kuvandikov, Z. Shodiev, U. Usarov, B. Khairullaev, and J. Akhtamov, *AIP Conf. Proc.*, **3304**, No. 1: 020009 (2025).
9. P. Giannozzi, S. Baroni, N. Bonini, M. Calandra, R. Car, C. Cavazzoni, D. Ceresoli, G. L. Chiarotti, M. Cococcioni, I. Dabo, A. Dal Corso, S. De Gironcoli, S. Fabris, G. Fratesi, R. Gebauer, U. Gerstmann, C. Gougoussis, A. Kokalj, M. Lazzeri, L. Martin-Samos, N. Marzari, F. Mauri, R. Mazzarello, S. Paolini, A. Pasquarello, L. Paulatto, C. Sbraccia, S. Scandolo, G. Sclauzero, A. P. Seitsonen, A. Smogunov, P. Umari, and R. M. Wentzcovitch, *J. Phys.: Condens. Matter*, **21**, No. 39: 395502 (2009).
10. P. Giannozzi, O. Baseggio, P. Bonfa, D. Brunato, R. Car, I. Carnimeo, C. Cavazzoni, S. de Gironcoli, P. Delugas, F. Ferrari Ruffino, A. Ferretti, N. Marzari, I. Timrov, A. Urru, and S. Baroni, *J. Chem. Phys.*, **152**, No. 15: 154105 (2020).
11. P. Giannozzi, O. Baseggio, P. Bonfa, D. Brunato, R. Car, I. Carnimeo, C. Cavazzoni, S. de Gironcoli, P. Delugas, F. Ferrari Ruffino, A. Ferretti, N. Marzari, I. Timrov, A. Urru, and S. Baroni, *J. Chem. Phys.*, **152**, No. 15: 154105 (2020).
12. W. V. Maresch and A. Mottana, *Contrib. Mineral. Petrol.*, **55**, No. 1: 69 (1976).
13. Mindat.org, *Rhodonite Mineral Data* (2025).
14. Y. Proidak, V. Gladkykh, and A. Ruban, *Sci. Innov.*, **18**, No. 2: 100 (2022).
15. J. K. Desmarais, G. Vignale, K. Bencheikh, A. Erba, and S. Pittalis, *Phys. Rev. Lett.*, **133**, No. 13: 136401 (2024).
16. D. B. Nguyen, C. Cárdenas, J. Lira, J. P. Perdew, K. A. Jackson, and J. E. Peralta, *J. Chem. Phys.*, **162**, No. 14: 144102 (2025).

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Dual-Mode Pulsed Plasma Source

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This paper presents the design and operating principle of a newly developed dual-mode pulsed plasma source capable of operating either as a pulsed vacuum-arc plasma source or as a pulsed magnetron sputtering system. Switching between the two operating modes is achieved by changing the magnetic-field configuration within the plasma source, modifying the activation sequence of the trigger plasma injectors and the energy supplied to the trigger discharges, as well as adjusting the voltages applied to the main electrodes of the source. The proposed technical solution simplifies significantly the design of ion-plasma deposition systems intended for hybrid (combined) coating technologies. Experimental evaluation of the dual-mode pulsed plasma source in a hybrid coating-deposition process, in which the vacuum-arc mode is employed during the initial ion-cleaning stage and the magnetron mode during the subsequent coating-deposition stage, demonstrates that the deposited coatings exhibit high adhesion to the substrate and are free of cathode-material microdroplets, indicating the high quality of the deposited coatings.

Key words: vacuum-arc discharge, magnetron sputtering system, magnetic-field configuration, trigger injector, hybrid coating-deposition technologies.

У даній статті описано конструкцію та принцип дії розробленого дворежимного імпульсного плазмового джерела, здатного працювати як імпульсне вакуумно-дугове джерело плазми, так і як імпульсна магнетронна

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розпорошувальна система. Перехід між режимами роботи забезпечується зміною конфігурації магнетного поля у робочому об'ємі плазмового джерела, зміною послідовності спрацьовування пускових плазмових інжекторів та енергії, що вкладається в пускові розряди, а також зміною напруг живлення, які подаються на основні електроди джерела. Запропоноване технічне рішення значною мірою спрощує конструкцію йонно-плазмових установок, призначених для реалізації гібридних (комбінованих) технологій нанесення покриттів різного призначення. Практичні випробування дворежимного імпульсного плазмового джерела в гібридній технології нанесення покриттів, за якою на першому етапі (йонне очищення) використовувався вакуумно-дуговий режим роботи, а на другому етапі (безпосереднє нанесення покриття) — магнетронний режим, показали, що сформовані покриття характеризуються високою адгезією до основи та не містять мікрочастинок матеріалу катоди, що свідчить про їхню високу якість.

Ключові слова: вакуумно-дуговий розряд, магнетрона розпорошувальна система, конфігурація магнетного поля, пусковий інжектор, гібридні технології одержання покриттів.

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1. INTRODUCTION

Among the most widely used PVD methods for producing coatings of various purposes are the vacuum-arc method (arc-PVD) [1, 2] and the method employing magnetron sputtering systems (mss-PVD) [3, 4]. Both methods belong to ion-plasma technologies, and each of them possesses specific advantages and disadvantages. The principal devices used for coating deposition by ion-plasma methods are technological vacuum-arc plasma sources (arc evaporators) in vacuum-arc systems and magnetron sputtering systems (magnetrons) in magnetron-based technologies.

In recent years, so-called hybrid (combined) methods [5–8] have increasingly been employed in ion-plasma systems to obtain coatings of the highest quality. Such methods involve the application of several different surface treatment techniques within a single technological process, utilizing the most advantageous features of each method.

The main advantages of the vacuum-arc coating-deposition method are the high adhesion of the deposited surface layer to the substrate and the high processing productivity. Its principal disadvantage is the presence of microparticles of cathode material within the deposited coatings, typically in the form of microdroplets with sizes $\geq 2 \mu\text{m}$, which is inherent to the physical nature of vacuum-arc discharges [9–11]. The use of plasma filters [12] makes it possible to reduce the number of microparticles reaching the treated surface; however, this approach does not eliminate completely the problem.

Coatings deposited by the magnetron method do not contain cathode-material microparticles that ensures their structural uniformity. However, their adhesion to the substrate and the productivity of the process are lower than those in the vacuum-arc method. This is due to the lower degree of ionization of atoms sputtered from the cathode target and the lower main discharge currents compared with the vacuum-arc method.

At present, the implementation of hybrid coating-deposition methods combining vacuum-arc and magnetron techniques within a single technological process requires ion-plasma systems equipped with at least one technological vacuum-arc plasma source and one magnetron sputtering system. Such an expansion of functional capabilities can be realized, for example, in 'Bulat' systems by installing a magnetron on the vacuum chamber. The development of systems for hybrid PVD processes based on the 'domino' principle is described in [13].

Improvement in coating quality achieved by the hybrid method combining vacuum-arc and magnetron technologies is realized as follows. During the first stage (ion cleaning), a vacuum-arc plasma source is employed simultaneously with the application of a high negative potential to the products. This ensures high coating adhesion to the substrate and heats the products to the required temperature, thereby improving the conditions for coating formation. After completion of ion cleaning, the second stage begins, during which the voltage applied to the substrate holder is reduced, the magnetron sputtering system is activated, and the coating is deposited by a flux of sputtered particles free from cathode material microparticles.

A significant disadvantage of such a combined method is the increased complexity of the equipment required for its implementation, since the coating-deposition system must incorporate two different types of particle sources responsible for coating formation.

The aim of this work is to develop the design of a pulsed plasma source capable of operating, depending on the selected mode, either as a pulsed vacuum-arc plasma source or as a magnetron sputtering system.

2. DESIGN OF THE DUAL-MODE PULSED PLASMA SOURCE

2.1 Basic Requirements for the Design of the Dual-Mode Pulsed Plasma Source

During the development of the dual-mode pulsed plasma source design, it was necessary to satisfy a number of requirements that would enable the plasma source to generate a plasma flux, while operating in two modes: either vacuum-arc discharge mode or magnetron discharge mode.

The principal requirements for vacuum-arc evaporators operating in a steady-state mode are presented in Refs. [1, 2]. When a vacuum-arc source operates in a pulsed mode, an additional requirement arises, namely, ensuring uniform cathode wearing.

As regards pulsed magnetron sputtering systems [3, 4], the operating requirements largely coincide with those for vacuum-arc plasma sources. The major difference between these types of plasma sources lies in the different magnetic field configurations required for their operation, as well as in the voltages that must be applied to the main electrodes of the devices and the operating modes of the trigger plasma injectors.

Thus, by creating conditions under which both vacuum-arc and magnetron discharges can be sustained sequentially within a single device through variation of the magnetic field configuration and other parameters, the implementation of the hybrid method can be significantly simplified.

2.2 Design of the Pulsed Plasma Source

The design of the dual-mode pulsed plasma source, which satisfies the requirements for operation in the mode of a pulsed vacuum-arc plasma source and, upon modification of the magnetic-field configuration and the operating mode of the trigger plasma injectors, provides transition to the mode of a pulsed magnetron sputtering system, is shown in Fig. 1.

The pulsed plasma source consists of an anode 1, at one end of which a consumable disc cathode 2 with an electromagnet 3 is coaxially mounted, while at the opposite end a substrate holder 4 with the treated products 5 is installed. By means of flange 6 and insulating gasket 7, the cathode is vacuum-tightly connected to the anode. The anode is surrounded by electromagnet 8 (the power supplies of electromagnets 3 and 8 are not shown in Fig. 1). The trigger plasma injectors TI_1-TI_{12} are installed within the anode body near cathode 2.

Each trigger plasma injector consists of an igniting electrode 9 (*IE*) and a cathode electrode 10 (*CE*), both made in the form of rectangular cross-section rods and placed inside a ceramic housing 11. The housing of each trigger plasma injector is vacuum-tightly integrated into the anode. The pulsed power supply unit PPS_0 of the main discharge between the anode and cathode includes a storage capacitor C_0 connected to the main power supply MPS_0 , and a switching element S_0 , the control terminal of which is connected to the control unit *CU*. The trigger plasma injectors TI_1-TI_{12} are powered by pulsed power supply units PPS_1-PPS_{12} (the units PPS_1-PPS_{12} and their connections to the arc power supply *APS* and the magnetron power supply *MPS* are shown schematically in Fig. 1).

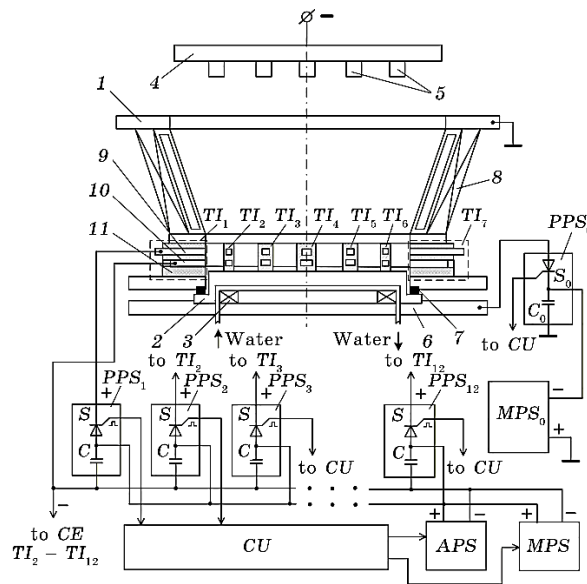


Fig. 1. Schematic diagram of the dual-mode pulsed plasma source and its power supply system: 1 is anode, 2 is cathode, 3 is solenoid, 4 is substrate holder, 5 are products, 6 is flange, 7 is insulator, 8 is solenoid, 9, 10 are electrodes of the trigger plasma injector, 11 is ceramic housing of the injector, TI_1 – TI_{12} are trigger plasma injectors, PPS_0 is pulsed power unit for the main discharge between the anode and cathode, PPS_1 – PPS_{12} are pulsed power units for the trigger plasma injectors (shown schematically), MPS_0 , APS, and MPS are power supplies, CU is control unit, CE is cathode electrode of the trigger plasma injector.

2.3 Features of the Components and Units of the Dual-Mode Pulsed Plasma Source

Unlike the plasma injectors used in vacuum-arc plasma sources for arc excitation, which typically have a cylindrical shape [13], the developed dual-mode pulsed plasma source employs trigger injectors of rectangular shape, as shown in Fig. 2.

In this case, the electrodes of the trigger injector have a rod-shaped configuration and are fabricated from the same material as the cathode. The increased cross-sectional area of the cathode electrode of the injector, compared with that of the igniting electrode, is explained by the formation of a cathode spot of the trigger discharge on its surface [14], which results in more intensive wearing compared with the igniting electrode. Such design solutions ensure the absence of impurities in the plasma generated by the dual-mode pulsed plasma source and provide a long service life of the main discharge ignition system.

During the fabrication of the trigger injector, electrodes were vac-

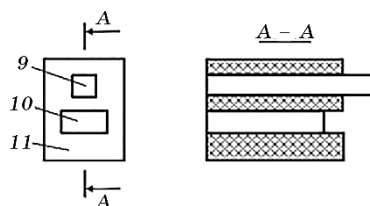


Fig. 2. Design of the trigger plasma injector: 9 is igniting electrode (IE), 10 is cathode electrode (CE), 11 is ceramic housing.

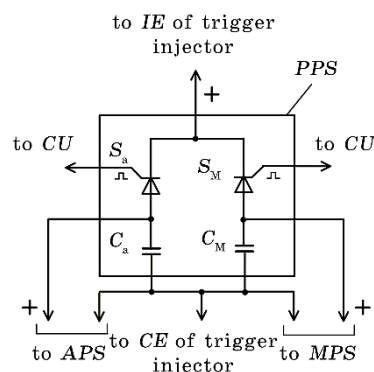


Fig. 3. Electrical circuit diagram of the trigger plasma-injector power supply unit.

uum-tight mounted into a housing made of 22KhS ceramic using sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$), following the procedure described in [15]. The trigger injectors were likewise vacuum-tight mounted into the anode, using the same technological procedure.

Each operating mode of the pulsed plasma source requires the corresponding operation of the trigger injectors, which ensure reliable excitation of either the vacuum-arc discharge or the magnetron discharge between the anode and cathode of the device. The operating mode of the trigger injectors is determined by the control unit *CU*, which regulates the operation of the plasma injector power supply units PPS_1 – PPS_{12} . The electrical circuit employed in the design of the trigger plasma injector power supply units is shown in Fig. 3.

3. OPERATING PRINCIPLES OF THE DUAL-MODE PULSED PLASMA SOURCE

After the preliminary surface preparation, the products 5 are mounted on the substrate holder 4. The vacuum chamber (not shown in Fig. 1) is then sealed and evacuated to a pressure of approximately 10^{-3} Pa. Sub-

sequently, the pulsed plasma source power supply system is activated. This includes the power supplies MPS_0 , APS , and MPS , which charge the capacitors C_0 , C_a , and C_m located in the pulsed units PPS_0 and PPS_1-PPS_{12} , respectively. In the present study, the capacitances of C_0 , C_a , and C_m were of 100.0 μF , 40.0 μF , and 2.0 μF , respectively. These capacitors were charged to the following voltages: 800 V for C_0 , 500 V for C_a , and 700 V for C_m .

The coating-deposition process subsequently begins with the ion-cleaning stage, during which the pulsed plasma source operates in the vacuum-arc mode. Upon receiving a command from the control unit CU, the power supplies of electromagnets 3 and 8 are energized, thereby, generating a combined and co-ordinated magnetic field.

The pulsed vacuum arc is ignited by means of trigger plasma injectors TI_1-TI_{12} . A triggering voltage is applied to the igniting electrode 9 (IE) of each injector from the pulsed units PPS_1-PPS_{12} (supplied by capacitors C_a ; Fig. 3) through the switching elements S_a (TB161-80-12 thyristors), which are controlled by the CU (Fig. 1).

The cathode electrodes 10 (CE) of the trigger plasma injectors TI_1-TI_{12} are permanently connected to the second terminals of capacitors C_a and C_m . Application of the triggering voltage to a plasma injector initiates a surface discharge along the dielectric separating its electrodes. As a result, a cathode spot is formed on the cathode electrode of the injector, generating a plasma plume in the vicinity of the working surface of cathode 2. Simultaneously, upon activation of the switching elements S_a , the control unit also switches on the main switching element S_0 (TB2-160-10 thyristor). Consequently, a voltage is established between anode 1 and cathode 2. In the presence of the plasma plume near the cathode surface, this voltage initiates a pulsed vacuum-arc discharge between the anode 1 and cathode 2 of the device.

The pulse repetition frequency of the vacuum-arc discharge was of 800 Hz, while the peak arc current reached 450 A. To increase the service life of the arc ignition system and to ensure uniform wearing of the cathode 2, the trigger plasma injectors were activated sequentially in a cyclic manner according to the algorithm implemented in the control unit. The energy supplied to each trigger discharge was of 5 J.

The cathode spots generated by the pulsed arc discharge transform the cathode material into a plasma state. The resulting cathode-material ions are accelerated by the bias voltage applied to the substrate holder 4 and the products 5 during this stage (the bias voltage was of 1000 V) and bombard the product surfaces, thereby providing simultaneous surface cleaning and heating. This process continues until the products 5 are heated to the required temperature (of approximately 500°C for P6M5 high-speed steel) and their surfaces become sufficiently cleaned and activated for the deposition of high-quality coatings. Once these conditions are achieved, the ion-cleaning stage is ter-

minated, and the process proceeds to the coating-deposition stage.

The coating-deposition process was carried out with the pulsed plasma source operating in the magnetron discharge mode. To switch to this operating mode, the power supplies of electromagnets 3 and 8 were reconfigured by the control unit *CU* to generate an opposing magnetic field. Simultaneously, the bias voltage applied to the substrate holder was reduced to 150 V, while the pressure in the vacuum chamber was increased to $2 \cdot 10^{-1}$ Pa by introducing the reactive gas (nitrogen).

The magnetron discharge between anode 1 and cathode 2 was initiated, using trigger plasma injectors TI_1 – TI_{12} . A triggering voltage from pulsed units PPS_1 – PPS_{12} was applied to the injector electrodes through the switching elements S_m (TCh-50-9 thyristors), which were controlled by the *CU*. This resulted in the simultaneous activation of all plasma injectors and the discharge of the energy stored in capacitors C_m into the trigger discharge. Under these operating conditions, the energy delivered to the trigger discharge of each injector was of 0.49 J.

Unlike the ion-cleaning mode, all trigger plasma injectors were activated simultaneously, producing a plasma cloud uniformly distributed over the working surface of the cathode 2. At the same time, similarly to the ion-cleaning stage, a voltage from the pulsed power unit PPS_0 was applied between anode 1 and cathode 2 by switching on the element S_0 in response to a command from the control unit. In the presence of the plasma cloud near the cathode surface and the magnetic field of the required configuration, these conditions resulted in the ignition of a pulsed magnetron discharge between anode 1 and cathode 2 of the plasma source.

The pulse repetition frequency of the magnetron discharge was 1000 Hz, while the peak discharge current reached 25 A. The atoms and ions sputtered from cathode 2 in the magnetron discharge were transported toward the substrate holder and deposited onto the surfaces of the products 5, thereby, forming the coating. The deposition process continued until a coating thickness of 2 μ m was achieved.

4. RESULTS AND DISCUSSION

Characterization of the coatings deposited using the proposed pulsed plasma source demonstrated that their adhesion to the substrate was comparable to that of conventional vacuum-arc coatings. At the same time, the deposited coatings were free of cathode material microdroplets, which are typically observed in vacuum-arc deposition processes. Furthermore, the coatings consisted exclusively of the cathode material without detectable impurities. These results indicate the high quality of the deposited coatings.

The proposed pulsed plasma source was used to deposit coatings on 15 batches of products under the operating conditions described above.

Throughout the experimental campaign, no failures were observed in the ignition of either the pulsed vacuum-arc discharge or the pulsed magnetron discharge, demonstrating the high reliability of the discharge ignition system.

Thus, the proposed design successfully achieves the intended technical objectives. Specifically, it extends the functional capabilities of the pulsed plasma source by enabling stable operation in both the magnetron discharge mode and the vacuum-arc discharge mode. In addition, the proposed ignition system provides enhanced reliability of the main discharge initiation in both operating modes and contributes to the deposition of high-quality coatings.

5. CONCLUSIONS

A pulsed plasma source capable of operating in both the pulsed vacuum-arc mode and the pulsed magnetron sputtering mode has been developed. Transition between the two operating modes is achieved by modifying the magnetic-field configuration within the plasma source, changing the activation sequence of the trigger plasma injectors and the energy supplied to the trigger discharges, as well as adjusting the voltages applied to the main electrodes. These operating conditions are implemented by the power supply circuit of the plasma source.

The design features of the dual-mode pulsed plasma source are presented, with particular emphasis on the trigger plasma injectors and their operating principles in each discharge mode.

Experimental evaluation of the dual-mode pulsed plasma source in a hybrid coating-deposition process, in which the plasma source operated in the vacuum-arc mode during the initial ion-cleaning stage and in the magnetron mode during the subsequent coating-deposition stage, demonstrated that the deposited coatings exhibited high adhesion to the substrate and were free of cathode material microdroplets. These results confirm the high quality of the deposited coatings.

The proposed plasma source significantly simplifies the design of ion-plasma coating systems intended for hybrid (combined) coating technologies by enabling both ion cleaning and coating deposition to be performed using a single plasma source operating in two different discharge modes.

AUTHORS' CONTRIBUTIONS

S. Iu. O. proposed and developed the operating principle of a plasma source capable of operating in two modes: as a vacuum-arc plasma source and, by changing the magnetic-field configuration, as a magnetron sputtering system. Sh. Yu. V. developed the design of the plasma

source, prepared the technical drawings, and provided further details of the operating principle of the developed plasma source. F. K. V. investigated the characteristics of the developed plasma source. All authors approved the final version of the manuscript.

REFERENCES

1. A. A. Andreev, L. P. Sablev, and S. N. Hryhorev, *Vakuumno-Dugovyye Pokrytiya* [Vacuum-Arc Coatings] (Kharkov: NSC 'Kharkov Institute of Physics and Technology': 2010) (in Russian).
2. Y. Y. Aksenov, A. A. Andreev, V. A. Belous, V. E. Strelnytskyi, and V. M. Khoroshykh, *Vakuumnaya Duga: Istochniki Plazmy, Osazhdenie Pokrytiy, Poverkhnostnoye Modifitsirovanie* [Vacuum Arc: Plasma Sources, Coating Deposition, Surface Modification] (Kiev: Naukova Dumka: 2012) (in Russian).
3. A. I. Kuzmichev, *Magnetronnyye Raspylitelnyye Sistemy. Kniga 1. Vvedenie v Fiziku i Tekhniku Magnetronnogo Raspyleniya* [Magnetron Sputtering Systems. Book 1. Introduction to the Physics and Technology of Magnetron Sputtering] (Kiev: Avers: 2008) (in Russian).
4. P. J. Kelly and J. W. Bradley, *Journal of Optoelectronics and Advanced Materials*, **11**, No. 9: 1101 (2009).
5. K. Yamamoto, S. Kujime, and K. Takahara, *Surface and Coating Technology*, **200**: 1383 (2005).
6. R. Shishov, D. Dochev, I. Dermendzhiev, V. Zaharieva, and M. Ilieva, Vacuum Metallization Equipment Combining Magnetron Sputtering and Arc Evaporation Methods (VME-MS/ARC), *Second National Conference with International Participation 'Metal Science, Novel Materials, Hydro- and Aerodynamics 2012'* (Ruse University 'Angel Kanchev': 2012).
7. D. Geng, Yu X. Xu, and Q. Wang, *Surface and Coatings Technology*, **456**: 129235 (2023).
8. R. Zhang, Q. Wang, Yu. Xu, L. Li, and K. Ho Kim, *Lubricants*, **13**, No. 9: 390 (2025).
9. A. Anders, *Cathodic Arcs – From Fractal Spots to Energetic Condensation* (Springer: 2008).
10. V. M. Khoroshikh, *PSE*, **2**, No. 4: 184 (2004) (in Russian).
11. P. M. Martin, *Handbook of Deposition Technologies for Films and Coatings, Third Edition: Science, Applications and Technology* (William Andrew: 2010).
12. P. J. Martin and A. Bendavid, *Thin Solid Films*, **394**, Iss. 1–2: 1 (2001).
13. J. Vetter, J. Müller, and G. Erkens, *IOP Conference Series: Mater. Sci. Eng.*, **39**: 012004 (2011).
14. Iu. O. Sysoiev, *Tekhnologiya Mashynobuduvannya. Zabezpechennya Ehfektivnosti Protseiv Otrymannya Vakuumno-Dugovykh Pokryttiv* [Engineering Technology: Ensuring the Efficiency of Vacuum-Arc Coating Processes] (Kharkiv: National Aerospace University 'Kharkiv Aviation Institute': 2021) (in Ukrainian).
15. Iu. O. Sysoiev, Yu. V. Shyrokyi, and K. V. Fesenko, *Metallofiz. Noveishie Tekhnol.*, **47**, No. 10: 1027 (2025).

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Relationship between Structural Characteristics and Physical-Mechanical Properties of Hafnium-Diboride Films Deposited by RF Magnetron Sputtering

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A comparative analysis of the structure and physical-mechanical characteristics of hafnium-diboride films synthesized by means of RF magnetron sputtering system is performed. As shown, changing the design features of the magnetron sputtering system (MSS) changes the structure formation parameters and, accordingly, the properties of the synthesized films. Two series of samples are obtained: before and after making changes to the MSS design. In the first series of experiments, films with the growth texture of the (00.1) plane are formed at a positive bias potential applied to the substrate, have a strong growth texture and high physical-mechanical characteristics: nanohardness of 41 GPa and elastic modulus of 396 GPa. For the second series of samples, textured hafnium-diboride films with the most intense texture (00.1) are formed with a grounded-substrate holder, while the resulting films have the best physical-mechanical characteristics: nanohardness of 46 GPa and elastic modulus of 306 GPa. As shown, hafnium-diboride films with a strong growth texture normal to the (00.1) plane have the highest physical-mechanical properties, regardless of the deposition conditions during magnetron sputtering.

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Key words: coatings, films, magnetron sputtering, nanohardness, modulus of elasticity.

Проведено порівняльну аналіз структури та фізико-механічних характеристик плівок дибориду Гафнію, синтезованих за допомогою ВЧ-магнетронної системи розпорошення. Показано, що зміна конструктивних особливостей магнетронної розпорошувальної системи (МРС) змінює параметри структуроутворення та, відповідно, властивості синтезованих плівок. Одержано дві серії зразків: до та після внесення змін у конструкцію МРС. У першій серії експериментів плівки з текстурою росту площини (00.1) формуються за прикладеного до підкладинки позитивного потенціалу зміщення, мають сильну текстуру росту та високі фізико-механічні характеристики: нанотвердість у 41 ГПа і модуль пружності у 396 Па. Для другої серії зразків текстуровані плівки дибориду Гафнію з найбільш інтенсивною текстурою (00.1) формуються із заґрунтованим підкладинкотримачем; водночас, одержані плівки мають найліпші фізико-механічні характеристики: нанотвердість у 46 ГПа і модуль пружності у 306 ГПа. Показано, що плівки дибориду Гафнію з сильною текстурою росту, нормальною до площини (00.1), мають найвищі фізико-механічні властивості, незалежно від умов осадження під час магнетронного розпорошення.

Ключові слова: покриття, плівки, магнетронне розпорошення, нанотвердість, модуль пружності.

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1. INTRODUCTION

Film coatings of transition-metal diborides (CrB_2 , HfB_2 , TiB_2 , TaB_2 , etc.) having high hardness with a relatively small elastic modulus are promising for use as the heat-resistant and wear-resistant protective coatings in various industries [1–4]. Due to the high melting point, the most widely used method for applying such coatings is magnetron sputtering, mainly, on direct current and RF magnetron sputtering. By changing the parameters of magnetron sputtering (working pressure, discharge power, substrate temperature, distance between the target and the substrate, bias potential), it is possible to change the energy of particles, which participate in the deposition and formation of films on the substrate surface in a wide range. The review [5] provides a detailed analysis of the sputtering parameters and their influence on the formation of the structure and, accordingly, their physical-mechanical characteristics. This makes it possible to obtain coatings of various structural states, from amorphous to nanocrystalline ones, with the (00.1)-plane growth texture and the composition from substoichiometric MeB to superstoichiometric MeB_n . As shown, the superstoichiometry n values for transition-metal diboride films with the

(00.1)-plane growth texture vary widely from 2.07 to 4.5 for TiB_2 [6–11] and 2.7 to 3.84 for TaB_2 [10–12]. However, the best hardness values, more than 40 GPa, were possessed by superstoichiometric films with the B/Me ratio within 2–3, which was noted for titanium [6, 8, 13, 14] and tantalum [9, 10, 13, 15, 16] diborides.

A characteristic feature of transition-metal diboride films is that boron-superstoichiometric films with a strong (0001)-plane growth texture had higher hardness values of 43.9 GPa (HfB_2), 44 GPa (TaB_2), 43 GPa (CrB_2), 48.5 GPa (TiB_2) compared to stoichiometric films ($HV = 33$ GPa, 36 GPa, 37 GPa, and 35 GPa, respectively) [6, 9–17]. It should also be noted that, of this class of compounds, only vanadium-diboride films with a (0001)-plane growth texture did not show such high characteristics. This was associated with a high percentage of oxide phases in the film [18, 19].

Among all nanostructured coatings of transition-metal diborides, hafnium-diboride coatings are of particular interest. Hafnium diboride has the highest melting point among diborides, high chemical resistance, and high hardness. Functional coatings based on it are capable of operating under extreme conditions, while maintaining the above-mentioned properties. Some of the promising areas of application of these coatings are nuclear energy and aerospace, namely, the creation of hypersonic aircraft. The study of the properties of nanostructured coatings of hafnium diboride began relatively recently [20] and remains relevant to this day [21].

A comparative analysis of the influence of structural characteristics on the properties of transition-metal boride films showed that films with a strong growth texture by the (0001) plane and high hardness values are formed for various magnetron sputtering systems at various magnetron sputtering parameters, both with direct current and with RF sputtering [8, 22]. This is due to the design features of various magnetron systems.

In this work, a study was conducted of the relationship between the structure and physical-mechanical characteristics of hafnium-diboride films synthesized using an RF magnetron sputtering system.

