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ORIGINAL STUDY

EFFECT OF COMPOSITION AND CONSOLIDATION METHOD ON THE MICROSTRUCTURE, HARDNESS, AND TRIBOLOGICAL PROPERTIES OF SiO₂–NiCr COMPOSITE PELLETS PRODUCED BY SPARK PLASMA SINTERING AND HOT PRESSINGSerikkhan Almira^{1*}, Kenesbekov Aidar¹, Kussainov Temirlan¹¹Institute of Composite Materials LLP, Ust-Kamenogorsk, Kazakhstan*Corresponding author: almiraserikkhan@mail.ru

Abstract. This study investigated the effects of the SiO₂–NiCr composition and thermomechanical consolidation method on the microstructure, phase composition, mechanical properties, and tribological behavior of composites produced by spark plasma sintering (SPS) and hot pressing. The samples were characterized by X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray fluorescence analysis, Vickers hardness measurements, and tribological testing. The composites exhibited a heterogeneous cermet structure, with Ni and Cr distributed in the metallic constituent and SiO₂ forming the oxide phase. The maximum Vickers hardness, 2020.2 ± 165.9 HV, was recorded for the hot-pressed sample with an SiO₂/NiCr ratio of 70/30. The minimum mean coefficient of friction (0.135) was obtained for the SPS-produced sample with a 50/50 composition. The results demonstrate that both composition and consolidation method substantially affect the microstructure, phase composition, hardness, and tribological properties of SiO₂–NiCr composites.

Keywords: spark plasma sintering, hot pressing, SiO₂–NiCr, cermet composite, microstructure, phase composition, hardness, coefficient of friction.

1. Introduction

In powder metallurgy, spark plasma sintering (SPS) and hot pressing are among the most effective methods for consolidating powder materials under uniaxial pressure at elevated temperature; however, they differ in the mode of heat supply and the thermomechanical processing conditions. During SPS, heating is generated by a pulsed electric current passing through the graphite tooling and, depending on the electrical conductivity of the material, through the powder compact. This provides a high heating rate, short holding time, and limited grain growth [1–4]. For dielectric SiO₂, consolidation efficiency is governed by heat-transfer conditions and the temperature–time regime [5], whereas temperature and pressure are the key factors controlling the density, grain size, and mechanical properties of nickel-based materials [6]. Unlike SPS, hot pressing consolidates the powder mixture by external heating. The feasibility of producing bulk Ni–SiO₂ composites by this method has been demonstrated previously [7, 8].

Published data indicate that consolidation parameters, including temperature, pressure, heating rate, and holding time, determine densification kinetics, microstructure formation, and phase retention [1–6]. Therefore, both the nominal composition and the consolidation method must be considered when examining the structure and functional properties of SiO₂–NiCr composites.

Studies of bulk SiO₂–NiCr composites produced by SPS and hot pressing remain scarce. The most closely related investigations concern Ni–SiO₂ composites prepared by mechanical milling [9], sol–gel processing [10, 11], spray pyrolysis [12], and hot pressing [7, 8, 18]. These studies show that composite composition, consolidation conditions, and heat treatment determine the degree of densification, metallic-particle size, particle distribution in the silica matrix, and phase state of the material [7–13]. Hot pressing may also produce a transition layer at the Ni/SiO₂ interface, indicating interfacial interaction at elevated temperatures [8].

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Studies of Ni–SiO₂ composites show that increasing the metallic fraction changes the size and spatial distribution of Ni particles [9, 10, 12–14], whereas a more uniform distribution of SiO₂ can improve the microhardness and wear resistance of the material [15–17]. These relationships cannot be transferred directly to the SiO₂–NiCr system because Cr affects consolidation, microstructure formation, and the resulting functional properties.

Despite extensive research on Ni–SiO₂ composites, little information is available on the influence of composition and consolidation method on the microstructure, phase composition, mechanical properties, and tribological behavior of bulk SiO₂–NiCr composites. The aim of this study was therefore to determine how the nominal SiO₂/NiCr ratio and consolidation method—spark plasma sintering or hot pressing—affect the microstructure, phase composition, mechanical properties, and tribological behavior of the resulting composites.

2. Materials and Methods

SiO₂–NiCr composite samples were prepared from powder mixtures with nominal component ratios of 30/70, 50/50, and 70/30. Two series were produced for each composition: series M by spark plasma sintering (SPS) and series L by hot pressing (HP). Series M samples were consolidated in a graphite die under the simultaneous action of uniaxial pressure and a pulsed electric current. Series L samples were produced by hot pressing under elevated temperature and uniaxial pressure with external heat supply. The SPS and hot-pressing parameters, including temperature, pressure, heating rate, holding time, and processing atmosphere, are listed in Table 1.

Table 1. Processing conditions for the SiO₂–NiCr composite samples.

Sample	SiO ₂ /NiCr, wt.%	Method	Temperature, °C	Pressure, MPa	Heating rate, °C/min	Holding time, min	Atmosphere
M-1	30/70	SPS	1000	50	100	10	Vacuum
M-2	50/50						
M-3	70/30						
L-1	30/70	HP	1100	30	10	60	
L-2	50/50						
L-3	70/30						

The surface microstructure and morphology of the samples were examined by scanning electron microscopy (SEM) using a TESCAN VEGA4 LMH microscope (TESCAN, Brno, Czech Republic). Energy-dispersive X-ray spectroscopy (EDS) was performed using an Xplore 30 system (Oxford Instruments, Oxford, United Kingdom). X-ray diffraction (XRD) analysis was conducted on a DX-27mini diffractometer (Dandong Haoyuan Instrument Co., Ltd., China) using Cu K α radiation ($\lambda = 0.154$ nm) at an operating voltage of 40 kV and a current of 30 mA. Measurements were collected over a 2θ range of 20–90°, with a step size of 0.05° and a counting time of 3 s per step. Phase identification was performed using HighScore software, version 4. The elemental composition was examined by X-ray fluorescence (XRF) analysis using an X-MET 8000 SMART portable analyzer. The Vickers hardness of the composite samples was measured using a FISCHERSCOPE HM2000 measurement system (Helmut Fischer GmbH, Germany). The indenter approach rate was 2 μ m/s, and the applied-load range was 0.1–2000 mN. Tribological tests were conducted on an Anton Paar TRB3 tribometer (Anton Paar, Buchs, Switzerland) in a ball-on-disc configuration in accordance with ASTM G133-95 and ASTM G99. Tests were performed under dry sliding conditions using a 6 mm diameter 100Cr6 steel ball as the counterbody. The normal load was 1 N, the linear sliding speed was 5 cm/s, and the wear-track radius was 4 mm, corresponding to a rotational speed of approximately 119 rpm.

3. Results and Discussion

3.1. Microstructure and Elemental Distribution

SEM images of the investigated pellets are presented in [Figs. 1](#) and [2](#). The surfaces of all samples exhibit heterogeneous contrast characteristic of combined metallic and oxide regions. The 50/50 compositions show a predominantly fine-grained surface relief, whereas the 70/30 pellets exhibit more pronounced height variations and local heterogeneity. A relationship between microstructure and the distribution of the metallic constituent within a silica medium has previously been reported for Ni–SiO₂ materials [\[6\]](#).

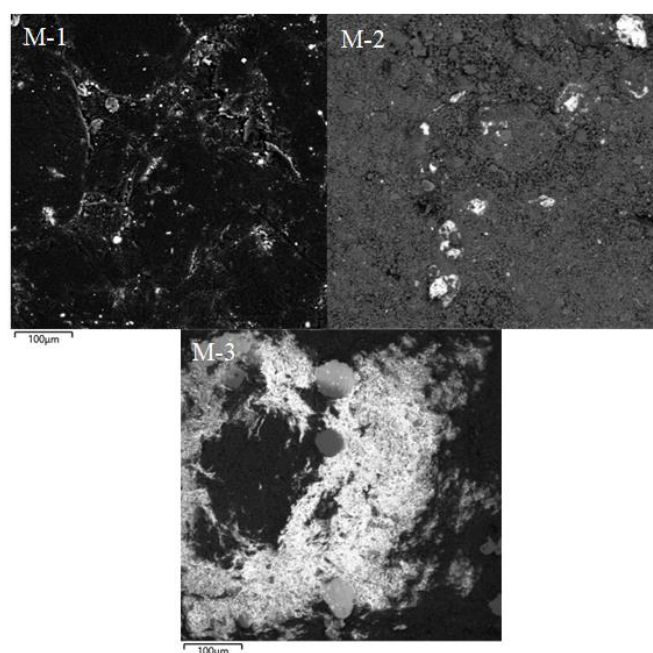


Fig. 1. SEM images of the surfaces of the series M SiO₂–NiCr composite pellets: M-1 (30/70), M-2 (50/50), and M-3 (70/30).

Sample M-1 (30/70) has a relatively dense and uniform surface. Local bright inclusions and minor surface irregularities are visible in some regions, but no pronounced network of large pores or extended cracks is observed. Smoother areas coexist with regions of increased roughness, indicating a certain degree of microstructural heterogeneity. The surface of M-2 (50/50) is more heterogeneous. The micrographs show numerous depressions, microdefects, and regions with different contrast. In some areas, the surface has a fine, granular morphology. Local cavities and discontinuities may indicate lower structural uniformity than in M-1. The most developed surface relief is observed for M-3 (70/30). Its surface contains pronounced irregularities, local protrusions and depressions, and regions with complex morphology. Some areas are markedly rougher, whereas others appear relatively smooth. This combination of distinct morphological regions indicates heterogeneous structure formation at this component ratio. Overall, the SEM observations show that changing the composition from 30/70 to 70/30 substantially affects surface morphology. Sample M-1 has a comparatively uniform and compact structure, whereas M-2 and particularly M-3 show increasingly pronounced surface relief and structural heterogeneity.

SEM images of the series L samples are presented in [Fig. 2](#) and reveal clear differences in surface morphology.

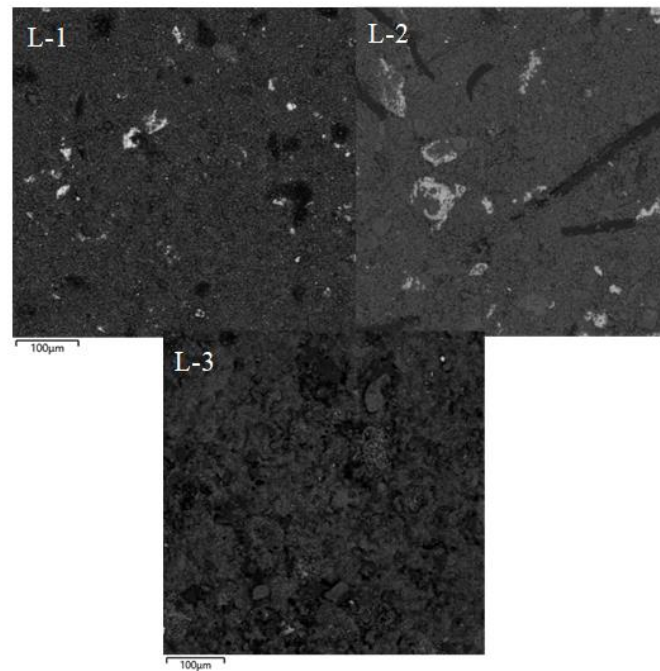


Fig. 2. SEM images of the series L SiO_2 –NiCr composite pellets: L-1 (30/70), L-2 (50/50), and L-3 (70/30).

Sample L-1 contains relatively smooth regions separated by extended crack-like defects and local depressions. Isolated protrusions and fine particles are also visible. These features are distributed nonuniformly: flat regions coexist with areas of pronounced relief. Sample L-2 has a finer, network-like surface texture. Against this background, individual rounded depressions and elongated dark bands are visible, corresponding to discontinuities or abrupt changes in topography. Local bright regions with irregular shapes are also distributed nonuniformly across the examined area. The surface of L-3 exhibits the most developed relief, consisting of tortuous ridges, protrusions, and numerous depressions of different shapes. Compared with L-1 and L-2, large smooth regions are less evident, and morphological heterogeneity extends over most of the field of view.

The differences between series M and L may be associated with their distinct heating modes - rapid current-assisted heating during SPS and external heating during hot pressing - as well as with differences in holding time and degree of densification. Thus, the L-1 to L-3 sequence shows a transition from a surface containing relatively smooth regions to a more complex and pronounced relief. The images permit a qualitative comparison of sample morphology; quantitative determination of roughness and porosity requires additional measurements. Elemental analysis is necessary to identify the chemical nature of regions with different brightness.

The EDS results must therefore be considered to identify the bright and dark regions and determine their chemical nature, because SEM contrast alone does not provide unambiguous phase or elemental identification. [Figures 3](#) and [4](#) present elemental distribution maps for the SiO_2 –NiCr pellets.

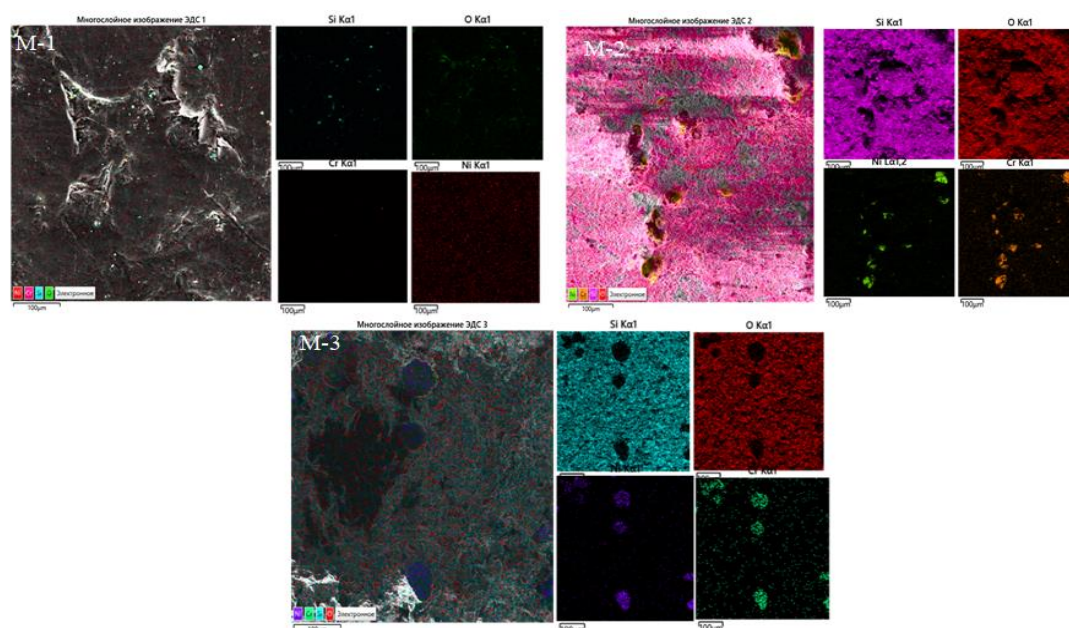


Fig. 3. EDS elemental maps of the series M SiO₂-NiCr composite pellets: M-1 (30/70), M-2 (50/50), and M-3 (70/30)

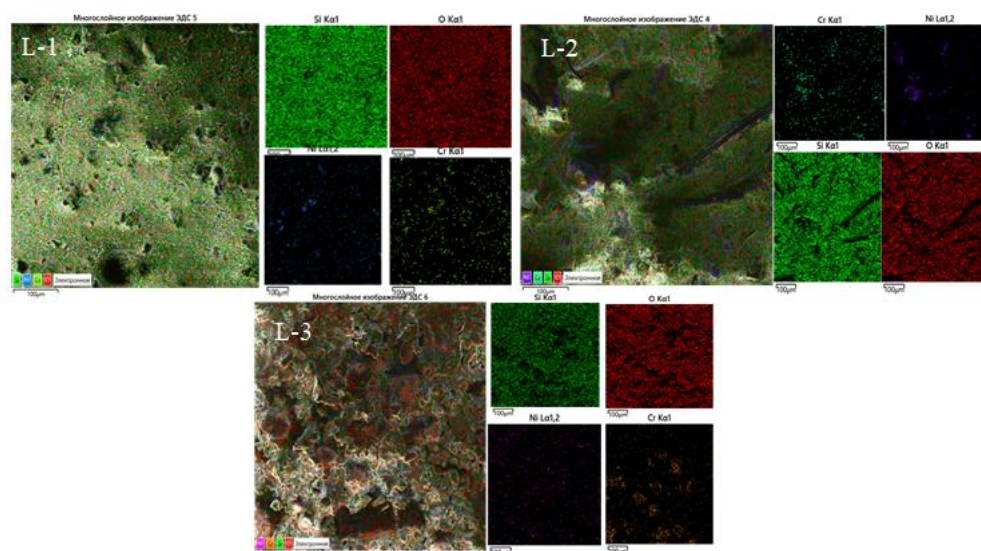


Fig. 4. EDS elemental maps of the series L SiO₂-NiCr composite pellets: L-1 (30/70), L-2 (50/50), and L-3 (70/30).