2. EXPERIMENTAL/THEORETICAL DETAILS

The horizontal RF magnetron sputtering system (MSS) based on an unbalanced planar magnetron designed for the use of standard round targets with a diameter of 120–125 mm and a thickness of up to 10 mm was used for deposition of hafnium-diboride films. The schematic diagram of scattering unit is shown in Fig. 1 [18]. The magnetron magnetic field with strength of about $4 \cdot 10^3$ A/m on the target surface is created by a set of permanent ring magnets (CoSm) with a steel pole core. The source of RF power is a generator with a frequency of

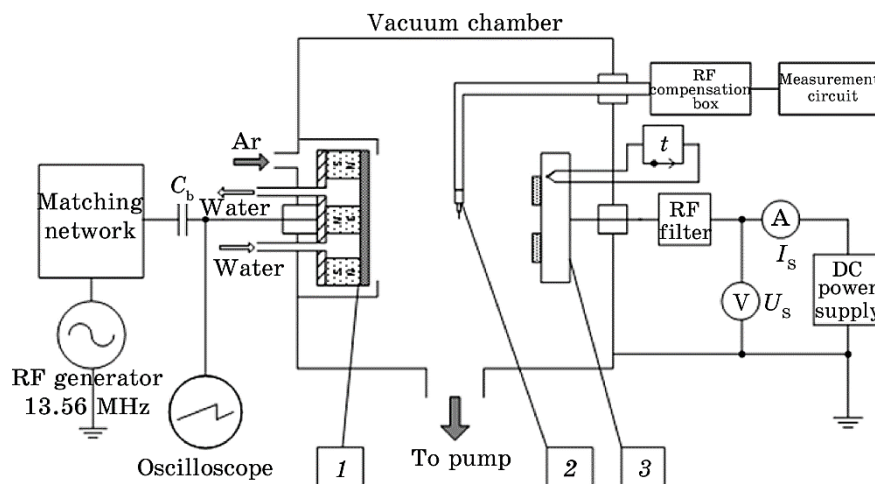


Fig. 1. Diagram of a magnetron sputtering system: 1—unbalanced magnetron, 2—Langmuir's probe, 3—substrate holder.

13.56 MHz and an adjustable power of up to 1 kW connected to the load, using a matching network, which is a L -circuit of reactive elements, and a blocking capacitor that ensures the occurrence of a negative self-bias on the target.

The substrate holder is electrically isolated from the installation body by ceramic insulators and designed for fastening plate substrates. A constant voltage source is connected to the substrate holder through an RF filter, allowing a bias potential in the range from -100 to $+100$ V to be applied to the substrate. The samples are heated, using a tungsten filament with a diameter of 2 mm; a chromel–alumel thermocouple provides temperature control up to 1100 K.

The sintered HfB_2 target was sputtered in Ar^+ plasma. Hafnium-diboride coatings were deposited on 12X18H10T stainless-steel substrates measuring $20 \times 10 \times 3$ mm, pre-polished to a surface roughness of $Ra = 0.25$ μm . The pressure of the residual gases in the chamber before spraying was of $3 \cdot 10^{-3}$ Pa. The duration of the deposition of each sample was of 60 min.

Two series of samples were obtained: the first series was obtained before modification of the magnetron sputtering system; the second series was obtained after modification of the MSS. The design changes were made in the modified installation: the designs of the magnetron and substrate holder were changed, the pumping system was changed, and a Langmuir's probe was installed to measure the parameters of RF plasma. In both cases, the working gas pressure during sputtering was of 0.65 Pa, the RF generator power was of 500 W, the bias potential applied to the substrate varied from $+25$ V to -25 V relative to ground

with a step of 25 V. The distance between the target and the substrate holder was $d_{s-t} = 60$ mm. The substrate temperature was maintained at 300°C.

Immediately before deposition, ion cleaning was carried out with argon ions for 3 min.

The thickness of the films was controlled by multibeam interferometry using an MII-4 interferometer; it was of approximately 1.5 μm .

X-ray diffraction studies of the structure of the material were performed on an automated diffractometer ДРОН-3. When recording, CuK_α radiation (wavelength of 0.154 nm) was used with Bragg–Brentano focusing θ – 2θ (2θ is the Bragg angle). The current and voltage values at the x-ray tube were of 20 mA and 40 kV. The samples were recorded with horizontal slits of 4 mm on the tube and of 1 mm on the detector in continuous recording mode at a speed of 1°/min with a range of 2θ -angles from 20° to 70°. The calculation of nanocrystallite sizes and microdeformations was carried out using the Scherrer's method.

Hardness and elastic modulus were measured by nanoindentation. For the first series of samples, nanoindentation was performed by means of the Nano Indenter II device from MTS Systems equipped with a diamond indenter sharpened in the shape of a trihedral pyramid (Berkovich's indenter). Nanoindentation of the second series of samples was performed by means of the Ultra-Nano Hardness Tester (UNHT) device from CSM Instruments equipped with a Berkovich's indenter too. Hardness and elastic modulus were found by analysing the indenter-unloading curve, using the Oliver–Pharr method [19].

3. RESULTS AND DISCUSSION

Two series of samples were obtained: the first series was obtained before modification of the magnetron sputtering system; the second series was obtained after modification of the MSS. The following design changes were made in the modified setup:

- the magnetron design was changed: the central iron core (pole) was replaced with a permalloy core, which significantly changed the magnetron balance (Fig. 2) and allowed increasing the concentration of the discharge plasma between the target and the substrate holder;
- the discharge gap (between the target and the grounded screen) was changed from 8 to 6.5 mm in order to reduce capacitive losses;
- the area of the substrate holder was increased in order to increase the discharge capacity;
- the pumping system was improved and the Langmuir's probe was installed to measure the parameters of the RF plasma.

Before replacing the magnetron core, the magnetron magnetic-field line configuration was simulated, using the ANSYS Mechanical APDL

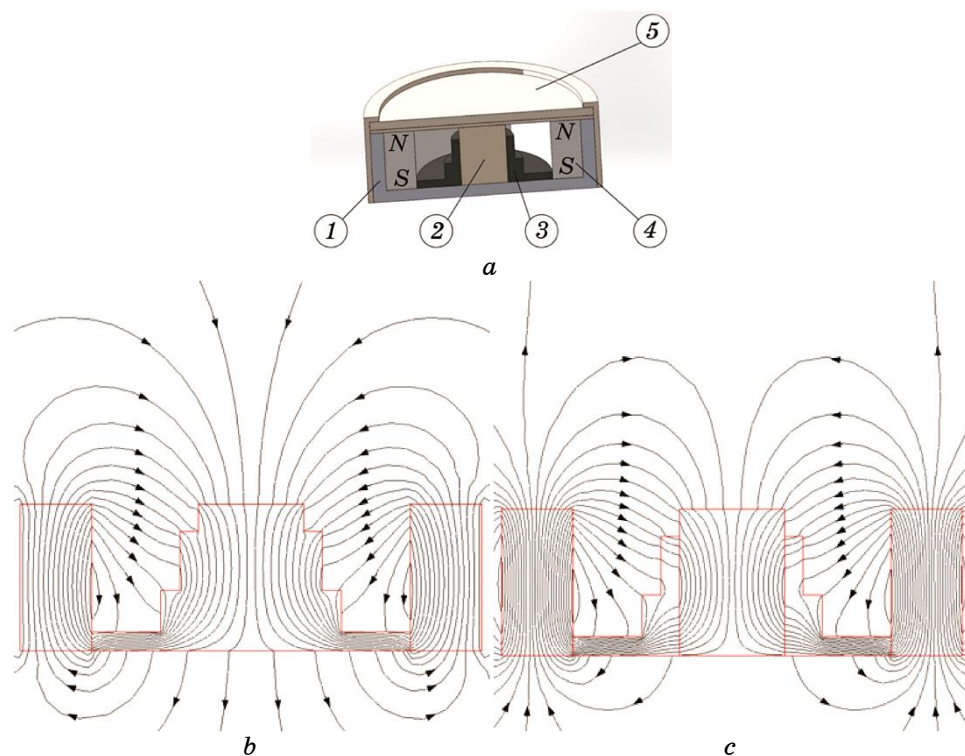


Fig. 2. Results of modelling the magnetic field of a magnetron: magnetron (1—stainless-steel body of the magnetron, 2—core, 3—steel core holder, 4—CoSm ring magnet, 5—HfB₂ target) (a); magnetic field of a magnetron with a steel core (b); magnetic field of a magnetron with a permalloy core (c).

package, the results of which are shown in Fig. 2. As can be seen, such a replacement leads to a change in the magnetron balance from type I to type II, according to Ref. [20]. Such a replacement leads to a significant change in the charged particle flows in the magnetron glow-discharge plasma during film deposition. Thus, when using a magnetron of the second type, there is an increase in the charged particle flows to the substrate (ions and electrons, depending on the bias potential on the substrate), and there is also an increase in the bias potentials on the surfaces coming into contact with the plasma discharge. This leads to an increase in the energy transferred by the charged particles to the growing film and, accordingly, to a modification of the structure and preferred orientation of the coating.

X-ray structural studies of the first series of samples showed (Fig. 3, a) that, with a grounded substrate holder, nanocluster films of hafnium diboride are formed. Applying a positive bias potential leads to the formation of films with a growth texture (00.1). Applying a nega-

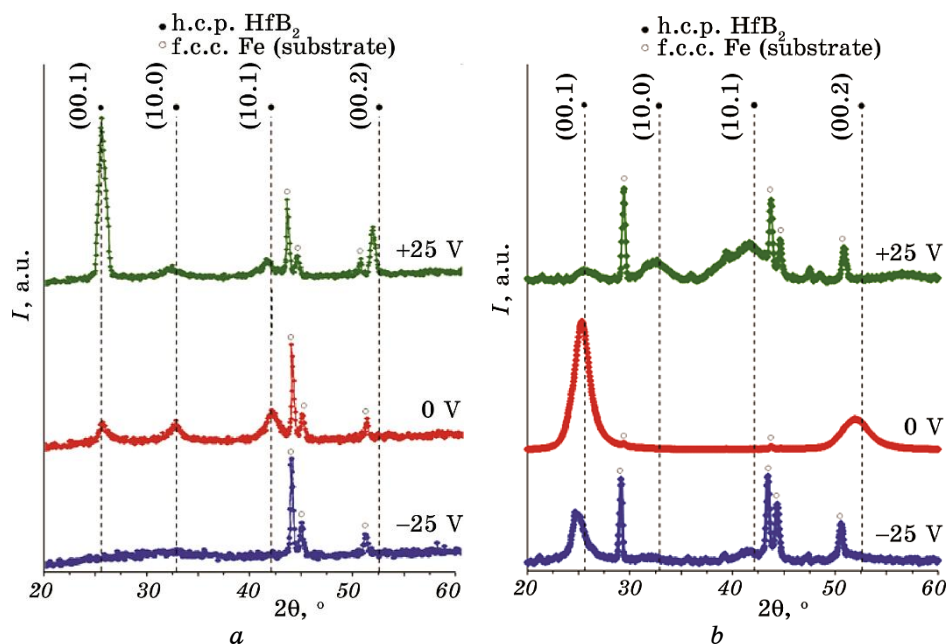


Fig. 3. Diffraction patterns of hafnium diboride films of samples obtained: in the first experiment [22] (a), in the second experiment (the diffraction pattern of the sample deposited with the substrate holder grounded is shown on a scale of 1:10) [18, 23] (b).

tive bias potential of -25 V leads to the formation of amorphous-like films. Changing the design features of the MSS leads to a significant change in the parameters of formation of the texture of the synthesized films.

For the second series of samples, with a grounded substrate holder, textured hafnium-diboride films with the most intense texture (00.1) are formed. Applying a negative bias potential of -25 V (Fig. 2, b) leads to a decrease in the growth texture by the (00.1) plane [22, 23]. Significant structural changes occur, when applying a positive bias potential of $+25$ V to the substrate holder (Fig. 2, b). Nanocluster films are formed [22, 23] with the growth texture being completely absent.

Calculation of the texture coefficient allows us to study the development of texture as a function of the change in bias voltage on the substrate. Texture coefficients for HfB_2 films were calculated, based on XRD studies of the obtained samples (Fig. 4), using the following formula [24]:

$$T_{(hk.l)} = \frac{I_{(hk.l)}}{I_{(00.1)} + I_{(10.0)} + I_{(10.1)}}, \quad (1)$$

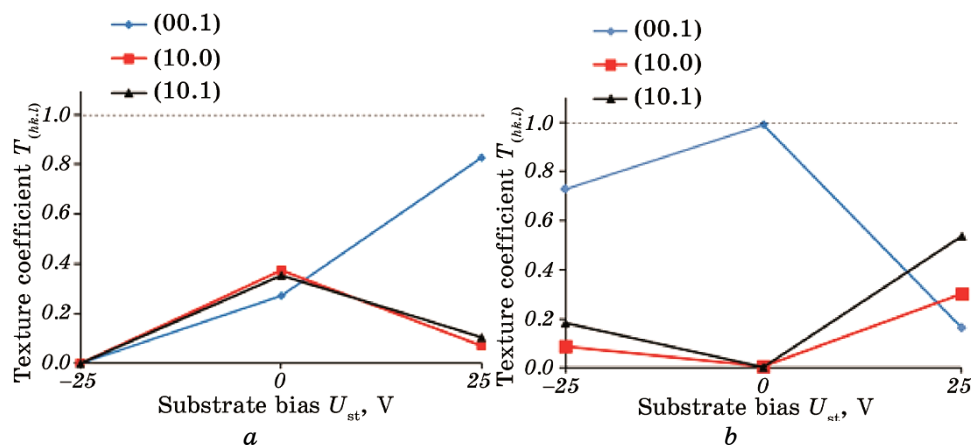


Fig. 4. Change in texture coefficients depending on bias voltage: for samples of the first series (a); for samples of the second series (b).

where $I(hk.l)$ represents the intensity of all the main orientations of the HfB_2 phase.

The sample from the second series obtained with a grounded substrate holder has the highest texture coefficient. In addition, the following have a high texture coefficient: the sample from the first series obtained with a bias potential of +25 V; the sample from the second series obtained with a bias potential of -25 V.

Based on the load-unload curves, the nanohardness and elastic modulus of the samples were calculated, using the Oliver-Pharr method (Fig. 5).

For the samples of the first series, the films with a strong growth texture by the (0001) plane had the best physical-mechanical properties: nanohardness of 41 GPa and elastic modulus of 396 GPa (Fig. 5, a). When the substrate holder is grounded, nanocrystalline films of hafnium diboride are formed, which leads to a decrease in hardness and elastic modulus (26 GPa and 254 GPa, respectively). Applying a negative bias potential of -25 V leads to the formation of amorphous-like films that ultimately leads to an even more-significant decrease in the hardness (13 GPa) and elastic modulus (187 GPa).

For the samples of the second series, the films with a strong growth texture by the (0001) plane also had the best physical-mechanical characteristics: nanohardness of 46 GPa and elastic modulus of 306 GPa (Fig. 5, b). For films with a less-pronounced growth texture, a decrease in the nanohardness value to 36 GPa and a decrease in the elastic modulus to 271 GPa are observed, which have already occurred previously for TaB_2 [25], TiB_2 [3, 26], CrB_2 [27] films. For hafnium-diboride nanocluster films obtained at a bias potential of +25 V, a significant

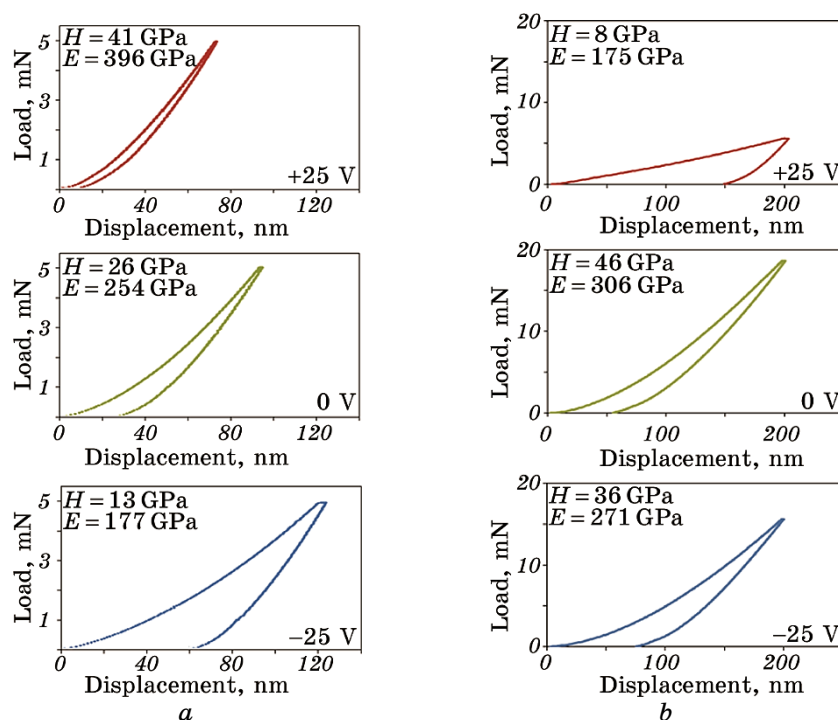


Fig. 5. Load–unloading curves, as well as nanohardness and elastic modulus obtained by nanoindentation of samples: first series (a); second series (b).

decrease in hardness and elastic modulus is observed (8 GPa and 175 GPa, respectively).

The calculations of the nanocrystallite sizes, carried out using the Scherrer's method, are presented in Table 1. A distinctive feature is that the nanocrystallite size of the highly-textured hafnium-diboride films from the first series is higher compared to similar highly-textured films from the second series.

Comparison of our results with those obtained by other authors, when depositing the transition-metal diboride films, using RF and DC magnetron sputtering systems, shows that each MSS has its own values of deposition parameters, at which films with the best physical-mechanical characteristics are formed.

Thus, in works [6, 13, 14], superstoichiometric-TiB_{2.4} films with the best physical-mechanical characteristics ($HV = 48.5$ GPa, $E \cong 480$ GPa) were obtained at a bias potential of -50 V, substrate temperature $T_s \cong 550^\circ\text{C}$ and current density $j = 1$ mA/cm². Highly-textured superstoichiometric titanium-diboride films with the composition $B/Me \cong 2.3\text{--}2.4$ having a nanohardness of 48.6 ± 3.1 GPa and elastic modulus of 562 ± 18 GPa were obtained by other authors [8, 32] at a

TABLE 1. Production modes, lattice parameters and mechanical properties of the studied HfB₂ coatings (*—JCPDS file, card No. 38-1398).

Sample series	Substrate bias potential U_s , V	Substrate temperature T , °C	Lattice parameters, nm			Nanocrystallite size D , nm	Nanohardness H , GPa	Modulus of elasticity E , GPa	Degree of texture
			a	c	c/a				
			3.1424*	3.476*	1.11*				
1	+25	300	3.230	3.400	1.05	25	41	396	strong
	0	300	3.160	3.460	1.09	10	26	254	—
	−25	300	3.179	3.560	1.10	—	13	177	—
2	+25	300	3.179	3.362	1.06	4	8	175	—
	0	300	3.210	3.494	1.09	8	46	306	relatively strong
	−25	300	3.182	3.379	1.06	15	36	271	strong

bias potential of -100 V and a substrate temperature of 175°C , which correlate with the above results. However, as shown in the works [34, 35] by Berger *et al.*, it is possible to form highly-textured titanium-diboride films with the best physical-mechanical characteristics ($H = 54 \pm 9$ GPa, $E = 600 \pm 85$ GPa) even at a positive bias potential of $+50$ V. In the work [17], highly-textured hafnium-diboride films of HfB_{2.4} with the best physical-mechanical characteristics ($HV = 44$ GPa and $E \cong 400$ GPa) were obtained by RF magnetron sputtering at a bias potential of $+50$ V and a substrate temperature of 500°C .

It is obvious that, regardless of the type of MSS (DC, RF) and its design features, there will always be a combination of deposition parameters (discharge power, working gas pressure, bias potential and temperature on the substrate, *etc.*) that will produce transition-metal diboride films with the highest degree of growth texture normal to the (00.1) plane and high physical-mechanical characteristics.

The analysis of the obtained results shows that the hardness of refractory compounds depends on their structural state. At the same time, the degree of texture affects the physical-mechanical characteristics of all transition-metal diborides having the AlB₂ structural type [4, 22].

4. CONCLUSION

The influence of structure on the physical-mechanical properties of hafnium-diboride films obtained by means of RF magnetron sputtering was studied. As shown, depending on the sputtering parameters, films are formed in various structural states from amorphous to nanocrystalline ones with a growth texture normal to the plane (0.01).

The values of hardness and elastic modulus varied from 8 GPa and 175 GPa to 46 GPa and 306 GPa, respectively. As shown, a change in the magnetron design leads to a change in the RF magnetron sputtering parameters. Hafnium-diboride films with the highest degree of growth texture normal to the plane (00.1) and high physical-mechanical properties (nanohardness of 41 GPa and elastic modulus of $\cong 400$ GPa) were obtained on unmodified MSS at a bias potential of +25 V. Modification of MSS led to a change in the values of the deposition parameters, as a result of which hafnium-diboride films with the highest degree of growth texture normal to the plane (00.1) and high physical-mechanical properties (nanohardness of 46 GPa and elastic modulus of 306 GPa) were obtained with a grounded substrate holder (bias potential of 0 V). A distinctive feature is that the size of nanocrystallites of highly-textured hafnium-diboride films from the first series is higher in comparison with similar highly-textured films from the second series, while the degree of texture for samples from the second series is higher, that probably leads to an increase in nanohardness to 46 GPa. It is obvious that the change in the design features of the MSS leads to a significant change in the parameters of the formation of the structural characteristics and texture of the synthesized films. The results obtained correlate with the results for highly-textured films of transition-metal diborides.

A comparative analysis of magnetron sputtering parameters for hafnium-diboride films with the data obtained for films of other transition-metal diborides showed that, regardless of the type of magnetron sputtering (DC, RF) and its design features, there will always be a combination of sputtering parameters (discharge power, working gas pressure, bias potential and temperature on the substrate, *etc.*), at which transition-metal diboride films with the highest degree of growth texture normal to the (00.1) plane and the best physical-mechanical properties are obtained.

Thus, it can be concluded that, if, at any sputtering parameters, highly-textured hafnium-diboride films are obtained, in particular, then, they will have *a priori* the highest physical-mechanical characteristics.

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REFERENCES

1. R. A. Andrievski, *Russian Chem. Rev.*, **84**: 540 (2015).
2. R. A. Andrievski, *Physics-Uspekhi*, **60**: 276 (2017).
3. M. Magnuson, L. Hultman, and H. Högberg, *Vacuum*, **196**: 110567 (2022).
4. M. Mikula, *Nanocomposite and Nanocrystalline Materials and Coatings. Ad-*

- vanced Structured Materials* (Eds. A. D. Pogrebnjak, Y. Bing, and M. Sahul) (Singapore: Springer: 2024), p. 1.
5. O. A. Goncharov, I. S. Kolinko, G. V. Kornich, O. V. Khomenko, and D. V. Shyrokorad, *Powder Metallurgy and Metal Ceramics*, **62**: 312 (2023).
 6. P. H. Mayrhofer, C. Mitterer, J. G. Wen, J. E. Greene, and I. Petrov, *Appl. Phys. Lett.*, **86**: 131909 (2005).
 7. C. Fuger, R. Hahn, A. Hirle, T. Wojcik, P. Kutrowatz, F. Bohrn, O. Hunold, P. Polcik, and H. Riedl, *Mater. Research Lett.*, **11**: 613 (2023).
 8. M. Mikula, B. Grančič, T. Roch, T. Plecenik, I. Vávra, E. Dobročka, A. Šatka, V. Buršiková, M. Držik, M. Zahoran, A. Plecenik, and P. Kúš, *Vacuum*, **85**: 866 (2011).
 9. V. Šroba, T. Fiantok, M. Truchlý, T. Roch, M. Zahoran, B. Grančič, P. J. Švec, Š. Nagy, V. Izai, P. Kúš, and M. Mikula, *J. Vacuum Sci. Technol. A*, **38**: 033408 (2020).
 10. K. Viskupová, B. Grančič, T. Roch, L. Satrapinskyy, M. Truchlý, M. Mikula, V. Šroba, P. Ďurina, and P. Kúš, *Surf. Coat. Technol.*, **421**: 127463 (2021).
 11. B. Grančič, M. Pleva, M. Mikula, M. Čaplovičová, L. Satrapinskyy, T. Roch, M. Truchlý, M. Sahul, M. Gregor, P. Švec, M. Zahoran, and P. Kúš, *Surf. Coat. Technol.*, **367**: 341 (2019).
 12. A. A. Goncharov, V. A. Konovalov, G. K. Volkova, and V. A. Stupak, *Phys. Met. Metallogr.*, **108**: 368 (2009).
 13. F. Kunc, J. Musil, P. H. Mayrhofer, and C. Mitterer, *Surf. Coat. Technol.*, **174–175**: 744 (2003).
 14. P. H. Mayrhofer and M. Stoiber, *Surf. Coat. Technol.*, **201**: 6148 (2007).
 15. A. A. Goncharov, S. N. Dub, A. V. Agulov, and V. V. Petukhov, *J. Superhard Mater.*, **37**: 422 (2015).
 16. A. A. Goncharov, A. N. Yunda, H. Komsta, and P. Rogalski, *Acta Phys. Pol. A*, **132**: 270 (2017).
 17. A. A. Goncharov, S. N. Dub, and A. V. Agulov, *Phys. Met. Metallogr.*, **114**: 95 (2013).
 18. A. A. Goncharov, V. V. Petukhov, D. N. Terpii, P. I. Ignatenko, and V. A. Stupak, *Inorganic Mater.*, **41**: 696 (2005).
 19. A. A. Goncharov, V. V. Petukhov, and D. S. Chaika, *Phys. Met. Metallogr.*, **100**: 222 (2005).
 20. S. Jayaraman, J. E. Gerbi, Y. Yang, D. Y. Kim, A. Chatterjee, P. Bellon, G. S. Girolami, J. P. Chevalier, and J. R. Abelson, *Surf. Coat. Technol.*, **200**: 6629 (2006).
 21. S. K. Shanmugham, A. le Febvrier, J. Palisaitis, P. O. E. Persson, R. J. W. Frost, D. Primetzhofer, I. Petrov, H. Högberg, J. Birch, J. Rosen, P. Eklund, and S. Nayak, *J. Applied Phys.*, **136**: 115304 (2024).
 22. A. Goncharov, A. Yunda, I. Kolinko, and O. Maksakova, *High Temperature Material Processes*, **27**: 31 (2023).
 23. A. A. Goncharov, A. N. Yunda, V. V. Buranich, I. V. Shelest, and V. B. Loboda, *J. Nano- Electronic Phys.*, **10**: 03002 (2018).
 24. W. C. Oliver and G. M. Pharr, *J. Mater. Research*, **7**: 1564 (1992).
 25. B. Window and N. Savvides, *J. Vacuum Sci. Technol. A*, **4**: 196 (1986).
 26. A. A. Goncharov, A. V. Agulov, V. A. Stupak, and V. V. Petukhov, *Inorganic Mater.*, **47**: 592 (2011).
 27. A. A. Goncharov, A. N. Yunda, A. D. Pogrebnjak, I. V. Shelest, V. V. Buranich,

- and V. B. Loboda, *2018 IEEE 8th Int. Conf. Nanomaterials: Application & Properties (NAP) (Dec. 28–30, 2018, Zatoka, Ukraine)*, p. 1.
28. A. A. Goncharov, A. V. Zykov, A. N. Yunda, I. V. Shelest, and V. V. Buranich, *Metallofiz. Noveishie Tekhnol.*, **42**, No. 6: 815 (2020).
 29. V. Buranich, A. Pogrebnyak, P. Budzynski, I. Shelest, A. Pruszyński, D. Chocyk, A. Goncharov, and A. Yunda, *SN Applied Sci.*, **2**: 563 (2020).
 30. T. F. Zhang, B. Gan, S.-M. Park, Q. M. Wang, and K. H. Kim, *Surf. Coat. Technol.*, **253**: 115 (2014).
 31. A. A. Goncharov, V. A. Konovalov, S. N. Dub, V. A. Stupak, and V. V. Petukhov, *Phys. Met. Metallogr.*, **107**: 285 (2009).
 32. F. Lofaj, T. Moskaiewicz, G. Cempura, M. Mikula, J. Dusza, and A. Czyrska-Filemonowicz, *J. Eur. Ceram. Soc.*, **33**: 2347 (2013).
 33. K. P. Budna, P. H. Mayrhofer, I. Neidhardt, É. Hegedűs, I. Kovács, L. Tóth, B. Pécz, and C. Mitterer, *Surf. Coat. Technol.*, **202**: 3088 (2008).
 34. M. Berger, L. Karlsson, M. Larsson, and S. Hogmark, *Thin Solid Films*, **401**: 179 (2001).
 35. M. Berger, E. Coronel, and E. Olsson, *Surf. Coat. Technol.*, **185**: 240 (2004).

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Application of the Process of Self-Propagating High-Temperature Synthesis for the Fabrication of Modifying Composite Ceramic–Metal Powders

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The processes based on self-propagating high-temperature synthesis (SHS) for the fabrication of two types of modifying composite materials (MCM) of a charge containing boron: Ti, B, C, Fe₂O₃, Al, PT-NA-01, and a charge, in which silicon (SiO₂) and aluminium (Al₂O₃) oxides in the form of refractory clay of the PGOSA-0 brand are included instead of expensive boron (B). The thermodynamic evaluation of the possibility of interplay between the components is carried out using the Gibbs energy. The phase compositions of the deposited coatings with composite materials (CM) based on PG-10N-01 alloy and their microhardness are presented. The results of studies of the wear resistance of CM during the friction against fixed abrasive particles show 1.5–2.4 times higher wear resistance compared to the PG-10N-01 alloy. As established, the deposited CM modified by the MCM, which contains silicon (SiO₂) and aluminium (Al₂O₃) oxides instead of boron (B), has higher wear resistance, when it works in an abrasive environment. This is due to the content of chromium, titanium, silicon, and boron carbides in its structure. The morphology of the friction surfaces is also in a good agreement with the results of friction tests upon fixed particles of the deposited coatings PG-10N-01 and CM.

Key words: composite material, boride, SHS process, surfacing, wear resistance.