EDS mapping of the SiO₂-NiCr composite pellet surfaces revealed a heterogeneous but spatially correlated distribution of the principal elements. Si and O signals occur across nearly the entire examined area, indicating the formation of a continuous SiO₂-containing matrix. Regions of increased Si and O intensity largely coincide, confirming their joint presence in the oxide constituent. In the series M samples, Ni and Cr are distributed mainly as isolated local regions and finely dispersed inclusions. M-1 exhibits the most heterogeneous distribution of Si and O, together with individual regions enriched in NiCr constituents. In M-2, the Si-O-containing phase is distributed more uniformly, whereas Ni and Cr are concentrated primarily in individual particles and agglomerates. M-3 retains a continuous Si and O distribution but also contains local regions jointly enriched in Ni and Cr, indicating that discrete NiCr-alloy particles remain within the oxide matrix. The series L samples likewise contain a predominantly uniform SiO₂-bearing phase. In L-1, Si and O signals cover most of the surface, while Ni and Cr occur as dispersed inclusions. L-2 has a comparatively homogeneous Si-O matrix with local accumulations of metallic constituents. L-3 exhibits developed heterogeneous relief and individual agglomerates; Si and O are distributed across the entire examined area, whereas Ni and Cr are localized primarily in discrete regions. Overall, increasing the SiO₂

fraction from 30 to 70% produces a more pronounced continuous oxide matrix, while Ni- and Cr-rich regions become more localized. The partial spatial coincidence of Ni and Cr signals indicates that particles of the initial NiCr alloy persist after mixing and pressing. Local regions with elevated metallic-element concentrations indicate incomplete homogenization of the powder mixture and possible agglomeration. EDS maps provide mainly qualitative information on elemental distribution; quantitative comparison requires analysis of the corresponding EDS spectra. [Table 2](#) presents the XRF results, which describe the relative proportions of the elements detected by this method. Ni is the dominant element in every composition, and Cr is the second major metallic constituent. The elevated W content in the 70/30 sample and trace amounts of Sn, Ti, Pd, Cu, Zn, Nb, Mo, Au, and Zr may reflect the composition of the initial NiCr powder, processing-related contamination, or calibration effects. Because Si and O were not quantified by XRF in this series, [Table 2](#) does not represent a complete mass balance and should be interpreted together with the EDS and XRD results.

Table 2. Relative elemental composition of the samples determined by XRF analysis, wt.%.

Sample	Ni	Cr	W	Fe	Sn	Ti	Pd	Cu	Zn	Nb	Mo	Au	Zr	Pb
M-1	83.61	15.37	0.07	0.49	–	0.10	0.06	0.06	0.03	–	0.03	–	–	–
M-2	80.84	13.31	3.44	1.08	0.73	0.22	0.06	0.05	0.03	0.02	0.01	–	–	–
M-3	70.94	13.80	9.70	1.51	1.82	0.91	0.22	0.01	0.03	–	0.02	0.68	0.11	–
L-1	80.38	14.54	0.60	3.57	0.23	0.16	–	0.05	0.01	–	0.17	–	–	–
L-2	82.16	13.18	1.78	1.85	0.48	0.10	–	0.06	–	0.01	0.04	–	–	–
L-3	69.90	16.36	2.68	3.27	1.36	0.19	–	0.05	0.07	0.02	0.05	0.16	0.03	4.87

3.2. Phase Composition

The XRD patterns of series M and L are presented in [Figs. 5](#) and [6](#). All patterns contain intense reflections provisionally assigned to a Ni-containing metallic phase, together with reflections from Cr- and SiO₂-containing constituents. Changes in relative peak intensity reflect the effects of nominal composition, local heterogeneity, and processing method; however, they cannot be used to quantify phase fractions without Rietveld refinement. The phase state of both series may have changed under the applied temperature and pressure, while differences between series M and L may result from the mode of heat supply, heating rate, and holding time during SPS and hot pressing.

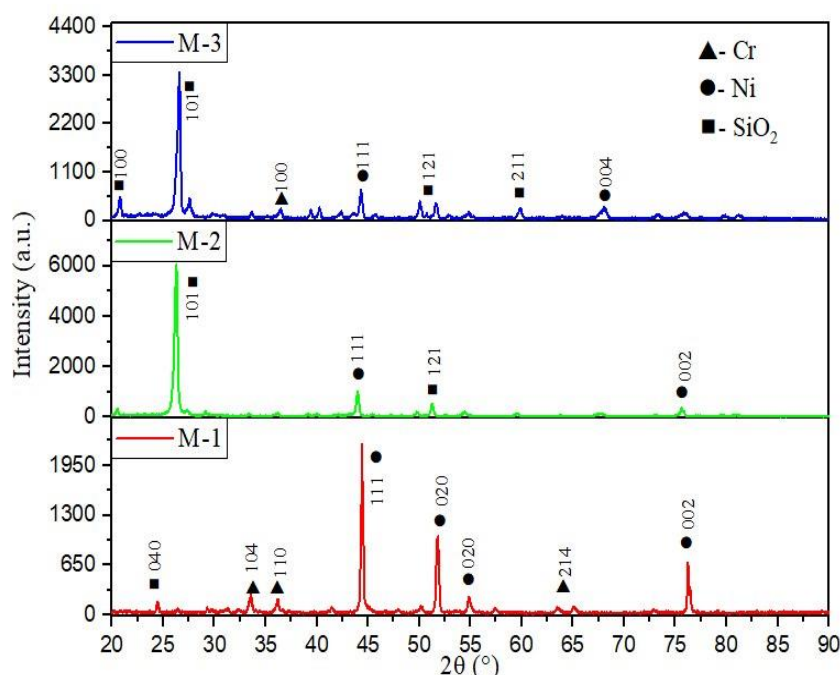


Fig. 5. XRD patterns of the SiO₂-NiCr composite samples produced by spark plasma sintering (SPS): M-1 (30/70), M-2 (50/50), and M-3 (70/30).

Figure 5 presents the XRD patterns of the series M samples produced by spark plasma sintering. For M-1 (30/70), the most intense diffraction maximum occurs at $2\theta \approx 44\text{--}45^\circ$ and corresponds to the Ni(111) reflection identified in the figure. Additional Ni reflections occur at approximately 52° , 55° , and $75\text{--}76^\circ$, whereas less intense Cr peaks are observed near $33\text{--}37^\circ$ and $63\text{--}65^\circ$. The SiO₂ reflection is substantially weaker, consistent with the greater proportion of the metallic NiCr constituent in this composition. The diffraction pattern changes markedly for M-2 (50/50). The intense SiO₂(101) reflection at $2\theta \approx 26\text{--}27^\circ$ becomes dominant. The Ni(111) reflection near $44\text{--}45^\circ$ and an additional Ni-containing maximum near $75\text{--}76^\circ$ are retained. The stronger SiO₂ peak relative to M-1 reflects the increased oxide fraction. For M-3 (70/30), the SiO₂(101) reflection at $26\text{--}27^\circ$ is again the most intense. Additional SiO₂ peaks are present at approximately $20\text{--}21^\circ$, $50\text{--}52^\circ$, and $59\text{--}60^\circ$, while the metallic Ni and Cr reflections have lower relative intensities. Thus, increasing the SiO₂ content from 30 to 70 wt.% in the SPS series changes the diffraction pattern from one dominated by Ni-Cr to one dominated by SiO₂.

Figure 6 presents the XRD patterns of the series L samples produced by hot pressing.

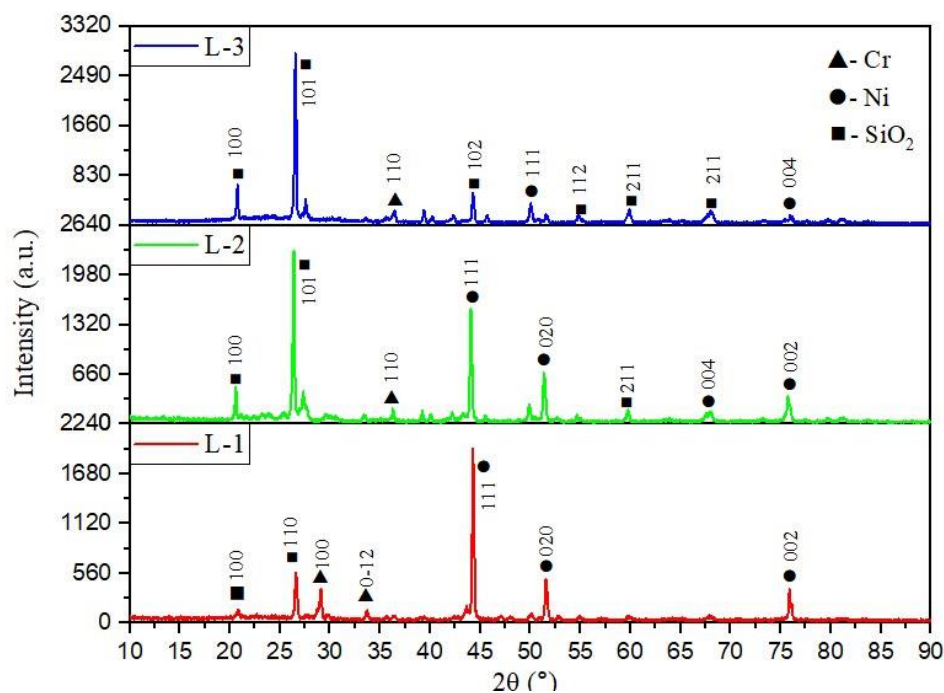


Fig. 6. XRD patterns of the SiO₂-NiCr composite samples produced by hot pressing (HP): L-1 (30/70), L-2 (50/50), and L-3 (70/30).

[Figure 6](#) presents the XRD patterns of the series L samples produced by hot pressing at different SiO₂/NiCr ratios. The diffraction pattern of L-1 (30/70) is dominated by reflections from the metallic constituent. The most intense maximum occurs at $2\theta \approx 44\text{--}45^\circ$ and, according to the indexing shown in the figure, corresponds to Ni(111). Additional Ni-containing reflections occur at approximately $51\text{--}52^\circ$ and $75\text{--}76^\circ$, while less intense reflections assigned to Cr and SiO₂ appear at approximately $26\text{--}36^\circ$. For L-2 (50/50), the intensity of the SiO₂ reflection at $2\theta \approx 26\text{--}27^\circ$, indexed as SiO₂(101), increases substantially. The pronounced Ni(111) peak near $44\text{--}45^\circ$ and additional metallic reflections at higher angles remain visible. Thus, both oxide and metallic constituents are evident in the diffraction pattern of the equiatomic SiO₂/NiCr composition. For L-3 (70/30), the SiO₂(101) maximum near $26\text{--}27^\circ$ becomes dominant. Additional SiO₂ reflections occur at approximately $20\text{--}21^\circ$, $39\text{--}40^\circ$, and $50\text{--}60^\circ$, whereas the relative intensities of the Ni and Cr reflections decrease. Overall, increasing the SiO₂ content from 30 to 70 wt.% systematically strengthens its diffraction reflections and reduces the relative contribution of the metallic Ni-Cr constituent.

3.3. Mechanical Properties

[Figure 7](#) presents the instrumented indentation loading–unloading curves for the series M samples with different component ratios: M-1 (30/70), M-2 (50/50), and M-3 (70/30). At the same maximum load, the curve shape and indentation depth differ substantially, indicating differences in resistance to local deformation.

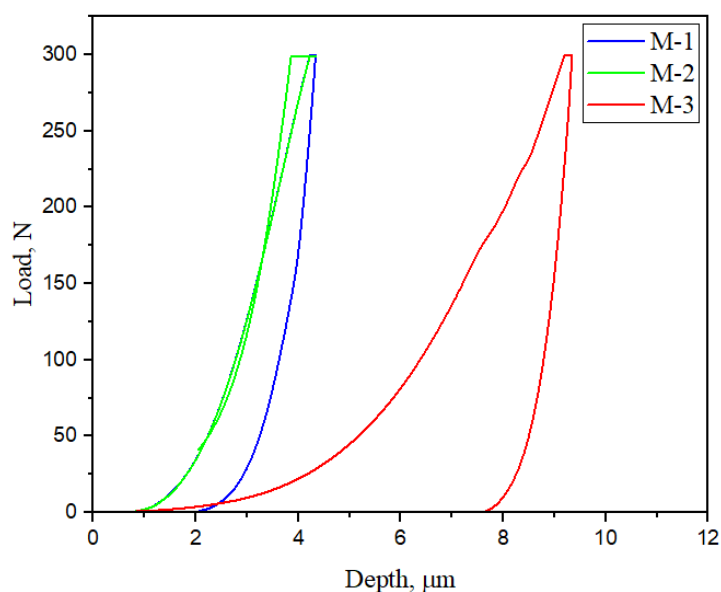


Fig. 7. Instrumented indentation loading–unloading curves for the series M samples: M-1 (30/70), M-2 (50/50), and M-3 (70/30).

The maximum indentation depth is approximately 4.1 μm for M-2 (50/50) and is slightly greater, approximately 4.3–4.4 μm , for M-1 (30/70). The close positions of the M-1 and M-2 curves indicate comparable indentation resistance, although M-2 shows a slightly smaller penetration depth.

The most pronounced deformation is observed for M-3 (70/30), for which the maximum indentation depth reaches approximately 9.2–9.4 μm . The marked shift of the M-3 curve toward greater depths indicates lower resistance to local deformation and a larger plastic contribution. Based on the loading–unloading curves, indentation resistance in series M decreases in the following order: M-2 (50/50) > M-1 (30/70) >> M-3 (70/30).

[Figure 8](#) presents the instrumented indentation loading–unloading curves for the series L samples with different component ratios: L-1 (30/70), L-2 (50/50), and L-3 (70/30). The curves demonstrate a pronounced effect of composition on indentation depth and the elastic–plastic response of the material.

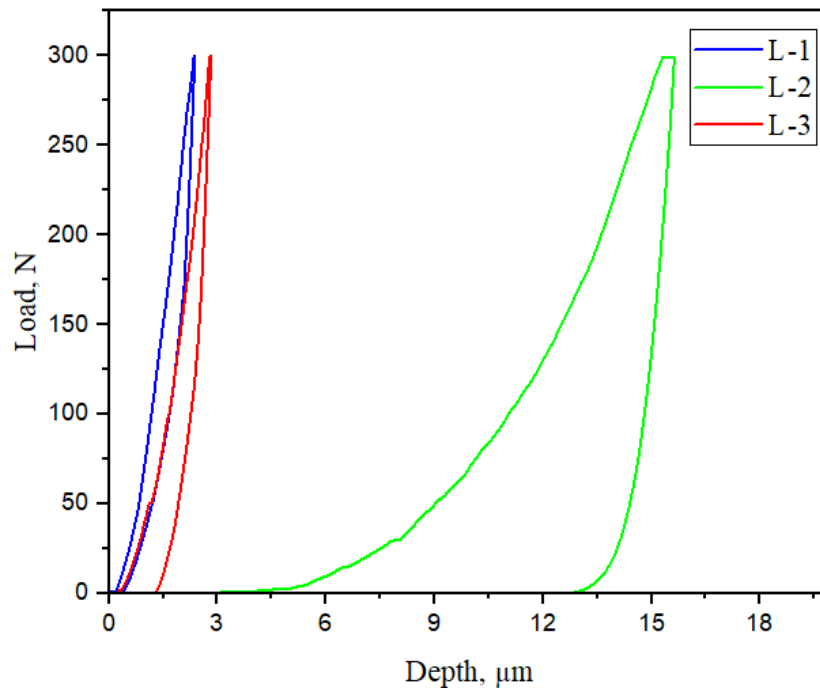


Fig. 8. Instrumented indentation loading–unloading curves for the series L samples: L-1 (30/70), L-2 (50/50), and L-3 (70/30).

The lowest maximum indentation depth, approximately 2.3–2.4 μm , is observed for L-1 (30/70). For L-3 (70/30), the depth is slightly greater at approximately 2.8–2.9 μm , indicating somewhat lower resistance to local deformation than L-1. The greatest increase in indentation depth occurs for L-2 (50/50). At the maximum load, the indentation depth reaches approximately 15–16 μm , considerably exceeding the values for L-1 and L-3. This behavior indicates substantially greater deformability and lower resistance to local indentation in L-2. Accordingly, indentation resistance in series L decreases in the following order: L-1 (30/70) > L-3 (70/30) >> L-2 (50/50).

Comparative analysis of the instrumented indentation results ([Table 3](#)) shows that the mechanical properties of the SiO_2 –NiCr composites vary nonmonotonically with composition and consolidation conditions.

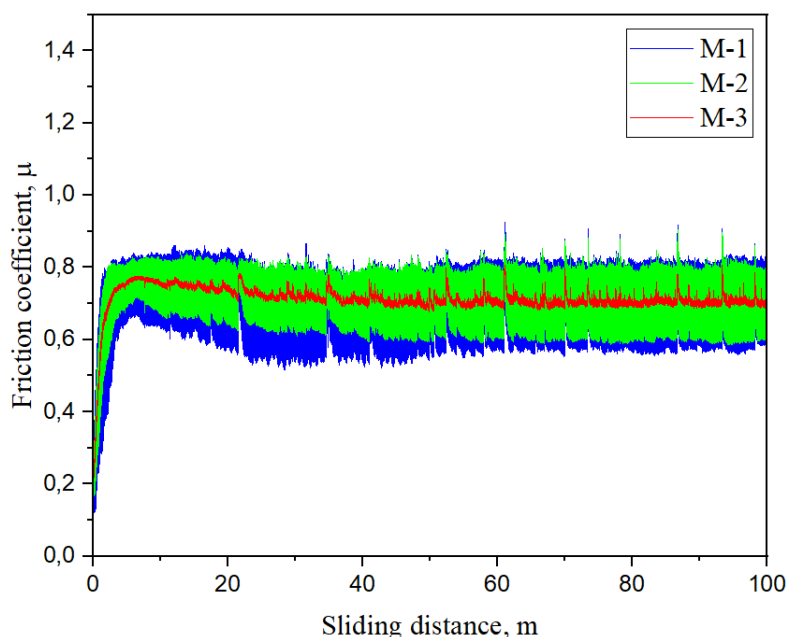
Table 3. Mechanical properties of the series M and L samples.

Sample	HM, N/mm ²	HV	E, GPa
M-1	663.3 ± 231.8	93.8 ± 36.0	19.3 ± 6.4
M-2	131.6 ± 10.7	15.6 ± 1.3	8.9 ± 2.5
M-3	525.7 ± 75.9	73.7 ± 12.1	17.0 ± 2.8
L-1	2020.2 ± 165.9	361.0 ± 52.9	43.3 ± 3.9
L-2	47.5 ± 2.2	5.5 ± 0.3	3.6 ± 1.1
L-3	1550.4 ± 566.0	235.0 ± 87.8	42.3 ± 16.0

The highest mean values were obtained for L-1 (30/70): the Vickers hardness was 361.0 ± 52.9 HV, the Martens hardness was 2020.2 ± 165.9 N/mm², and the indentation modulus was 43.3 ± 3.9 GPa. The lowest values were recorded for L-2 (50/50): 5.5 ± 0.3 HV, 47.5 ± 2.2 N/mm², and 3.6 ± 1.1 GPa, respectively. Based on mean hardness, the samples follow the sequences M-1 > M-3 > M-2 and L-1 > L-3 > L-2. At SiO₂/NiCr ratios of 30/70 and 70/30, the series L samples exhibit higher hardness and indentation modulus than the corresponding series M samples, whereas the opposite relationship is observed for the 50/50 composition. The relative advantage of each consolidation method therefore depends on the component ratio. Among the investigated samples, L-1 combines the 30/70 composition and hot-pressing conditions to provide the highest mean hardness and indentation modulus.

3.4. Tribological Behavior

Figures 9 and 10 show the coefficient of friction as a function of sliding distance for the series M and L samples with different component ratios: M-1 and L-1, 30/70; M-2 and L-2, 50/50; and M-3 and L-3, 70/30. In every case, the initial portion of the test corresponds to a running-in stage with a rapid change in the coefficient of friction, followed by steady-state or quasi-steady-state sliding.

**Fig. 9.** Coefficient of friction as a function of sliding distance for M-1 (30/70), M-2 (50/50), and M-3 (70/30).

The series M samples enter the quasi-steady-state regime comparatively rapidly. After the initial increase, the coefficient of friction of M-1 (30/70) varies mainly within approximately 0.58–0.83, with noticeable high-frequency fluctuations persisting throughout the test. M-2 (50/50) exhibits a similar friction level; after running-in, the values generally remain near 0.62–0.78. M-3 (70/30) produces a smoother curve: after the initial stage, the coefficient of friction stabilizes mainly within approximately 0.69–0.75 and then changes only slightly up to a sliding distance of 100 m. Thus, the component ratio in series M affects both the magnitude of the coefficient of friction and the stability of the frictional contact. Within this series, M-3 exhibits the smoothest tribological curve and the smallest fluctuation amplitude.

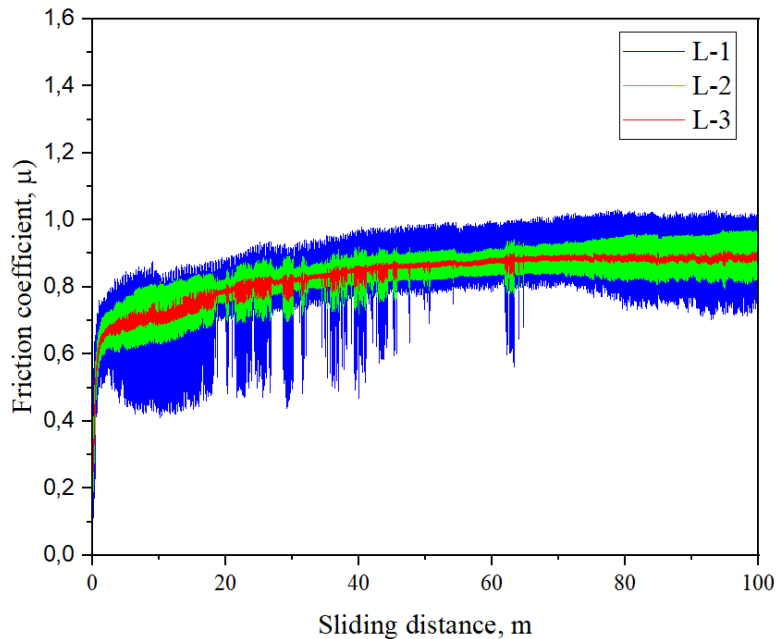


Fig. 10. Coefficient of friction as a function of sliding distance for L-1 (30/70), L-2 (50/50), and L-3 (70/30).

The coefficient of friction follows a different evolution in series L. After the initial running-in stage, it does not immediately reach a constant level but continues to increase gradually with sliding distance. L-1 (30/70) exhibits the largest signal fluctuations. Sharp, short-term decreases in the coefficient of friction occur in several regions, particularly between approximately 15 and 45 m, while the upper level of the curve approaches 1.0 by the end of the test. These fluctuations indicate unstable frictional-contact conditions and possible periodic changes in the state of the contacting surfaces during sliding. L-2 (50/50) exhibits a more uniform increase in the coefficient of friction. After an initial rise to approximately 0.65–0.70, the value increases gradually and reaches approximately 0.90–0.96 in the final part of the test. The fluctuations are less pronounced than for L-1, although the coefficient of friction continues to increase throughout the investigated sliding distance. L-3 (70/30) exhibits the most stable behavior within series L. After a short running-in period, the coefficient of friction rises smoothly from approximately 0.65–0.70 to 0.87–0.90 and then changes only slightly. Compared with L-1 and L-2, the L-3 curve has a smaller fluctuation amplitude, indicating a more stable friction process.

Comparison of series M and L shows that the series M samples generally enter the quasi-steady-state friction regime more rapidly, whereas series L exhibits a longer period of gradual increase in the coefficient of friction. In both series, the 70/30 compositions (M-3 and L-3) produce the smoothest tribological curves. The presented dependences support conclusions primarily about the magnitude and stability of the coefficient of friction. A quantitative assessment of wear resistance additionally requires the wear volume or wear rate, wear-track profile, and post-test surface morphology.

4. Conclusions

A comparative investigation was conducted to determine the effects of the SiO₂/NiCr ratio and consolidation method on the structure, mechanical properties, and tribological behavior of the composite pellets. The following conclusions were drawn.

1. The samples produced by spark plasma sintering and hot pressing exhibit a heterogeneous cermet structure. Si and O are distributed over most of the examined surface and form a SiO₂-containing matrix, whereas Ni and Cr are localized mainly in discrete particles and agglomerates. As the SiO₂ fraction increases from 30 to 70 wt.%, the oxide matrix becomes more pronounced and the metallic constituent becomes more localized.

2. According to the XRD results, reflections from the Ni–Cr-containing constituent dominate the diffraction patterns at an SiO₂/NiCr ratio of 30/70. Increasing the SiO₂ content to 50 and 70 wt.% strengthens the SiO₂(101) reflection at $2\theta \approx 26\text{--}27^\circ$ and decreases the relative intensity of the metallic reflections. This trend is observed in both sample series, irrespective of the consolidation method.

3. The mechanical properties vary nonmonotonically and are governed by the combined effects of composition and processing method. The highest values were obtained for hot-pressed L-1 with a 30/70 composition: the Vickers hardness was 361.0 ± 52.9 HV, the Martens hardness was 2020.2 ± 165.9 N/mm², and the indentation modulus was 43.3 ± 3.9 GPa. The lowest values were recorded for L-2 with a 50/50 composition: 5.5 ± 0.3 HV, 47.5 ± 2.2 N/mm², and 3.6 ± 1.1 GPa, respectively. Hot pressing produced higher mechanical properties for the 30/70 and 70/30 compositions, whereas SPS produced higher values for the 50/50 composition.

4. Tribological testing showed that the series M samples enter the quasi-steady-state friction regime more rapidly and generally exhibit lower coefficients of friction than the series L samples. The hot-pressed samples show a gradual increase in the coefficient of friction with sliding distance. The most stable tribological curves in both series were obtained for the 70/30 compositions, M-3 and L-3. Conclusions regarding wear resistance require additional measurements of wear volume or wear rate and analysis of wear-track morphology.

5. The optimum composition and consolidation method therefore depend on the required functional properties. The combination of an SiO₂/NiCr ratio of 30/70 and hot pressing provides the highest hardness and indentation modulus, whereas increasing the SiO₂ content to 70 wt.% promotes more stable friction behavior. These results demonstrate that the structure and properties of SiO₂–NiCr composites can be tailored by controlling the component ratio and consolidation conditions.

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Conflict of interest

The authors declare that they have no competing interests.

References

1. Munir Z.A., Quach D.V., Ohyanagi M. (2011) Electric Current Activation of Sintering: A Review of the Pulsed Electric Current Sintering Process. *Journal of the American Ceramic Society*, 94(1), 1–19. <https://doi.org/10.1111/j.1551-2916.2010.04210.x>
2. Orrù R., Licheri R., Locci A.M., Cincotti A., Cao G. (2009) Consolidation/synthesis of materials by electric current activated/assisted sintering. *Materials Science and Engineering R: Reports*, 63(4–6), 127–287. <https://doi.org/10.1016/j.mser.2008.09.003>
3. Guillon O., Gonzalez-Julian J., Dargatz B., Kessel T., Schierning G., Räthel J., Herrmann M. (2014) Field-Assisted Sintering Technology/Spark Plasma Sintering: Mechanisms, Materials, and Technology Developments. *Advanced Engineering Materials*, 16(7), 830–849. <https://doi.org/10.1002/adem.201300409>
4. Saheb N., Iqbal Z., Khalil A., Hakeem A.S., Al Aqeeli N., Laoui T., Al-Qutub A., Kirchner R. (2012) Spark Plasma Sintering of Metals and Metal Matrix Nanocomposites: A Review. *Journal of Nanomaterials*, 2012, 983470. <https://doi.org/10.1155/2012/983470>
5. Masai H., Kimura H., Kitamura N., et al. (2022) Densification in transparent SiO₂ glasses prepared by spark plasma sintering. *Scientific Reports*, 12, 14761. <https://doi.org/10.1038/s41598-022-18892-4>

6. Borkar T., Banerjee R. (2014) Influence of spark plasma sintering processing parameters on microstructure and mechanical properties of nickel. *Materials Science and Engineering A*, 618, 176–181. <https://doi.org/10.1016/j.msea.2014.08.070>
7. Roy S., Chatterjee A., Chakravorty D. (1993) Glass-metal nanocomposites in bulk form by sol-gel route followed by hot pressing. *Journal of Materials Research*, 8(4), 689–692. <https://doi.org/10.1557/JMR.1993.0689>
8. Doyle R.A., Rycroft S.F.W.R., Dewhurst C.D., et al. (1998) Effect of interfacial reactions of oxide particle-reinforced nickel-matrix composites on thermally induced strains. *Materials Science and Engineering A*, 252(1), 144–148. [https://doi.org/10.1016/S0921-5093\(98\)00515-2](https://doi.org/10.1016/S0921-5093(98)00515-2)
9. Corrias A., Ennas G., Musinu A., Paschina G., Zedda D. (1997) Iron-silica and nickel-silica nanocomposites prepared by high energy ball milling. *Journal of Materials Research*, 12(10), 2767–2772. <https://doi.org/10.1557/JMR.1997.0368>
10. Ennas G., Mei A., Musinu A., Piccaluga G., Pinna G., Solinas S. (1998) Sol-gel preparation and characterization of Ni-SiO₂ nanocomposites. *Journal of Non-Crystalline Solids*, 232–234, 587–593. [https://doi.org/10.1016/S0022-3093\(98\)00430-X](https://doi.org/10.1016/S0022-3093(98)00430-X)
11. Narváez L., Domínguez O., Martínez J.R., Ruiz F. (2003) Preparation of (Ni-B)/SiO₂, Ni/SiO₂ and NiO/SiO₂ nanocomposites. *Journal of Non-Crystalline Solids*, 318(1–2), 37–42. [https://doi.org/10.1016/S0022-3093\(02\)01877-X](https://doi.org/10.1016/S0022-3093(02)01877-X)
12. Kim H.-S., Kim C.S., Kim S.-G. (2006) Preparation of nickel oxide- and nickel-silica nanocomposites by spray pyrolysis. *Journal of Non-Crystalline Solids*, 352(21–22), 2204–2212. <https://doi.org/10.1016/j.jnoncrysol.2006.02.040>
13. Peng K., Zhou L., Hu A., Tang Y., Li D. (2008) Synthesis and magnetic properties of Ni-SiO₂ nanocomposites. *Materials Chemistry and Physics*, 111(1), 34–37. <https://doi.org/10.1016/j.matchemphys.2008.02.039>
14. Walia R., Pivin J.C., Chawla A.K., Jayaganthan R., Gautam Y.K., Chandra R. (2012) A comprehensive study of structural and magnetic properties of sputter deposited nickel-silica thin films. *Materials Science and Engineering B*, 177(13), 1073–1079. <https://doi.org/10.1016/j.mseb.2012.05.004>
15. Li R., Hou Y., Liu B., Wang D., Liang J. (2016) Electrodeposition of homogeneous Ni/SiO₂ nanocomposite coatings from deep eutectic solvent with in-situ synthesized SiO₂ nanoparticles. *Electrochimica Acta*, 222, 1272–1280. <https://doi.org/10.1016/j.electacta.2016.11.101>
16. Rouhollahi A., Fazlollahzadeh O., Dolati A., Ghahramanifard F. (2018) Effects of different surfactants on the silica content and characterization of Ni-SiO₂ nanocomposites. *Journal of Nanostructure in Chemistry*, 8, 139–152. <https://doi.org/10.1007/s40097-018-0259-4>
17. Ratajski T., Kalembe-Rec I., Indyka P., Kac S., Kot M., Dubiel B. (2018) Microstructural characterization of SiO₂/Ni nanocomposites electrodeposited from a sulphate bath modified by PEI. *Materials Characterization*, 142, 478–491. <https://doi.org/10.1016/j.matchar.2018.06.011>
18. Fang S., Wang H., Yang J., Yu B., Lu S. (2016) Formation of Si nanowires by the electrochemical reduction of SiO₂ with Ni or NiO additives. *Faraday Discussions*, 190, 433–449. <https://doi.org/10.1039/C5FD00222B>