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Досліджено процеси саморозповсюджуваної високотемпературної синтези для одержання двох типів модифікувальних композиційних матеріалів (МКМ) з шихти, що містить бор: Ti, B, C, Fe₂O₃, Al, ПТ-НА-01, і шихти, в якій замість дорогого бору (B) включено оксиди Силіцію (SiO₂) й Алюмінію (Al₂O₃) у вигляді вогнетривкої глини марки ПГОСА-0. Термодинамічну оцінку можливості перебігу взаємочину між компонентами проводили з використанням Гіббсової енергії. Наведено фазові склади нагрітих покриттів композиційними матеріалами (КМ) на основі стопу ПГ-10Н-01 та їхню мікротвердість. Результати досліджень зносостійкості КМ під час тертя об закріплені абразивні частинки показали у 1,5–2,4 рази більшу зносостійкість порівняно зі стопом ПГ-10Н-01. Встановлено, що нагрітий КМ, модифікований МКМ, який містить оксиди Силіцію (SiO₂) й Алюмінію (Al₂O₃) замість бору (B), має вищу зносостійкість під час роботи в умовах абразивного середовища. Це зумовлено вмістом у його структурі карбідів Хрому, Титану, Силіцію та Бору. Морфологія поверхонь тертя також добре узгоджується з результатами випробувань на тертя об закріплені частки нагрітих покриттів ПГ-10Н-01 і КМ.

Ключові слова: композиційний матеріал, борид, саморозповсюджувана високотемпературна синтеза, нагрівання, зносостійкість.

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1. INTRODUCTION

To strengthen and restore machine parts, various methods of surfacing have become the most widespread, including arc surfacing with consumable and non-consumable electrodes, as it has a number of advantages: low cost of the equipment used, insignificant penetration of the surfacing surface, which ensures stable properties of the surfacing layer throughout its thickness.

Currently, there are many materials available for surfacing machine parts represented by flux-cored and composite wires, coated electrodes, and surfacing tapes, but the amount of research into the development of materials, which work under abrasive wear conditions, is relatively small.

The resistance of materials to abrasive wear depends significantly on their structural and phase composition, which is determined not only by their chemical composition, but also depends on the surfacing method and mode, weld pool crystallization conditions, and heat treatment modes of the welded products.

The basis of the structure of wear-resistant alloys is the matrix, in which there are strengthening phases (carbides, nitrides, borides, etc.). The alloy matrix performs two functions: it serves as a structural element, on which wear resistance depends, and it contributes to the reliable retention of the hardening phases to prevent their crumbling under the influence of abrasives and impacts. The type of matrix, its con-

dition and properties determine the likelihood of crack initiation, growth and consolidation in the metal under the influence of various factors and the environment, which affects the wear rate of the alloy.

To achieve high wear resistance of parts operating under abrasive wear conditions, there are two options for creating wear-resistant alloys. The first is to create materials with a metastable structure. Such materials are capable of absorbing energy introduced from the outside and dissipating it through structural and phase transformations, primarily through the $\gamma \rightarrow \alpha$ transformation. However, such alloys have little resistance to abrasive wear at temperatures up to 550°C, which is associated with thermal weakening of the alloy matrix [1]. The second option is to develop materials that have a thermally stable structure reinforced with a large amount of solid phase [2]. Materials with such a structure are the most promising for resisting abrasive wear at elevated temperatures.

In addition, the wear resistance of surfacing materials depends significantly on the type and amount of the carbide phase in the alloys. Most often, the hardening phase should contain the following carbides: Fe_3C , Mn_3C , Cr_7C_3 , W_2C , WC , VC , TiC , B_4C , Mo_2C , etc., as well as carbides, iron nitrides and alloying elements. At present, metal, non-metallic and composite materials, monolithic and porous, with both homogeneous and heterogeneous structures, are used in machine friction units.

To obtain a modifying composite material in the form of a sinter, it is possible to use traditional methods of powder metallurgy, which include the stages of preparing a charge of powder components, pressing, and sintering [3]. The main disadvantages of powder metallurgy methods include the use of expensive large-size equipment, the duration and energy intensity of the production cycle.

A promising method for obtaining new materials, including composite materials, is self-propagating high-temperature synthesis (SHS), which is based on the local initiation of highly exothermic reactions between the starting reagents, which makes it possible to generate a significant amount of heat to propagate the front of physical and chemical transformations with the formation of synthesis products [4–10].

The advantages of SHS technologies over conventional metallurgical production technologies are minimal material losses during the synthesis process, simplification and reduction of the number of technological operations, and energy savings. SHS technologies are based on three main operations: preparation of the initial charge, the SHS reaction itself, and processing and analysis of the resulting product. This method belongs to the *in situ* methods of obtaining composite materials and has all the advantages of such methods [11, 12].

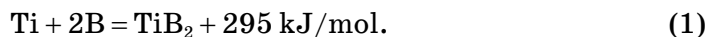
The aim of this work is to study the possibility of obtaining a com-

posite modifying material, using the SHS process to increase the abrasive wear resistance of deposited coatings with an alloy of the Ni–Cr–B–Si system, using oxides instead of expensive boron in the charge.

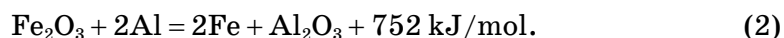
2. MATERIAL AND RESEARCH METHODS

At the beginning of the study of the possibility of obtaining a composite modifying material (MCM) by the SHS method to increase the abrasive wear resistance of deposited coatings with an alloy of the Ni–Cr–B–Si system, the following powder materials were selected: Ti, C, B, Al, Fe_2O_3 , PT–NA–01. Titanium reacting with carbon forms titanium carbide (TiC) and generates a large amount of heat (310 kJ) [13]. As a result of the heat released, the temperature of solid and molten reaction products reaches 2500–3500 K, so, the reaction does not depend on external heating sources and can propagate as a combustion wave that generates energy for its propagation. Despite the high temperature, the transition of any of the components of the mixture to the gas phase is insignificant and can be neglected.

Boron is one of the most effective and economical microalloying elements in steel. In most cases, the minimum concentration of boron in the metal to obtain a positive result is about one thousandth of a percent. The uniqueness of boron lies in the fact that, with such a low content in steel, it is able to exert an effect on its properties equivalent to that of a much larger number of alloying elements such as Cr, Mo, Ni, *etc.* Therefore, to obtain a composite material, we synthesize titanium diboride:



In addition, to increase the thermal effect in the synthesis of titanium carbide and titanium diboride, thermosetting nickel aluminide powder PT–NA–01, aluminium powder (powder) PAP-1 of GOST 5494-95, and iron oxide Fe_2O_3 were introduced into the mechanical mixture. As a result of the thermite reaction, molten iron and aluminium oxide are formed, a large amount of energy is released, and the temperature reaches approximately 2500°C [2]:



The selected components were mixed, and the resulting powder mixture was subjected to mechanical activation in a ball mill. In the experiments, the volume of the mill drum was of 160 cm³, the diameter of the balls was of 4–5 mm, and the weight of the balls was of 200 g. The time of the mechanical activation process varied from 2 to 6 minutes.

The CM was prepared in two stages: 1) mixing of Ti, B, C, Fe_2O_3 , Al, PT-NA-01 powders and their joint mechanical activation, addition of a binder—Metylan glue, cylinder formation and drying, initiation of the SHS process, during which titanium boride particles, chromium, *etc.* are synthesized; 2) crushing the material obtained during sintering and mixing the resulting powder with the matrix material—powder grade PG-10N-01 TUU 322-19-004-96, mechanical activation of the resulting powder mixture, adding liquid glass of GOST 13078 to the mechanically activated powder mixture until it becomes a paste [2].

The prepared paste was applied to the surface of the steel plate (steel 20) prepared for surfacing, and, after drying, surfacing was performed with a graphite electrode with a diameter of 10 mm, surfacing current of 80–120 A, and direct polarity. Arc surfacing was performed, using an inverter power source Paton VDI-200R DC TIG.

In addition, the composition of the initial charge of the second composite-modifying material was studied, which differed from the first in that instead of expensive boron (B); silicon oxides SiO_2 and aluminium oxides Al_2O_3 were included in the charge in the form of refractory clay of the PGOSA-0 brand.

The microstructure was studied using a metallographic microscope. The microhardness of the coatings was measured by the PMT-3 device in accordance with DST 9450.

Comparative wear tests of the deposited samples were performed on a friction machine of the MI type according to the ‘disk–pad’ scheme in industrial oil of the I-20 grade under the following conditions: average circumferential sliding speed of 0.42 m/s, specific pressure on the pad under normal wear of 8.0 MPa, friction surface area of 1.8 cm². The wear rate was determined by the formula

$$U_w = \frac{G_1 - G_2}{\gamma Ft} [\text{mm}^3/(\text{cm}^2 \cdot \text{s})], \quad (3)$$

where G_1 , G_2 are the weights of the sample before and after the test [mg], γ is the density of the material [mg/mm³], F is the sample area [cm²], t is the test time [s].

The materials were tested for abrasive wear under friction against fixed abrasive particles in accordance with GOST 17367. The abrasive particles used were green silicon carbide, grade 64C, with a grain size of 8 H. The relative wear resistance of the materials was estimated by the formula

$$\varepsilon = \frac{\Delta l_e}{\Delta l_t} (d_e/d_t)^2, \quad (4)$$

where Δl_e , Δl_t are the absolute linear wear of the reference and test

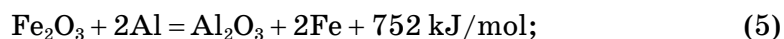
specimens [mm], d_e , d_t are their actual diameters [mm], respectively.

The phase composition of the deposited layers was determined using a DRON-3 diffractometer in monochromatic CuK_α radiation in the angular interval $2\theta = 10\text{--}80^\circ$. X-ray diffraction patterns were recorded in a discrete mode. The samples were prepared for analysis by grinding with abrasive paper and polishing to achieve the required surface roughness. The data from the ASTM file were used to decipher the diffractograms.

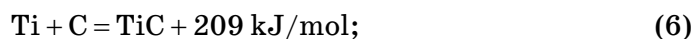
3. RESULTS AND DISCUSSION

The study of the process of producing CMC from a charge consisting of powders of Ti, B, C, Fe_2O_3 , Al, PT-NA-01, involved the use of reactions of iron reduction by aluminium from oxide, carbonization, and titanium-boride formation. In other words, this composition of powdered materials was intended to carry out three combined reactions of the SHS process:

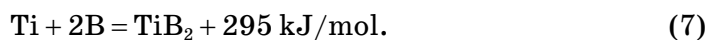
– thermite reaction



– carbonization reaction



– boron-formation reaction



These reactions, which have large thermal effects, can proceed in the combustion mode independently of each other. The relative content of components corresponded to stoichiometric Eqs. (5)–(7). The combustion of a cylindrical sample formed from a mixture of powders with the addition of a binding adhesive of the ‘Methylan’ brand was carried out in air in an open reactor. The initiation of the SHS process was carried out after drying the sample for 72 hours from a heated nichrome spiral. The combustion front of the powder mixture moved uniformly along the length of the cylindrical sample from the place of its initiation without emission of powder components and reaction products. As a result of the SHS process, a sinter was obtained (Fig. 1), which was easily crushed.

Further, the obtained MCM in the amount of 10 and 20% was added to the filler powder of PG-10N-01 grade and, according to the above method, a paste was obtained, which was applied to the surface of the steel plate (steel 20) prepared for surfacing, and after drying, surfac-



Fig. 1. A speck.

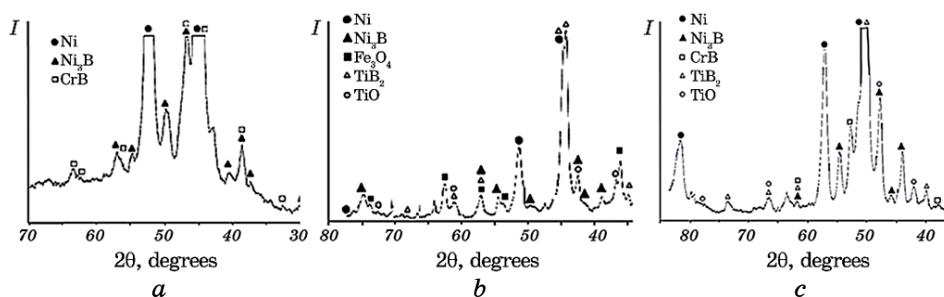


Fig. 2. X-ray diffractogram of deposited coatings: PG-10N-01 (a); CM (10% MCM + 90% PG-10N-01) (b); CM (20% MCM + 80% PG-10N-01) (c).

ing was performed with a graphite electrode with a diameter of 10 mm.

In the previous work [2], the following phase composition was established: titanium diboride (TiB_2), nickel boride (Ni_3B), titanium oxides (TiO) and iron oxides (Fe_3O_4) for the CM containing 10% CMC, and titanium diboride (TiB_2), chromium boride (CrB), nickel boride (Ni_3B) and titanium oxide (TiO) for the CM (20% CMC + 80% PG-10H-01). Aluminium oxide (Al_2O_3) and titanium carbide (TiC), which corresponds to Eqs. (5) and (6), were not detected (Fig. 2).

The absence of aluminium oxide is explained by a higher temperature, which reaches 2500°C as a result of a thermite reaction, than, the melting point of aluminium oxide of 2050°C , that leads to its melting and removal from the deposited CM in the form of a slag phase. Titanium carbide has a higher melting point of $3260 + 150^\circ\text{C}$ compared to the temperature of solid and molten products of the titanium carbide formation reaction, which reaches 2500–3500 K.

However, x-ray phase analysis did not reveal titanium carbide in the deposited layer, which can be explained by its low percentage in the initial CM powder mixture and the preparation of the sample surface for the study. The thermodynamic assessment of the possibility of interaction between the components was carried out using the Gibbs energy, since any reaction or process is accompanied by a change in the Gibbs energy (ΔG) of the system, the value of which is a measure of the ability

of the reaction to develop in the conditions under consideration: the greater the decrease in the Gibbs energy, the greater the affinity between the reacting substances.

The Gibbs energy is calculated according to the expression

$$\Delta G = \Delta H - T\Delta S \text{ [kJ/mol]}, \quad (8)$$

where ΔH is the enthalpy, T is the temperature, ΔS is the entropy.

$$\Delta H_{\text{reaction}} = \sum \Delta H_{\text{products}}^0 - \sum \Delta H_{\text{reactants}}^0 \text{ [kJ/mol]}. \quad (9)$$

Using the reference data of the standard enthalpies of substances, given that the enthalpies of formation of simple substances are zero, we find:

$$\Delta H_{\text{reaction}} = \Delta H_{\text{TiC}}^0 - \Delta H_{\text{Ti}}^0 - \Delta H_{\text{C}}^0 = -209 - 0 - 0 = -209 \text{ kJ/mol}. \quad (10)$$

$$\Delta S_{\text{reaction}} = \sum \Delta S_{\text{products}}^0 - \sum \Delta S_{\text{reactants}}^0 \text{ [J/(mol·K)]}. \quad (11)$$

Using the reference data of the standard entropies of substances, we find:

$$\Delta S_{\text{reaction}} = \Delta S_{\text{TiC}}^0 - \Delta S_{\text{Ti}}^0 - \Delta S_{\text{C}}^0 = 24.7 - 30.63 - 5.74 = -11.67 \text{ J/(mol·K)}. \quad (12)$$

$$\Delta G = \Delta H - T\Delta S = -209 + 298 \cdot (11.67/1000) = -205.52 \text{ kJ/mol}. \quad (13)$$

At $T = 298^\circ\text{C}$, $\Delta G < 0$, and the reaction is spontaneous, *i.e.*, the reaction will proceed in the forward direction. Thus, the thermodynamic calculation of the change in the Gibbs energy confirms the possibility of the formation of titanium carbide TiC.

The average microhardness of the CM coating (10% MCM + 90% PG-10N-01) is of 740 *HV*, and, for the CM coating (20% MCM + 80% PG-10N-01), it is of 978 *HV* that exceeds the average microhardness of the PG-10N-01 coating, which is of 520 *HV*. The microhardness distribution of the CM containing 20% CMC is more stable compared to the coating containing 10% CMC. This can be explained by a more uniform distribution of solid particles in the matrix material PG-10N-01 with an increase in their concentration [2].

The second composite modifying material was investigated, which was obtained from a charge consisting of powders of Ti, C, Fe_2O_3 , Al, PT-NA-01, silicon oxide SiO_2 and aluminium oxide Al_2O_3 in the form of refractory clay of the PGOSA-0 brand. This composition, unlike the previous one, instead of expensive boron (B), includes aluminium and silicon oxides, which are solid refractory substances ($\alpha\text{-Al}_2\text{O}_3$ with 20.6–29.4 *HV*, 2050°C, and SiO_2 with 8 *HV*, 1710°C) with high wear

and corrosion resistance. Such a composition of powder materials provided for the implementation of two combined reactions of the SHS process: a thermite reaction (5) and a carbonization reaction (6).

As a result, the following phase composition was determined by XRD analysis: γ -Ni, nickel boride (Ni_3B), titanium carbides (TiC), boron (B_4C), chromium (Cr_3C_2), and silicon (SiC) for the CM containing 10% of MCM [5]. Figure 3 shows the microstructure of the deposited CM coating (10% MCM–90% PG-10N-01).

The obtained results confirm the possibility of the formation of titanium carbide TiC during the SHS process. In addition, the deposited coating contains carbides of boron (B_4C), chromium (Cr_3C_2), and silicon (SiC) formed during the arc surfacing of CM from the matrix material of the self-fluxing alloy PG-10N-01 TUU 322-19-004-96 (chemical composition: Cr—14–20%, B—2.8–4.2%, Si—4.0–4.5%, Fe—3–7%, C—0.6–1.0%, Ni—base).

It should also be noted that the deposited coating does not contain aluminium and silicon oxides, which are part of the original composition of the charge. This can be explained by the self-fluxing effect of the PG-10N-01 matrix material, since the boron and silicon included in its composition form fusible eutectics with nickel with a melting point of 950–1080°C during heating, and also restore oxide films on the surface of the sample to form borosilicate slags that float to the surface of the deposited layer. Taking into account the high melting point of aluminium oxide (2054°C) and silicon oxide (1710°C), as well as their low density of 3.99 and 2.65 g/cm³, respectively, it can be concluded that most of them float to the surface in the form of a slag phase.

The average microhardness of the deposited CM layer (10% MCM + 90% PG-10N-01) is of 660 HV, and that of the CM layer (20%

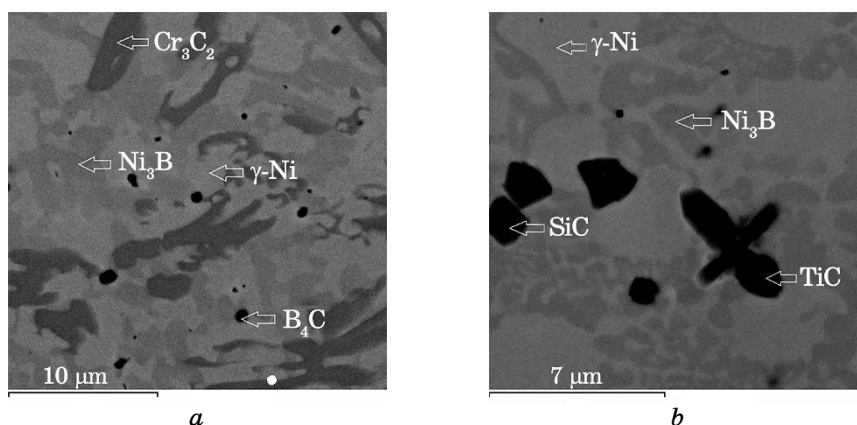


Fig. 3. Microstructure and phase composition (wt.%) of deposited coatings: PG-10N-01 (*a*); 10% MCM + 90% PG-10N-01 (*b*).

MCM + 80% PG-10N-01) is of 720 *HV*, which also exceeds the average microhardness of the PG-10N-01 coating, which is of 510 *HV*.

The combustion of the CMC compositions of the first and second types occurred evenly, with no emissions of the initial components and reaction products, and the addition of CMC up to 10% improves the technological properties of the arc surfacing process and contributes to a better formation of the deposited layer [14].

According to the results of tests for wear resistance of materials deposited by the arc method (Fig. 4), the abrasive wear resistance of CM (10% MCM + 90% PG-10N-01) of the first type is 1.5 times higher than that of CM (10% MCM + 90% PG-10N-01) of the second type, and 1.8 times higher than that of the self-fluxing alloy PG-10N-01.

With an increase in the content of CMC in the CM, the wear resistance of the deposited coatings during operation in an abrasive environment increase. Thus, at a content of 20% MCM, the wear resistance of the deposited layer of the first type of the deposited CM increases by 1.7 times, and the second type by 2.4 times.

The morphology of the friction surfaces is also in a good agreement with the results of friction tests on the fixed abrasive particles of the deposited coatings PG-10N-01 and CM (Fig. 5).

During wear under the influence of fixed abrasive particles, quite deep parallel grooves were found on the coating made of PG-10N-01 alloy (Fig. 5, *a*). The relief of the wear surface is uneven. Shallow, small, and evenly spaced friction scratches are observed on the surfaces of the deposited coatings made of CM (Fig. 5, *b* and *c*). This is explained by the introduction of CMCs, which contribute to the forma-

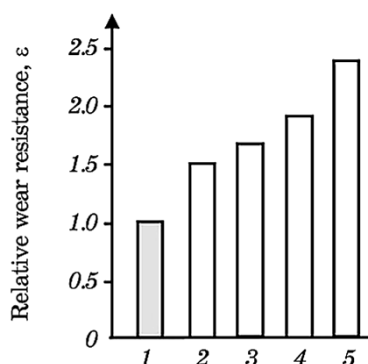


Fig. 4. Wear resistance of materials deposited by the arc method in the process of friction under the influence of fixed abrasive particles: 1—PG-10N-01 alloy, 2—CM (10% CMC + 90% PG-10N-01) of the first type, 3—CM (20% CMC + 80% PG-10N-01) of the first type, 4—CM (10% CMC + 90% PG-10N-01) of the second type, 5—CM (20% CMC + 80% PG-10N-01) of the second type.

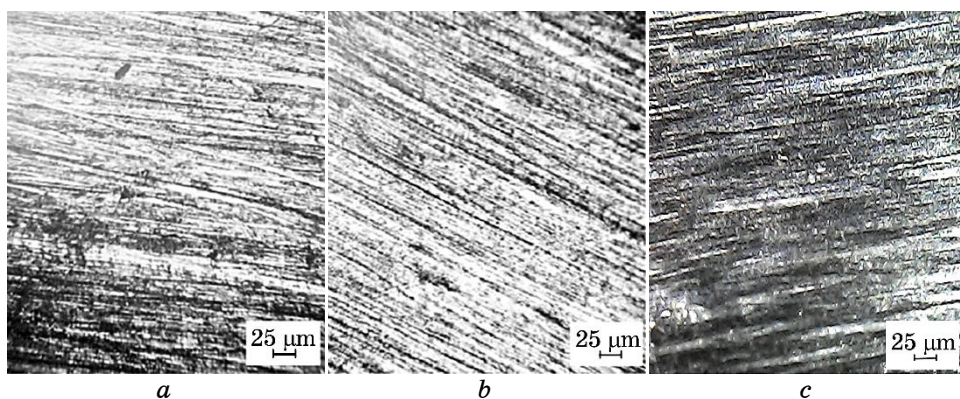


Fig. 5. Morphology of friction surfaces during wear against fixed abrasive particles of deposited coatings: PG-10N-01 (*a*), CM (10% MCM + 90% PG-10N-01) of the first type (*b*), CM (10% MCM + 90% PG-10N-01) of the second type (*c*).

tion of new crystallization centres during surfacing and, as a result, leads to the grinding of the structure. The plastic matrix based on nickel PG-10N-01 redistributes stresses, providing conditions in which solid components occupy a favourable position. This eliminates the local increase in contact pressure and reduces the conditions for the detachment of hardwearing particles.

4. CONCLUSIONS

The results of studies of the wear resistance of CM consisting of the matrix material of the nickel-based alloy PG-10N-01, modified CMC of the first and second types, which differ in that the second type, instead of the boron component, introduced silicon oxides SiO_2 and aluminium oxides Al_2O_3 in the form of refractory clay, showed increased wear resistance during operation in an abrasive environment compared to the coating with PG-10N-01 alloy. CM of the first type contains solid inclusions in the structure in the form of titanium diboride (TiB_2), nickel borides (Ni_3B) and chromium (CrB), titanium oxides (TiO) and iron (Fe_3O_4), which provides an average microhardness of 740–978 *HV* and increased wear resistance in an abrasive environment. The wear resistance of the deposited layer of the first type of CM (10% MCM + 90% PG-10N-01) at friction against fixed abrasive particles is 1.5 times higher, and that of the first type of CM (20% MCM + 80% PG-10N-01) is 1.7 times higher compared to the PG-10N-01 alloy.

The second type of CMC contains Cr_3C_2 , B_4C , TiC , SiC , and nickel boride (Ni_3B) carbides in its structure. The average microhardness of

the deposited layer of 10% MCM + 90% PG-10N-01 is of 660 HV, and that of 20% MCM + 80% PG-10N-01 is of 720 HV. The wear resistance of the deposited layer of 10% MCM + 90% PG-10N-01 composition is 1.8 times higher, and that of 20% MCM + 80% PG-10N-01 composition is 2.4 times higher in comparison with the layer deposited with self-fluxing alloy PG-10N-01.

The second type of welded cermets has higher wear resistance, when operating in abrasive environments. This is due to the content of chromium, titanium, silicon, and boron carbides in its structure.

Thus, the research results indicate the expediency of using oxides in the charge instead of expensive boron, when using the SHS process for producing CMC.

REFERENCES

1. M. Yu. Osipov, *Innovative Materials and Technologies in Metallurgy and Mechanical Engineering*, **1**: 52 (2014).
2. S. O. Luzan, V. A. Bantkovskiy, and A. S. Luzan, *Metallofiz. Noveishie Tekhnol.*, **44**, No. 4: 531 (2022).
3. R. M. German, *Powder Metallurgy and Particulate Materials Processing* (Princeton, USA: Metal Powder Industries Federation: 2005).
4. A. Makino, *Prog. Energy Combust. Sci.*, **27**: 1 (2001).
5. S. O. Luzan and P. A. Sytnykov, *Herald of Khmelnytskyi National University. Technical Sciences*, **323**, No. 4: 194 (2023).
6. S. O. Luzan and P. A. Sytnykov, *Funct. Mater.*, **30**, No. 4: 526 (2023).
7. V. I. Savuliak, *Syntez Znosostykykh Kompozytsiynykh Materialiv ta Poverkhnevyykh Shariiv z Ehkzotermichnykh Komponentiv* [Synthesis of Wear-resistant Composite Materials and Surface Layers of Exothermic Components] (Vinnitsia: UNIVERSUM-Vinnitsia: 2002) (in Ukrainian).
8. S. O. Luzan and P. A. Sytnykov, *Visnyk Kremenchutskoho Natsionalnoho Universytetu im. Mykhaila Ostrohradskoho*, **4**: 88 (2022) (in Ukrainian).
9. B. Trembach, Y. Silchenko, O. Balenko, D. Hlachev, K. Kulahin, H. Heiko, O. Bellorin-Herrera, S. Khabosha, O. Zakovorotnyi, and I. Trembach, *Int. J. Adv. Manuf. Technol.*, **134**: 309 (2024).
10. B. O. Trembach, Yu. A. Silchenko, M. G. Sukov, N. B. Ratska, Z. A. Duriagina, I. V. Krasnoshapka, O. V. Kabatskyi, and O. M. Rebrova, *Mater. Sci.*, **59**: 733 (2024).
11. A. Chrysanthou, *Adv. in Sci. and Techn.*, **63**: 273 (2010).
12. E. G. Dyadko, *Samorasprostranyayushchiysya Vysokotemperaturnyi Sintez Kompozitsionnykh Materialov na Osnove Karbida Titana i Oksida Alyuminiya dlya Abrasivnoy i Magnitno-Abrasivnoy Obrabotki* (Self-Propagating High-Temperature Synthesis of Composite Materials Based on Titanium Carbide and Aluminum Oxide for Abrasive and Magnetic-Abrasive Machining) (Ph.D. Thesis Abstract) (Kiev: Frantsevich Institute for Problems in Materials Science: 1988) (in Russian).
13. S. A. Luzan, A. Y. Sydashenko, and A. S. Luzan, *Tekhnichniy Servis Ahropromyslovoho, Lisovoho ta Transportnoho Kompleksiv*, **6**: 152 (2016) (in Ukrainian).

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Detection and Minimizing of Surface Blistering in Continuously Cast and Hot-Rolled Mild Steel Coils

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This study investigates the root courses of surface blistering and explores methods to minimise it in hot-rolled coil steels containing 0.1–0.2 C, 0.4–1.6 Mn, 0.05 Si (wt.%). Surface voids are believed to form during continuous casting, to develop into surface cracks during hot rolling, and to result in product rejection. These defects are found in the central part of the coils on the side corresponding to the small radius of the continuous casting machine (CCM). Metallographic analysis of samples reveals the defect-proliferation

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depth ranging from 100 to 300 μm , decarburisation around the defected zones, and grain coarsening of ferrite to ASTM No. 7 (32–45 μm) from ASTM No. 9–10 (11–16 μm) in the non-defected microstructure. In addition to decarburisation, chemical analysis reveals oxidation and presence of non-metallic inclusions in the defected zones. Oxidation indicates moisture ingress during casting, which is linked to a loss of seal integrity at the CCM, particularly, following a leak between the steel and copper water-cooled plate of the mould. Indentation traces observed on the coils' surface indicate the formation of a so-called 'metallic skull' due to the seal damage. The analysis of slag-forming mixtures reveals the maximum allowable limit of moisture content approaching 0.50%, although the moisture content in the coal used for carburisation of steel is ranging from 1% to 1.2% that is well below the limit of 9%. Based on the obtained results, a set of measures is developed to reduce the defect formation; this set includes: strict control of the moisture content in the mould powders, slag-forming mixtures, and ferroalloys; mandatory testing of mould seal integrity before casting; additional monitoring of tundish drying and the prevention of casting without a protective pipe.

Key words: continuous casting, mild steel, surface blister, microstructure characterisation, moisture control, mould powder.