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ORIGINAL STUDY

EFFECT OF THERMOCYCLIC ELECTROLYTE-PLASMA TREATMENT PARAMETERS ON THE STRUCTURAL-PHASE STATE AND TRIBOCORROSION BEHAVIOR OF 12KH1MF STEEL

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Abstract. In this work the influence of regimes of thermocyclic electrolyte-plasma treatment (TEPT) on structure-phase state, microhardness and tribocorrosion behavior of 12Kh1MF heat resistant low alloy steel for power plant steam pipelines and boilers is investigated. TEPT was performed in 10 % aqueous solution of Na₂CO₃. Voltage varied from 300 V (30 A) to 150 V (8 A) every 5 s. Three regimes were considered at maximum surface temperature from 800 to 900 °C. Samples were studied by SEM, X-ray phase analysis, Vickers microhardness test, ball-on-disk tribology test and neutral salt spray corrosion tests. TEPT has a layered structure - thermocyclic hardening zone, heat affected zone and base material. Hardened layer thickness is from 400 to 600 μm according to regimes. Ferrite pearlite initial structure transforms into martensite (α'-Fe) with Fe₃C carbides. Consequently, microhardness increases from 205 to 345-373 HV 0.1 (almost 1.7-1.8 times). Friction factor reduces from 0.627 to 0.596-0.617; wear intensity decreases up to 1.8-fold. Corrosion speed is reduced from 0.0024 to 0.0013-0.0019 mm / year. The optimal balance between friction and corrosion resistance was achieved in regime no 2. The maximum values of hardness and wear resistance were obtained for regime no 3. The obtained results indicate the effectiveness of TEPT on localized surface hardening of 12Kh1MF steel components working in abrasive and corrosive conditions.

Keywords: thermocyclic electrolyte-plasma treatment, 12Kh1MF steel, heat-resistant steel, martensite, microhardness, wear resistance, corrosion resistance.

1. Introduction

Heat-resistant structural steels are widely used in power engineering and other industries because they keep their properties at high temperatures and pressures. In thermal power plants, they are used for steam pipelines, turbine components and other parts. During long service, however, their structure gradually degrades and the steel loses strength [1]. Such steel is usually restored by conventional heat treatment, which acts on the whole volume of the part. However, many components need a hard, wear-resistant surface while the core stays ductile so that the assembly works reliably [2]. For this reason, researchers are developing hardening methods that change only the near-surface layer and keep the ductility of the bulk material. Electrolyte-plasma treatment, which is based on electric-discharge phenomena, is one such method [3]. The combination of them allows to increase strength, wear resistance and thermal stability of constructional steels, increase their operational lifetime in harsh environments.

The research on the electric-discharge techniques for materials processing progresses rapidly from theoretical to practical point of view. This resulted in development of highly effective and inexpensive technologies like: - Electrical discharge machining (EDM), [4, 5]; Electrospark machining (ESM), [6]; Electrolyte plasma hardening (EPH), [7]; Electrolyte plasma anodic treatment (EPAT), [8], Electrolyte plasma chemical thermal treatment (EPCTT) [9]. The mentioned technologies find wide application in different fields of industry, including power and mining industries.

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Garba et al. [10] showed that EDM is widely used in mould and die making, mechanical engineering, and the nuclear, aerospace and biomedical industries. Its main advantage is high precision in making complex parts. However, EDM has important drawbacks: a low material removal rate, poor surface quality, long processing times and high cost, and it can only process electrically conductive materials. These drawbacks limit its industrial use.

Korzhyk, Tyurin and Kolisnichenko [11]. Theoretical and experimental investigations of modification of metals by passing an electric current through a liquid - electrolyte electrode have been conducted in [11]. On its basis there were created complex hardening methods and devices for oxidation, alloying, structure changing of surface layer, not influencing on body part. Such technologies significantly increase the physical and mechanical characteristics of material - strength, resistance to wear and fatigue under friction.

Berladir et al. [12] compared nitrocarburising with cyclic heating and conventional isothermal nitrocarburising. They found that the thermocyclic process increases resistance to cavitation damage by 0.6–1.5 times. It also shortens treatment time, refines the grain structure and improves the mechanical properties of structural steel.

Among them, thermocyclic electrolyte-plasma treatment (TEPT) is one of the prospective technologies. The combination of thermal, electric and plasma actions on metal in an electrolyte – a current passing via the electrolyte into its surface; active components of the electrolyte are changing its surface - leads to high hardening of the surface layer. Repeated switching between heating and cooling results in substantial structure modification and enhancement of mechanical characteristics. This renders it suitable for industrial application.

Analysis of the literature revealed that electric discharge methods are able to substitute traditional thermal processing in order to enhance service characteristics of various steels. Process conditions for cathode electrolyte plasma treatment by means of thermocycling remain unexplored. The objective of this paper was investigation of influence of modes of thermocycle electrolyte plasma treatment on structure - phase composition and tribocorrosion behavior of 12Kh1MF construction steel.

2. Materials and methods

The investigated material was 12Kh1MF steel - heat resistant low alloy steel, which is used in power engineering as pipelines, boilers and other parts of high temperature operation under aggressive conditions. Composition by mass percent according to GOST 20072-74: C 0.10–0.15 %, Si 0.17–0.37 %; Mn 0.40–0.70 %; Cr 0.90–1.20 %; Mo 0.25–0.35 %; V 0.15–0.30 %, max. P 0.035 %; S 0.030 %; remainder – Fe.

Thermocyclic electrolyte-plasma treatment (TEPT) was performed in the electrolyte-plasma treatment device of the Surface Engineering and Tribology Research Center, Sarsen Amanzholov East Kazakhstan University. The device has a power source and an electrochemical cell (Fig. 1).

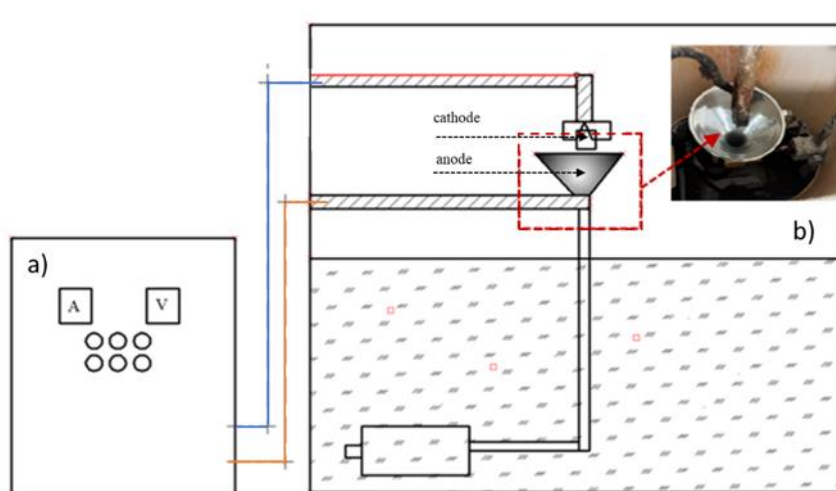


Fig. 1. Schematic diagram of the unit for thermocyclic electrolyte-plasma treatment: a) power supply; b) electrolytic cell.

The process works as follows. At a low voltage of about 150 V, ordinary electrolysis takes place in the cell. When the voltage is raised to 300 V, gas is released intensively at the electrode, and a vapour-gas envelope

forms around the sample surface. The electric field strength in this envelope reaches 10^4 – 10^5 V/cm. At a temperature of about 100 °C, this field ionises the vapour and causes emission of ions and electrons, which keeps a stable electric discharge going and produces an electrolyte plasma. The aqueous electrolyte serves as both the heating and the cooling medium.

An aqueous solution of sodium carbonate (Na_2CO_3) was used as the electrolyte. Our earlier studies [13, 14] showed that a higher Na_2CO_3 concentration increases the electrical conductivity of the electrolyte, which leads to high current density and intense heating. Compared with a 15% solution, a 10% solution gives a lower current density, so heating is smoother, the surface does not melt, and the heating process is easier to control. The electrolyte for the present study was thus 10 wt.% Na_2CO_3 in distilled water.

The TEPT regime parameters are given in Table 1. Each thermocycle consisted of four stages: heating at 300 V (30 A) for 5 s, holding at 150 V (8 A) for 5 s, heating at 300 V for 4 s and holding at 150 V for 5 s, giving 19 s per cycle. Regimes No. 1, No. 2 and No. 3 consisted of one, two and three cycles, respectively.

Table 1. Results of corrosion tests of samples before and after TEPT.

Stage within one cycle		Voltage U, V	Current I, A	Duration t, s
1 – heating		300	30	5
2 – holding		150	8	5
3 – heating		300	30	4
4 – holding		150	8	5
Regime	Number of cycles	Total treatment time, s	Max. surface temperature, °C	Electrolyte temperature, °C
No. 1	1	19	800	36
No. 2	2	38	850	37
No. 3	3	57	900	39

Metallographic analysis was performed after grinding and polishing on the Metapol - 2000 P device. Etching with a 4 % solution of HNO_3 in ethanol was used to show the structure. Structure of raw and processed specimens studied using TESCAN MIRA SEM at x60 and x1000 magnification.

The phase composition before and after TEPT was determined by X-ray phase analysis on an X'Pert Pro diffractometer. Scans were recorded in the 2θ range of 10–90° with a step of 0.02°.

Cross-sectional microhardness was determined using METOLAB 502 microhardness meter, Vickers diamond pyramid indenter (136° angle of opposite faces), by the method of GOST 9450-76. Load - 1 N (HV0.1), duration of holding under load - 10 s.

Tribological tests were carried out on a TRB3 tribometer using the ball-on-disk method. The load was 6 N, the sliding speed was 2 cm/s, the sliding distance was 60 m, and the wear track radius was 3 mm. The counterbody was a 6 mm ball of 100Cr6 steel (equivalent to ShKh15).

Resistance to corrosion was determined in HUD-E808 salt spray chamber according to GOST 34388-2023. Neutral 5 % NaCl solution; $T=35$ °C, $t=8$ h. Quantitative assessment of corrosion damage performed according to GOST 9.908-85 by determining the mass loss per unit surface area $\Delta m(\text{g}/\text{m}^2)$, which is calculated by formula (1).

$$\Delta m = \frac{m_0 - m_1}{S}, \quad (1)$$

where m_0 is the sample mass before testing, g; m_1 is the sample mass after testing and removal of corrosion products, g; S is the sample surface area, m^2 .

3. Results and discussion

Fig. 2 presents SEM micrographs of transversal sections from samples of 12Kh1MF steel after TEPT at a concentration of the 10 % Na_2CO_3 electrolyte in different modes (No. 1, No. 2 and No. 3) prior to treatment - the structure is ferrite - pearlite; after TEPT - the surface layer is predominantly martensitic, as a result of rapid cooling of the heated surface with an electrolyte solution. The transformation into martensite accounts for increased microhardness values and improved tribocorrosion behavior (Table 3).

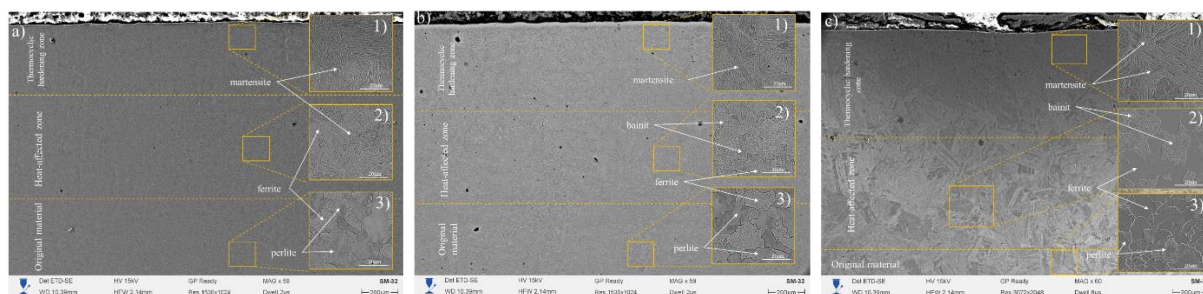


Fig. 2. SEM images of cross-sections of 12Kh1MF steel after TEPT in regimes No. 1 (a), No. 2 (b) and No. 3 (c): 1) thermocyclic hardening zone; 2) heat-affected zone; 3) base material.

In any case, TEPT results in formation of a zonal structure comprising three areas with different microstructure and hardness - thermocyclic hardening layer, heat affected layer, and basic metal (Fig. 2). At the end of regime No. 1 the thickness of hardening layer reaches approximately 400 μm and comprises martensite (Fig. 2a, area 1), while below it there is an HAZ where there is a smooth change from martensite to initial ferrite – pearlite structure (Fig. 2a, areas 2 and 3); at the end of regime No. 2 - a hardening layer up to ~ 500 μm thick (Fig. 2b, zone 1) is formed as well, mainly consisting of martensite; in the heat affected zone there are bainite and ferrite present (Fig. 2b, zones 2 and 3); Regime No. 3 results in the largest depth of hardness, roughly 600 μm . Here, the transition from martensite through bainite to heat affected zone occurs (Fig. 2c). With increasing heat input of the regime, the thickness of martensite increases accordingly to approx. 600 μm .

In order to determine phases, X - ray phase analysis was carried out for surface of specimens before and after TEPT (Fig. 3). Before treatment there is only α - Fe in the steel that corresponds to ferrite - pearlite microstructure. Following the TEPT all specimens contain martensite (α' - Fe) plus cementite (Fe_3C), hardening agent of steels. Results are consistent with the SEM data.