Це дослідження спрямовано на аналізі причин і шляхів усунення дефектів типу «розкатаний газовий пухир» на гарячекатаних рулонах низьковуглецевої криці марок SAE 1008 і S235, що виявився впродовж шістьох днів виробництва на стані 1700 одного з металургійних підприємств групи «Метінвест». Продукція, на якій було виявлено дефекти, складала 3327 тон, з яких 88,3% припадали на сляби, розлиті на машині безперервного лиття заготовок (МНЛЗ) № 3, і лише 11,7% — на сляби з МНЛЗ-2. Особливо дефекти зосереджувалися в центральній частині рулонів з боку, що відповідає малому радіусу МНЛЗ, а також на рулонах, розлитих із застосуванням кристалізатора № 2 упродовж п'ятих днів. Металографічна аналіза зразків показала, що глибина поширення дефектів сягала 100–300 мкм, а структура металу поблизу дефектних зон характеризувалася повним зневуглецюванням і укрупненням зерна фериту до 6–7 номера за шкалою ASTM (32–45 мкм), тоді як нормальне зерно у непошкоджених зонах мало розмір 9–10 номера (11–16 мкм). На основі хемічного складу, визначеного відповідно до стандарту ISO 4967:2013, було підтверджено наявність окиснених зон у вигляді скупчень неметалевих включень в зоні дефектів. Це вказує на окиснення металу через потрапляння вологи у процесі розливання, що було пов'язане з пошкодженням герметичності кристалізатора № 2 на МНЛЗ-3, зокрема внаслідок виявленої течії між крицевою та мідною водоохолоджуваною плитою кристалізатора. У технологічному журналі також було зафіксовано сліди втискувань, які вказують на утворення так званого «металевого королька» як причину пошкодження герметичності. Аналіза складу шлакоутворювальних сумішей показала, що під час розливання вміст вологи у шлакоутворювальних сумішах становив від 0,20% до 0,50%, що відповідає максимально допустимій межі у 0,50%. Контроль також виявив, що вологість вугілля марки АС, яке використовувалося для навуглецювання криці, варіювалася від 1% до 1,2%, що не перевищує граничні 9%. Проте

об'єднання вологих матеріалів і порушення герметичності створювало умови для окиснення металу. На основі одержаних даних було розроблено комплекс заходів для пониження дефектності прокату, включаючи: суворий контроль вологості шлакоутворювальних сумішей і феростопів, обов'язкове тестування герметичності кристалізаторів перед розливом, додатковий контроль просушування проміжних ковшів і недопущення розливу без захисної труби.

Ключові слова: безперервне лиття, низьковуглецева криця, розкатаний газовий пухир, характеристика мікроструктури, контроль вологості, шлакоутворювальна суміш.

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1. INTRODUCTION

Intense commercial competition stimulates metallurgical companies to further improve the technological processes and implement the quality management system [1]. Surface appearance is one of the key quality parameters for many steel grades, such as structural and automotive; therefore, defect monitoring and prevention become a critical part of the whole production process. Various processing factors throughout the production cycle may influence the formation of surface defects, such as rolled-in scale or oxide layers, with processing routes playing a crucial role in determining both surface quality and mechanical properties [2]. In spite of the measures undertaken, the rolled-steel surface may suffer from a number of defects, such as foreign matter (rolled-in scale), roll marks (patches), scratches (long marks), crazing (short multiple marks), pitted surface, and inclusions. These defects are closely related to a steelmaking technology, continuous casting and hot rolling parameters, and the condition of equipment [3, 4].

Surface defects result from multiple causes such as incorrect set-up of the ladle–tundish–mould system, heat transfer within the mould, cooling water sprays, friction between strand and mould, misalignment of the casting machine, straightening strains, and phase transformations [5]. Effective hydrodynamic optimization of tundish flow, including control of turbulence zones and metal receiver design, enhances inclusion removal efficiency, homogenizes the melt, and reduces the risk of gas-bubble entrapment during solidification [6]. Meanwhile, the hydrodynamic conditions in the mould, particularly, the submerged entry nozzle (SEN) design, are essential for maintaining uniform metal flow and minimizing defects during continuous casting [7]. To minimize the defect formation, several measures have been undertaken:

- improvement of the ladle-shroud designs to optimise the molten-metal flow during casting [8, 9];

- correction of the mould oscillation frequency and heat-flux distribution to the slab geometry and steel grade [10–13];
- selection of the casting speed with respect to the high-temperature mechanical properties of a steel grade to minimise temperature fluctuations [14, 15];
- a thorough descaling to prevent rolling of the scale into the surface [16];
- reduction of the sources for formation of non-metallic inclusions and optimisation of the inclusion removal from the liquid steel into the slag before continuous casting [17, 18];
- argon protection of the liquid steel melt during transfer from the ladle to the tundish and to the mould [19, 20];
- optimisation of the caster geometry (nozzle port angle, size, and shape, mould width and thickness), casting speed, argon gas injection, nozzle submergence depth [21];
- improvement of the roll-surface quality and control of their wear [22];
- application of increased cooling rates during strip coiling [23];
- application of various defect detection methods, like electromagnetic sensors, radiography, magnetic particle inspection, and artificial intelligence [24–27].

During ladle treatment and continuous casting, oxygen, nitrogen, and hydrogen can dissolve in the melt. When the melt solidifies, the solubility of gases decreases and CO, H₂, N₂ containing bubbles may form. The addition of aluminium and calcium facilitates steel deoxidation, reducing the risk of gas-bubble formation, while the introduction of yttrium after aluminium may lead to increased losses due to the oxidation and the formation of complex-oxide inclusions [28]. If the steel is pre-oxidized or contains a high oxygen level, this increases the likelihood of reoxidation during casting, negatively affecting the quality of the cast product. The gas-bubble formation depends on the pressure: under low pressure, the gas formation is easier. The lowest pressure in the liquid pool is at the meniscus. Therefore, gas bubbles can form most easily in the mould near the strand surface. The bubbles may be engulfed by the solidifying shell, so, that pores appear near the surface. Nucleation of gases can take place also in the solid strand due to the solubility decrease with temperature or presence of microporosity and cracks [29]. The phase structure influences the bubble formation. For example, the solubility in ferrite is lower than in austenite due to the more close-packed b.c.c. crystal structure (ferrite) compared to the f.c.c. crystal structure (austenite). In medium carbon steel with ferrite–pearlite microstructure, the pearlite colony boundaries and the prior austenite grain boundaries may act as the void nucleation sites [30]. In case of an increased P concentration, Fe(PO) may form around 980°C; then, it will melt and vaporize under high temperature to result

in blistering of scale. Gas holes and cracks can be found in scales at a temperature of $> 1050^{\circ}\text{C}$ due to formation of CO [31]. Subsurface bubbles may develop into surface cracks, especially, in presence of inter-metallic inclusions [32]. Cracks and gas bubbles may close *via* solid-state diffusion, if their surfaces are not oxidised [33–38]. This requires increased compressive strains and stresses. For example, in work [39], during 50% reduction of a 150 mm thick slab, the 10–25 mm diameter voids closed after the fourth pass for a seven-pass rolling schedule and after the second pass for the five-pass rolling schedule. This means that a larger strain per pass provides a stronger void closer. The extent of void closure was observed, increasing toward the slab centre line from the slab edge. During void closure, the local compressive stresses around voids may reach significant values of > 2500 MPa; this drives the void healing process [40]. Effective closure of internal cavities using the shape of the tool and compression modes can reduce the number of subsurface voids, which are potential precursors to surface defects after hot deformation [41].

The reoxidation of liquid steel, that may prevent the void closure, can occur through contact with gaseous atmosphere, slag with high oxygen potential, ferroalloys, refractory phases during secondary steel refinement, cracks in the refractory walls, and during transfer of liquid steel from a ladle to a tundish and to a mould [42–47]. Oxygen pickup increases precipitation of non-metallic inclusions [48, 49] and scale growth, weakens the austenite grain boundaries, and facilitates crack nucleation during hot rolling and cooling [28, 50–52]. Decreasing the oxygen/gas ratio during slab reheating was shown to reduce oxidation and defect formation [53]. If not prevented on the stages of steel making and hot rolling, defects remain on the cold rolled steel [54, 55].

This paper is concerned with investigation and prevention of defects at one of the leading strip mills in Ukraine, namely, 1700 Hot Rolling Mill (HRM) of the Metinvest Group. ‘Rolled-out gas voids’ observed on SAE 1008 and S235 steel coils were believed to originate from continuous casting. These defects were located in the central part of the coils, primarily, on the side corresponding to the small radius of the continuous casting machine. ‘Rolled-out gas void’ defects can be completely removed by surface grinding; however, this slows down the product despatch. As a result of defect removal, the thickness of the finished product may fall below the minimum permissible tolerances, leading to the rejection of the product as scrap and economic losses. The occurrence of such defects not only affects production efficiency, but also introduces significant financial and operational risks, emphasizing the need for predictive modelling and risk assessment in metallurgical enterprises [56, 57]. Investigations conducted in this paper aimed to identify the causes of the ‘rolled-out gas void’ defects on the

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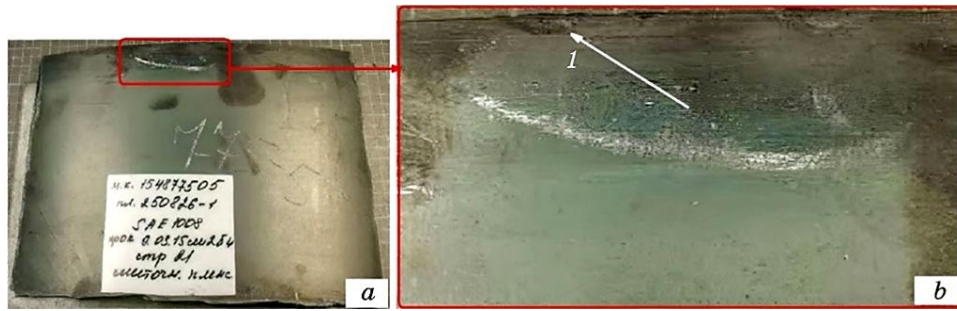


Fig. 1. Photoimage of sample No. 1 (SAE 1008 steel) with a blister (1) defect on the edge (a) and its enlarged view, revealing delamination (b).

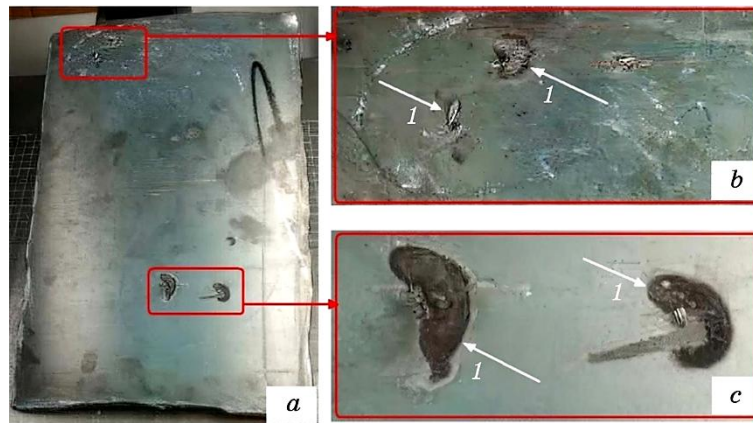


Fig. 2. Photoimage of sample No. 2 (SAE 1008 steel) with blister (1) defects (a), affected area (enlarged) (b), and delamination (enlarged) (c).

ering a 10 mm wide zone (Fig. 1). Directly along the edge, two small 10–15 mm long delamination were found. On sample No. 2 (Fig. 2, a), the entire surface of the rolled product is affected by blisters (Fig. 2, b). Small delamination has also been found extending along the rolling direction up to 20 mm long, accompanied by a dark haze (Fig. 2, c). On sample No. 3, the entire surface is affected by small delamination accompanied by a dark haze (Fig. 3). On this sample, the defects are located on one rolled surface.

Sampling for metallographic investigations was carried out in accordance with the ISO 377:2017 standard ‘Steel and steel products—Location and preparation of samples and test pieces for mechanical testing’. The metallographic study of defect was made in accordance with the ISO 4967:2013 standard ‘Steel—Determination of content of non-metallic inclusions—Micrographic method using standard dia-

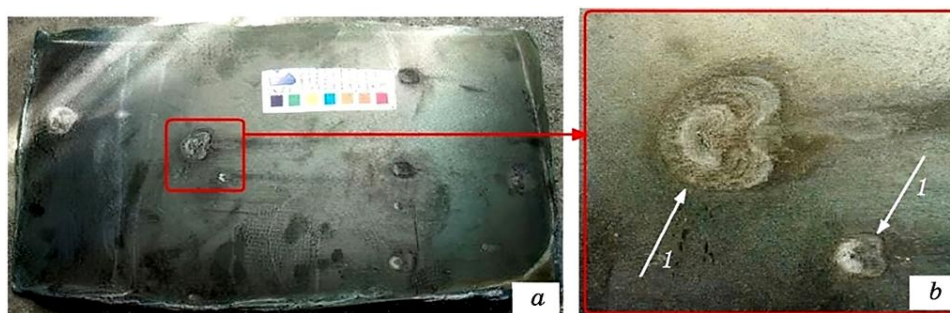


Fig. 3. Photoimage of sample No. 3 (steel S235) with a blister (1) defect (a) and delamination (enlarged) (b).

grams'. The study included determination of the chemical composition of the samples, microstructural analysis of defects, using microsections made in the longitudinal and transverse directions. The microstructure of the rolled products was determined both in the defect areas and for the sound material. The sample etching was carried out using 4% alcohol solution of HNO_3 . The grain size was measured, using optical imaging on a MIM-7 metallographic microscope (optical magnification up to $\times 2000$, bright-field and dark-field observation) following the ASTM E112 'Standard Test Methods for Determining Average Grain Size'.

3. RESULTS AND DISCUSSION

3.1. Structure of the Defects

The macrostructure of a delamination in steel SAE 1008 is shown in Fig. 4, *a*. A coarse cluster of iron oxides was revealed in the delamination cavity. The thickness of delamination was of 80–200 μm . The delaminated metal is diffusively oxidized, completely decarburized; the ferrite grain is enlarged to number 6–7 (32–45 μm). The depth of the decarburized layer at the defect site was measured to be of 100–300 μm (Fig. 4, *b*). The delamination walls with a haze had a thickness of 26–70 μm . The delamination cavity was oxidized, accompanied by oxide accumulations. The exfoliated metal was diffusively oxidized, completely decarburized; the ferrite grain was enlarged to number 6–7 (32–45 μm). The depth of the decarburized layer was of 100–234 μm .

Blisters revealed on samples Nos. 1 and 2 had a structure similar to delamination (Figs. 4 and 5). In line with oxide accumulations, the metal was decarburized in sample No. 1 to a depth of 275 μm , and in sample No. 2—to 296 μm (Fig. 5, *b*). The ferrite-grain size increased to the number 6–7 (32–45 μm). In areas not affected by defects, the de-

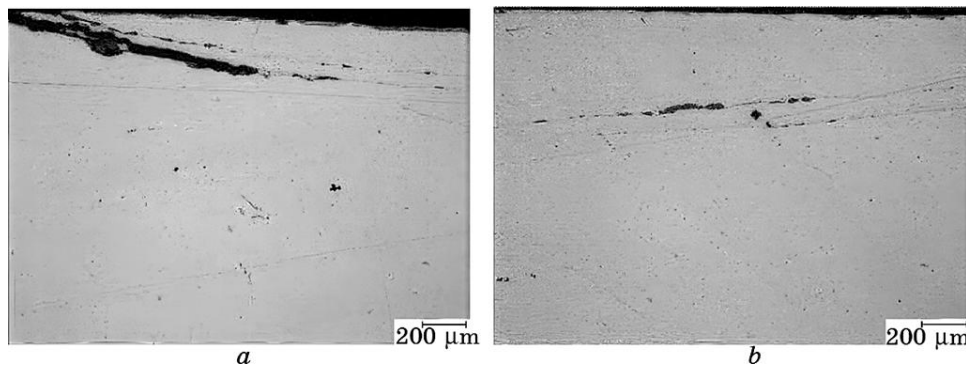


Fig. 4. The macrostructure of the walls of delamination (*a*) and blisters (*b*) in sample No. 1 (magnification $\times 50$).

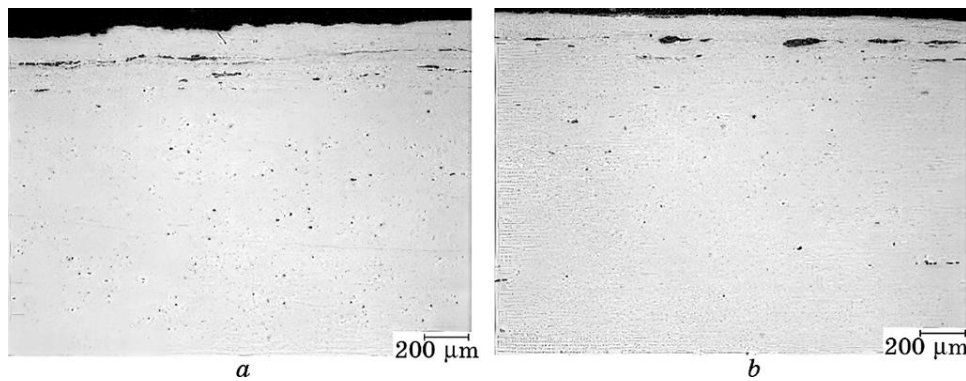


Fig. 5. The macrostructure of the blister walls in sample No. 2 (magnification $\times 50$).

carburized surface of the rolled product was absent on both samples (Figs. 6 and 7). The ferrite-grain number was 9–10 (11–16 μm).

The results of the study indicate that blisters and metal delamination in the rolled products of SAE 1008 steel grade (samples Nos. 1, 2) were associated with defects formed during continuous casting. The absence of decarburization and grain coarsening on the rolled-product surface away from the defects indicates the absence of metal overheating.

The macrostructure of the delamination walls with a haze in steel S235 is shown in Fig. 8. There are no clusters of non-metallic inclusions along the walls of the delamination. Clusters of iron oxides were found in the root of the delamination and along its length. The thickness of the delamination was of 11–50 μm . The delaminated metal was diffusively oxidized, partially decarburized; the ferrite-grain size was

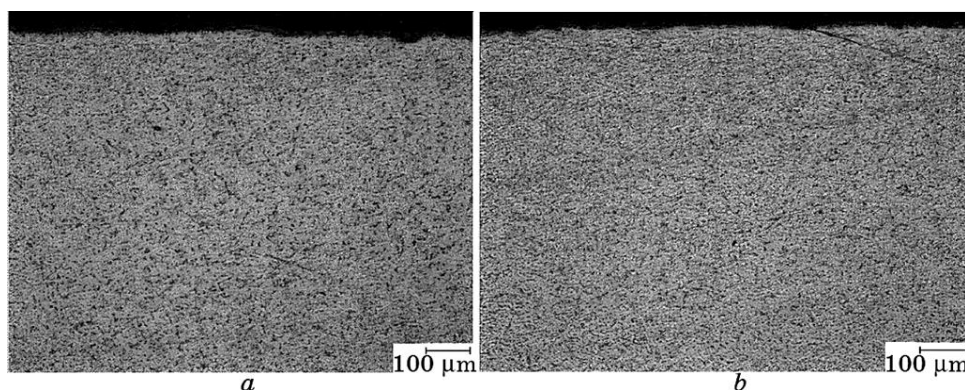


Fig. 6. Microstructure of sample No. 1 through rolled-metal thickness (magnification $\times 100$).

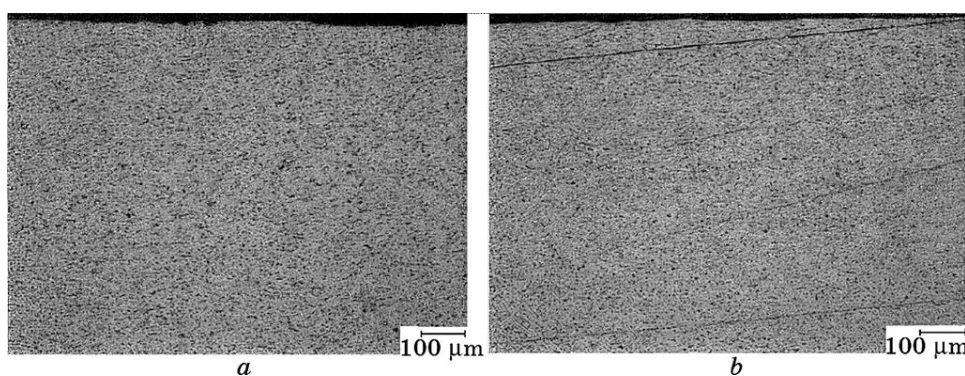


Fig. 7. Microstructure of sample No. 2 through rolled-metal thickness (magnification $\times 100$).

slightly coarsened to the number 8 ($22\text{ }\mu\text{m}$). The depth of the decarburized layer at the defect site was up to $20\text{ }\mu\text{m}$ (Fig. 9). In areas not affected by defects, there is no decarburization of the rolled-product surface. The ferrite-grain size number was of 9–10 ($11\text{--}16\text{ }\mu\text{m}$). The results of the study indicate that defects in the rolled products of steel S235 (sample No. 3) were formed during the rolling of oxidized subcutaneous blowholes in continuously cast slab. As for samples Nos. 1 and 2, the absence of decarburization and grain coarsening on the surface of the rolled products away from the defects indicates the absence of metal overheating.

As a result of metallographic investigation, the following preliminary conclusions can be made: the chemical composition of the samples corresponds to the heat composition and the company regulations for steel grades S235 and SAE 1008; defects on the surface of rolled steels

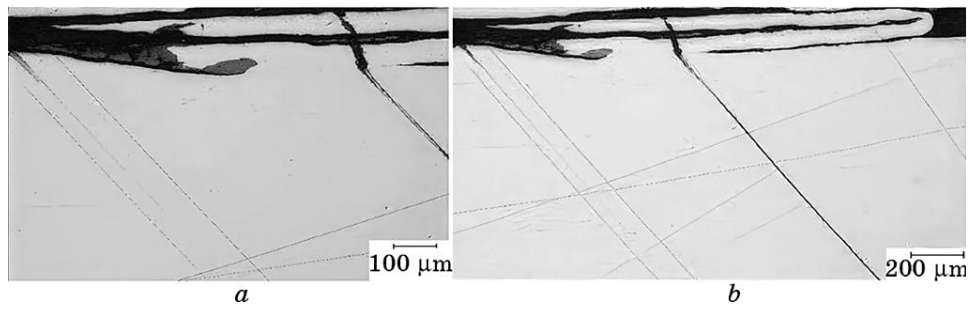


Fig. 8. Microstructure of delamination walls in sample No. 3 (magnification $\times 100$ (a), magnification $\times 200$ (b)).

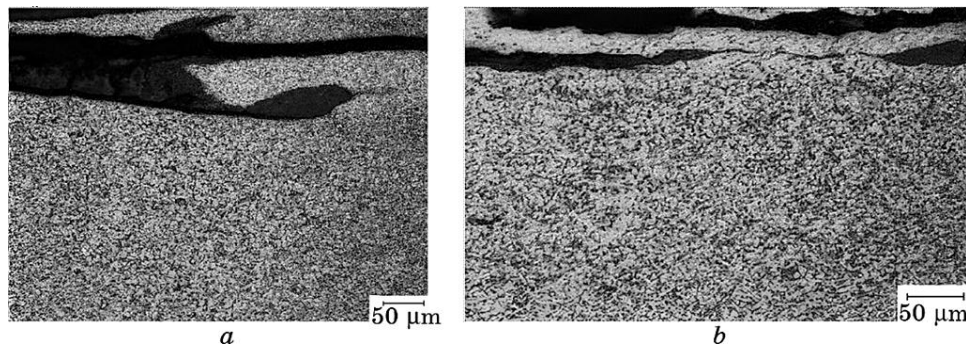


Fig. 9. Microstructure of delamination walls in sample No. 3 (magnification $\times 200$).

S235 and SAE 1008 were formed following opening of oxidized subcutaneous blowholes, which appeared during continuous casting; no signs of metal overheating were detected.

3.2. Root Cause of Blisters

Table 2 summarizes the main known reasons for the formation of subcutaneous blowholes in continuously cast slabs.

The analysis of the process documentation and heats' certificates, performed for those heats, where the blisters were detected, and verification of the processing parameters' correspondence to the required values have shown the following.

(i) Insufficient killing of steel. The average content of total and acid-soluble Al in the steel was of 0.033% and 0.030%, respectively (the standard requirement is at least of 0.02%). The oxygen concentration in the steel before being transported to continuous casting machine (CCM) No. 3 (measured, using an electrochemical oxygen probe, which

TABLE 2. Subcutaneous blowholes, reasons for their formation and methods of prevention.

Key issues	Information on methodology, origins and effects
Detection of the defects	are revealed on sulphur prints (according to Bauman), after etching or peeling, subcutaneous blowholes are clearly visible on the first slabs and usually disappear after the third slab
Key reasons for formation	insufficient steel killing, high gas content in steel (O_2 , N_2 , H_2), wet mould powder metal blow-off by oxygen in the tundish or casting without a ladle shroud,
Additional causes	overheated metal, undried tundish, use of wet ferroalloys
Suggested methods of prevention	improvement of steel killing technology, vacuum treatment of steel, use of dry materials for the tundish lining, use of dry ferroalloys
Impact on the rolled product quality	blowholes and shells can roll out and cause surface cracks during rolling

determines electromotive force proportional to oxygen activity and provides results within 1 minute with accuracy in ppm) is on average of 2.94 ppm, according to the technological instructions of the enterprise, which allow no more than 3 ppm. Thus, no violations of the ‘insufficient metal killing’ criterion were observed.

(ii) Overheated metal. The steel temperature during continuous casting was measured, using an Optris CT Laser pyrometer. This device determines temperature by analysing the infrared radiation emitted by the molten metal. It provides immediate and accurate readings (temperature range: 250–1600°C, accuracy: $\pm 0.75\%$, response time: < 1 second). The measured temperature corresponded to the technological instructions. No ‘hot’ heats were noted. Thus, no violations of the ‘overheated metal’ criterion were observed.

(iii) Wet mould powder. In CCM No. 1 and CCM No. 2, the heats were cast using the mould powder T-4-2(i); in CCM No. 3, the heats were cast using the mould powder ATST-SP627, GS-MC22MA, STSP285-22. The composition of the mould powders is given in Table 3. The actual water content in the mould powder was of 0.20–0.49% (according to the technical instructions, the water content in the mould powder should not exceed 0.5%). Thus, no violations of the ‘wet mould powder’ criterion were observed.

(iv) Metal blow-off by oxygen in the tundish or casting without ladle

shroud. No metal blow-off by oxygen in the tundish was detected; argon consumption in the stopper corresponded to the standardized indicators (the standard for CCM Nos. 1 and 2 is of 0.32–0.51 m³/h, and for CCM No. 3, it is of 0.72 m³/h). During one of the heats, casting a 9 m long slab without a ladle shroud was noted (according to the technical instructions, slabs no longer than 2.5 m can be cast without a ladle shroud). Thus, a local violation of the requirement was detected due to the presence of the reason ‘casting without a ladle shroud’.

(v) Insufficiently dried lining of the tundishes. Drying and heating of tundishes at CCM No. 3 were carried out in accordance with the specified temperature conditions, which were maintained automatically by changing the consumption of natural gas and air. The total drying and preheating time was of 2 hours 45 minutes. Heating to 1100°C took 45 min, after which the temperature was held for 2 hours. During the six-day period under study, six tundishes were dried and heated for CCM No. 3. For all tundishes, the drying and heating time has been increased. Therefore, no inconsistencies were detected for the criterion ‘insufficient drying of lining’.

(vi) Increased gas content (N, H, O) in liquid steel. The steel chemical-composition adjustment, degassing, killing, and reduction of the impurity content in it were performed in the ladle-refining unit (LRU). The hydrogen content in the metal was not measured. Metal oxidation in the LRU before delivery to the continuous casting machine was on average of 2.94 ppm and did not exceed 3 ppm; this is recommended by the technological instructions of the enterprise. At the same time, in Ref. [59], the authors recommended the values of liquid steel oxidation to be within 3–5 ppm. In liquid steel studied in Ref. [45], a fluctuation in the oxygen activity was of 0.6–3.2 ppm, mass fraction of Al was of 0.01–0.1 wt.%, and the CaO/Al₂O₃ ratio was of 60/40–65/35 in the temperature range of 1.520–1.560°C. The average nitrogen content in the steel was of 60 ppm (ranging from 40 to 80 ppm) that also corresponded to the ‘Technological Instruction’ (TI) and is within the known standards [60].

(vii) Leaks in cooling systems. In the heat certificates, a leak in the boiler of BOF No. 2 was noted on the heat dates. During the retraining period, namely, when replacing the mould and the accompanying mandatory inspection of the connection cable and the mould cross-sectional change system, a water leak at CCM No. 3 was discovered in the upper part of mould No. 2 on the narrow wall. No water leaks occurred during the operation. This was noted in the technological casting log, and an inspection report was drawn up for the narrow wall of the mould No. 2 after disassembling and disconnecting the copper working walls from the steel part. The following was revealed during the inspection:

(a) the fastening bolts were tightened according to the instructions (force 60 Nm); they did not have visible defects; their length was main-

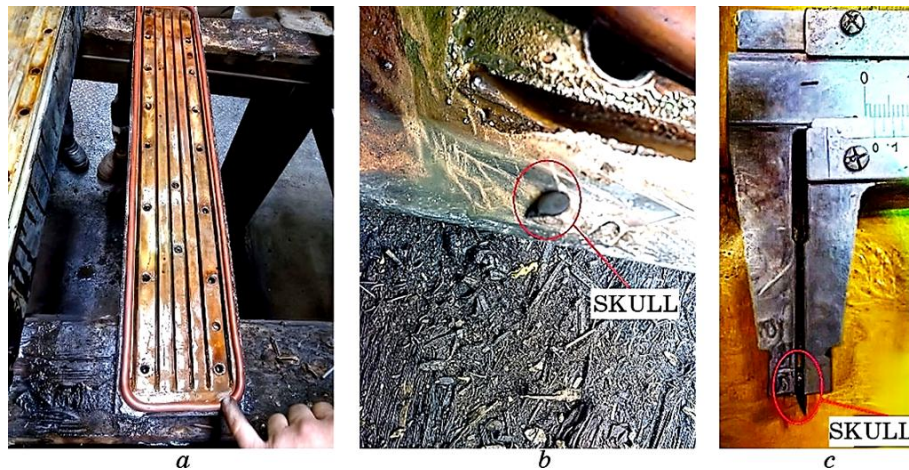


Fig. 10. Photo of the mould wall (*a*) and the skull (*b*, *c*).

tained as required;

(b) on the narrow wall between the steel and water-cooled copper wall on the narrow side (far end), where a water leak was detected, a metal skull was found, and a dent corresponding in shape to the skull was also found (Fig. 10);

(c) on the date of acceptance of mould No. 2, there were no comments about the hydraulic test of the copper plates.