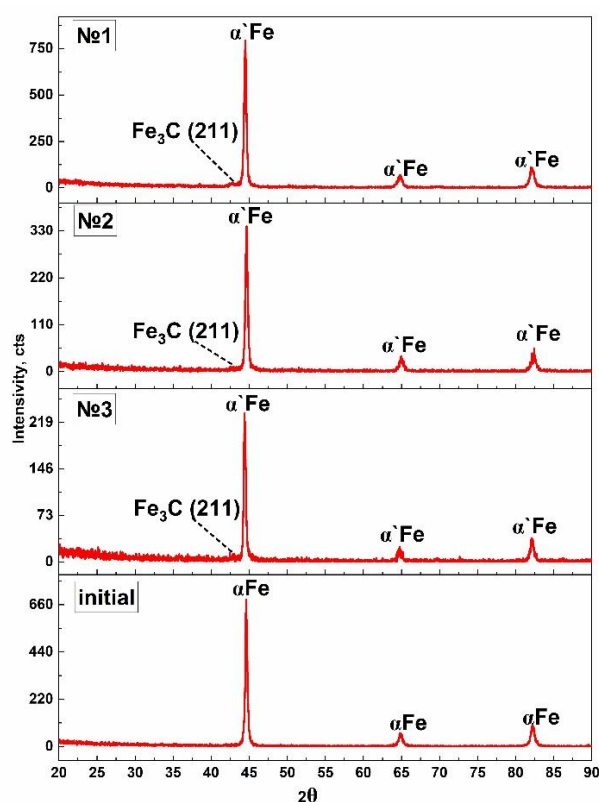


Fig. 3. X-ray diffraction patterns of 12Kh1MF steel before and after TEPT in different regimes.

The surface microhardness results are given in Table 3. Microhardness rose from 205 $\text{HV}_{0.1}$ in the initial state to 345, 365 and 373 $\text{HV}_{0.1}$ after regimes No. 1, No. 2 and No. 3. This is about 1.7–1.8 times higher and

is due to the formation of martensite. Microhardness increases slightly from regime to regime, following the growth of the hardened layer.

Figure 4 shows the results of the tribological tests. The friction coefficient of the untreated steel is 0.627. After TEPT, it decreases to 0.617, 0.596 and 0.613 for regimes No. 1, No. 2 and No. 3, respectively. The lowest value is for regime No. 2, about 5% below the initial state. Wear resistance improves much more. Wear intensity falls from $4.7 \cdot 10^{-4}$ to $4.4 \cdot 10^{-4}$, $3.0 \cdot 10^{-4}$ and $2.7 \cdot 10^{-4}$ mm³/(N·m), which means wear resistance is 1.1, 1.5 and 1.8 times higher, respectively. Wear resistance increases along with microhardness, so the harder martensitic layer resists wear better.

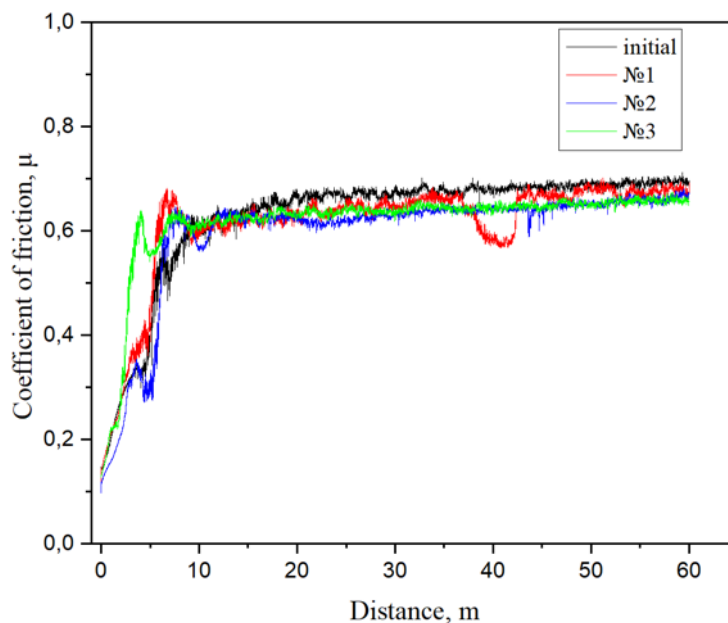


Fig. 4. Tribological test results before and after TEPT.

The results of the salt-spray corrosion tests are presented in Table 3. Losses in mass per unit surface were determined using eq. (1) - they reduced following treatment in each case – approximately 32 % for regime No. 1; 43 % for regime No. 2 and 20 % for regime No. 3. The corrosion rate follows a similar pattern. It drops from 0.0024 mm/year for the non-treated steel to 0.0016, 0.0013 and 0.0019 mm/year respectively. Best corrosion protection is provided by regime No. 2. Regime No. 3 has the largest amount of heat input, so it probably results in greater residual stresses. This may account for localised corrosion, and thus explains why regime No. 3 is more susceptible to corrosion than regime No. 2 despite having a higher hardness value.

Table 2. Results of corrosion tests of samples before and after TEPT.

Sample	Mass before testing, g	Mass after testing, g	Loss of mass, g	Loss of mass per unit area Δm , kg/m ²
Initial	9.5391	9.5299	0.0092	0.0191
Cycle No.1	11.2293	11.2198	0.0095	0.0129
Cycle No.2	8.4946	8.4883	0.0063	0.0109
Cycle No.3	7.0022	6.9941	0.0081	0.0152

Table 2 summarises all the results. The regimes affect the properties in different ways. Regime No. 3 gives the thickest hardened layer and the highest microhardness and wear resistance. Regime No. 2 gives the lowest friction coefficient and the best corrosion resistance, together with high hardness and wear resistance. Overall, a 10% Na₂CO₃ electrolyte combined with regimes No. 2 and No. 3 provides steady power supply and regulated heat treatment, resulting in an optimal set of mechanical and tribocorrosion characteristics for 12Kh1MF steel.

Table 3. Phase composition, microhardness, tribological and corrosion properties of samples before and after TEPT.

Sample	Phase composition	Microhardness, HV _{0.1}	TRB3		Corrosion rate, mm/year
			μ	Wear intensity, mm ³ /N×m	
Initial	α Fe	205±30	0.627	0.0004706	0.0024
Cycle No.1	α' Fe, Fe ₃ C	345±30	0.617	0.0004391	0.0016
Cycle No.2	α' Fe, Fe ₃ C	365±34	0.596	0.0003047	0.0013
Cycle No.3	α' Fe, Fe ₃ C	373±31	0.613	0.0002672	0.0019

4. Conclusion

Based on the results, the following conclusions were drawn:

1. TEPT of 12Kh1MF steel in an electrolyte of 10% Na₂CO₃ in distilled water produces a zonal structure in the surface layer. It consists of a thermocyclic hardening zone, a heat-affected zone and the base material. The hardened layer grows with the number of cycles, from about 400 μ m for regime No. 1 to about 600 μ m for regime No. 3.
2. X-ray phase analysis and SEM showed that the initial ferrite-pearlite structure (α -Fe) turns into martensite (α' -Fe) with cementite (Fe₃C) in all regimes. As a result, microhardness rises from 205 to 345–373 HV_{0.1}, which is about 1.7–1.8 times higher.
3. TEPT improves the tribological properties of the steel. The friction coefficient falls from 0.627 to 0.596–0.617, and wear intensity drops from $4.7 \cdot 10^{-4}$ to $2.7 \cdot 10^{-4}$ mm³/(N·m), which means wear resistance is up to 1.8 times higher. Wear resistance increases along with microhardness and the thickness of the hardened layer.
4. Salt spray tests showed better corrosion resistance after treatment. The corrosion rate fell from 0.0024 to 0.0013–0.0019 mm/year, a reduction of 21–46%. Regime No. 2 was the most corrosion-resistant. The weaker result for regime No. 3 is likely due to higher residual stresses caused by its greater heat input.
5. The recommended TEPT regime for 12Kh1MF steel is a 10% aqueous Na₂CO₃ electrolyte, with the voltage alternating between 300 V (30 A) and 150 V (8 A), and 2–3 thermocycles, each lasting 19 s, at a maximum surface temperature of 850–900 °C. Regime No. 2 gives the best balance of hardness, low friction and corrosion resistance. Regime No. 3 gives the highest hardness and wear resistance. The choice between them depends on the service conditions of the part.

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Conflict of interest statement

The authors declare that they have no competing interests.

References

1. Zrilić M., Aleksić R. (2003) The effect of long-term exposure to elevated temperature on steam line steel properties. *Thermal Science*. 7(1), 33–46.
2. Zakharova I., Studenikin D. (2024) Effect of heat treatment processes on the characteristics of welded joints in the production of pipes for critical environments. *Nauka ta vyrobnytstvo*. 28, 79–85. [in Ukrainian]
3. Bolshakov V.I., Gubenko S.I. (2004) Metallurgy and heat treatment of metals. *Dnepropetrovsk: PGASA*, 145 p. [in Russian]
4. Yurikov Yu.V. (2000) Improving the efficiency of electrical discharge restoration of shaft journals. *Cand. Diss. of the Techn. Science degree*, Voronezh. [in Russian]
5. Takuntsov K.V., Zaitsev A.N. (2001) Electrical discharge-electrochemical machining: current state, problems, prospects. *Advanced technologies of physicochemical dimensional machining and formation of operational properties of metals and alloys*, Ufa, 9–13. [in Russian]
6. Koch O., Ehrfeld W., Michel F. (2001) Recent progress in micro-electro discharge machining. Part 1: Technology. *Proceedings of the International Symposium on Electromachining (ISEM XIII)*, Bilbao, Spain, 117–127.

7. Fujino M., Okamoto N., Masuzawa T. (1995) Development of multi-purpose microprocessing machine. *Proceedings of the International Symposium on Electromachining (ISEM XI)*, Lausanne, Switzerland, 613–620.
8. Stavitsky B.I. (2000) Electrospark machining of materials: Lazarenko's method at the turn of the century. 5, 25–40. [in Russian]
9. Baizhan D., Rakhadilov B., Zhurerova L., Tyurin Y., Sagdoldina Zh., Adilkanova M., Kozhanova R. (2022) Investigation of changes in the structural-phase state and the efficiency of hardening of 30CrMnSiA steel by the method of electrolytic plasma thermocyclic surface treatment. *Coatings*. 12(11), 1696. <https://doi.org/10.3390/coatings12111696>
10. Garba E., Abdul-Rani A.M., Yunus N.A., Aliyu A.A., Gul I.A., Al-Amin M., Aliyu R. (2023) A review of electrode manufacturing methods for electrical discharge machining: current status and future perspectives for surface alloying. *Machines*. 11(9), 906. <https://doi.org/10.3390/machines11090906>
11. Korzhyk V., Tyurin Yu., Kolisnichenko O. (2021) Surface modification of metal products by electrolyte plasma: monograph. *Kharkiv: PC Technology Center*, 180 p. <https://doi.org/10.15587/978-617-7319-47-3>
12. Berladir K., Hovorun T., Gusak O. (2021) Strengthening of the NKV type centrifugal pump's shaft by chemical-thermocycling treatment. *Advanced Manufacturing Processes II. Lecture Notes in Mechanical Engineering*. Springer, Cham, 525–535. https://doi.org/10.1007/978-3-030-77719-7_52
13. Sulyubayeva L.G., Buitkenov D.B., Baizhan D.R., Berdimuratov N.E., Raisov N.S., Zhumabekov A.Zh. (2025) Influence of thermocyclic electrolyte-plasma treatment on mechanical properties of U9 tool steel. *Bulletin of the University of Karaganda. Physics Series*. 30, 1(117), 77–88. <https://doi.org/10.31489/2025PH1/77-88>
14. Popova N.A., Zhurerova L.G., Nikonenko E.L., Skakov M.K. (2016) Phase transformations in 30KhGS steel under electrolyte-plasma nitrocarburizing. *Bulletin of Tomsk State University*. 1, 60–70. [in Russian]

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ORIGINAL STUDY

HARDNESS AND POST-SCRATCH SURFACE MORPHOLOGY OF Cr_3C_2 -NiCr COATINGS AFTER ULTRASONIC NANOCRYSTAL SURFACE MODIFICATIONA.Y. Kussainov¹, I.K. Abizhanova¹, M.T. Kaliaskarova¹, Y.S. Molbossynov¹, N.M. Magazov¹¹D. Serikbayev East Kazakhstan Technical University, Ust-Kamenogorsk, Kazakhstan*Corresponding author: mkaliaskarova@edu.ektu.kz

Abstract. The hardness and post-scratch surface morphology of Cr_3C_2 -NiCr coatings subjected to ultrasonic nanocrystal surface modification (UNSM) were examined for detonation-sprayed (DS) and high-velocity oxy-fuel (HVOF) deposits. Four treated samples per deposition route were evaluated at static loads of 20 and 30 N and vibration amplitudes of 20 and 30 μm . Instrumental hardness was measured at a load of 300 mN with a dwell time of 10 s. The DS samples exhibited hardness values of 820–970 HV, whereas the HVOF samples covered the wider range of 546–1165 HV. The highest values were recorded for DS-T1 at 20 N/20 μm and HVOF-T4 at 30 N/30 μm . SEM fields acquired after scratch testing showed heterogeneous surface contrast and crack-like discontinuities in both coating series. These observations provide qualitative evidence of the post-test surface condition but do not isolate damage caused by UNSM or establish a ranking of scratch resistance. Available mean friction coefficients are reported as supplementary observations for six treated samples. The results demonstrate a sample-dependent hardness response and emphasize the need to assess local mechanical properties together with surface integrity.

Keywords: Cr_3C_2 -NiCr; detonation spraying; HVOF; ultrasonic nanocrystal surface modification; instrumental hardness; scanning electron microscopy; scratch damage.

1. Introduction

Cr_3C_2 -NiCr coatings combine hard chromium-carbide particles with a metallic binder and are used to protect surfaces exposed to sliding and abrasive contact. Their response depends on how the carbide phase is supported within the deposited structure. In HVOF- and HVOF-sprayed chromium-carbide coatings, the cohesion within and between deposited regions influences fragment removal and carbide pull-out during wear [1]. The deposition route and its operating parameters therefore affect the performance of the coating as a composite, beyond the hardness of its individual constituents. For detonation-sprayed Cr_3C_2 -NiCr, changes in barrel filling have been shown to alter phase composition, hardness and tribological response [2].

Post-deposition treatment offers an additional means of modifying this structure. For example, pulse-plasma treatment of detonation-sprayed Cr_3C_2 -NiCr produced changes in phase composition and surface morphology together with increased hardness and wear resistance [3]. Such results demonstrate the potential of post-treatment, while also showing that its effect must be assessed for the particular coating and processing method. Thermal modification and repeated mechanical impact act through different pathways; improvements obtained with one treatment do not establish the outcome of another.

Ultrasonic nanocrystal surface modification (UNSM) applies repeated impacts through a contacting tool under a static load. Research on thermally sprayed ceramic coatings has examined changes in hardness, roughness and resistance to adhesive failure following this treatment [4]. More directly relevant studies of HVOF Cr_3C_2 -NiCr have investigated its friction and wear in fuel-lubricated contacts [5] and its scratch response after UNSM [6]. These studies motivate the use of ultrasonic treatment for chromium-carbide

coatings, but also highlight the importance of considering several responses. A mechanically modified binder and a carbide-rich region need not accommodate repeated contact in the same manner.

Hardness and resistance to cracking address complementary aspects of coating performance. Indentation characterizes local resistance to penetration, whereas scratch testing introduces a moving contact and allows examination of cracking and other damage along the loaded region. Work on HVOF Cr_3C_2 -NiCr-based coatings with cobalt additions has used scratch morphology and critical loads to characterize failure [7]. Dynamic impact studies of chromium-carbide coatings have likewise related contact durability to the binder and deposited microstructure [8]. These findings support combining hardness measurements with observations of damage, rather than using maximum hardness as the sole criterion for post-treatment selection.

The present study examines the hardness and qualitative resistance to cracking under scratch loading of UNSM-treated DS and HVOF Cr_3C_2 -NiCr coatings. Static loads of 20 and 30 N and vibration amplitudes of 20 and 30 μm are considered. SEM observations are used to describe the post-scratch crack morphology, while published as-sprayed hardness values [9] provide reference context. Available friction coefficients are included as secondary observations. The objective is to establish the measured hardness response across the treated samples and describe the accompanying damage morphology without equating qualitative scratch observations with a measured fracture-toughness parameter.