Therefore, it can be concluded that the leak in the cooling system was caused by the presence of a metal skull between the steel and water-cooled copper walls of the mould that led to the damage of the seal. Pressure testing (10 atmospheres, 15 minutes) of the copper plates before commissioning did not reveal any defects, but, during the operation of the mould, this defect appeared. This is confirmed by the detection of a dent and a water leak and corresponds to previously obtained results.

Analysing this case allows us to conclude that leaks or ruptures in the cooling system of the continuous casting mould can lead to the formation of subsurface gas bubbles in the solidified slab. When cooling water accidentally enters the molten metal, it instantly vaporizes, generating high-pressure steam that may become trapped in the solidifying shell and form gas inclusions. Moreover, sudden contact between water and the hot surface of the strand can cause localized uneven cooling, which may lead to microcracks and destabilization of the solidification front. These conditions promote the entrapment of gases and the development of internal voids or blister-like defects in the cast product (slab).

The manuscript [61] states that the cooling systems have a critical

impact on the formation of defects. In CCM steelmaking industry practice, verifying the tightness of mould cooling systems by conducting hydraulic pressure tests is a mandatory procedure. However, excessive pressure during such tests can lead to deformation of copper walls, coating damage, and potential leakage in sealing zones [62]. Reference [62] also notes that failure to adhere to recommended testing parameters may alter the mould geometry and affect the formation of the initial solid shell. Therefore, water leaks such as those described in the carried-out study may be an indirect consequence of improper pre-commissioning pressure-testing procedures. Although the problem of cooling water leakage in continuous casting systems is well known and poses a serious threat to process stability, it remains insufficiently covered in scientific literature. The relevance of this issue is further supported by patent US4182397A [63], which proposed an engineering solution to minimize leakage risks through the use of dovetail-shaped channels and corrosion-resistant fastening elements. Thus, received findings not only confirm the potential link between the cooling water leakage and the formation of subsurface gas bubbles (blisters), but also expand the current scientific knowledge by emphasizing the critical role of mould tightness control as a key factor in preventing defects in continuously cast slabs.

3.3. Root Cause of Gas Saturation

During the study period, scheduled measurements of hydrogen content were undertaken in the tundish, during casting of steel grade S235 on continuous casting machine CCM No. 3. The measurements were taken on five heats, using the HydriS electrochemical probe, in accordance with the plant's standard practice for hydrogen-sensitive grades. The results of hydrogen-content measurements, oxidation values, and comments on the rolling quality of these heats in HRM-1700 are presented in Table 3.

Table 3 shows that the actual hydrogen content in steel at the continuous casting machine was of 3.2–4.9 ppm, while the oxidation of the metal in the ladle-refining unit did not exceed 3 ppm. A number of measures were taken to identify the causes and prevent increased gas saturation of the metal.

(i) CCM No. 3 control sampling was made of argon for water content in the gas. The water content in argon was determined to be of 0.0023% that complies with the required $< 0.0070\%$.

(ii) Three samples of AM-grade coal (used for the carbon-restoration treatment during tapping from the BOF) were taken to determine their water content. The water content in AM-grade coal was within the range of 1–1.2% (with a requirement of no more than 9%).

(iii) The duration of thermostatic treatment of slabs has been in-

TABLE 3. Gas saturation measurements and characteristics of the rolled product quality.

Oxidation and hydrogen content for S235 (1.305) CCM No. 3 for different heats		Notes on the mill	Total, tonnes	Valid, tonnes	Rejected, tonnes
Oxidation on LRU, ppm	Hydrogen at CCM, ppm				
2.9	3.6	–	Heats didn't rolled on HRM-1700		
2.7	3.2	Width divergence (+)	132.3	132.3	0
2.8	4.4	Indentations	141.75	141.75	0
2.8	3.7	–	Heats didn't rolled on HRM-1700		
2.9	4.9	–			

creased for heats cast on CCM No. 2 and CCM No. 3 for orders from HRM-1700 (at least 48 hours in the BOF shop warehouse). Slabs in standby mode were stored in the BOF shop slab warehouse.

(iv) Nine control samples of mould powder were taken, which were used for casting (ATST-SP627, GS-MC22MA, STSP285-22, except for the T-4-2(i), since the entire batch was produced) at all continuous casting machines to determine the water content. The actual water content was of 0.20–0.50%.

(v) In HRM-1700, selective peeling of slabs of current heats of similar grades was carried out. The amount of metal rolled with the blister defect was of 3 312.5 tonnes. From this amount, 42.4 tonnes (1.2%) of rolled steel were additionally rejected due to the defects 'telescope' and 'protruding coils'. These imperfections are not related to the presence of blisters, but depend on the condition and settings of the mechanical equipment of the rolling shop [64].

(vi) The analysis of several heats cast and rolled in the same period did not detect the blister defect. Dependences of the defect formation on the processing parameters (liquid steel treatment, casting, and rolling) in adjacent heats and heats with the defect were not detected.

Considering the sufficient killing of the steel, absence of overheating before rolling and the secondary oxidation in the continuous casting machine, and the nitrogen content being in the required range of 40–80 ppm, the reason for the formation of the largest number of blister defects was the ingress of water from the mould cooling system to the slab surface.

3.4. Development of Methods for Blistering Prevention

The analysis of results obtained in this work suggested the following

methods for prevention of blisters on the rolled steel surface: perform tests of incoming AM- and AF-coals, which are used for carbon-restoration treatment of steel, for water, ash and volatile contents, eliminate the possibility of mixing coals of the AM- and AF-grades with non-coking energy coal during storage, drying and other technological operations, eliminate the possibility of using solid-slag mixtures during steel tapping from the BOF with a shelf life of more than 24 hours, eliminate the production of steel grades, which are going by 'warm' charge to HRM-1700, on BOFs with leaking boiler, use wet ferroalloys only after calcination, conduct mandatory oxidation measurements on the LRU for low-silicon (semi-killed) steel grades cast on continuous casting machines, perform moulds' monitoring for leaks during acceptance and installation on the continuous casting machine, test each batch of mould powder for water content with an increased number of samples (2 or more), conduct weekly monitoring of argon supplied to the continuous casting machine for water content, eliminate using tundishes with violation of the heating and drying schedule.

Following implementation of these measures and prior to publication of this paper, 2.692 tonnes of hot rolled coils have been produced without defects. All this steel was supplied to the electric-resistance welding (ERW) pipe mill that produced pipes fully complying with the standard requirements, demonstrating the effectiveness of the measures undertaken to minimise the defect formation. To further ensure the defect-free production, hot-rolled coils were shipped to the ERW mill with recommendations to maintain a lower manufacturing speed, thereby, eliminating the probability for blister-defects' formation. In addition, the experiments involving etching and galvanizing of hot-rolled steel coils were carried out in the cold-rolling shop. The results confirmed that the galvanized coils met all regulatory quality requirements, showcasing the effectiveness of the implemented measures in mitigating the defects.

4. CONCLUSIONS

The analysis of steel making, casting, and hot-rolling technology at HRM-1700 mill of the Metinvest established the primary cause of the blister defects being the ingress of water during steel casting following a loss of seal integrity in the moulds of continuous casting machines.

The water content in the mould powders was measured to be of 0.20–0.50% that is approaching maximum permissible limit of 0.50%. Therefore, stricter quality control of the mould powders was recommended to prevent formation of defects.

A comprehensive set of preventive methods was developed, including enhanced control of ferroalloy humidity, thorough checks of the

casting machines mould seals, and additional requirements for tundishes' heating. Following implementation of the developed methods, 98.8% of the hot-rolled 2.692 tonnes of steel met quality standards. This confirms the effectiveness of the measures undertaken to reduce the defects' formation.

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AUTHORS' CONTRIBUTIONS

V. V. Kukhar: supervised the research project, validated the effectiveness of preventive methods at the HRM-1700 mill, wrote the paper. O. M. Smirnov: support on the development of the technologies proposed in the paper, details on the technologies, wrote the paper. A. G. Kostryzhev: conducted high-resolution microscopy and micro-analysis of surface defects, interpreted metallographic data regarding grain coarsening and oxide clusters, wrote the paper. K. Ye. Pismarov: coordinated sample preparation and metallographic-investigation procedures, managed the chemical-composition verification according to ISO standards. V. Ye. Ukhin: collected the data, analysis of results and correlation with industrial data, wrote the paper. O. D. Rud: performed physical-metallurgy analysis of the 'metallic skull' and seal damage, investigated the root causes of subsurface gas-bubble formation. M. S. Goryuk: collected industrial production data and monitored casting parameters at CCM No. 3, assisted in the investigation of water leaks in the mould cooling system. S. V. Semiriagin: analysed the moisture content in mould powders and slag-forming mixtures, correlated the defect formation with argon consumption and ladle shroud usage. Yu. P. Skorobagatko: evaluated the gas saturation (hydrogen and oxy-

gen content) in liquid steel, monitored the drying and heating schedules for tundish preparation. A. Yu. Semenko: collected the data, conceived and designed the analysis, wrote the paper.

REFERENCES

1. V. Kukhar, O. Kurpe, A. Prysiaznyi, O. Khliestova, V. Burko, E. Balalayeva, and N. Yelistratova, *IOP Conf. Ser.: Mater. Sci. Eng.*, **1037**: 012024 (2021).
2. V. Zurnadzhy, V. Stavrovskaya, Y. Chabak, I. Petryshynets, B. Efremenko, K. Wu, V. Efremenko, and M. Brykov, *Materials*, **17**: 1958 (2024).
3. R. I. L. Guthrie and M. M. Isac, *Metals*, **12**: 862 (2022).
4. M. W. Ashour, F. Khalid, A. A. Halin, L. N. Abdullah, and S. H. Darwish, *Arab. J. Sci. Eng.*, **44**: 2925 (2019).
5. A.-H. Bui and V.-H. Nguyen, *J. Met. Mater. Miner.*, **30**, No. 1: 80 (2020).
6. A. Smirnov, A. Verzilov, A. Semenko, Yu. Skorobogatko, and D. Hoida, *Metall. Res. Technol.*, **120**, No. 3: 310 (2023).
7. V. Smirnov, V. Ukhin, S. Semiriagin, A. Semenko, and Yu. Skorobogatko, *Metall. Res. Technol.*, **121**: 106 (2024).
8. J. Zhang, S. Yang, Y. Chen, Z. Chen, J. Zhao, J. Li, and Z. Jiang, *JOM*, **70**: 2886 (2018).
9. J. Zhang, Q. Liu, S. Yang, Z. Chen, J. Li, and Z. Jiang, *ISIJ Int.*, **59**, Iss. 7: 1167 (2019).
10. Z. Wei, Z. Wang, D. Zhang, Y. Liu, X. Wang, and M. Yao, *Metall. Mater. Trans. B*, **55**: 3773 (2024).
11. H. Zhang and W. Wang, *Metall. Mater. Trans. B*, **47**: 920 (2016).
12. W. Tong, S. Luo, W. Wang, and M. Zhu, *Metall. Mater. Trans. B*, **55**: 727 (2024).
13. H. Lei, J. Liu, G. Tang, H. Zhang, Z. Jiang, and P. Lv, *JOM*, **75**: 914 (2023).
14. P. Lyu, W. Wang, and H. Zhang, *Metall. Mater. Trans. B*, **48**: 247 (2017).
15. M. L. S. Zappulla and B. G. Thomas, *IOP Conf. Ser.: Mater. Sci. Eng.*, **861**: 012016 (2020).
16. B. Sun, Z.-Y. Liu, and G.-D. Wang, *J. Iron Steel Res.*, **31**, No. 10: 1417 (2010).
17. L. D. Way, *Mater. Sci. Technol.*, **17**, Iss. 10: 1175 (2001).
18. S. Zhao, J. Lyu, G. Song, J. Wang, D. Xu, and Z. Li, *Metall. Res. Technol.*, **120**, No. 4: 327 (2023).
19. R. Schlatter, *Steel Times*, **214**: 432 (1986).
20. C.-J. Zhang, Y.-P. Bao, M. Wang, and L.-C. Zhang, *Int. J. Miner. Metall. Mater.*, **24**: 47 (2017).
21. P. H. Dauby, *Rev. Metall.*, **109**, No. 2: 113 (2012).
22. P. Pathak, G. Das, and S. K. Jha, *Recent Advances in Manufacturing Processes* (Eds. R. Kumari, J. D. Majumdar, and A. Behera), (Singapore: Springer: 2022), p. 153.
23. O. Kurpe, V. Kukhar, E. Klimov, and A. Prysiaznyi, *Solid State Phenom.*, **291**: 63 (2019).
24. M. Sun, *2023 Proc. 42nd Chin. Control Conf. (CCC)*, p. 6998.
25. C. Zhang, K. Peng, and J. Dong, *Control Eng. Practice*, **107**: 104698 (2021).
26. V. Vasan, N. V. Sridharan, S. Vaithiyanathan, and M. Aghaei, *Heliyon*, **10**, Iss. 19: e38498 (2024).

27. S. Wackenrohr, S. Herbst, P. Wöbeking, G. Gerstein, and F. Nürnberger, *J. Manuf. Mater. Process.*, **7**: 142 (2023).
28. D. Kalisz, P. L. Żak, S. Semiryagin, and S. Gerasin, *Materials*, **14**, Iss. 23: 7113 (2021).
29. S. Louhenkilpi, *Treatise on Process Metallurgy* (Eds. S. Seetharaman, R. Guthrie, A. McLean, S. Seetharaman, and H. Y. Sohn) (Elsevier: 2024), Ch. 1.10, p. 343.
30. N. Ali, L. Zhang, H. Zhou, A. Zhao, C. Zhang, and Y. Gao, *Mater. Design*, **209**: 109978 (2021).
31. C. Wang, Y. Yu, L. Wang, Z.-G. Pang, L. Chen, and H.-L. Jiao, *J. Iron Steel Res.*, **28**, No. 3: 74 (2016).
32. G. Das, S. Ghosh, S. Ghosh Chowdhury, S. Ghosh, S. Das, and D. K. Bhattacharaya, *Eng. Fail. Analysis*, **10**, Iss. 3: 363 (2003).
33. W. Shen, G. Cheng, and X. Liu, *Ironmaking and Steelmaking: Processes, Products and Applications*, **52**, Iss. 1: 24 (2024).
34. M. Pietrzyk, R. Kawalla, and H. Pircher, *Steel Research*, **66**, Iss. 12: 526 (1995).
35. M. Vlado, R. Bidulský, L. Gulová, K. Machová, J. Bidulská, J. Valíček, and J. Sas, *High Temp. Mater. Proc.*, **30**: 105 (2011).
36. L.-W. Zhang, W.-F. Shen, C. Zhang, Q.-H. Xu, and Y. Cui, *Proc. Eng.*, **207**: 532 (2017).
37. S.-H. Zhang, W.-H. Tian, L.-Z. Che, W.-J. Chen, Y. Li, L.-W. Wan, and Z.-Q. Yin, *J. Iron Steel Res. Int.*, **31**: 2236 (2024).
38. S. Moir and J. Preston, *J. Mater. Process. Technol.*, **125–126**: 720 (2002).
39. S. Ganguly, X. Wang, K. Chandrashekhara, M. F. Buchely, S. Lekakh, R. J. O'Malley, D. Bai, and Y. Wang, *Steel Res. Int.*, **92**, Iss. 2: 2000293 (2021).
40. L. Hua, Y. Liu, D. Qian, L. Xie, F. Wang, and M. Wu, *Mater. Characterization*, **185**: 111736 (2022).
41. O. E. Markov, A. S. Khvashchynskyi, A. V. Musorin, M. A. Markova, A. A. Shapoval, and N. S. Hrudkina, *Int. J. Adv. Manuf. Technol.*, **122**: 1383 (2022).
42. H. Tanaka, R. Nishihara, R. Miura, R. Tsujino, T. Kimura, T. Nishi, and T. Imoto, *ISIJ Int.*, **34**, Iss. 11: 868 (1994).
43. H. Goto and K. Miyazawa, *ISIJ Int.*, **38**, Iss. 3: 256 (1998).
44. C. Shi, J. Li, and S. Yang, *Electroslag Remelting Towards Clean Steel* (Singapore: Springer: 2023), p. 73.
45. K. Riyahimalayeri, P. Ölund, and M. Selleby, *Steel Res. Int.*, **84**, Iss. 2: 136 (2013).
46. L. Wang, H.-G. Lee, and P. Hayes, *Steel Res.*, **66**, Iss. 7: 279 (1995).
47. H. Yang, H. Olia, and B. G. Thomas, *Metals*, **11**: 116 (2021).
48. Z. W. Hou, M. Jiang, E. J. Yang, S. Y. Gao, and X. H. Wang, *Metall. Mater. Trans. B*, **49**: 3056 (2018).
49. J. H. Park and Y.-B. Kang, *Steel Res. Int.*, **95**, Iss. 11: 2300598 (2024).
50. J. G. Duh and C. J. Wang, *J. Mater. Sci.*, **25**: 2063 (1990).
51. S. Zhang, M. Long, H. Zhang, S. Ai, D. Chen, P. Liu, and H. Duan, *J. Mater. Res. Technol.*, **11**: 2049 (2021).
52. D. Lee, Y.-S. Kim, T. T. T. Trang, W.-T. Cho, M.-H. Kang, J. S. Lee, C. H. Yim, and Y.-U. Heo, *Eng. Failure Analysis*, **166**: 108895 (2024).
53. A. Bahrami, M. K. Khouzani, S. A. Mokhtari, S. Zareh, and M. Y. Mehr, *Met-*

- als*, **9**, Iss. 7: 801 (2019).
54. S. K. Dhua, *J. Failure Analysis Prevention*, **19**: 1023 (2019).
55. N. F. Garza-Montes-de-Oca, K. Y. Pineda-Arriaga, M. J. Gaona-Martinez, J. H. Ramirez-Ramirez, F. A. Perez-Gonzalez, and R. Colas, *J. Failure Analysis Prevention*, **23**: 298 (2023).
56. O. Kruzhilko, N. Volodchenkova, V. Maystrenko, B. Bolibrukh, V. P. Kalinchyk, A. Zakora, A. Feshchenko, and S. Yeremenko, *Arch. Min. Sci.*, **67**: 179 (2022).
57. K.-K. Lai, S.-W. Lin, H.-W. Lo, C.-Y. Hsiao, and P.-J. Lai, *Sustainability*, **15**, Iss. 24: 16583 (2023).
58. W. Glitscher, *La Metall. Ital.*, **11–12**: 61 (2005).
59. B. G. Bartosiaki, J. A. M. Pereira, W. V. Bielefeldt, and A. C. F. Vilela, *J. Mater. Res. Technol.*, **4**, Iss. 3: 235 (2015).
60. S. Pal, A. Patra, and P. R. Padhee, *Process Modeling for Steel Industry* (I K International Publishing House: 2018).
61. F. R. Camisani-Calzolari, I. K. Craig, and P. C. Pistorius, *IFAC Proc. (2002)*, Vol. 35, Iss. 1, p. 149.
62. LMM Group. *Analysis of the Pressure Problem during the Installation of the Mold* (2020); <https://www.lmmgroupcn.com/analysis-of-the-pressure-problem-during-the-installation-of-the-mold/>
63. W. A. Schmucker and L. M. Dings Jr., *Continuous Casting Mold and Means for Securing Mold Liners Therein*, Patent of USA No. 4182397 (Published January 8, 1980).
64. J. K. Good and D. R. Roisum, *Winding: Machines, Mechanics and Measurements* (DEStech Publ. Inc.: 2008).

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Fundamentals of Stable Drawing Process of Steel Rod, Which Has Integrity Violations

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Using the developed method, the deformation regularities of artificial cone-shaped defects' models on wire rod and wire during their multiple drawing are experimentally determined for the first time. As found, for the most part, the smoothing of such defects occurs more intensively, the greater the total wire elongation and the initial values of the depth and width of the defects. These data are compared with the results of the previous similar investigations on wire deformation in the presence of defects of predominantly transverse orientation and predominantly longitudinal one relative to the drawing axis. As shown, the qualitatively obtained new results are closer to the 'transverse' defects' deformation patterns, and the quantitative differences can be explained by the presence of a volumetric scheme of the metal stress-strain state around the artificial surface violation of its continuity. The influence of the presence of such artificial formations ('defects') on the condition of uninterrupted wire drawing is determined. Attention is paid to the possible additional negative influence of 'defects' as stress concentrators on the stability of the drawing process. The results of the investigation may be useful in designing the routes for continuous multiple drawing of a work-piece with the probable presence of continuity defects on it, and in investigating the mechanism of deformation of such surface defects to obtain drawn wire of acceptable quality. A promising direction of research is to combine

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the data of existing work with known methods for calculating force conditions of deformation and thoroughly quantifying the parameters of designed routes for multiple stable wire drawing.

Key words: drawing, wire rod, wire, surface defects, deformation regularities.

З використанням розробленого методу уперше експериментально визначено закономірності деформування штучних моделей конусоподібних дефектів на катанці та дроті за їхнього багаторазового волочіння. Виявлено, що здебільшого має місце вигладжування таких дефектів тим інтенсивніше, чим більше сумарна витяжка дроту та початкові значення глибини та ширини дефектів. Дані зіставлено з результатами попередніх аналогічних досліджень під час деформування дроту за наявності на ньому дефектів переважно поперечної та переважно поздовжньої орієнтації відносно осі волочіння. Показано, що якісно одержані нові результати ближче до закономірностей деформування «поперечних» дефектів, а кількісні відмінності можуть бути пояснені наявністю об'ємної схеми напружено-деформованого стану металу в околі штучного поверхневого порушення його суцільності. Визначено вплив наявності таких штучних утворень («дефектів») на умову безобривного волочіння дроту. Приділено увагу щодо наявності вірогідного додаткового негативного впливу «дефектів» як концентраторів напружень на стабільність процесу волочіння. Результати дослідження можуть бути корисними під час проєктування маршрутів безобривного багаторазового волочіння заготовки з вірогідною наявністю на ній дефектів суцільності, для вивчення механізму деформування таких поверхневих дефектів з одержанням волоченого дроту прийнятної якості. Перспективним напрямом досліджень представляється об'єднання даних даної роботи з відомими методиками розрахунку силових умов деформування та ґрунтового кількісного визначення параметрів проєктованих маршрутів багаторазового стабільного волочіння дроту.

Ключові слова: волочіння, катанка, дріт, поверхневі дефекти, закономірності деформування.

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1. INTRODUCTION

Drawing as a method of metal forming by pressure consists of one or more times pulling a previously rolled or pressed workpiece through the drawing die hole. The die cross-sectional area is smaller than the workpiece cross-sectional area, and its configuration forms a given profile of the product: wire, thin-walled pipe, round in cross-section or profiled shaped bar. As a result, the cross-sectional area of the metal decreases and the length of the product increases.

As an initial workpiece for the production of cold-drawn steel wire with a final diameter of $d_e = 0.002\text{--}6$ mm, a wire rod with a nominal diameter of $d_0 = 5.5\text{--}6.5$ mm is usually used. The workpiece for drawing

in subsequent drawing cycles is the wire obtained after the previous passes.

The possibility of the process is assessed by the value of the safety factor coefficient, which is determined by the ratio of the wire tensile strength and the tensile stress ('drawing stress') at the drawing die exit, and which must be no less than a certain critical value of $[\gamma] = 1.34-2.10$ [1]:

$$\gamma = \sigma_{B1} / \sigma \geq [\gamma]. \quad (1)$$

The variation range of $[\gamma] = 1.34-2.10$ was determined experimentally for a standard workpiece, which is due to factors that destabilize the values of σ_{B1} and σ , and its smaller value corresponds to a larger wire diameter.

These force parameters are formed by the conditions in different sections of the deformation zone of die and depend on the metal strengthening, which is due to its chemical composition and the accumulated elongation μ_Σ while drawing [1]:

$$\mu_\Sigma = F_0 / F_{1n} = \prod_{i=1}^n \mu_i, \quad (2)$$

where F_0 , F_{1i} are the area of the initial and at the exit from the n -th drawing metal cross-section, n is the drawing multiplicity ($1 \leq i \leq n$), the drawing elongation in the i -th pass [1]:

$$\mu_i = F_{0i} / F_{1i}, \quad (3)$$

F_{0i} is the area of metal at the entrance of i -th die.

It is clear that for round cross-sections of wire rod and wire: $F_0 = \pi d_0^2 / 4$, $F_{0i} = \pi d_{0i}^2 / 4$, $F_{1i} = \pi d_{1i}^2 / 4$ (where d is the corresponding diameter). In addition, the drawing stress σ depends on the friction conditions and the geometry of the drawing.

Using the levels σ_{B1} and $[\gamma]$ according to (1) it is possible to obtain the value σ , and then by reverse calculation using one of the numerous known formulas [1, 2, 3, *etc.*] taking into account the dependence of the mechanical properties of the metal on μ_Σ , the geometry of the die and the friction conditions, determine the characteristics of the metal compression in the drawing, in particular μ_Σ , and also (if necessary)—the mode of multiple drawing. The value μ_i usually ranges from 1.05 to 1.5.

The main regulatory documents regulating the quality of steel wire rod are the standards: European EN 16120 (in four parts), American ASTM A510/A510M, Ukrainian ДСТУ 2770, ДСТУ 2651, ДСТУ 4738, ДСТУ 3684, Technical conditions of Ukraine ТУ У 27.1-00190319-1280, ТУ У 27.1-00190319-1314, *etc.* [4].

Such production in Ukraine is carried out at several large enterpris-

es (PJSC ‘Zaporizhzhia Steel Rolling Plant’, LLC ‘Dniprometiz TAS’, PJSC ‘Stalakanat’, LLC ‘Garant Metiz Invest’ [5] and at several dozen smaller firms.

The annual output of drawn steel wire in Ukraine and, accordingly, wire rod in the period 2015–2021 was 270–380 thousand tons, which is approximately 8–10% of the rolled products amount. The market value of steel wire is 21–24 thousand UAH per ton [6].

After the ^{Russian} invasion of Ukraine and the loss of control over some metallurgical enterprises, such production decreased by 15–35%. More accurate data (according to the Law of Ukraine ‘On Official Statistics’ of August 16, 2022, No. 2524-IX) are classified as confidential statistical information [7, 8].

Drawn steel wire is used to make ‘hardware’—metal products that are mostly used to fasten steel, wooden and plastic parts or components, and are also used for household purposes as tools [9].

In the production practice of hardware enterprises (drawing, mesh, electrode, nail, fastening, *etc.* shops of metallurgical enterprises), such products include mesh, cables, electrodes, ropes, bolts, screws, nuts, nails, cotter pins, *etc.*

The approximate structure of steel wire mass consumption is as follows: commercial wire of various general purposes—up to 59%, cables and ropes—10%, wire for reinforcing reinforced concrete structures—7%, nails and other fasteners—more than 5%, fibre—4%, steel mesh—3%, *etc.* [5].

2. ANALYSIS OF THE PROBLEM

Although each of the above-mentioned types of products has specific requirements, which are reflected in a large number of standards [10], manufacturers often use the cheapest low-carbon wire rod when manufacturing wire and hardware for general (non-responsible) purposes. The latter may have an expanded (compared to standards) range of variation of geometric and mechanical characteristics, as well as minor surface and volumetric defects that do not prevent its further use for its intended purpose. An example may be binding wire, nails, *etc.*

Production experience shows that defects (with typical sizes from fractions of a μm to several mm) and their location in the volume and (or) on the surface of the workpiece can significantly negatively affect the stability of the drawing process, causing breaks, premature failure of dies, *etc.* At the same time, such an impact is greater the more: the ratio of the defect sizes to the cross-sectional dimensions of the workpiece and wire [11], the difference between the strength properties of the base metal and the material of the defect itself [11].

Defective wire rod accounts for approximately 10% of its total output and is delivered in coils, which makes it difficult to identify devia-

tions from regulatory requirements. In fact, it should be scrapped with losses of the difference in the corresponding prices. At the same time, the price of scrap metal is 5–10 times lower than the price of standard wire rod, and the corresponding losses, in fact, are borne usually by the hardware company.

The standardization of the use of such a workpiece in practice is carried out by adopting specifications and (or) technical conditions for the workpiece and products made from it, agreed between the parties, with a corresponding price reduction.

Therefore, it becomes economically feasible to develop measures to ensure stable drawing of wire rod, which has non-critical deviations from the requirements of the standard, which do not prevent the production of high-quality final products. This has led to the feasibility of investigating the deformation patterns not only of the workpiece itself, but also of defects that may occur on it, as well as their impact on the stability of the drawing process.

Usually, fixing and identifying the type and size of specific defects is possible only on samples specially selected from the beginning or end of the wire rod coil, or in places of wire breaks (in case of violation of stable drawing regimes). Based on the number of detected violations, an expert conclusion is made on the possibility and feasibility of processing such wire rod into conditioned wire without changing the established deformation regime, and subsequently for the manufacture of hardware.

Nevertheless, at the same time, continuous drawing with regulated reduced compression of the workpiece must be ensured, usually in several passes to achieve the required wire size.

Obviously, this is ensured by defining μ_i , when performing, a relation similar to formula (1) [11]:

$$\gamma = \sigma_{B1} / \sigma \geq K_{\text{def}} [\gamma], \quad (4)$$

where $K_{\text{def}} (> 1)$ is the coefficient of the defect influence on the conditions of stable (continuous) drawing.

The quantitative assessment K_{def} in the investigation [11] took into account the following: the presence of a defect with K_{def} cross-sectional area (relative to the workpiece axis) reduces the area of defect-free metal compared to the area F_{1i} at the exit from the drawing die, as well as the strength of the wire regardless of the material strength that fills the volume of the defect and does not resist stretching due to a violation of the continuity of the material of the entire product; the drawing process destabilizes precisely the difference in strength $\sigma_{B,\text{def}}$ of the material filling the defect from σ_{B1} the base metal of the wire, since the latter depends on the total μ_{Σ} (2) and partial μ_i (3) drawing elongation.

Then, to ensure $K_{\text{def}} > 1$, this parameter was defined [11]: when

$\sigma_{B,def} \leq \sigma_{B1}$ according to the formula [11]:

$$K_{def,i} = \frac{F_{1i} \sigma_{B1}}{(F_{1i} - F_{def}) \sigma_{B1} + F_{def} \sigma_{B,def}} > 1, \quad (5)$$

when $\sigma_{B,def} \geq \sigma_{B1}$ according to the formula [11]:

$$K_{def,i} = \frac{(F_{1i} - F_{def}) \sigma_{B1} + F_{def} \sigma_{B,def}}{F_{1i} \sigma_{B1}} > 1. \quad (6)$$

Usually, as a result of preparing the workpiece for drawing by etching or alternating bending, its surface defects become hollow ($\sigma_{B,def} \rightarrow 0$). Then, from Eq. (5), it followed [11]:

$$K_{CID,i} = \frac{F_{1i}}{F_{1i} - F_{def,i}} > 1, \quad K_{def,i} = \frac{F_{1i}}{(F_{1i} - F_{def})} > 1, \quad (7)$$

and to determine the value K_{def} according to formulas (5)–(7), quantitative data on the regularities of the shape change of defects during the drawing process, taking into account their initial shape and location in the body of the workpiece relative to the axis of the latter, became necessary.

The shape, size, and location of defects in a workpiece cannot be predicted a priori.

Therefore, theoretical assessment of their influence on the stability of the drawing process is possible only by investigating the deformation process of simplified defect models, and experimental assessment is possible by creating artificial ‘defects’ on the workpiece: drilling and surface notches with different geometry, dimensions and orientation, followed by deformation, measurement of dimensions and corresponding analysis of the results.

The first logical step towards developing appropriate measures was to identify the most common defects in wire rod. These include [11–15, *etc.*]:

- non-metallic (oxide, slag) inclusion in the volume or (and) on the surface of the wire rod; the consequence of the presence of such a volumetric defect is the formation of discontinuity and (or) heterogeneity of the properties of the workpiece material, and the surface violation of the continuity of the metal, as a rule, exfoliates during the deformation process and leaves behind a shell with a depth of fractions of a millimetres to a through hole;
- delamination, which can originate during preliminary rolling, appear during deformation of wire rod areas with the presence of non-metallic inclusions or shrinkage, or serious segregation by chemical composition, which causes excessive internal stresses in the metal up to the appearance of cracks;

- longitudinal and transverse cracks, which may be the result of uneven deformation of the metal in terms of volume with the initial presence of surface defects, microscopic pores and bubbles, or overburning during preliminary heat treatment;
- delaminations ('captures'), which are a consequence of the loss of radial stability and subsequent rolling of the surface convexities of the workpiece during its deformation, and which, as a rule, turn into delamination or cracking during drawing;
- scratches and dents of different depths and directions relative to the wire rod axis, *etc.*

Given that the present work is a continuation of previous similar investigations under the same leadership, it became appropriate to make a certain generalization and analysis of the results already obtained. The emphasis is placed on the fact that in the deformation centre during wire drawing in the radial direction to its axis, compressive stresses and strains act, and in the longitudinal direction—tensile.

Such a scheme of the stress-strain metal state is dangerous from the point of view of initiating wire breakage under the action of the drawing force, especially in the presence and influence of wire continuity violations along the drawing axis [1]. In this case, the dimensions of these violations in the cross section perpendicular to the drawing axis (wire) must be taken into account.

As a part of the research conducted earlier and reflected in the works [16, 17], results were presented regarding the deformation patterns of one type of artificial defects.

In particular, in work [16], the deformation zone during drawing was theoretically considered with a defect model in the form of a through cylindrical drilling with a diameter of Δ_0 , located transversely to the axis of a round wire rod with a diameter of d_0 . Virtual 'deformation' occurred in a conical drawing die with a drawing elongation μ_i , and the metal received a total drawing elongation $\mu_{\Sigma i}$ at the exit from the sequence of draws, starting from the first one.

This model of a conditional 'defect' was presented: hollow with a tensile strength $\sigma_{B,def} = 0$, filled with a material with a tensile strength lower than that of the base metal ($\sigma_{B,def} < \sigma_{B1}$), filled with a material with a tensile strength close to the base metal ($\sigma_{B,def} \approx \sigma_{B1}$), filled with a material with a tensile strength greater than that of the base metal ($\sigma_{B,def} > \sigma_{B1}$).

During the theoretical analysis, the authors took into account that the wire rod (preform) during drawing is stretched along the longitudinal axis z and compressed radially along the axis ρ . This caused the deposition of the 'defect' along its 'height', which was equal to the current diameter d_{zi} in the drawing, and the initial circular cross-section of the 'defect' with a diameter Δ_0 acquired an elliptical shape with the current dimensions a_{zi} (along the metal axis) and b_{zi} (perpendicular to

the longitudinal axes of the wire and the ‘defect’ model).

Based on reasonable assumptions about the invariance of the cross-section of the ‘defect’ during metal deformation i -th gap [16] and the relationship between deformations and stresses [1], it was shown that for an arbitrary ratio $(\sigma_{B,def}/\sigma_{B1i})$ [16]:

$$b_{Zi} = \frac{\Delta_{Zi}}{1 - (\sigma_{B,def}/\sigma_{B1i})(\Delta_{Zi}/d_{Zi}) + \Delta_{Zi}/d_{Zi}}, \quad (8)$$

$$a_{Zi} = \frac{\Delta_0 \sqrt[4]{\mu_{\Sigma i}} [d_{Zi} + (1 - \sigma_{B,def}/\sigma_{B1i})\Delta_0 \sqrt[4]{\mu_{Zi}}]}{d_{Zi}} \left[1 + \frac{\Delta_0 \sqrt[4]{\mu_{Zi}}}{2(d_{Zi} - (\sigma_{B,def}/\sigma_{B1i})\Delta_0 \sqrt[4]{\mu_{Zi}})} \right]. \quad (9)$$

For the cross section of the metal exit and the ‘defect’ from the deformation centre, according to Eqs. (8) and (9), as well as formulas (2) and (5) from the source [16]:

$$\Delta_{1i} = \Delta_0 \sqrt{\mu_{\Sigma i}}, \quad (10)$$

after transformations, taking into account $F_0 = \pi d_0^2/4$, $F_{0i} = \pi d_{0i}^2/4$, $F_{1i} = \pi d_{1i}^2/4$, it turned out [16] with $d_{Zi} = d_{1i} = d_{0i}/\sqrt{\mu_{\Sigma i}}$ and arbitrary ratio $\sigma_{B,def}/\sigma_{B1i}$ [16]:

$$b_{Zi} = \frac{\Delta_0 \sqrt{\mu_{\Sigma i}}}{1 - (\sigma_{B,def}/\sigma_{B1i})(\Delta_0 \mu_{\Sigma i}/d_0) + \Delta_0 \mu_{\Sigma i}/d_0}, \quad (11)$$

$$a_{Zi} = \frac{\Delta_0 \sqrt[4]{\mu_{\Sigma i}} [d_0 + (1 - \sigma_{B,def}/\sigma_{B1i})\Delta_0 \mu_{\Sigma i} \sqrt{\mu_{\Sigma i}}]}{d_0} \left[1 + \frac{\Delta_0 \mu_{\Sigma i} \sqrt{\mu_{\Sigma i}}}{2(d_0 - (\sigma_{B,def}/\sigma_{B1i})\Delta_0 \mu_{\Sigma i} \sqrt{\mu_{\Sigma i}})} \right]; \quad (12)$$

with $\sigma_{B,def}/\sigma_{B1i} = 0$,

$$b_{1,0,i} = \Delta_0 d_0 \sqrt{\mu_{\Sigma i}} / (d_0 + \Delta_0 \mu_{\Sigma i}), \quad (13)$$

$$a_{1,0,i} = \Delta_0 \sqrt[4]{\mu_{\Sigma i}} [1 + \Delta_0 \mu_{\Sigma i} \sqrt{\mu_{\Sigma i}} / 2d_0]; \quad (14)$$

with $\sigma_{B,def}/\sigma_{B1i} = 1$,

$$b_{1,1,i} = \Delta_0 \sqrt{\mu_{\Sigma i}}, \quad (15)$$

$$a_{1,1,i} = \Delta_0 \sqrt[4]{\mu_{\Sigma i}} \left[1 + \frac{\Delta_0 \mu_{\Sigma i} \sqrt{\mu_{\Sigma i}}}{2 \cdot (d_0 - \Delta_0 \mu_{\Sigma i} \sqrt{\mu_{\Sigma i}})} \right]. \quad (16)$$

The results of calculations using formulas (11)–(13) were compared with experimental data [17].

In those experimental investigations, defect modelling was carried out by through-hole drilling with a diameter of $\Delta_0 = 2.0, 1.5$ and 1.0 mm perpendicular to the axis of the wire rod, which had a diameter of

$d_0 = 6.50$ mm. Modelling of the material properties of defects was performed using the following options: filled with rods made of aluminium, unhardened and pre-hardened low-carbon steel with known tensile strength values, as well as without filler.

The wire rod with holes was drawn through double dies in one pass to a diameter of $d_1 = 6.2$ mm ($\mu_i = \mu_s = 1.048$) in the presence of dry soap lubricant. After that, cylindrical samples were cut from the wire at the locations of the deformed 'defects', cut along the axis, made longitudinal sections of them, and measured the dimensions of the hole with a trammel.

It was found that, after drawing the workpiece, the initial round cross-sectional shape of the drilling-'defect' transformed into an elliptical one with dimensions a and b .

Comparison of experimental (index 'exp') and theoretical (index 'theor') values a revealed their satisfactory coincidence with a deviation of no more than 25% and the feasibility a of introducing a correction empirical coefficient A , which approximated the results of calculations and experiments with a deviation of no worse than 1% in the form of [17]:

$$a_{1i,\text{theor}} = A a_{1i,\text{exp}}, \quad (17)$$

were

$$A = 1.24 - 1.50 \frac{\Delta_0}{d_1} + 0.070 \frac{\sigma_{B,\text{def}}}{\sigma_{B \text{ Me.}}} + 0.25 \frac{\Delta_0}{d_1} \frac{\sigma_{B,\text{def}}}{\sigma_{B \text{ Me.}}}. \quad (18)$$

At the same time, the experimental data were more thorough.

At the analysis took into account that the cross section of the exit from the deformation zone and the deformation zone itself were at a certain distance from each other. As a result, if there is a 'defect' of a certain length a_i at the exit from the die, the metal in the deformation zone itself may not contain a defect. This situation is accompanied by a wire front end weakening and the preservation of the required drawing force.

What was important here it was that the size is 'responsible' for the weakening of the wire cross-section by the diameter d_{1i} at the exit from the drawing die in the presence of a defect according to Eq. (7) was precisely the size b_{1i} .

The data presented in Ref. [17] allowed us to assess the influence of the area of $F_{i,\text{def}} = b_{1i} d_{1i}$ a through-hole defect in the wire rod in the form of an initial cylindrical hole in the cross-section of the metal exit from the drawing die with the area $F_{1i} = \pi d_{1i}^2 / 4$ on the corresponding 'weakening' of the wire cross-section with a decrease in its reduced strength. In this case, it became possible to assume in a first approximation that the area of the conditionally cylindrical cross-section of the defect model with a diameter at the exit from the drawing is trans-

formed into an ellipse with axes a and b . From this, it follows: $\Delta_{1i}^2 = a_{1i} b_{1i}$ and $b_{1i} = \Delta_{1i}^2 / a_{1i}$.

Calculation of values $a_{1.0,i}$ according to formula (14) and adjustment of this quantity according to formulas (17) and (18) necessitated a corresponding change in the quantity b_i . Then, taking into account formula (10), after simple transformations, it is obtained [17]:

$$b_{1i} = \frac{\Delta_{0i}^2 \mu_{1i}}{A a_{1i, \text{theor}}}, \quad (19)$$

and it became appropriate to determine the defect influence coefficient K_{def} according to the simplified formula (7), which after simple transformations gave [17]:

$$K_{\text{def}} = \frac{\pi d_{1i}^2}{\pi d_{1i}^2 - 4 d_{1i} b_{1i}} > 1, \quad (20)$$

regulating according to (4) the corresponding reduction μ_i in the allowance.

Unlike volumetric defects, surface defects of a workpiece with different geometry and penetration depth (sometimes up to half its diameter), depending on the pressure treatment conditions, may disappear as a result of smoothing, remain or close, turning into a 'crack' [11].

To assess the influence of the size and orientation of surface defects on the results of their deformation and the conditions of stable multiple drawing of steel rod and wire, the results of relevant experimental investigations were presented using a specially developed method [11].

The drawing of prepared samples of wire rod with a diameter $d_0 = 6.5$ mm made of low-carbon steel was carried out through prefabricated double dies made of BK-6 alloy on a chain mill at a speed of 0.2 m/s for 4 passes along the corresponding routes: $6.2 \rightarrow 5.9 \rightarrow 5.45 \rightarrow 5.05$ mm (partial drawing elongation μ_i were $1.10 \rightarrow 1.10 \rightarrow 1.17 \rightarrow 1.16$).

Sodium soap powder was used as a technological lubricant.

On the surface of the workpiece, longitudinal and transverse (relative to the wire rod axis) notches with a depth $h_0 = 2.5, 1.6, 1.2$ and 1.0 mm and a size of $2.6\text{--}6.5$ mm in the direction perpendicular to the drawing axis were applied by milling. Another size of the notches was 1 mm. This was significant in the subsequent determination of the weakening of the wire profile at the exit from the drawing.

The notches were placed along the axis of the workpiece in 4 groups (according to the number of passes, respectively). The distance between the notches was at least of 10 mm, and between their groups, it was of 100 mm (Fig. 1) to prevent the mutual influence of these defect models during metal deformation.

From the wire rod and after each i -th pass from the wire rear end with a diameter d_{1i} , sections with a 'defects' group were cut off, then

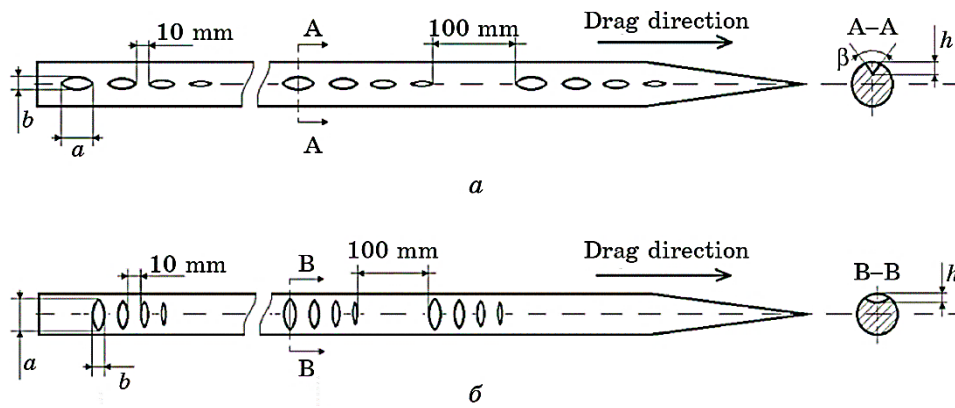


Fig. 1. Scheme of preparation of wire rod samples with groups of longitudinal (a) and transverse (b) surface notches (shown for three passes) [11].

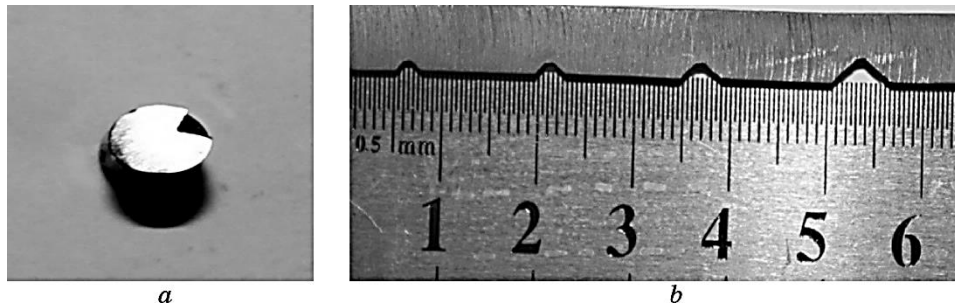


Fig. 2. Examples of deformed wire sections photographs: transverse with a longitudinal 'defect' applied (a), longitudinal with transverse 'defects' applied (in comparison with a fragment of the tool ruler) (b) [11].

each of them with the surrounding metal zone was separated, marked, cut at the place of their maximum depth and perpendicular to the wire axis, polished and photographed with a digital camera (Fig. 2).

The accuracy of such measurements was limited to visual determination of dimensions (within 0.5 mm). It became possible to increase the accuracy of measurements by transferring a digitized image of defects to a computer monitor screen.

The transition from enlarged computer images to the corresponding actual 'defects' sizes was carried out taking into account the 'conversion factor':

$$K_p = \frac{l_{\text{inst}}}{l_{\text{comp}}} < 1, \quad (21)$$

where l_{inst} , l_{comp} are respectively, the results of instrumental measure-

ment of the dimensions of the product in an accessible place (for example, the diameter d_0 of the wire rod using a micrometres with an accuracy of ± 0.01 mm) and measurements of the dimensions of the computer display of the same place using a tool ruler.

The regularities of deformation of surface defect models depending on the values of total drawing elongation $\mu_\Sigma = (d_0/d_i)^2$, drawing elongation $\mu_i = (d_{i-1}/d_i)^2$ in the i -th pass and (or) the corresponding change in the perimeter of the wire cross-section $\sqrt{\mu_i} = d_{i-1}/d_i$, as well as the number of compressions (passes) were judged by the change in the measured depth h and size b of the notch in the direction perpendicular to the drawing axis before (index ' $i-1$ ') and after (index ' i ') deformation in the pass (see cross-sections A-A and B-B in Fig. 1).

Accordingly, the ratio h_{i-1}/h_i reflected the intensity of the decrease in the depth of the 'defect' with increasing μ_Σ and μ_i ; an increase in the ratio $K_{1i} = b_{1i}/h_{1i}$ corresponded to the relative expansion (smoothing) of the 'defect', and a decrease K_{1i} —to its narrowing, *i.e.* the tendency to transform the notch into a longitudinal 'crack') after drawing in the next i -th pass. The ratio $K_{2i} = b_i d_{i-1}/(b_{i-1} d_i)$ also assessed the synchronicity of the change in the width of the 'defect' and the perimeter (diameter) of the wire. The value $K_{2i} = 1$ corresponded to the synchronous change in the width of the 'defect' and the perimeter of the product; the value $K_{2i} < 1$ corresponded to the tendency to transform the notch into a longitudinal 'crack', and $K_{2i} > 1$ —to the tendency to smooth the 'defect' relative to the perimeter (diameter) of the wire, which decreased in successive drawing passes. Additionally, it was possible to estimate the angle at the apex of the notch: $\beta = 2\arctg(b_i/2h_i)$ (Fig. 1).

The results obtained in the investigation [11] indicated the following.

With a longitudinal (relative to the workpiece axis) orientation of the notches (see Fig. 1, *a*), with increase μ_Σ the depth h_i and width b_i of the 'defect' decreased intensively, mainly in the first two passes, the more so, the larger their initial values were h_0 and b_0 on the wire rod, and then at $\mu_\Sigma > 1.2$ – 1.4 the intensity of their change decreased, and they asymptotically approached in magnitude.

Important here was the decrease in the magnitude K_{1i} and corresponding angle β_i with increasing extraction μ_Σ , which indicated a tendency for such a 'defect' to transform into a longitudinal 'crack' the more intense the greater the extraction μ_Σ and the smaller the initial depth h_0 of the 'defect'.

The non-monotonic nature of the change $K_{2i \text{ prod}} < 1$ from μ_Σ in the longitudinal orientation of the defects reflected the complex influence of strengthening and the stress state scheme of the metal in the deformation centre near the notch during wire deformation and, in general, confirmed the tendency for the longitudinal notch to transform into a 'crack'. This dependence was probably due to the complex influence of

the metal deformation conditions on the metal flow.

With the transverse orientation of the notches on the workpiece relative to the axis (see Fig. 1, *b*), a monotonic decrease in the depth h_i and width b_i of the ‘defects’ was also observed with the increase in μ_Σ . But in this case, the magnitude K_{1i} and the corresponding angle β_i values increased, which indicated a tendency to smooth out such ‘defects’. This was also consistent with the values $h_{i-1}/h_i > 1$ in the entire investigated range of the increase in the parameter $\sqrt{\mu_i} = d_{i-1}/d_i$. In this case, a more intensive smoothing of the transverse ‘defect’ became important in comparison with the decrease in the wire cross-sectional perimeter.

Also, in Ref. [11], the influence of surface defects of longitudinal and transverse orientation relative to the wire axis on the stability of the drawing process in the i -th passes was estimated using geometric relations by the coefficient value according to formula (7).

Accordingly, for the longitudinal orientation of the defect when considering the method of notches applying to the workpiece, its cross-sectional area was represented by a sector with width b_i , depth h_i and angle $\beta = 2\arctg(b_i/2h_i)$ under the segment (see view A–A in Fig. 1, *a*). Then, according to Ref. [18],

$$F_{\text{def.long},i} = \frac{r_i^2(\beta_i - \sin \beta_i)}{2}, \quad (22)$$

where $r_i = (h_i^2 + b_i^2/4)^{1/2}$, which after transformations gave:

$$F_{\text{long,seg},i} = \frac{1}{2}(h_i^2 + \frac{b_i^2}{4})(\beta_i - \sin \beta_i),$$

and according to (7) and taking into account $d_{1i} = 2r_i$, we obtained:

$$K_{\text{CID,long}} = \frac{1}{1 - (2h_i^2/(\pi d_{1i}^2) + b_i^2/(2\pi d_{1i}^2))(\beta_i - \sin \beta_i)}, \quad (23)$$

Similarly, for the defect transverse orientation on the wire, its cross-sectional view, which is perpendicular to the drawing axis, taking into account the notches method of applying to the workpiece, was approximately represented by a segment (see view B–B in Fig. 1, *b*). The area of this figure is determined by the formula (18) [18]:

$$F_{\text{def.tran},i} = \frac{r_i^2(\beta_i - \sin \beta_i)}{2}, \quad \beta_i = 2 \arccos((d_{1i} - 2h_i)/d_{1i}),$$

and, after simple transformations, we obtained:

$$K_{\text{CID,tran}} = \frac{1}{1 - (\beta_i - \sin \beta_i)/(2\pi)}, \quad (24)$$

Calculation according to formulas (23) and (24) showed that the impact K_{def} on the stability of the drawing process is most significant in the first passes, which requires, according to equation (4), an increase in the limiting safety factor according to formula (1) and a corresponding decrease by 5–30% of the drawing elongation μ_i .

Evaluation of the information presented above determined the feasibility of conducting additional investigations to identify deformation patterns in comparable conditions of surface defects with the same initial orientation of the axes relative to the rod axis and their influence on the condition (4) of continuous drawing. Also, the formula for estimating the coefficient K_{def} was subject to appropriate definition. This became the aim of the following experiments and analysis.

3. RESEARCH SECTION

To achieve the set purpose, we used 5 samples of wire rod, prepared in the same way for drawing according to the method presented in Ref. [11] and shown above.

On each wire rod sample made of low-carbon steel, groups of conical non-through holes (Fig. 3, *a*) with a depth $h_0 = 3.0$ mm and dimensions $b_0 = a_0 = 6.0$ mm; $h_0 = 2.0$ mm and $b_0 = a_0 = 4.0$ mm; $h_0 = 1.0$ mm and $b_0 = a_0 = 2.0$ mm were applied with a drill with an angle at the apex of 90°. Such models conditionally reflected defects: scale detachment, non-metallic surface inclusions, small shells, *etc.*, better than those ones oriented only along the axis or perpendicular to the drawing axis.

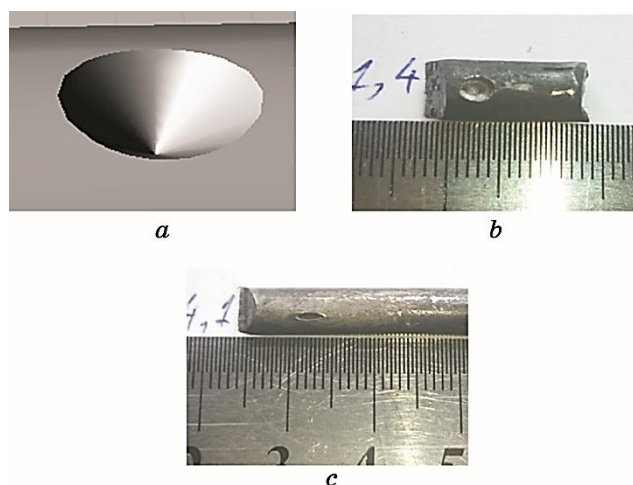


Fig. 3. General view: conical model of the surface ‘defect’ (magnification $\times 10$) (*a*), wire fragment after the first pass with a deformed defect (magnification $\times 2$) (*b*), the same after the fourth pass (*c*).

Prepared samples of wire rod with applied conical ‘models’ of surface defects were deformed on a chain drawing mill under the conditions presented above.

Then, after each i -th pass, a section with the next deformed ‘defect’ was cut from the rear end of the sample with a diameter d_{1i} (Fig. 3, *b*, *c*). Then, each of them was cut perpendicular to the axes of the wire and the ‘defect’ into templates, polished, photographed with a known magnification using a digital camera and transferred to the PC screen. The characteristic dimensions of the ‘defects’ were measured with an instrumental ruler of the second accuracy class with a division value of 0.5 mm and the results of similar measurements were averaged.

The negative impact of image boundaries blurring with increasing computer magnification on measurement accuracy was taken into account using the conversion factor K_κ , which was determined by the relationship:

$$K_\kappa = 10/N, \quad (25)$$

where N is the number of pixels per 10 mm of the template (photographed together with the samples).

This approach allowed us to reduce the relative error in measurements of the geometric parameters of ‘defects’ and the wire to 2%. Strictly speaking, the results obtained in such discrete investigations should be presented as bar charts. However, for the convenience of comparing the newly obtained data with similar ones, they were presented as lines on graphs that displayed the trends in the change of characteristic indicators during the deformation of surface defects depending on the values of the total drawing elongation $\mu_\Sigma = (d_0/d_i)^2$ in a similar way as in Ref. [11].

The analysis determined the next. Absolute values of depth h_i and width b_i (taking into account that the initial round base of the cylindrical ‘defect’ on the wire surface after the first drawing pass transformed into an elliptical figure with a width b_i and length a_i that changed in successive i -th drawing passes of the wire rod and wire (Fig. 4). The ratio h_{i-1}/h_i , which reflected the intensity of the decrease in the depth of the ‘defect’ with an increase in μ_Σ , the ratio b_i/h_i , which was consistent with the change in the angle β_i and reflected the tendency to smooth out or transform the conical ‘defect’ into a crack; the ratio $K_{2i} = b_i d_{i-1}/(b_{i-1} d_i)$ also assessed the synchronicity of the change in the width of the ‘defect’ with the perimeter (diameter) of the wire (Fig. 5).

4. DISCUSSION OF RESULTS

Analysis of the presented data indicated the following. In the presence of conical models of ‘defects’ on the wire rod with a change in the total

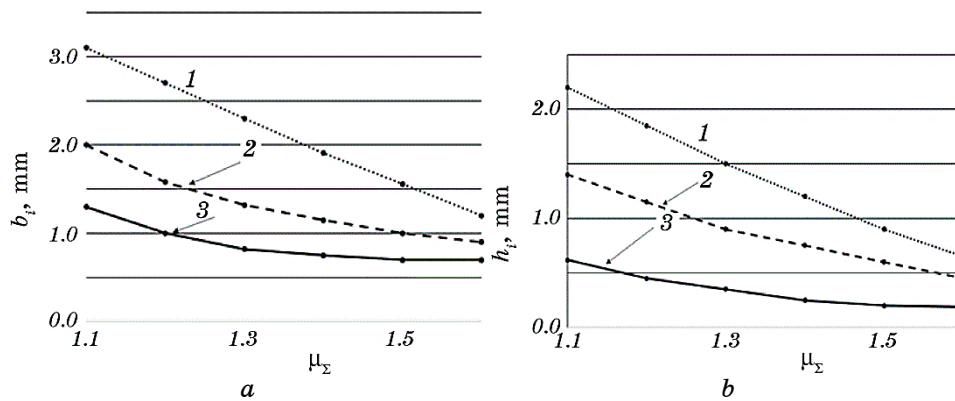


Fig. 4. Dependence of the total extraction depth (a) and width (b) of conical models of 'defects': 1— $h_0 = 3.0$ mm and $b_0 = a_0 = 6.0$ mm, 2— $h_0 = 2.0$ mm and $b_0 = a_0 = 4.0$ mm, 3— $h_0 = 1.0$ mm and $b_0 = a_0 = 2.0$ mm.

draw over several passes during drawing, the width b_i and depth h_i of the defect on the wire monotonically decreased the more intensively the larger these initial parameters on the wire rod were.

Qualitatively, this was consistent with the corresponding data of work [11] and the results of calculations using formulas (8)–(17). At the same time, a noticeable manifestation of the volumetric scheme of deformations and stresses was detected, as a result of which the quantitative data differed. In particular, the intensity of the change $h_{i-1}/h_i > 1$ and $b_i/h_i > 1$ indicated a positive trend in the smoothing of defects, the greater the larger μ_Σ was (see Fig. 4). To a greater extent, this was consistent with the 'behaviour' of transverse defects (see Ref. [11]). The values $K_{2i} = b_i d_{i-1} / (b_{i-1} d_i) \cong 1$ indicated an approximate synchrony of the change in the width of the 'defect' and the perimeter (diameter) of the wire (see Fig. 5), which was the largest at larger values h_0 , b_0 and decreased with a decrease in these parameters from μ_Σ indicates the influence of a predominantly volumetric scheme of metal deformation around the conical defect, in contrast to predominantly linear schemes with its longitudinal or transverse orientation relative to the drawing axis.

In further analysis, the influence of surface conical defects of the workpiece on the stability of the process of its multiple drawing by changing the geometric parameters in the drawing process, similar to previous investigations [11], was estimated by the value of the defect coefficient according to formula (7). It was taken into account that the geometric 'weakening' of the wire profile at the exit from the drawing was due to the corresponding sizes b_i , h_i and d_{1i} in the cross-section of the 'defect' and the wire.