2. Materials and methods

2.1. Coating preparation and reference states

Cr_3C_2 -NiCr deposits were produced by detonation spraying using a CCDS2000 system and by propane-fuelled HVOF spraying. The recorded HVOF settings were a propane pressure of 2.9 bar, oxygen pressure of 5.0 bar, compressed-air pressure of 3.2 bar and a spraying distance of 200 mm.

2.2. Ultrasonic treatment conditions

The treatment system used a nominal vibration frequency of 20 kHz, a spherical-ended hard-alloy tool and pneumatic application of a static load. The process geometry is illustrated in Fig. 1. Sample codes DS-T1–DS-T4 and HVOF-T1–HVOF-T4 identify the treated coatings by deposition route. The suffix identifies a specimen, not a unique load–amplitude combination. The documented amplitudes and static loads are listed in Table 1. The condition number is an identifier and does not represent a monotonic increase in treatment intensity.

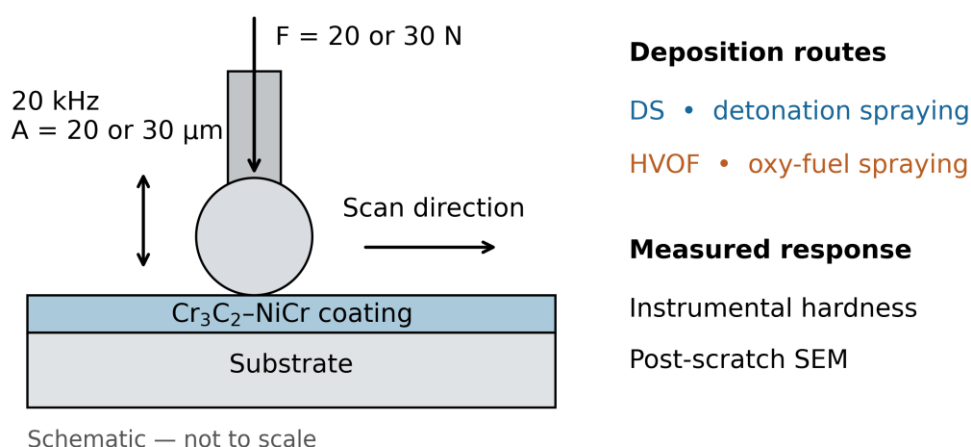


Fig. 1. Schematic of the UNSM contact and the two deposition routes. The drawing illustrates the process geometry and is not to scale.

Table 1. Sample designation and UNSM parameters for Cr₃C₂-NiCr coatings.

Sample	Route	Static load (N)	Amplitude (μm)
DS-T1	DS	20	20
DS-T2	DS	30	20
DS-T3	DS	20	30
DS-T4	DS	20	30
HVOF-T1	HVOF	20	20
HVOF-T2	HVOF	30	20
HVOF-T3	HVOF	20	30
HVOF-T4	HVOF	30	30

2.3. Mechanical and tribological characterization

Instrumental hardness was characterized using a FISCHERSCOPE HM2000 system controlled by WIN-HCU software. The applied indentation load was 300 mN with a dwell time of 10 s. The general coating characterization procedure involved indentation of polished cross-sections and averaging at least ten measurements per specimen. Scratch testing followed by scanning electron microscopy was used for a qualitative assessment of coating resistance to crack initiation and propagation under a moving contact. Eight fields were associated with the treated samples, with one field per sample. These images document the post-scratch condition rather than the surface immediately after UNSM. Original image fields and scale bars were retained. The images were assessed qualitatively for surface heterogeneity and visible discontinuities; no quantitative grain-size, porosity or crack-density analysis was performed. Supplementary dry sliding measurements were obtained at room temperature using an Anton Paar TRB³ tribometer in a ball-on-disc configuration.

3. Results and discussion

3.1. Hardness response to ultrasonic treatment

Figure 2 shows the hardness values of both coating series. The four DS conditions yielded 970, 860, 930 and 820 HV for T1–T4, respectively. The maximum occurred at T1 and the minimum at T4, with an overall span of 150 HV. T2 and T3 differed by 70 HV. Since their amplitude and load change simultaneously, this difference cannot be attributed to either parameter alone.

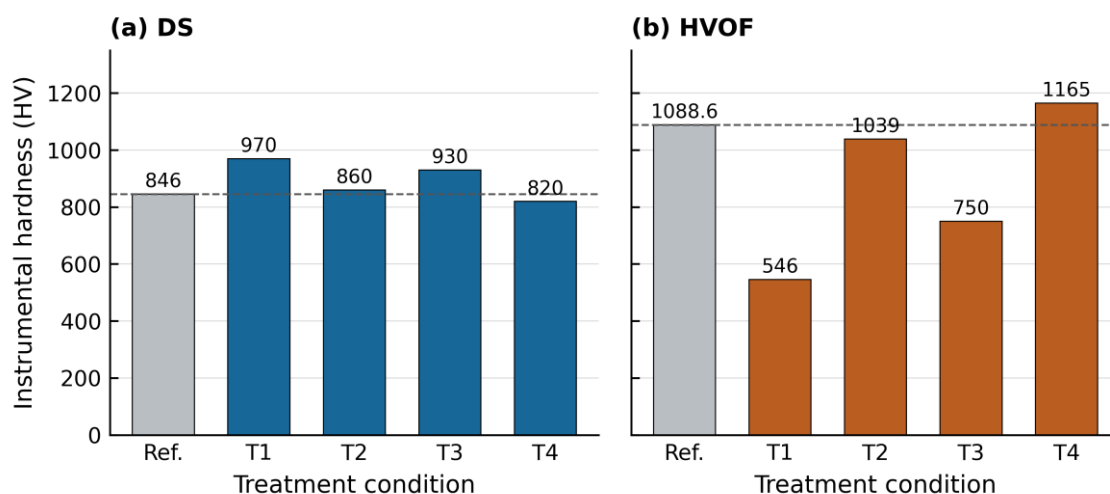


Fig. 2. Instrumental hardness for (a) DS and (b) HVOF coatings. Grey bars and dashed lines identify published as-sprayed references [9]; coloured bars represent the UNSM conditions.

The HVOF series yielded 546, 1039, 750 and 1165 HV for T1–T4. The range across the four conditions was 619 HV, substantially wider than the DS range. This is variation between processing conditions; it must not be confused with repeatability or scatter within a condition. The maximum HVOF hardness was measured at T4, despite the large hardness reduction observed at T1.

The published reference hardness values were 846 HV for DS and 1088.6 HV for HVOF [9]. Three treated DS samples were numerically above the DS reference, while only HVOF-T4 exceeded the HVOF reference. These comparisons do not establish an optimal setting. DS-T3 and DS-T4, both recorded at 20 N/30 μm , yielded 930 and 820 HV, respectively; the available record does not establish their status as independent processing replicates. Their different results prevent assigning all variation solely to the two listed treatment parameters.

A possible interpretation is competition between plastic deformation of the binder and accumulation of damage in the composite. Both the carbide distribution and cohesion between deposited regions can influence how repeated impacts are accommodated. This interpretation is consistent with the importance of cohesion in chromium-carbide coating wear [1], but the present hardness data alone do not identify a fracture mechanism. No quantitative grain-size, residual-stress or porosity measurements are used here to claim nanostructuring or densification.

3.2. Qualitative assessment of cracking resistance by scratch testing

Figure 3 presents the available SEM fields for all eight treated samples. Both series display spatially heterogeneous contrast with irregular dark discontinuities. Crack-like lines are visible in multiple HVOF and DS fields, including branched or intersecting features. Their appearance varies from one field to another. Dark regions alone cannot be identified unambiguously as open pores, detached material or compositional contrast without additional imaging or local chemical analysis.

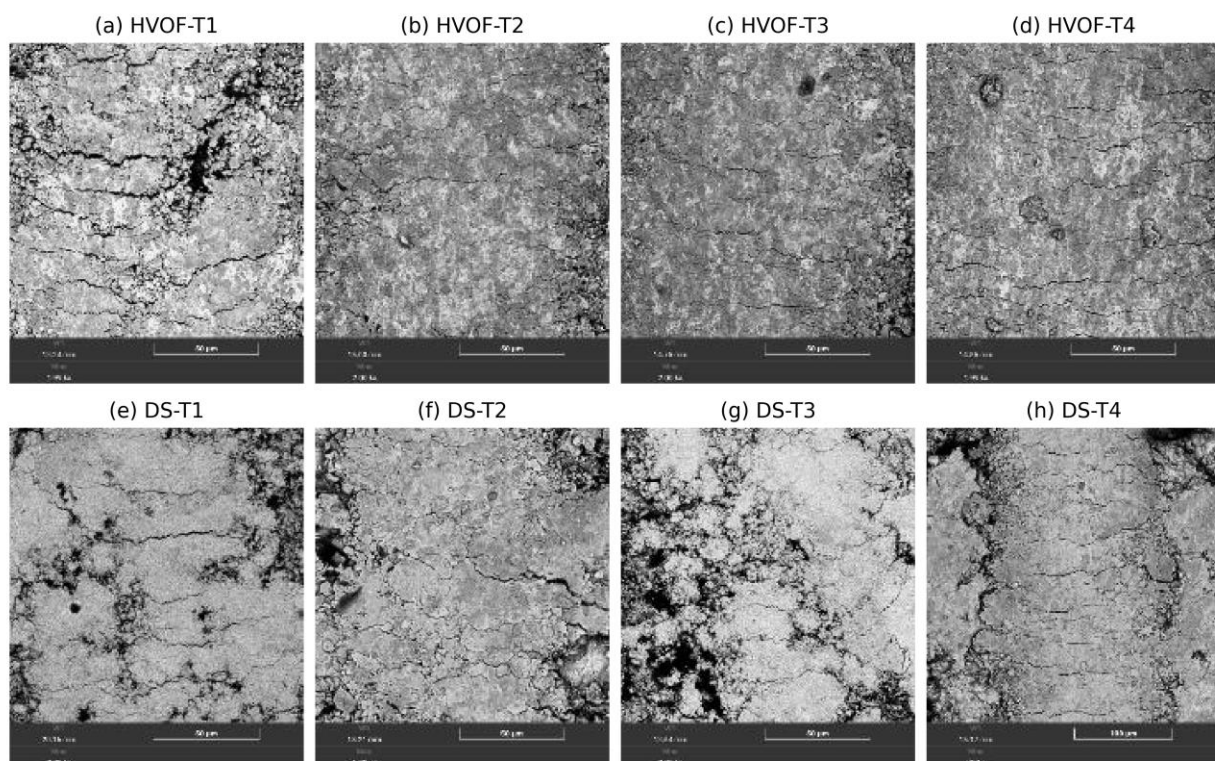


Fig. 3. SEM fields of Cr_3C_2 –NiCr coatings after scratch testing: (a–d) HVOF-T1–HVOF-T4 and (e–h) DS-T1–DS-T4.

Scratch-induced damage is evaluated here through the continuity, branching and spatial distribution of visible cracks. Extended crack-like features and locally connected discontinuities occur in the presented fields, indicating that cracking contributes to the response of both coating series under mechanical contact.

Differences between individual fields describe local damage morphology; they are not sufficient to rank the resistance to cracking of entire coating series.

3.3. Supplementary friction observations

The available mean friction coefficients are listed in [Table 2](#). The measured values span 0.678–0.726 for DS and 0.711–0.757 for HVOF. HVOF-T4 has the highest measured hardness and the highest available friction coefficient within its series, so the two criteria do not give the same sample ranking.

Table 2. Available mean friction coefficients after UNSM. NR denotes an unreported value.

Sample suffix	DS	HVOF
T1	0.678	NR
T2	NR	0.722
T3	0.726	0.711
T4	0.717	0.757

Published studies of UNSM-treated Cr_3C_2 –NiCr coatings under fuel-lubricated sliding [5,6] provide relevant context but involve a different contact environment. Their reported improvements cannot be transferred directly to the present observations.

4. Conclusion

The hardness, post-scratch surface morphology and frictional response of UNSM-treated Cr_3C_2 –NiCr coatings produced by detonation spraying and HVOF spraying were examined. The following conclusions were drawn:

1. The DS coatings exhibited hardness values of 820–970 HV, whereas the HVOF coatings showed a wider range of 546–1165 HV. The highest hardness in the DS series was 970 HV for DS-T1, treated at a static load of 20 N and a vibration amplitude of 20 μm . For HVOF coatings, the highest value was 1165 HV for HVOF-T4, treated at 30 N and 30 μm . These results demonstrate that the hardness response across the investigated treatment conditions differs between the two deposition routes.

2. SEM examination after scratch testing revealed crack-like discontinuities with varying continuity, branching and spatial distribution in both coating series. Extended and interconnected features characterized the local damage patterns under scratch loading. The joint examination of these features provided a qualitative characterization of cracking behaviour and complemented the hardness measurements.

3. The measured mean friction coefficients ranged from 0.678 to 0.726 for DS coatings and from 0.711 to 0.757 for HVOF coatings. DS-T1 combined the highest hardness in its series with the lowest measured friction coefficient of 0.678. HVOF-T4 exhibited both the highest hardness and the highest friction coefficient in its series, at 0.757. Thus, the relationship between hardness and friction depended on the coating series and treatment condition.

4. The combined assessment of hardness, post-scratch morphology and friction distinguished the mechanical and contact responses of the treated coatings. DS-T1 showed a favourable combination of hardness and friction within the DS series, while HVOF-T4 achieved the highest hardness among all investigated specimens. These findings identify distinct property combinations for the two deposition routes and provide a basis for selecting treatment conditions according to the required coating response.

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Conflict of interest

The authors declare that they have no competing interests.

References

1. Bolelli G, Berger LM, Börner T, Koivuluoto H, Matikainen V, Lusvarghi L, et al. Sliding and abrasive wear behaviour of HVOF- and HVOF-sprayed $\text{Cr}_3\text{C}_2\text{-NiCr}$ hardmetal coatings. *Wear*. 2016; 358–359:32–50. <https://doi.org/10.1016/j.wear.2016.03.034>.
2. Rakhadilov B, Kakimzhanov D, Dautbekov M, Sagdoldina Z, Adylkanova M, Abylkalykova R. Influence of spraying parameters on the structure and tribological properties of $\text{Cr}_3\text{C}_2\text{-NiCr}$ detonation coatings. *Advances in Tribology*. 2023;2023:6684656. <https://doi.org/10.1155/2023/6684656>.
3. Kakimzhanov D, Rakhadilov B, Sulyubayeva L, Dautbekov M. Influence of pulse-plasma treatment distance on structure and properties of $\text{Cr}_3\text{C}_2\text{-NiCr}$ -based detonation coatings. *Coatings*. 2023; 13(11):1824. <https://doi.org/10.3390/coatings13111824>.
4. Amanov A. Wear resistance and adhesive failure of thermal spray ceramic coatings deposited onto graphite in response to ultrasonic nanocrystal surface modification technique. *Applied Surface Science*. 2019; 477:184–197. <https://doi.org/10.1016/j.apsusc.2017.11.013>.
5. Amanov A, Berkebile S. Improvement in tribological behavior of thermal spray Cr_2O_3 and $\text{Cr}_3\text{C}_2\text{-NiCr}$ coatings by ultrasonic nanocrystal surface modification. *Materials Letters*. 2022; 314:131919. <https://doi.org/10.1016/j.matlet.2022.131919>.
6. Amanov A, Berkebile SP. Enhancement of sliding wear and scratch resistance of two thermally sprayed Cr-based coatings by ultrasonic nanocrystal surface modification. *Wear*. 2023; 512–513:204555. <https://doi.org/10.1016/j.wear.2022.204555>.
7. Ksiazek M, Łyp-Wrońska K. An HVOF-sprayed ($\text{Cr}_3\text{C}_2\text{-NiCr+Co}$) composite coating on ductile cast iron: microstructure, mechanical properties, and scratch resistance. *Materials*. 2024; 17(7):1484. <https://doi.org/10.3390/ma17071484>.
8. Daniel J, Grossman J, Houdková Š, Bystrianský M. Impact wear of the protective Cr_3C_2 -based HVOF-sprayed coatings. *Materials*. 2020; 13(9):2132. <https://doi.org/10.3390/ma13092132>.
9. Kussainov AY, Kaliaskarova MT, Abizhanova IK, Molbossynov YS, Bayatanova LB, Zhunisova GZh. Comparative analysis of WC-NiCr, $\text{Al}_2\text{O}_3\text{-NiCr}$, and $\text{Cr}_3\text{C}_2\text{-NiCr}$ metal-ceramic coatings applied by HVOF and detonation spraying methods. *Recent Contributions to Physics*. 2026; 97(2):94–103. <https://doi.org/10.26577/RCPH97220269>.