In Figure 6, *a*, the cross-section of the conical defect model is repre-

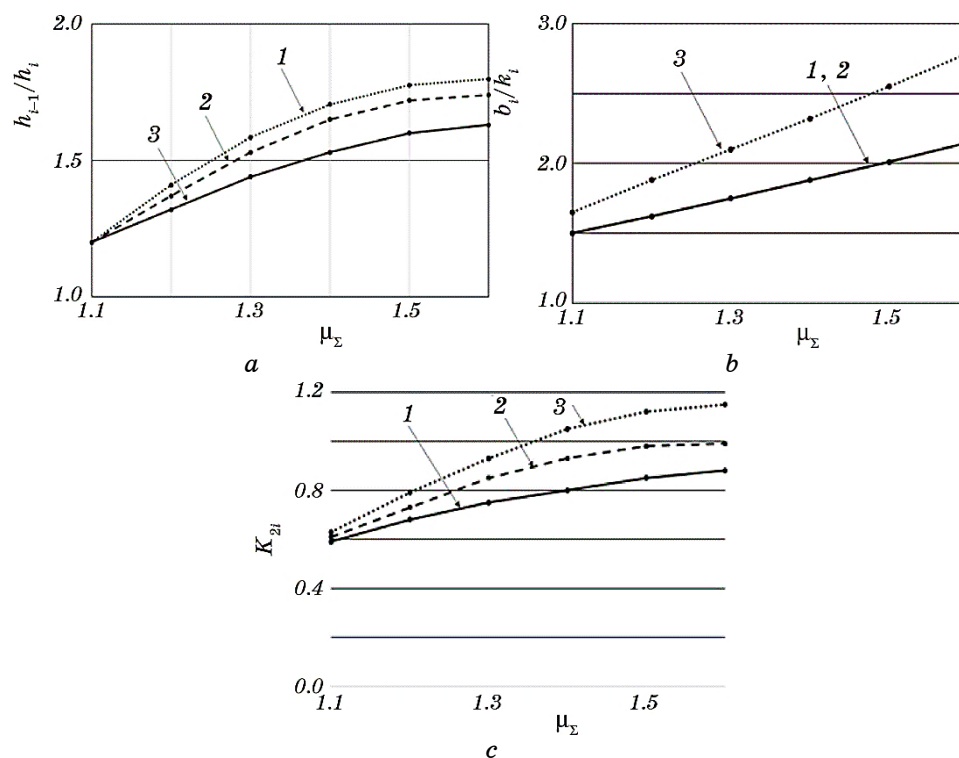


Fig. 5. Dependence of the intensity of the change in depth (a), relative width (b) and synchronicity of the change in the width of the 'defect' with the perimeter (diameter) of the wire (c) on the total draw for conical models of 'defects': 1— $h_0 = 3.0$ mm and $b_0 = a_0 = 6.0$ mm, 2— $h_0 = 2.0$ mm and $b_0 = a_0 = 4.0$ mm, 3— $h_0 = 1.0$ mm and $b_0 = a_0 = 2.0$ mm.

sented by a curvilinear triangle AKBC with width b_i , depth h_i and angle β_i under the corresponding arc AKB, on which the central angle α_i of the circle corresponding to the cross-section of the wire also rests. The area of the 'defect' consists of the sector AKB of the circle with diameter d_{1i} and the triangle ABC: $F_{\text{def.i.con}} = F_{\text{AKB}} + F_{\text{ABC}}$.

With this representation according to Ref. [18],

$$F_{\text{AKB}} = \frac{r_i^2 (\alpha_i - \sin \alpha_i)}{2},$$

where $r_i = d_{1i}/2$, angle $\alpha_i = 2\arcsin(b_i/d_{1i})$ in radians, $F_{\text{ABC}} = 0.5h_i b_i$, and after simple transformations taking into account (7) we obtained:

$$K_{\text{CID.con}} = \frac{\pi d_{1i}^2}{\pi d_{1i}^2 - 0.5 d_{1i}^2 (\alpha - \sin \alpha) - 2 h_i b_i}. \quad (26)$$

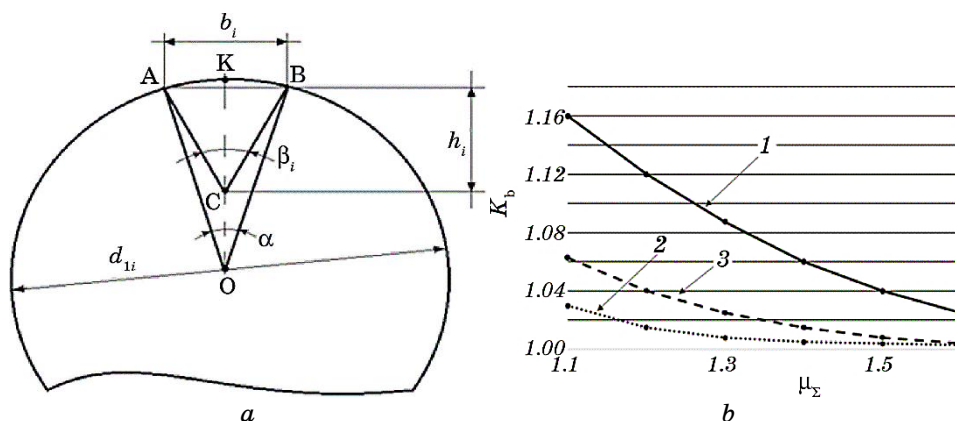


Fig. 6. Scheme for determining the area of the conical defect model in the cross-section of the wire at the exit from the drawing (*a*) and the dependence of the defect coefficient on the metal extraction during drawing (*b*): 1— $h_0 = 3.0$ mm and $b_0 = a_0 = 6.0$ mm, 2— $h_0 = 2.0$ mm and $b_0 = a_0 = 4.0$ mm, 3— $h_0 = 1.0$ mm and $b_0 = a_0 = 2.0$ mm.

Next, using the experimental values of b_i , h_i and d_{1i} (see Fig. 4), we determined the dependence of the parameter $K_{\text{CID.con}}$ on μ_Σ (Fig. 6, *b*).

The obtained data showed that, in agreement with the tendency to smooth out conical defects (see Figs. 4 and 5), their negative impact on the stability of the drawing process decreased with decreasing wire diameter, the deeper the initial ‘defect’ was. At the same time, the absolute values $K_{\text{CID.con}}$ were the smallest at small values h_0 . This, to some extent, is consistent with the data presented in Ref. [11] and calculations using formulas (18) and (19). The quantitative difference can be explained by the significant influence of the stress–strain state of the metal around the defect on its formation, while maintaining the recommendation regarding the need to weaken the deformation regime, *i.e.*, the values of μ_i , along the entire drawing route, the greater the depth of the defect and the value of μ_Σ , which is consistent with production practice.

The results of the research also showed that drawing of wire with surface conical defect models is accompanied by an increase in their longitudinal dimensions relative to the wire axis. At the same time, a difference was recorded between the relative elongation of the linear dimensions of defects closer to the axis and on the wire surface: on the surface, they were larger by 15–40%. The latter indicated the uneven deformation in the drawing of both the wire and the defects on it.

It should be noted that the presence of cone-shaped defects on the wire surface, as well as defects with a transverse location relative to the drawing axis, forms a stress concentrator for the acting longitudi-

nal drawing force. This can affect additionally negatively the stability of continuous drawing, which requires an even greater reduction in the deformations μ_i in the gaps and changes the right-hand side of inequality (4):

$$\gamma = \frac{\sigma_{B1}}{\sigma} \geq K_{\sigma} K_{\text{def}} [\gamma], \quad (27)$$

where $1 < K_{\sigma} < 3$ is the technical stress concentration coefficient for the conditions of plastic deformation of the material [19].

It can be argued that the preferred longitudinal orientation of the defect relative to the tensile force vector during drawing is less sensitive to stress concentration.

The obtained data may be useful in developing models of a stable (continuous) wire rod and wire drawing process.

5. CONCLUSIONS

Using the developed method, the deformation regularities of artificial models of cone-shaped defects on wire rod and wire during frequentative drawing were experimentally determined for the first time. It was found that, in most cases, the smoothing of such defects occurs more intensively, the greater the total wire drawing and the initial values of the depth and width of the defects.

The influence of defects on the condition of stable (unbroken) wire drawing is determined. The data are compared with the results of previous similar investigations on wire deformation in the presence of defects of predominantly transverse and predominantly longitudinal orientation relative to the drawing axis. It is shown that the qualitatively obtained new results are closer to the regularities of deformation of ‘transverse’ defects, and the quantitative differences can be explained by the presence of a volumetric scheme of the stress–strain state of the metal around the defect.

Attention is drawn to the probable additional influence of conical and ‘transverse’ defects as stress concentrators on the condition of continuous drawing, which is also advisable to take into account when designing routes for multiple deformation of the workpiece to obtain drawn wire of acceptable quality. A promising direction of research is to combine the data of existing work with known methods for calculating force conditions of deformation and thorough design of routes for multiple stable wire drawing.

REFERENCES

1. O. M. Ikumapayi, E. T. Akinlabi, P. Onu, and O. P. Abolusoro, *Mater. Today*

- Proc.*, **26**, Pt. 2: 1644 (2020).
2. G. Gottstein, M. Goerdeler, and G. V. S. S. Prasad, *Encyclopedia of Condensed Matter Physics* (Elsevier: 2005), p. 298.
3. Ya. Vasilev, D. Samokish, O. Bondarenko, and N. Mospan, *Sci. Innovation*, **18**, No. 3: 49 (2022).
4. R. Matthias, *Encyclopedia of Energy* (Elsevier: 2004), p. 695.
5. P. Montmitonnet, Y. K. Khalfalla, and K. Y. Benyounis, *Reference Module in Materials Science and Materials Engineering* (Elsevier: 2016), p. 5500.
6. O. M. Anisimova, *Naukovi Zapysky Natsional'noho Universytetu 'Ostroz'ka Akademiya'. Seriya 'Ehkonomika'*, **16**: 435 (2011) (in Ukrainian).
7. V. V. Venger and V. M. Osypov, *Ehkonomika i Prohnozuvannya*, No. 3: 69 (2017) (in Ukrainian).
8. T. Obolenska and D. Dovhan, *Visnyk L'vivs'koho Universytetu. Seriya 'Mizhnarodni Vidnosyny'*, **36**, Pt. 3: 169 (2015) (in Ukrainian).
9. L. Y. Jiang, *Membrane-Based Separations in Metallurgy* (Eds. L. Y. Jiang and N. Li) (Elsevier: 2017), p. 3.
10. A. M. Dolzhanskyi, O. S. Maksakova, K. O. Chornoivanenko, T. A. Ayupova, N. V. Polyakova, O. A. Bondarenko, and I. M. Lomov, *Tekhnichne Rehulyuvannya ta Kontrol' na Pidpryyemstvi. Vol. 2. Tekhnolohiyi ta Defekty Produktiyi Metalurgiyi* [Technical Regulation and Control at the Enterprise. Vol. 2. Technologies and Defects of Metallurgical Products] (Dnipro: Svidler A. L.: 2023) (in Ukrainian).
11. A. M. Dolzhanskyi, Ye. O. Petliovanyi, and K. L. Suslova, *Metalurgiyna ta Hirnychorudna Promyslovist'*, No. 1: 60 (2014); *ibidem*, No. 2: 39 (2014) (in Ukrainian).
12. J. Švejcár, M. Juliš, L. Klakurková, P. Gejdoš, and T. Zikmund, *Defect Diffusion Forum*, **405**: 217 (2020).
13. H. M. Baek, Y. Gwan, S. K. Hwang, Y.-T. Im, and I.-H. Son, *J. Mater. Processing Techn.*, **212**, Iss. 4: 776 (2012).
14. K. Yoshida, S. Norasethasopon, T. Shinohara, and R. Ido, *JSME Int. J. Series A Solid Mechanics Mater. Eng.*, **46**, Iss. 3: 365 (2003).
15. X.-S. Yao, Y.-D. Liu, and L. Zuo, *J. Northeast. Univ.*, **29**, No. 3: 332 (2008).
16. A. M. Dolzhanskyi, Ye. O. Petliovanyi, and I. M. Lomov, *Metalurgiyna ta Hirnychorudna Promyslovist'*, No. 4: 58 (2008) (in Ukrainian).
17. A. M. Dolzhanskyi, E. A. Petlevanyy, I. N. Lomov, O. B. Lomova, and O. S. Ermakova, *Udoskonalennya Protsesiv i Obladnannya Obrobky Tyskom v Metalurgiyi i Mashynobuduvanni* [Improving Processes and Processing Equipment by Pressure in Metallurgy and Mechanical Engineering] (Kramatorsk: 2008), p. 198 (in Ukrainian).
18. T. Yu. Kepych and O. H. Kutsenko, *Osnovy Teoriyi Podibnosti ta Analizu Rozmirnostey v Mekhanitsi* [Fundamentals of Similarity Theory and Dimensional Analysis in Mechanics] (Kyiv: Taras Shevchenko National University of Kyiv: 2004) (in Ukrainian).
19. G. G. Pysarenko, O. V. Voinalovych, A. V. Byalonovych, A. M. Mailo, and I. V. Lymanskyi, *Strength Mater.*, **56**: 1108 (2024).

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Роль пластичності в руйнуванні металів і сплавів

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В оглядовій статті механічна властивість пластичності розглядається під кутом зору впливу її на крихкість металевих матеріалів через втручання в механічний напружено-деформований стан (НДС) металевого тіла (МТ) під силовим навантаженням: поява пластичності обмежує пружний вид НДС на межі плинності МТ $\sigma_{0,2}$ і породжує пружно-пластичний вид НДС в межах напружень від $\sigma_{0,2}$ до S_K (істинне напруження руйнування зразка у «шийці»). Інтервал напружень $\Delta S = S_K - \sigma_{0,2}$ слугує резервом механічної стабільності (РМС) напруженого стану МТ з показником РМС у вигляді коефіцієнта $B_r = S_K / \sigma_{0,2}$ (зламостійкість МТ), що забезпечує пластичному МТ ресурс захисту від крихкого руйнування за $B_r > 1$. Пластичність породжує у МТ спосіб для захисту від швидкого руйнування — крихкості. Показник зламостійкості B_r слугує параметром кількісного виміру здатності МТ чинити опір руйнуванню МТ взагалі, в тому числі і під дією концентраторів напружень (КН) (надрізів, тріщин, тощо). В цій статті наведено результати аналізу експериментів з літературних джерел щодо ролі показника B_r (зламостійкості) в процесах руйнування конструкційних крих на зразках із КН за кімнатної та низьких температур випробувань.

Ключові слова: міцність, стабільність міцності, зламостійкість, перенапруження, концентратор перенапружень, крихкість, критичний параметер крихкості, субкритичне та надкритичне руйнування, захисний ре-

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курс пластичности.

In the review paper, the mechanical property of plasticity is considered from the standpoint of its effect on the brittleness of metallic materials due to influence on the mechanical stress-strain state (SSS) of a metal under a force load: the appearance of plasticity limits the elastic form of the SSS at the yield point of a metal $\sigma_{0.2}$ and generates the elastic-plastic form of the SSS within the stress range from $\sigma_{0.2}$ to S_K (the true fracture stress of a sample in 'neck'). Stress interval $\Delta S = S_K - \sigma_{0.2}$ is a mechanical-stability margin (MSM) of the stress state of a metal with a numerical characteristic of MSM in the form of a coefficient $B_r = S_K / \sigma_{0.2}$ (break resistance), which provides the ductile metal with a protection margin against brittle fracture at $B_r > 1$. Plasticity creates within the metal a means to protect against rapid destruction (brittleness). The break-resistance coefficient B_r is a parameter for quantitative measurement of the metal ability to resist fracture of a metal in general, including under the action of stress raisers (SR) (notches, cracks, *etc.*). This paper presents the results of the analysis of experiments from literary sources on the role of break-resistance coefficient B_r in the processes of fracture of structural steels for specimens with stress raisers at both room and low test temperatures.

Key words: strength, strength stability, break resistance, overstrain, overstrain concentrator, brittleness, critical parameter of brittleness, subcritical and supercritical fracture, protective resource of plasticity.

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1. ВСТУП

Властивість пластичності притаманна металевим матеріалам завдяки особливостям металічного типу міжатомових зв'язків, зумовлених наявністю в металах так званого «електронного газу» [1]. Пластична деформація починається за критичного напруження умовної межі плинності $\sigma_{0.2}$, що залежить від структури полікристалічного стопу, температури й динаміки навантаження [2] та завершується відривом після досягнення енергетичної нестабільності деформованої структури металу за напруження S_K (істинне напруження руйнування у «шийці» зразка) [3, 4]. Отже, в металевих матеріалах руйнування без початку пластичної деформації в принципі неможливе; тому закономірно постає питання про фізичну сутність тієї ролі, яку пластичність відіграє в явищі руйнування взагалі і в крихкому виді руйнування зокрема.

В традиційній методології дослідження крихкості металевих матеріалів широко використовують концепцію енергоємності руйнування за динамічного навантаження на вигин спеціальних призматичних зразків із надрізом певної гостроти з визначенням роботи руйнування («ударної в'язкості») KCV (надріз типу Шарпі) або KCU (надріз Менаже) [5]. Але з точки зору ролі пластичності в показниках ударної в'язкості KCV і KCU [Дж/м²] така методологія не

є адекватною, оскільки в показнику роботи руйнування вплив самої пластичності істотно притіняється впливом показника міцності ($\sigma_{0,2}$), особливо у високоміцних стопах, в результаті чого критерій крихкості у такій методології доводиться коригувати у відповідності до класу міцності стопу [6].

В даній роботі поставлену задачу пропонується розглядати, виходячи з уявлень про стан механічної стабільності напруженого металевго тіла (МТ), закладених у попередніх роботах авторів цієї концепції [7–11]. В концепції механічної стабільності роль пластичності у руйнуванні металів однозначно відображається у спеціальному показнику резерву (ресурсу) стабільності пластичного стану (РСПС) (зламостійкості металу) B_r [8]:

$$B_r = S_K / \sigma_{0,2}; \quad (1)$$

S_K — істинне напруження руйнування у «шийці» зразка, $\sigma_{0,2}$ — умовна межа плинності зразка за залишкової деформації $\varepsilon_{0,2} = 0,002$.

2. МЕХАНІЧНА СТАБІЛЬНІСТЬ МЕТАЛІВ

Механічна стабільність тіла, що знаходиться під дією прикладених сил, означає стійке збереження діючого деформованого стану (напружено-деформованого стану — НДС) тіла в процесі його подальшого навантаження, яке закінчується або руйнуванням тіла (крихкість твердих тіл), або переходом до пластичної деформації (металеві матеріали). У металевих матеріалах стабільність механічного стану порушується двічі: спочатку порушується стабільність пружного стану тіла на умовній межі плинності ($\sigma_{0,2}$), вдруге стабільність пластичного (насправді пружньо-пластичного) НДС порушується у вигляді руйнування тіла за напруження S_K в результаті енергетичної нестабільності деградованої через пластичну деформацію структури металу [3]. Енергетична стабільність структури, заповненої масою субмікроскопічних зародкових тріщин (ЗТ) [3, 4], порушується за принципом, встановленим А. А. Гріффітом в моделі руйнування твердого тіла з еліптичною порожниною, що діє як концентратор напружень (КН) [12].

Фізичною причиною порушення стабільності пластичного деформованого стану (ПДС) металевих тіл є накопичення пружної енергії в результаті деформаційного зміцнення металу, коли за напруження S_K відбувається розрив міжатомових зв'язків зі спонтанним вивільненням накопиченої пружної енергії тіла, тобто повна ліквідація стабільності ПДС з одночасним припиненням пластичного деформування МТ і фіксацією показника пластичності зразка ψ_K .

Другий рівень механічної стабільності, що проявляється в інтервалі напружень $\Delta S = S_K - \sigma_{0,2}$, в попередніх роботах визначався як

деформаційний резерв міцності у вигляді коефіцієнта B_r (коефіцієнт зламостійкості) за формулою (1) [8–11]. Оскільки резерв міцності є наслідком прояву пластичності металу, то за своїм фізичним змістом величину ΔS і похідну від неї B_r можна трактувати як певний ресурс стабільності пластичного стану, який віддаляє момент енергетичної нестабільності деформаційно деградованої структури металу (момент руйнування за S_K) від можливого швидкого руйнування на межі плинності $\sigma_{0,2}$.

Отже, параметер B_r за (1) представляє собою кількісний вимір захисного ресурсу стабільності пластичного стану або захисний ресурс пластичності (ЗРП) в процесі руйнування металу взагалі і в крихкому руйнуванні зокрема. Зламостійкість B_r своєю величиною відображає міру захисту металу від крихкості, що стало предметом систематичних досліджень в роботах [8–11, 13–16]. Зрештою, саме в рамках концепції механічної стабільності в роботі [11] було представлено механізм руйнівальної дії КН як результат повного вбирання ЗРП (B_r) в зоні пластичності в околі вістря КН, що призводить до передчасного руйнування тіла з КН за номінальних напружень $\sigma_{NF} < S_K$ і до крихкого руйнування за $\sigma_{NF} < \sigma_{0,2}$ (рис. 1).

В роботі [14] роль показника зламостійкості B_r розглядалася в контексті впливу базових механічних властивостей (міцності та пластичності) на номінальне напруження руйнування σ_{NF} на зразках з КН різного типу у надрізів або тріщин втоми. Було показано, що застосування зламостійкості B_r уможливорює однозначно прогнозувати умову переходу до крихкого руйнування зразка з даним видом КН, якщо попередньо експериментально було визначено руйнівальну силу α_{efc} для тестового КН у вигляді критичного показника B_{rc} , за якого тестовий КН реалізує руйнування на рівні межі плинності металу $\sigma_{NF} = \sigma_{0,2}$ (рис. 2):

$$\alpha_{efc} = B_{rc} = (S_{KC}/\sigma_{0,2C}) \Big|_{\sigma_{NF}=\sigma_{0,2}}. \quad (2)$$

Умова (2) означає, що знайдено такий критичний для даного виду КН рівень $B_r = B_{rc}$, за якого весь ЗРП цілком вбирається у зоні пластичності КН і в результаті реалізує стан крихкості металу за номінального (середнього) напруження $\sigma_{NF} = \sigma_{0,2}$. Тим самим виявляється можливість відмічати перехід до крихкості МТ за механічною характеристикою B_r , сформованою з наперед відомих показників механічних властивостей металу — $\sigma_{0,2}$ і S_K , що мають бути заданими вже в його сертифікаційних показниках і не потребують додаткового визначення (рис. 2). Такий критичний показник B_{rc} є індивідуальною характеристикою кожного типу КН, але не сталою константою для геометрії даного КН, як, наприклад, Нейберів коефіцієнт концентрації напружень α_σ , а жорстко пов'язаною з наявним рівнем B_r стійкою кореляційною залежністю у вигляді співвід-

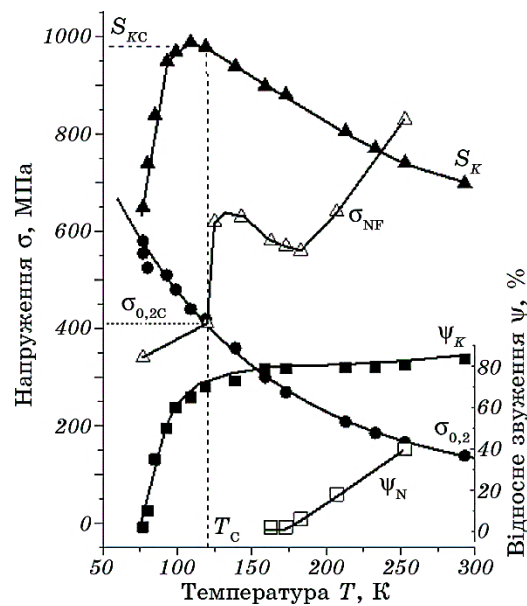


Рис. 1. Температурні залежності механічних властивостей α -Fe для гладких зразків ($\sigma_{0,2}$ — межа плинності, S_K — істинне напруження руйнування, ψ_K — відносне звуження за розриву у шийці) і зразків з тріщиною втоми за триточкового вигину (σ_{NF} — середнє номінальне напруження руйнування в місці руйнування зразка, ψ_N — відносне звуження в нетто-перерізі зразка), $\sigma_{0,2C}$, S_{KC} — значення відповідних міцностей за критичної температури T_c [17].

Fig. 1. Temperature dependence of the mechanical properties of α -Fe for unnotched specimens ($\sigma_{0,2}$ is yield strength, S_K is true fracture stress, ψ_K is reduction in area, when breaking) and specimens with a fatigue crack under three-point loading (σ_{NF} is the average nominal fracture stress of specimen, ψ_N is reduction in area of the specimen net cross-section), $\sigma_{0,2C}$, S_{KC} are the values of corresponding strengths at critical temperature T_c [17].

ношення $B_r/B_{rC} = f(B_r)$ [16]:

$$B_r / B_{rC} = 0,806 + 0,194 B_r \quad (3)$$

з коефіцієнтом кореляції $R = 0,71$. Рівняння (3) відображає зв'язок між B_r і B_{rC} для руйнування зразків у вигляді призматичних балок товщиною $t = 18$ мм з нанесеною тріщиною втоми за триточкового вигину [17]. Для циліндричних зразків з кільцевим надрізом [18] кореляційне співвідношення B_r/B_{rC} виконується з більш високим коефіцієнтом кореляції $R = 0,99$ [16]:

$$B_r / B_{rC} = 0,436 + 0,915 B_r. \quad (4)$$

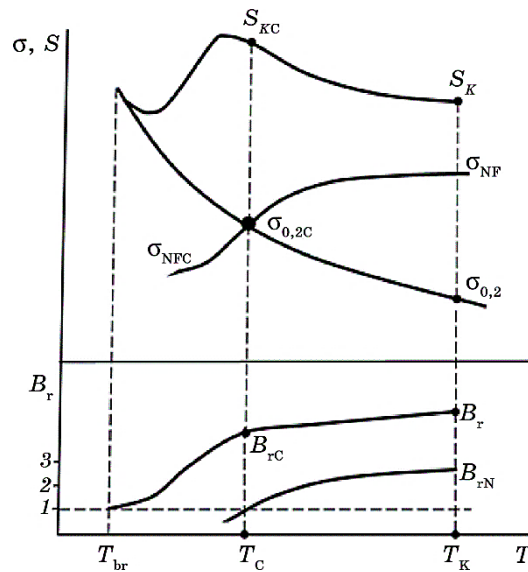


Рис. 2. Температурні залежності базових механічних характеристик $\sigma_{0,2}$, S_K і зламостійкостей для гладких зразків (B_r) і зразків з КН (B_{rN}) (схема).

Fig. 2. Temperature dependences of basic mechanical characteristics $\sigma_{0,2}$, S_K and break resistances for smooth specimens (B_r) and specimens with stress raisers (SR) (B_{rN}) (scheme).

З порівняння залежностей (3) і (4) видно, що для крихких матеріалів ($B_r = 1$) в межах похибки критичний рівень B_{rc} для всіх видів КН практично однаковий: $B_{rc} \cong 1$, тобто у крихкому матеріалі будь-який КН призведе до певної втрати тривкої здатності: $\sigma_{NF} < \sigma_{0,2}$. Але для металів ($B_r > 1$) величина B_{rc} залежить як від типу КН, так і від захисного ресурсу пластичності (B_r).

Аналіза причин такого впливу пластичності на умови руйнування металів під дією КН потребує більш детального розгляду.

3. ДВА ВИДИ МЕХАНІЗМУ ВИЧЕРПАННЯ ЗАХИСНОГО РЕСУРСУ ПЛАСТИЧНОСТІ ЗА ОКРИХЧЕННЯ МЕТАЛІВ

Захисний від крихкості ресурс пластичності, зосереджений в показнику зламостійкості B_r , може бути ввібраним до рівня $B_r = 1$, коли метал переходить від пластичного до крихкого стану, двома принципово різними способами — шляхом підвищення «межі плинності» початкової недеформованої структури металу $\sigma_{0,2}$, тобто за рахунок зміни нижньої межі резерву стабільності B_r за (1), або шляхом пониження верхнього рубежу пластичного стану — напруження руйнування S_K . Напруження початку ($\sigma_{0,2}$) і кінця (S_K)

ресурсу пластичності незалежні одне від другого, різні за своєю фізичною природою і тому змінюються за різними закономірностями.

Перший вид механізму вбирання ЗРП (B_r) яскраво проявляється в результаті низькотемпературного зростання $\sigma_{0,2}$ за охолодження криць (рис. 1, 2). Але можливий також механічний вид підвищення $\sigma_{0,2}$ через тристороннє розтягування в умовах складного НДС (СНС) або за структурних змін межі плинності $\sigma_{0,2}$ в процесах старіння, радіаційного опромінення, тощо, а також внаслідок динамічного навантаження зразка (ударні випробування криць).

Взагалі перший вид механізму вбирання ЗРП базується на високій чутливості показника $\sigma_{0,2}$ до впливу чинників: температури, виду НДС, швидкості навантаження, а також структурного стану металу, що змінюється в процесі пластичного деформування. Слід підкреслити, що головна ланка першого виду механізму зосереджена в початковій стадії пластичності ($\sigma_{0,2}$), в той час як другий вид зменшення ЗРП на шляху до крихкості металу регулюється кінцевою стадією пластичності — руйнуванням металу (S_K). Принципова відмінність першої та другої ланок механізму вичерпання ЗРП пов'язана з різною фізичною природою напружень початку пластичності ($\sigma_{0,2}$) і кінця пластичного стану металу (S_K).

Критерій плинності полікристалічного металу базується на суто усередненому (номінальному) напруженні $\sigma_{0,2}$ за залишкової макроскопічної деформації $\varepsilon_{0,2} = 0,2\% = 0,002$ і є за природою силовим (не енергетичним) критерієм [2]. Руйнування, навпаки, є суто локальним, мікроскопічним, навіть наномасштабного виміру явищем [19–21], що спричиняється енергетичною нестабільністю, і тому руйнування набуває характеру спонтанного, автокаталітичного процесу глобального масштабу. Критерій нестабільності у самому зародку руйнування описується в термінах локальних параметрів НДС [3, 4], що важко застосувати в інженерній практиці; тому в параметрі резерву стабільності міцності B_r (1) використовується практично доступна величина S_K — номінальне (середнє) напруження руйнування у шийці зразка, як така, що макроскопічно відображає момент закінчення стадії пластичного деформування.

Але найбільш поширеним на практиці є гібридний варіант вичерпання захисного ресурсу пластичності, що спостерігається за звичайного в'язкого руйнування зразка металу за кімнатних температур. Тут в процесі деформування пластичність одночасно виконує дві протилежні функції — запобігає швидкому руйнуванню металу на межі плинності $\sigma_{0,2}$ (захисна роль пластичності від крихкості металу) й одночасно породжує передумови для закінчення пластичності (передумови руйнування) внаслідок деформаційної деградації полікристалічної структури металу (зародкові тріщини, пори, розлами твердих частинок структури, тощо); накопичення деформаційних пошкоджень елементів структури готує базу для подаль-

шої енергетичної нестабільності та руйнування металу. Деформаційне зміцнення металу забезпечує достатній рівень густини пружної енергії за напруження S_K :

$$W_\varepsilon = S_K^2 / (2E); \quad (5)$$

W_ε означає середній граничний рівень густини накопиченої енергії в об'ємі деформованого тіла в зоні розриву за умовного квазіоднорідного напруження S_K у шийці зразка, де фактично спостерігається певна неоднорідність НДС [22, 23].

Саме такий гібридний механізм вбирання ЗРП реалізується в локальній зоні пластичності поблизу вістря КН (надрізу, тріщини), де критичний рівень номінальної густини пружної енергії $W_{\varepsilon C}$ за номінального напруження в нетто-перерізі надрізаного зразка σ_{NF} складає

$$W_{\varepsilon C} = \sigma_{NF}^2 / (2E), \quad (6)$$

а залишковий резерв стабільності міцності зразка з КН B_{rN} по аналогії з (1) —

$$B_{rN} = \sigma_{NF} / \sigma_{0,2}. \quad (7)$$

4. АНАЛІЗА ЕКСПЕРИМЕНТІВ З ОКРИХЧЕННЯ КРИЦЬ ПІД ДІЄЮ КН В МОДЕЛЮ ЗРП

Вище було з'ясовано, що властивість пластичності металів відіграє головну регульовальну роль у механізмі руйнування як в крихкому, так і у в'язкому варіантах процесу. Але ключовим моментом у проблемі визначення критичних параметрів крихкості є вибір адекватної механічної характеристики, відповідальної за регульовальну роль пластичності. Відразу зазначимо, що найбільш поширена на практиці традиційна методика визначення так званої ударної в'язкості за Шарпі (KCV) або Менаже (KCU) [5], за якою в основу критерію крихкості покладено умовний параметер роботи руйнування зразка з КН, не спроможна адекватно оцінити роль пластичності в руйнуванні, оскільки в критичному показнику критерію крихкості KCV_c або KCU_c не виокремлюється внесок в енергію руйнування роботи пластичної деформації із внеску міцності металу. В результаті вказана методологія потребує певної коректури для критичних показників роботи руйнування відповідно до класу міцності металу [6].

Концепція механічної стабільності, яку представлено в циклі робіт [8–11], ґрунтується на розгляді прямого внеску пластичності в формування параметра зламостійкості (B_r), що безпосередньо відпо-

відає за міру захисту металу від крихкості під дією КН за рахунок створюваного пластичністю певного ресурсу захисту від руйнування.

Далі в короткому огляді буде представлено основний підсумок з результатів застосування концепції захисного ресурсу пластичності для дослідження закономірностей у формуванні критичних параметрів крихкості конструкційних криць в умовах концентрації напружень.

В роботі [16] аналізували результати досліджень в роботах [17, 18], з яких було вираховано параметри ЗРП B_r і B_{rc} для зразків з кільцевими надрізами та тріщинами втоми на певному наборі конструкційних криць (табл. 1). З таблиці 1 видно, що для кожного виду криці є власний індикатор крихкого руйнування за критичної температури T_c у вигляді коефіцієнта холодостійкості K_{cr} , який по

ТАБЛИЦЯ 1. Критичні параметри крихкості криць B_{rc} і K_{cr} для КН різного виду (група 1 — надріз із $D = 8$ мм [18], група 2 — тріщина втоми [17]) в залежності від базової зламостійкості B_r (C_B — вміст Бору).

TABLE 1. Critical parameters of brittleness of steels, B_{rc} and K_{cr} , for stress raisers (SR) of different types (group 1 is notch with $D = 8$ mm [18], group 2 is fatigue crack [17]) depending on the basic break resistance B_r (C_B is boron contents).

№	Криця, оброблення	Тип КН	$\sigma_{0,2}$, МПа	$\sigma_{0,2C}$, МПа	B_r	$B_{rc} (\alpha_{efc})$	K_{cr}
1	Ст. 30	Група 1. К1[18] кільцевий надріз розтягнення	350	800	2,97	1,31	2,27
2	30ХГСА (з. в., 200°C)		1400	1600	1,58	1,5	1,05
3	30ХГСА (ізот. з., 300°C)		1500	1600	1,8	1,45	1,24
4	30ХГСНА (ізот. з., 200°C)		1450	1700	1,77	1,57	1,13
5	30ХГСНА (ізот. з., 300°C)		1170	1400	2,1	1,36	1,54
6	10Х2СВА		1600	1700	1,83	1,59	1,15
7	У8 (з. а., 400°C)	Група 2. К2[17] вигин з тріщиною	1180	1300	1,58	1,54	1,03
8	α -Fe		140	350	5,0	2,77	1,85
9	У8 (відпал)		340	450	2,8	2,15	1,30
10	Ст. 3сп		160	400	3,1	2	1,55
11	10ХСНД		310	420	2,9	2,5	1,16
12	АК35		1027	1100	2,3	2	1,15
13	ЗШ 12ХН2МДФ (легув. Бором $C_B = 0,0$)		640	700	1,97	1,81	1,09
14	ЗШ 12ХН2МДФ ($C_B = 0,001$)		640	820	1,8	1,46	1,23
15	ЗШ 12ХН2МДФ ($C_B = 0,0022$)		640	977	2,0	1,5	1,33
16	ЗШ 12ХН2МДФ ($C_B = 0,004$)		650	968	2,17	1,67	1,30

суті є показником частини залишкової, не увібраної в зоні КН базової зламостійкості B_r за температури T_K :

$$K_{cr} = B_r / B_{rc}. \quad (8)$$

Встановлено, що показник холодостійкості має закономірний зв'язок із ЗРП (B_r), залежний від виду КН (рис. 3) (див. формули (3) і (4)). З формул (3) і (4) визначимо критичний параметер зламостійкості B_{rc} :

$$B_{rc} = B_r / (a + bB_r), \quad (9)$$

де a і b залежать від виду КН.

Кореляційні залежності (3) і (4) дають змогу виконувати розрахункові визначення критичної температури крихкості T_c через експериментально встановлену температурну залежність базової ($\sigma_{0,2}$) і граничної (S_K) міцності (рис. 1) без додаткових випробувань зразків з КН. Це — суттєва методологічна перевага концепції ЗРП перед традиційною методикою ударної в'язкості, що потребує додаткових випробувань зразків з КН в широкому інтервалі температур [5, 6].

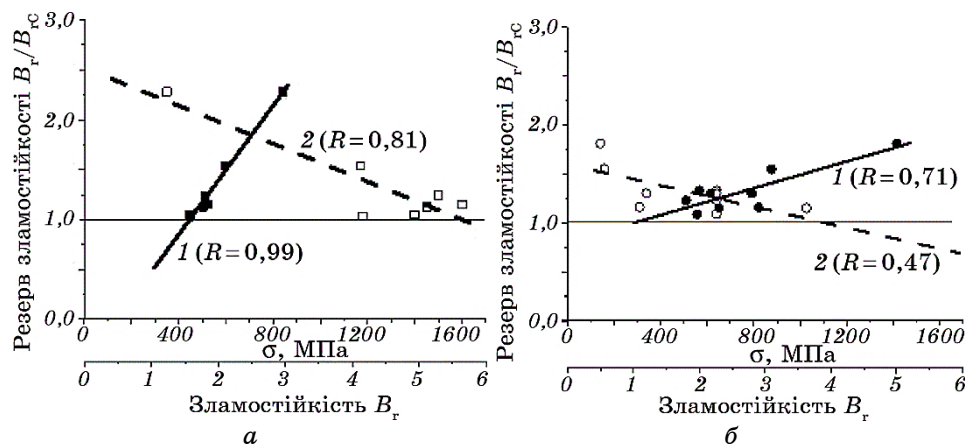


Рис. 3. Зміна параметра холодостійкості B_r/B_{rc} від властивостей міцності $\sigma_{0,2}$ та зламостійкості B_r за випробувань зразків з КН: К1 (п. 1–7, табл. 1, $\square$ і $\blacksquare$) (а), К2 (п. 8–16, табл. 1, $\circ$ і $\bullet$) (б); лінія 1 — залежність для B_r/B_{rc} шкали B_r , лінія 2 — те саме для шкали $\sigma_{0,2}$; R — коефіцієнт кореляції.

Fig. 3. Change in the cold brittleness parameter B_r/B_{rc} due to strength properties $\sigma_{0,2}$ and resistance to break B_r at testing specimens with stress raisers (SR): K1 (p. 1–7, Table 1, $\square$ and $\blacksquare$) (a), K2 (p. 8–16, Table 1, $\circ$ and $\bullet$) (b); line 1 is dependence for B_r/B_{rc} of the B_r scale, line 2 is the same for the $\sigma_{0,2}$ scale; R is correlation coefficient.

Використання параметра зламостійкості B_r уможливорює радикально спростити методологію атестації металевих стопів на крихкість за кімнатної температури T_K без визначення температури крихкості T_C , виходячи із заздалегідь експериментально визначеного порогового показника руйнівальної ефективності КН α_{ef}^{th} за (2) за руйнування зразка з КН за умови $\sigma_{NF} = \sigma_{0,2}$, але за кімнатної температури випробувань $T_K \cong 300$ К. Тоді в'язкі матеріали з $B_r \geq \alpha_{ef}^{th}$ за кімнатної температури T_K руйнуються запевне за $\sigma_{NF} > \sigma_{0,2}$ ($B_{rN} > 1$), а крихкі (для даного виду КН), у яких $B_r < \alpha_{ef}^{th} = B_{rC}(T_K)$, руйнуються нижче межі плинності стопу $\sigma_{NF} < \sigma_{0,2}$ ($B_{rN} < 1$, див. поз. 19–26 табл. 2 з роботи [13]) (рис. 4).

На рисунку 4 наведено зміну параметра залишкової зламостійкості B_{rN} у зразка з КН, що представляє залишкову тривку здатність σ_{NF} за формулою (7) зразка з КН для різних видів КН, що досліджувались у роботах різних авторів за кімнатної температури T_K [13, 17, 18]. З рисунку 4 видно, що за кімнатної температури T_K лише на зразках з тріщиною втоми вдається реалізувати умови крихкого руйнування ($B_{rN} \leq 1$) виключно на зразках лише високоміцних

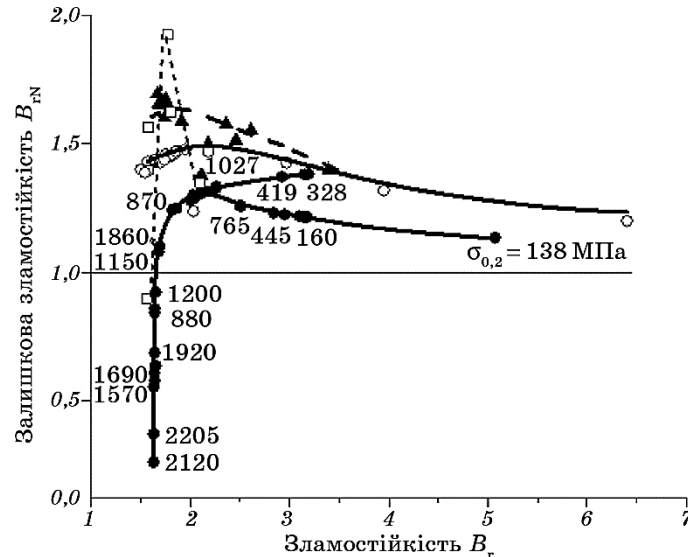


Рис. 4. Залежність залишкової зламостійкості B_{rN} від початкової (B_r) для зразків з різними видами КН із $T_K \cong 300$ К: зразки з тріщиною втоми [13] (●), з КН по [17] (○, ▲), з КН по [18] (□). Цифри — значення $\sigma_{0,2}$ за $T_K \cong 300$ К.

Fig. 4. Dependence of the residual fracture toughness of B_{rN} on the initial (B_r) for specimens with different types of stress raisers (SR) at $T_K \cong 300$ K: fatigue cracked specimens [13] (●), with SR according to [17] (○, ▲), with SR according to [18] (□). Numbers are values of $\sigma_{0,2}$ at $T_K \cong 300$ K.

криць інструментального класу (табл. 2), в той час як звичайні криці конструкційного класу з показником $B_{rN} \geq 2$, в тому числі й високоміцні (поз. 1, АК-35, $\sigma_{0,2} = 1027$ МПа, $B_r = 2,26$) з тріщиною втоми, руйнуються цілком в'язко за виключенням криці, що зазнала водневого окрихчення за міцності $\sigma_{0,2} = 880$ МПа (поз. 6, табл. 2), у якої $B_{rN} = 0,81$.

ТАБЛИЦЯ 2. Механічні властивості криць і зразків з тріщиною за вигину за $T_K \cong 300$ К [13]: Г — гартування, ПО — пічне охолодження, ВП — відпуск, H_2 — водневе середовище, ЗШ — зварний шов, ПД — порошковий дріт, КФ — керамічний флюс, C_B — вміст Бору, ВТМО — високотемпературне термічне оброблення, $B_{rCO} = \sigma_{CO}/\sigma_{0,2}$. Цифри посилянь в колонці КС наведено в роботі [13].

TABLE 2. Mechanical properties of steels and specimens with crack at bending at $T_K \cong 300$ K [13]: Г is quenching, ПО is furnace cooling, ВП is tempering, H_2 is hydrogen environment, ЗШ is weld seam, ПД is flux cored wire, КФ is ceramic flux, C_B is boron contents, ВТМО is high-temperature heat treatment, $B_{rCO} = \sigma_{CO}/\sigma_{0,2}$. Reference numbers in the КС column are given in the work [13].

№	КС	Оброблення	$\sigma_{0,2}$, МПа	ψ_K , %	S_K , МПа	B_r	σ_{NF} , МПа	B_{rN}
1	2	3	4	5	6	7	8	9
1	АК-35 [2]	Стан поставки	1027	75,0	2321	2,26	1820	1,83
2	α -Fe [2]	Г 1323 К, 2 год. ПО	138	83,8	700	5,07	290	2,10
3	10ХСНД [2]	Г 1373 К, 2 год. ПО	312	72,0	920	2,95	590	1,88
4	Ст. 3 сп. [2]	Г 1373 К, 2,75. ПО	160	71,7	502	3,14	307	1,92
5		Г 1373 К, 1 год. + ВП 1023 К, 16 год., 923 К, 10 год. 10Х15Н27Т3В2МР [3]	870	23,0	1583	1,82	1220	1,40
6		Г 1373 К, 1 год. + ВП 1023 К, 16 год, 923 К, 10 год. + H_2	880	10,0	1443	1,64	713	0,81
7	15Х12Н2МФАВ [4]	Г 1393 К, 1 год. + ВП 953 К 2 год	940	62,0	1983	2,11	1580	1,68
8	03Х12Н10МТ [4]	1273 К, 1 год, 1023 К, 2 год. + ВП 773 К 2 год.	940	79,0	2670	2,84	1740	1,85
9	03Х12Н10МТ [4]	Г 1373 К, 15 хв., 1023 К, 2 год. + ВП 773 К, 2 год.	930	76,0	2325	2,50	1655	1,78
10		ПД-АНЗО, 1 шар	430	67,5	1360	3,16	2830	1,92
11	СШ Х75 [2]	ПД-АНЗО, 2 шара	361	60,7	1444	3,17	700	1,94
12		ПД-АНЗО, 3 шара	404	51,9	1252	3,10	776	1,92
13		КФ 48-АНК-54, ВП, $C_B = 0,0$	622	59,9	1244	2,00	970	1,56
14	СШ 12ХН2МДФ [2]	те ж, ВП, $C_B = 0,001$	623	55,6	1160	1,86	890	1,43
15		те ж, ВП, $C_B = 0,0022$	628	68,1	1281	2,04	1005	1,60
16		те ж, ВП, $C_B = 0,004$	642	67,9	1400	2,18	1117	1,74

Продовження Таблиці 2.

Continuation of Table 2.

1	2	3	4	5	6	7	8	9
17	20X [5]	ВП 473 К	1150	53,9	1932	1,68	1276	1,11
18		Г 1113 К + ВП 423 К, 2 год.	1860	16,5	3050	1,64	930	1,50
19		Г 1113 К + ВП 473 К, 2 год.	1920	46,7	3150	1,64	1152	0,60
20	50X [5]	Г 1113 К + ВП 673 К, 2 год.	1560	48,6	2530	1,64	1248	0,80
21		Г 1113 К + ВП 773 К, 2 год.	1200	57,2	1980	1,65	1080	0,90
22	40C2X [5]	ВТМО + ВП 473 К	1760	53,0	2870	1,63	774	0,44
23		ВТМО + ВП 573 К	1690	55,0	2770	1,65	900	0,53
24	60C2X [5]	ВТМО + ВП 573 К	2205	38,0	3594	1,63	433	0,21
25		ВТМО + ВП 773 К	1570	40,0	2575	1,64	723	0,46
26	ШХ15 [5]	З 1133 К + ВП 473 К	2120	3,6	3460	1,63	1502	0,07
27	24ХН0МФА [5]	Г 1153 К + ВП 913 К, 15 год.	765	73,5	1920	2,51	1362	1,78
28	65Ф [5]	Стан поставки	700	28,0	1183	1,69	800	1,14
29	10ХСНД [6]	Вздовж прокату	419	69,7	1223	2,92	1020	2,43
30	10ХСНД [6]	Поперек прокату	445	73,7	1400	3,15	1175	2,64
31	12Г2МФТ [6]	Вздовж прокату	571	65,6	1290	2,26	1050	1,84
32		Поперек прокату	602	58,4	1282	2,03	975	1,62
33	15ХСНД [7]	Стан поставки	328	68,0	1046	3,19	880	2,68

З таблиці 2 випливає висновок, що пороговим показником ефективності КН α_{ef}^{th} за температури $T_K = 300$ К для зразка з тріщиною втоми (за регламентом у зразків для випробувань на тріщиностійкість K_{IC} по [24]) є величина

$$\alpha_{ef}^{th} = B_{rc}(T_K) = 1,66. \quad (10)$$

Відповідно, для зразків з надрізами різної гостроти пороговий показник руйнівальної ефективності КН має бути меншим за 1,66, тобто некритичним для криць з $B_r \geq 2$, яким крихкість від КН за $T_K \cong 300$ К запевне не загрожує, і тому для криць конструкційного класу схильність до крихкості можна виявляти лише за низькотемпературних випробувань на температуру T_c .

Отже, для криць конструкційного класу достатньо встановити вимогу на рівень зламостійкості $B_r \geq 2$, щоб бути впевненим за силою надійності у виробках за будь-якого регулярного конструктивного концентратора напружень (наприклад, нарізки на болтових кріпленнях, зубчаті шестерні, тощо), за виключенням експлуатації в умовах довготривалих циклічних навантажень, за яких можлива загроза втомного руйнування.

5. ЗАКІНЧЕННЯ

В твердих тілах з крихкого матеріалу (скло, граніт, тощо) руйнування напруженого тіла спричиняються енергетичною нестабільністю дефектів структури або порожнин у тілі, де виникають розірвані міжатомові зв'язки. Граничний енергетичний рівень крихкого матеріалу $W_{\text{ср}}$ регулюється його міцністю у відповідності з наявною структурою матеріалу.

В металевих матеріалах властива їм пластичність різко сповільнює темп накопичення пружної енергії до небезпечного граничного рівня $W_{\text{ср}}$, чим завдяки ЗРП (B_r) запобігає швидкому (крихкому) руйнуванню металевих тіл на межі плинності $\sigma_{0,2}$, віддаляє момент енергетичної нестабільності деформованої структури металу до напруження в'язкого руйнування S_K . Тому інтервал напружень $\sigma_{0,2} - S_K$ створює для металу специфічний захисний ресурс пластичного стану, що віддаляє стан енергетичної нестабільності (руйнування) до більш високої межі — S_K . Створений в інтервалі напружень $\Delta S = S_K - \sigma_{0,2}$ деформаційний резерв базової міцності $\sigma_{0,2}$ являє собою особливу механічну характеристику, що відображає пластичність металу у суто силовому вимірі коефіцієнта зламостійкості $B_r = S_K / \sigma_{0,2}$.

Захисна сила ЗРП залежить від пластичності металу (ψ_K) і темпу деформаційного зміцнення за Холломоновою закономірністю, є ключовим чинником у визначенні параметрів крихкого стану в тілах з неоднорідними видами НДС (вигин, надрізи, тріщини в МТ), де відбувається суто локалізоване вбирання ЗРП за рахунок локального перезміцнення в зоні КН за номінального (середнього) напруження в тілі σ_{NF} на рівні межі плинності металу $\sigma_{0,2}$ або навіть нижче $\sigma_{0,2}$.

Критична ситуація руйнування МТ на межі плинності металу $\sigma_{0,2}$ визначає не лише критичну температуру крихкості T_c для тіла з КН, але й критичний рівень зламостійкості $B_{rc} = S_{Kc} / \sigma_{0,2c}$ для даного виду КН, що стає знаковою величиною для характеристики самого концентратора напружень, його руйнівальної ефективності у вигляді критичного коефіцієнта руйнівальної ефективності КН $\alpha_{\text{ефс}} = B_{rc}(T_c)$.

Кожен вид КН має свій власний показник $\alpha_{\text{ефс}}$, але не фіксований на певній величині B_{rc} , тому що енергетична нестабільність (руйнування) не забезпечується лише пружним ресурсом резерву міцності $\Delta S = S_K - \sigma_{0,2}$, а й сумарним напруженням резерву міцності ΔS разом з базовою міцністю $\sigma_{0,2}$, тобто показником граничної міцності S_K . Тому B_{rc} за T_c для кожного виду КН має свою власну усталену закономірність «дрейфу» B_{rc} зі зміною S_K металу. Але доля критичної ефективності КН $\alpha_{\text{ефс}}$ в наявній зламостійкості металу у вигляді співвідношень (3), (4) для B_r / B_{rc} є стабільною, властивою для даного виду КН, що дає змогу здійснювати розрахунковий підхід до ви-

значення критичних параметрів крихкого стану (T_c, B_{rc}) зразка з наперед відкаліброваним видом КН лише за базовим показником зламостійкості металу B_r за формулами (3), (4).

Саме цей результат досліджень з проблеми крихкого руйнування металевих стопів під дією КН, на думку авторів роботи, може свідчити про розкриття ключової ролі захисного ресурсу пластичності B_r (як наслідок самої пластичності металу) в процесах руйнування металевих виробів, елементів конструкцій і металевих споруд.

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ЦИТОВАНА ЛІТЕРАТУРА

1. Ф. Зейтц, *Современная теория твердого тела* (Москва: Гостехиздат: 1949) (пер. з англ.).
2. А. Х. Котрелл, *Дислокации и пластическое течение в кристаллах* (Москва: Металлургия: 1958) (пер. з англ.).
3. С. А. Котречко, Ю. Я. Мешков, *Предельная прочность. Кристаллы, металлы, конструкции* (Київ: Наукова думка: 2008).
4. Ю. Я. Мешков, Т. Н. Сердитова, *Разрушение деформированной стали* (Київ: Наукова думка: 1989).
5. *ГОСТ 9454-78. Металлы. Метод испытания на ударный изгиб при пониженных, комнатной и повышенных температурах* (Москва: Издательство стандартов: 1978).
6. *ПНАЭ Г-7-002086. Нормы расчета на прочность оборудования и трубопроводов атомных энергетических установок.*
7. Ю. Я. Мешков, С. А. Котречко, А. В. Шиян, *Механическая стабильность металлов и сплавов* (Київ: Наукова думка: 2014).
8. Ю. Я. Мешков, Г. П. Зіміна, *Металофіз. новітні технол.*, **45**, № 8: 1029 (2023).
9. Ю. Я. Мешков, Г. П. Зіміна, *Металофіз. новітні технол.*, **46**, № 4: 355 (2024).
10. Ю. Я. Мешков, Г. П. Зіміна, *Металофіз. новітні технол.*, **46**, № 10: 1031 (2024).
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12. А. А. Griffith, *Phil. Trans. A*, **221**, Iss. 562–593: 163 (1920).
13. А. В. Шиян, Ю. Я. Мешков, Ю. А. Полупшин, *Сталь*, № 6: 39 (2019).
14. А. В. Шиян, Ю. Я. Мешков, *Металофіз. новітні технол.*, **41**, № 6: 775 (2019).
15. А. В. Шиян, Ю. Я. Мешков, *Сталь*, № 1: 45 (2019).
16. Ю. Я. Мешков, С. О. Котречко, К. Ф. Сорока, *Металофіз. новітні технол.*, **44**, № 10: 1377 (2022).
17. А. В. Шиян, *Физическая природа локального напряжения хрупкого разрушения сталей и сварных швов* (Автореф. дис. ... канд. физ.-мат. наук) (Київ: Институт металлофизики АН УССР: 1990).
18. П. Ф. Кошелев, С. Е. Беляев, *Прочность и пластичность конструкционных*

- материалов при низких температурах* (Москва: Машиностроение: 1967).
19. A. N. Stroth, *Proc. Roy. Soc. A*, **223**: 404 (1954).
 20. Ю. Я. Мешков, *Физические основы разрушения стальных конструкций* (Киев: Наукова думка: 1981).
 21. Ю. Я. Мешков, Г. А. Пахаренко, *Структура металлов и хрупкость стальных изделий* (Киев: Наукова думка: 1985).
 22. Н. Н. Давиденков, Н. И. Спиридонова, *Заводская лаборатория*, № 11: 583 (1945).
 23. П. Бриджмен, *Исследование больших пластических деформаций и разрыва* (Москва: Издательство иностранной литературы: 1955) (пер. з англ.).

REFERENCES

1. F. Seitz, *Sovremennaya Teoriya Tverdogo Tela* [The Modern Theory of Solids] (Moskva: Gostekhizdat: 1949) (Russian translation).
2. A. H. Cottrell, *Dislokatsii i Plasticheskoe Techenie v Kristallakh* [Dislocations and Plastic Flow in Crystals] (Moskva: Metallurgiya: 1958) (Russian translation).
3. S. O. Kotrechko and Yu. Ya. Meshkov, *Predel'naya Prochnost'. Kristally, Metally, Konstruktsii* [Ultimate Strength. Crystals, Metals, Structures] (Kiev: Naukova Dumka: 2008) (in Russian).
4. Yu. Ya. Meshkov and T. N. Serditova, *Razrushenie Deformirovannoy Stali* [Destruction of Deformed Steel] (Kiev: Naukova Dumka: 1989) (in Russian).
5. *GOST 9454-78. Metally. Metod Ispytaniya na Udarnyy Izgib pri Ponizhennykh, Komnatnoy i Povyshennykh Temperaturakh* [Metals. Impact Test Method at Low, Room and High Temperatures] (Moskva: Izdatel'stvo Standartov: 1978) (in Russian).
6. *PNAEh G-7-002086. Normy Raschyota na Prochnost' Oborudovaniya i Truboprovodov Atomnykh Ehnergeticheskikh Ustanovok* [Standards for Calculating the Strength of Equipment and Pipelines of Nuclear Power Plants] (in Russian).
7. Yu. Ya. Meshkov, S. O. Kotrechko, and A. V. Shiyan, *Mekhanicheskaya Stabilit'nost' Metallov i Splavov* [Mechanical Stability of Metals and Alloys] (Kiev: Naukova Dumka: 2014) (in Russian).
8. Yu. Ya. Meshkov and H. P. Zimina, *Metallofiz. Noveishie Tekhnol.*, **45**, No. 8: 1029 (2023) (in Ukrainian).
9. Yu. Ya. Meshkov and H. P. Zimina, *Metallofiz. Noveishie Tekhnol.*, **46**, No. 4: 355 (2024) (in Ukrainian).
10. Yu. Ya. Meshkov and H. P. Zimina, *Metallofiz. Noveishie Tekhnol.*, **46**, No. 10: 1031 (2024) (in Ukrainian).
11. Yu. Ya. Meshkov, H. P. Zimina, K. F. Soroka, and T. V. Melnychenko, *Metallofiz. Noveishie Tekhnol.* **47**, No. 7: 753 (2025) (in Ukrainian).
12. A. A. Griffith, *Phil. Trans. A*, **221**, Iss. 562–593: 163 (1920).
13. A. V. Shiyan, Yu. Ya. Meshkov, and Yu. A. Polushkin, *Stal'*, No. 6: 39 (2019) (in Russian).
14. A. V. Shiyan and Yu. Ya. Meshkov, *Metallofiz. Noveishie Tekhnol.*, **41**, No. 6: 775 (2019) (in Russian).
15. A. V. Shiyan and Yu. Ya. Meshkov, *Stal'*, No. 1: 45 (2019) (in Russian).
16. Yu. Ya. Meshkov, S. O. Kotrechko, and K. F. Soroka, *Metallofiz. Noveishie Tekhnol.*, **44**, No. 10: 1377 (2022) (in Ukrainian).
17. A. V. Shiyan, *Fizicheskaya Priroda Lokal'nogo Napryazheniya Khrupkogo*

- Razrusheniya Staley i Svarnykh Shvov* [The Physical Nature of the Local Stress of Brittle Fracture of Steels and Welds] (Thesis of Disser. for Cand. Phys.-Math. Sci.) (Kiev: Institute for Metal Physics, A.S. of UkrSSR: 1990) (in Russian).
18. P. F. Koshelev and S. E. Belyaev, *Prochnost' i Plastichnost' Konstruktsionnykh Materialov pri Nizkikh Temperaturakh* [Strength and Plasticity of Structural Materials at Low Temperatures] (Moskva: Mashinostroenie: 1967) (in Russian).
 19. A. N. Stroth, *Proc. Roy. Soc. A*, **223**: 404 (1954).
 20. Yu. Ya. Meshkov, *Fizicheskie Osnovy Razrusheniya Stal'nykh Konstruktsiy* [Physical Foundations of Destruction of Steel Structures] (Kiev: Naukova Dumka: 1981) (in Russian).
 21. Yu. Ya. Meshkov and G. A. Pakhareno, *Struktura Metallov i Khrupkost' Stal'nykh Izdeliy* [Structure of Metals and Brittleness of Steel Products] (Kiev: Naukova Dumka: 1985) (in Russian).
 22. N. N. Davidenkov and N. I. Spiridonova, *Zavodskaya Laboratoriya*, No. 11: 583 (1945) (in Russian).
 23. P. W. Bridgman, *Issledovanie Bol'shikh Plasticheskikh Deformatsiy i Razryva* [Studies in Large Plastic Flow and Fracture] (Moskva: Izdatel'stvo Inostrannoy Literatury: 1955) (Russian translation).

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