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ORIGINAL STUDY

EFFECT OF FLAME-SPRAYING TEMPERATURE ON THE PROPERTIES OF Ni–Cr–B–Si-BASED SELF-FLUXING COATINGS

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Abstract. The present study investigates the effect of flame-spraying process parameters on the microstructure and mechanical properties of Ni–Cr–B–Si-based self-fluxing coatings. Corrosion-resistant AISI 321 austenitic chromium–nickel steel was used as the substrate material. The coatings were deposited by flame spraying using a Metal Coat 6PM-II system. To vary the thermal conditions of the spraying process, the oxygen-to-acetylene (O_2/C_2H_2) ratio was adjusted to 1.0, 1.1, and 1.2. The spraying parameters were monitored using an Accuraspray 4.0 diagnostic system. The resulting coatings were characterized in terms of microstructure, porosity, microhardness, surface roughness, and adhesion strength. Increasing the O_2/C_2H_2 ratio resulted in densification of the coating structure and a decrease in porosity from $1.97 \pm 0.13\%$ to $0.90 \pm 0.09\%$. Concurrently, the surface roughness decreased from $4.70 \pm 0.09 \mu m$ to $2.99 \pm 0.11 \mu m$, while the adhesion strength increased from $22.14 \pm 4.3 MPa$ to $35.02 \pm 3.9 MPa$. The highest average microhardness of 1051.7 HV was obtained at an O_2/C_2H_2 ratio of 1.1. Among the investigated spraying conditions, the coating produced at $O_2/C_2H_2 = 1.2$ exhibited the lowest porosity and surface roughness together with the highest adhesion strength. The results demonstrate that controlling the O_2/C_2H_2 ratio provides an effective means of tailoring the microstructural and mechanical characteristics of flame-sprayed Ni–Cr–B–Si-based self-fluxing coatings.

Keywords: flame spraying; self-fluxing alloy; Ni–Cr–B–Si; AISI 321; mechanical properties.

1. Introduction

Metal components operating in the power engineering industry are exposed to severe degradation factors, including elevated temperatures, oxidizing environments, high-velocity gas flows, and erosive attack [1]. Under such operating conditions, high-temperature corrosion develops on metallic surfaces, resulting in material loss, changes in component geometry, and deterioration of mechanical properties. Consequently, the degradation rate of equipment increases, its service life is shortened, and maintenance and repair costs rise.

One effective approach to addressing this problem is the formation of corrosion- and wear-resistant protective coatings on component surfaces. According to previous studies, nickel-based Ni–Cr–B–Si self-fluxing alloys are considered promising materials for coatings intended for high-temperature applications [2]. Chromium enhances oxidation and corrosion resistance, whereas boron and silicon promote the self-fluxing behavior of the alloy and facilitate the formation of a denser coating structure.

Various technologies are currently employed for the deposition of protective coatings, including plasma spraying, high-velocity oxy-fuel (HVOF) spraying, laser cladding, and flame spraying. Plasma and HVOF spraying can provide coatings with low porosity and high adhesion strength [3]. However, these processes generally require sophisticated equipment, relatively high energy consumption, and precise control of processing parameters. In addition, the high gas consumption, equipment cost, and maintenance requirements associated with HVOF technology may limit its economic feasibility for certain industrial applications [4].

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Flame spraying provides an alternative approach that enables coatings to be deposited using comparatively simple and cost-effective equipment. In this process, powder particles are heated by an oxy-acetylene flame and accelerated toward the substrate surface. The major advantages of this technology include relatively simple equipment design, operational flexibility, ease of process control, and its applicability to the repair and restoration of power engineering components [5].

Previous studies have demonstrated that as-sprayed NiCrBSi coatings produced by flame spraying may exhibit relatively high porosity and limited adhesion strength to the substrate. For example, subsequent remelting has been reported to reduce coating porosity from approximately 15% to 3%, while induction remelting can further decrease porosity to approximately 1% [6]. These findings demonstrate that coating quality depends not only on the chemical composition of the feedstock material but also on the appropriate selection and optimization of spraying parameters. Therefore, controlling the thermal conditions during flame spraying represents an important approach for improving coating formation without necessarily introducing additional post-deposition processing [7].

Accordingly, the aim of the present study is to investigate the flame-spray deposition of Ni–Cr–B–Si-based self-fluxing coatings intended to protect power engineering components against high-temperature corrosion and to determine the influence of the principal processing parameters on coating properties [8]. Particular attention is given to the effect of flame temperature on coating porosity, microhardness, surface roughness, and adhesion strength to the substrate [9].

The distinctive feature of this study is the systematic evaluation of the structural and mechanical characteristics of Ni–Cr–B–Si-based self-fluxing coatings through controlled variation of the flame-spraying parameters [10,11]. The relationships between spraying temperature and coating porosity, microhardness, surface roughness, and adhesion strength are investigated to identify processing conditions that provide a favorable combination of coating integrity and mechanical performance. The obtained results are intended to establish effective flame-spraying regimes for producing protective coatings suitable for components operating under high-temperature corrosive conditions.

The scientific and practical significance of this study lies in establishing technologically accessible and economically viable deposition conditions for producing protective coatings capable of improving the high-temperature corrosion resistance and extending the service life of power engineering components [12].

2. Materials and methods

Corrosion-resistant AISI 321 austenitic chromium–nickel stainless steel was used as the substrate material. Prior to flame spraying, the substrate surfaces were prepared to promote uniform coating formation and improve adhesion between the coating and the substrate. The specimens were initially ground using abrasive papers ranging from P100 to P600, followed by sandblasting. After mechanical surface preparation, the specimens were ultrasonically cleaned in ethanol for 10 min at a frequency of 40 kHz and a temperature not exceeding 35 °C to remove residual abrasive particles and processing contaminants. The cleaned specimens were subsequently dried and prepared for the flame-spraying process.

Ni–Cr–B–Si-based self-fluxing powder coatings were deposited using a Metal Coat 6PM-II flame-spraying system. A schematic representation of the experimental setup is shown in Figure 1. The spraying system consisted of oxygen and acetylene cylinders equipped with pressure regulators, a compressed-air supply and control unit, gas supply lines, a flame-spray gun, and an MPF-700 powder feeder. Oxygen and acetylene were supplied separately to the flame-spray gun, where their combustion generated a high-temperature flame required for heating and melting the powder particles and accelerating them toward the substrate surface.

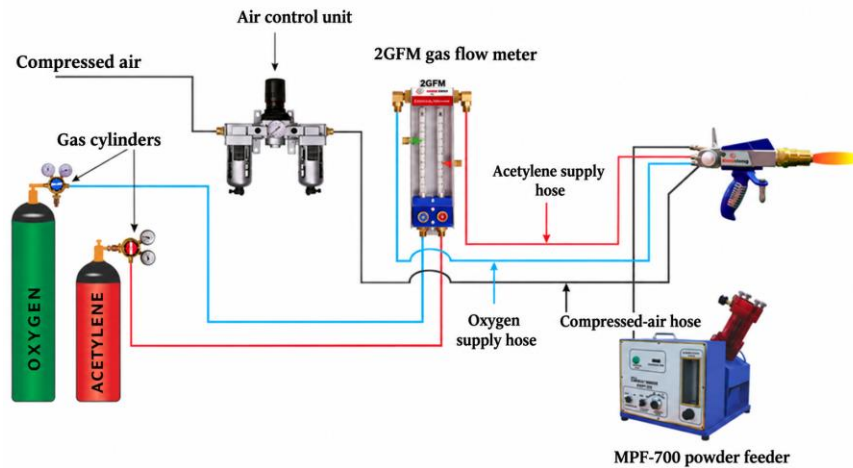


Fig. 1. Schematic diagram of the powder flame-spraying setup.

The thermal conditions of the flame-spraying process were controlled by varying the oxygen-to-acetylene (O_2/C_2H_2) ratio. Three O_2/C_2H_2 ratios of 1.0, 1.1, and 1.2 were investigated. The flame temperatures corresponding to the selected O_2/C_2H_2 ratios are presented in [Table 1](#).

The spraying process parameters were monitored using an Accuraspray 4.0 system. This system enabled non-contact monitoring of the thermal characteristics during flame spraying and ensured the stability and reproducibility of the selected processing conditions.

Table 1. Flame-spraying parameters.

Spraying condition	Flame temperature, °C	O_2/C_2H_2 ratio	Spray distance, mm	Carrier gas flow rate, NLPM
1	3800	1.0	150	20
2	4200	1.1		
3	4400	1.2		

Prior to coating deposition, the prepared AISI 321 steel substrates were preheated using the gas flame without powder feeding. Once the substrate reached the specified temperature, powder feeding was initiated using the MPF-700 powder feeder, and the coatings were deposited at the selected O_2/C_2H_2 ratios. The substrate temperature was monitored throughout the spraying process to maintain stable processing conditions for each experiment.

To objectively evaluate the effect of the O_2/C_2H_2 ratio on coating formation, the other principal spraying parameters were maintained constant. The microstructure, porosity, hardness, adhesion strength, and tribological properties of the deposited coatings were characterized using a comprehensive set of experimental techniques. Cross-sectional microstructural characterization was performed using an Olympus BX53M optical metallographic microscope. The obtained metallographic images were used to evaluate the microstructural features of the coatings, coating uniformity, the integrity of the coating–substrate interface, and porosity.

The adhesion strength of the coatings was determined using a Shimadzu AGS-X 100 kN universal testing machine. The tests were performed in accordance with ASTM C633, using a tensile method in which the coating was loaded normal to the substrate surface until failure occurred. The adhesion strength was calculated as the ratio of the maximum load at failure to the bonded area and was expressed in MPa.

The tribological properties of the coatings were evaluated using an Anton Paar TRB³ tribometer (Anton Paar GmbH, Austria) in a *ball-on-disk* configuration. A 100Cr6 steel ball with a diameter of 6 mm was used as the counterbody. The tests were conducted under a normal load of 10 N, a linear sliding velocity of 5 cm/s (0.05 m/s), and a wear-track radius of 2 mm. The total sliding distance was 100 m. During testing, the coefficient of friction was continuously recorded as a function of sliding distance to evaluate the friction and

wear behavior of the coatings. To ensure the reproducibility of the experimental results, tribological tests were performed three times ($n = 3$) for each coating under identical conditions. The coefficient of friction and wear rate are reported as the mean values obtained from three independent measurements.

3. Results and discussion

Prior to coating deposition, the AISI 321 steel substrates were preheated using a gas flame, followed by powder spraying under the selected processing conditions. Visual inspection confirmed the formation of continuous coatings over the substrate surface under all investigated spraying conditions.

To evaluate the effect of the flame-spraying conditions on the structural characteristics of the Ni–Cr–B–Si coatings, microscopic examination of the coating cross-sections was performed. Figure 2 presents the cross-sectional microstructures of the coatings deposited under different thermal conditions, together with the results of the quantitative porosity analysis.

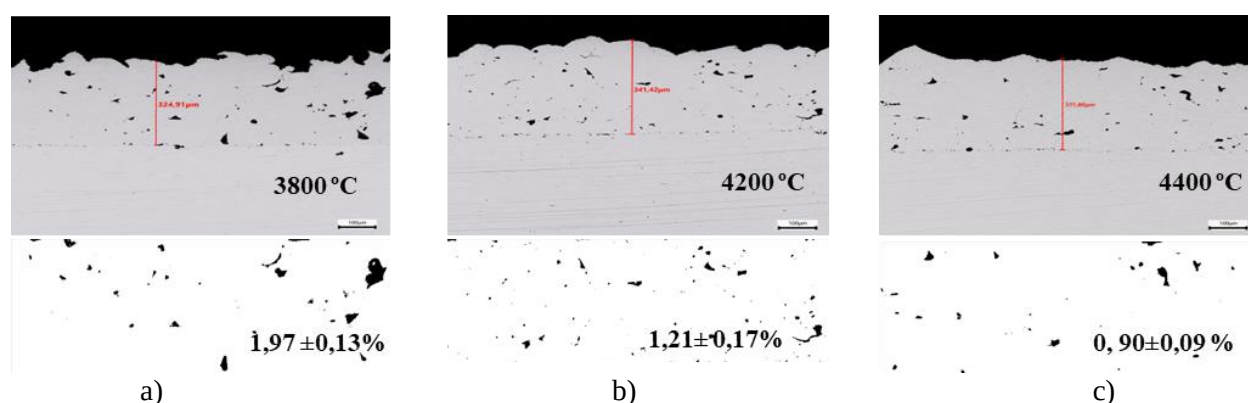


Fig. 2. Cross-sectional microstructure and porosity of Ni–Cr–B–Si coatings obtained at different flame-spraying temperatures: (a) 3800 °C – $1.97 \pm 0.13\%$; (b) 4200 °C – $1.21 \pm 0.17\%$; and (c) 4400 °C – $0.90 \pm 0.09\%$.

Microstructural analysis showed that, under all investigated spraying conditions, the coating formed as a continuous layer over the substrate surface. At the same time, increasing the flame temperature had a pronounced effect on the internal defect structure of the coatings. At a flame temperature of 3800 °C, the coating exhibited a porosity of $1.97 \pm 0.13\%$, which was the highest value among the investigated conditions. The corresponding microstructure contained clearly visible dark pores distributed throughout the coating.

When the flame temperature was increased to 4200 °C, the coating porosity decreased to $1.21 \pm 0.17\%$, indicating a substantial reduction compared with that obtained at 3800 °C. A further increase in temperature to 4400 °C resulted in a porosity of $0.90 \pm 0.09\%$, which was approximately 2.2 times lower than that of the coating deposited at 3800 °C ($1.97 \pm 0.13\%$).

The observed decrease in porosity may be associated with changes in the thermal state of the powder particles as the flame temperature increases. Higher flame temperatures can promote a greater degree of particle melting, thereby facilitating particle deformation upon impact with the substrate and improving contact between the resulting splats. Consequently, interlamellar voids may be reduced, leading to the formation of a denser coating structure. However, since direct confirmation of this mechanism would require independent measurements of the in-flight particle temperature and velocity, this explanation should be regarded as a plausible mechanism underlying the observed microstructural changes.

Thus, within the investigated range, increasing the flame temperature from 3800 to 4400 °C resulted in progressive coating densification and a decrease in overall porosity.

The densification of the coating structure was accompanied by changes in its mechanical characteristics. The microhardness values of the coatings obtained at different O_2/C_2H_2 ratios are summarized in Table 2.

At an O_2/C_2H_2 ratio of 1.0, the average coating microhardness was 954.1 HV. Increasing the gas ratio to 1.1 resulted in an increase in microhardness to 1051.7 HV. When the O_2/C_2H_2 ratio was further increased to 1.2, the microhardness slightly decreased to approximately 1015.0 HV, although it remained higher than that obtained at an O_2/C_2H_2 ratio of 1.0.

These results indicate that the microhardness did not exhibit a monotonic dependence on the O_2/C_2H_2 ratio. The highest average microhardness was obtained at $O_2/C_2H_2 = 1.1$. Under this condition, the relatively low porosity and improved structural integrity of the coating may have contributed to the higher hardness. However, despite the further decrease in porosity at $O_2/C_2H_2 = 1.2$, a slight reduction in microhardness was observed, indicating that coating hardness is not governed solely by porosity. Other factors, including phase composition, oxide formation, and the melting and subsequent solidification behavior of the sprayed particles, may also contribute to the observed variation in microhardness.

To evaluate the influence of the flame-spraying parameters on the surface condition of the coatings, the surface roughness was measured, and the corresponding results are presented in [Table 2](#).

The coating deposited at $O_2/C_2H_2 = 1.0$ exhibited a surface roughness of $4.70 \pm 0.09 \mu\text{m}$, whereas a value of $4.95 \pm 0.13 \mu\text{m}$ was obtained at $O_2/C_2H_2 = 1.1$. The difference between these two conditions was relatively small. However, increasing the O_2/C_2H_2 ratio to 1.2 resulted in a pronounced decrease in surface roughness to $2.99 \pm 0.11 \mu\text{m}$.

This trend is consistent with the porosity results: the coating deposited at $O_2/C_2H_2 = 1.2$ exhibited both the lowest porosity ($0.90 \pm 0.09\%$) and the lowest surface roughness ($2.99 \pm 0.11 \mu\text{m}$). This behavior may be attributed to the enhanced melting of the powder particles under the higher thermal input, which can promote more effective deformation and spreading upon impact with the substrate. Improved particle flattening may consequently reduce surface irregularities and contribute to the formation of a more homogeneous and relatively smooth coating surface.

To assess the influence of the flame-spraying parameters on the mechanical integrity of the coating–substrate system, the adhesion strength of the coatings was determined. The corresponding results are presented in [Table 2](#).

Table 2. Mechanical and surface characteristics of Ni–Cr–B–Si coatings obtained under different flame-spraying conditions.

O_2/C_2H_2 ratio	Temperature, °C	Microhardness, HV	Surface roughness, R_a , μm	Adhesion strength, MPa
1,0	3800	954,1	954,1	954,1
1,1	4200	1051,7	1051,7	1051,7
1,2	4400	1015,0	1015,0	1015,0

A systematic increase in coating adhesion strength was observed with increasing flame temperature. At 3800 °C, the adhesion strength was $22.14 \pm 4.3 \text{ MPa}$, whereas increasing the temperature to 4200 °C resulted in an increase to $27.84 \pm 5.4 \text{ MPa}$. The highest adhesion strength of $35.02 \pm 3.9 \text{ MPa}$ was obtained at 4400 °C. Thus, increasing the flame temperature from 3800 to 4400 °C resulted in an approximately 58% increase in the mean adhesion strength.

The improvement in adhesion strength may be associated with the progressive densification of the coating structure. In particular, as the flame temperature increased from 3800 to 4400 °C, the coating porosity decreased from $1.97 \pm 0.13\%$ to $0.90 \pm 0.09\%$. Under higher-temperature spraying conditions, an increased degree of particle melting and deformation upon impact may increase the effective contact area between the deposited particles and the substrate, thereby promoting mechanical interlocking and improving the integrity of the coating–substrate interface.

Overall, the obtained results demonstrate that variation in the O_2/C_2H_2 ratio during flame spraying has a combined effect on the microstructural, surface, and mechanical characteristics of Ni–Cr–B–Si coatings. Within the investigated range, increasing the O_2/C_2H_2 ratio from 1.0 to 1.2, accompanied by an increase in flame temperature, resulted in a decrease in coating porosity from $1.97 \pm 0.13\%$ to $0.90 \pm 0.09\%$, a reduction in surface roughness from 4.70 ± 0.09 to $2.99 \pm 0.11 \mu\text{m}$, and an increase in adhesion strength from 22.14 ± 4.3 to $35.02 \pm 3.9 \text{ MPa}$. In contrast to these monotonic trends, the microhardness reached its maximum average value of approximately 1051.7 HV at $O_2/C_2H_2 = 1.1$.

These findings indicate that the investigated coating properties do not respond uniformly to changes in the O_2/C_2H_2 ratio. The coating deposited at $O_2/C_2H_2 = 1.2$ exhibited the lowest porosity and surface roughness together with the highest adhesion strength, whereas the maximum average microhardness was achieved at $O_2/C_2H_2 = 1.1$. Therefore, selection of the flame-spraying conditions should take into account the specific performance requirements of the coating rather than relying on a single property as the optimization criterion.

4. Conclusion

In this study, Ni–Cr–B–Si-based self-fluxing coatings were deposited onto AISI 321 austenitic stainless steel substrates by flame spraying, and the effect of varying the O_2/C_2H_2 ratio from 1.0 to 1.2 on their microstructure and properties was investigated. Increasing the gas ratio and the corresponding thermal input was found to promote progressive densification of the coating structure.

As the O_2/C_2H_2 ratio increased from 1.0 to 1.2, the coating porosity decreased from $1.97 \pm 0.13\%$ to $0.90 \pm 0.09\%$, while the surface roughness decreased from $4.70 \pm 0.09 \mu\text{m}$ to $2.99 \pm 0.11 \mu\text{m}$. At the same time, the adhesion strength increased from $22.14 \pm 4.3 \text{ MPa}$ to $35.02 \pm 3.9 \text{ MPa}$. These changes may be attributed to improved melting and deformation of the powder particles under higher thermal input, resulting in reduced structural defects and improved interfacial contact between the coating and the substrate.

The dependence of microhardness on the O_2/C_2H_2 ratio was non-monotonic. The highest average microhardness of 1051.7 HV was obtained at $O_2/C_2H_2 = 1.1$, whereas a slightly lower value of 1015.0 HV was measured at $O_2/C_2H_2 = 1.2$. This behavior indicates that the mechanical response of the coatings is governed not only by porosity but may also be affected by microstructural and phase transformations occurring during the spraying process.

Among the investigated conditions, the coating deposited at $O_2/C_2H_2 = 1.2$ exhibited the lowest porosity ($0.90 \pm 0.09\%$), the lowest surface roughness ($2.99 \pm 0.11 \mu\text{m}$), and the highest adhesion strength ($35.02 \pm 3.9 \text{ MPa}$), whereas the maximum microhardness was achieved at $O_2/C_2H_2 = 1.1$. Overall, the results demonstrate that controlling the O_2/C_2H_2 ratio provides an effective technological approach for tailoring the microstructure and functional properties of flame-sprayed Ni–Cr–B–Si-based self-fluxing coatings.

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Conflict of interest

The authors declare that they have no competing interests.

References

- 1 Simunovic, K., Havrlisan, S., Saric, T., & Vukelic, D. (2020). Modeling and optimization in investigating thermally sprayed Ni-based self-fluxing alloy coatings: A review. *Materials*, **13**(20), 4584. DOI: <https://doi.org/10.3390/ma13204584>.
- 2 Sidhu, T. S., Prakash, S., & Agrawal, R. D. (2006). Hot corrosion behaviour of HVOF-sprayed NiCrBSi coatings on Ni- and Fe-based superalloys in Na_2SO_4 –60% V_2O_5 environment at 900 °C. *Surface and Coatings Technology*, **201**(3–4), 792–800. DOI: [Hot corrosion behaviour of HVOF-sprayed NiCrBSi coatings on Ni- and Fe-based superalloys in Na2SO4–60% V2O5 environment at 900 °C - ScienceDirect](https://doi.org/10.1016/j.surfcoat.2005.12.001)
- 3 Medabalimi, S., Rokkala, U., Gudala, S., Behera, N., Ramesh, M. R., & Dejene, M. (2025). Wear and frictional behaviour of partially oxidized and plasma sprayed NiCr and NiCrBSiFe coatings. *Scientific Reports*. DOI: [Wear and frictional behaviour of partially oxidized and plasma sprayed NiCr and NiCrBSiFe coatings | Scientific Reports](https://doi.org/10.1038/s41598-025-00000-0)
- 4 Korobov, Yu., & Shalimov, M. (2014). Energy efficiency by thermal spraying. *WIT Transactions on Ecology and the Environment*, **190**, 455–466. DOI: <https://scispace.com/pdf/energy-efficiency-by-thermal-spraying-2lf770i297.pdf>
- 5 Kazamer, N., Muntean, R., Vălean, P. C., Pascal, D. T., Mărginean, G., & Șerban, V.-A. (2021). Comparison of Ni-based self-fluxing remelted coatings for wear and corrosion applications. *Materials (Basel)*, **14**(12), 3293. DOI: <https://doi.org/10.3390/ma14123293>.
- 6 Buitkenov, D., Raisov, N., Alimbekuly, T., & Alibekova, B. (2025). Study of the tribological properties of self-fluxing nickel-based coatings obtained by gas-flame spraying. *Crystals*, **15**, 862. DOI: <https://doi.org/10.3390/cryst15100862>.

- 7 Vălean, P. C., Kazamer, N., Muntean, R., Pascal, D. T., Kilic, Y., Mărginean, G., & Șerban, V. A. (2018). Investigations on the characteristics of thermally sprayed NiCrBSi coatings fused by flame and inductive processing. *IOP Conference Series: Materials Science and Engineering*, **416**, 012002. DOI: [Open Access proceedings Journal of Physics: Conference series](#)
- 8 Buitkenov, D., Sulyubayeva, L., Baizhan, D., Raisov, N., Tleubergenova, G., & Bimakhan, N. (2026). Study of the influence of detonation spraying parameters on the structure and properties of self-fluxing coatings of the Ni–Cr–Fe–Si–B–C system. *Applied Sciences*, **16**(15), 7637. DOI: [10.3390/app16157637](#).
- 9 Rachidi, R., El Kihel, B., & Delaunois, F. (2019). Microstructure and mechanical characterization of NiCrBSi alloy and NiCrBSi-WC composite coatings produced by flame spraying. *Materials Science and Engineering: B*, **241**, 13–21. DOI: [Microstructure and mechanical characterization of NiCrBSi alloy and NiCrBSi-WC composite coatings produced by flame spraying - ScienceDirect](#)
- 10 Bergant, Z., Trdan, U., & Grum, J. (2014). Effect of high-temperature furnace treatment on the microstructure and corrosion behavior of NiCrBSi flame-sprayed coatings. *Corrosion Science*, **88**, 372–386. DOI: [Effect of high-temperature furnace treatment on the microstructure and corrosion behavior of NiCrBSi flame-sprayed coatings - ScienceDirect](#)
- 11 Habib, K. A., Cano, D. L., Serrano-Mira, J., Rayón Encinas, E., & Dosta, R. S. (2024). Impact of microstructure on remelting parameters and mechanical behavior of thermally sprayed NiCrBSi coating. *Journal of Thermal Spray Technology*, **33**, 290–307. DOI: [Impact of Microstructure on Remelting Parameters and Mechanical Behavior of Thermally Sprayed NiCrBSi Coating | Journal of Thermal Spray Technology | Springer Nature Link](#)
- 12 Czupryński, A., Adamiec, J., Adamiak, M., Żuk, M., Kříž, A., Mele, C., & Kciuk, M. (2023). High-temperature corrosion of flame-sprayed power boiler components with nickel alloy powders. *Materials*, **16**(4), 1658. DOI: [High-Temperature Corrosion of Flame-Sprayed Power Boiler Components with Nickel Alloy Powders | MDPI](#)

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ORIGINAL STUDY

CALCULATION AND EXPERIMENTAL STUDY OF THE REAL CONTACT AREA, CONTACT PRESSURE, AND COEFFICIENT OF FRICTION

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Abstract. The study investigates the formation of the real area of contact and the corresponding contact pressure during the tribological interaction of rough surfaces. Particular attention is given to the distinction between the nominal area, contour area, and real area of direct contact, taking into account the waviness and roughness of the surface profile. A calculation approach based on the geometric and statistical characteristics of the surface microrelief is used to estimate the transition from the nominal contact area to the actual areas involved in load transfer. The contact pressure in the zones of direct interaction is evaluated considering the deformation of surface asperities and their mechanical characteristics. The calculated parameters of the real contact are compared with the experimental characteristics obtained during tribological testing. The analysis demonstrates the importance of considering the actual contact conditions when interpreting tribological test results and evaluating the stress state of contacting surfaces. The proposed approach provides a basis for a more physically justified assessment of contact interaction and the subsequent analysis of friction and wear processes.

Keywords: real contact area; nominal contact area; contact pressure; surface roughness; waviness; asperities; friction; wear; tribological interaction.

1. Introduction

Friction and wear are among the key factors determining the reliability, durability, and serviceability of machine and mechanism components. During the relative motion of contacting surfaces, a complex set of interrelated physicomachanical processes occurs within the contact zone, including elastic and plastic deformation of asperities, destruction of surface layers, changes in surface microgeometry, and formation of wear debris. The combination of these processes generates a complex stress–strain state in the contact zone and directly determines the nature of frictional interaction [1]. Therefore, establishing the regularities governing real contact formation is of considerable importance for evaluating the tribological characteristics of materials.

Classical tribometric test configurations, such as ball-on-disk and pin-on-disk, are widely used for the experimental investigation of friction and wear processes. These configurations make it possible to determine the coefficient of friction, wear volume, and wear intensity under different conditions of relative motion and loading. However, the tribological characteristics recorded during testing are the result of processes occurring directly within the real contact zones, whose geometry and state differ substantially from the parameters determined by the initial nominal geometry of the mating surfaces.

The theoretical description of contact interaction is traditionally based on the classical Hertz theory, which allows the main characteristics of contact between elastic bodies to be determined as a function of their geometry, mechanical properties, and applied load. At the same time, the application of idealized contact models to real tribological systems requires consideration of the characteristics of the surface layer. D. H.

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Buckley emphasized the significant role of surface microgeometry, surface films, and adhesive interactions in the formation of real contact. This indicates the need to consider the contact surface as a multilevel system in which the geometrical parameters of the mating surfaces are complemented by the characteristics of their surface microrelief [1].

Real machine-component surfaces are characterized by a combination of irregularities of different scales, heights, shapes, and spatial distributions. Therefore, the interaction between mating surfaces does not occur over the entire nominal area but through a set of localized contact regions formed by the most prominent elements of the surface microrelief. This approach is particularly important when investigating the surfaces of hardened components. The formation of a hardened surface layer is accompanied by changes not only in hardness and mechanical properties but also in the microgeometry of the material. Surface roughness and waviness parameters determine the distribution of material along the profile height and, consequently, the conditions under which individual asperities are successively brought into contact. Under applied loading, asperities may undergo elastic, elastic–plastic, or plastic deformation, resulting in changes in the real contact area together with variations in pressure within the local interaction zones.

Consequently, the use of only nominal pressure, determined as the ratio of the applied load to the geometrical contact area, does not provide a complete characterization of the stress state of the real contact. A more informative approach is the sequential consideration of the nominal, contour, and real contact areas, which makes it possible to evaluate changes in contact pressure during the transition from the macroscopic geometrical level to the level of direct interaction between asperities [2]. In this context, the real contact pressure can be considered one of the key parameters linking surface microgeometry with the mechanics of contact interaction.

Changes in the real contact area and corresponding contact pressure are directly reflected in the nature of frictional interaction. During actual sliding, individual microcontacts continuously form, deform, and break down, accompanied by load redistribution and changes in the interaction conditions of the surface layers. This, in turn, determines the development of tangential force and the coefficient of friction and, during prolonged interaction, the nature and intensity of wear.

Thus, a more complete description of the tribological behavior of contacting surfaces requires consideration not only of their nominal geometry and average roughness parameters but also of the regularities governing the formation of the real contact area and real contact pressure, taking into account the surface microgeometry [3]. Such an approach makes it possible to relate the geometrical characteristics of real contact to its stress–strain state and subsequent friction and wear processes.

The aim of this study is to establish the regularities governing the formation of the real contact area and real contact pressure during the tribological interaction of rough surfaces, taking into account their microgeometry, and to investigate the relationship of these characteristics with the contact stress state, coefficient of friction.

2. Materials and methods

Surface roughness and waviness were measured before and after TEPT using a Surtronic S-100 [S-116 / S-128] portable contact profilometer (Taylor Hobson, AMETEK, UK). The instrument was equipped with a skidded inductive pickup and a diamond stylus with a tip radius of 5 μm and a stylus force of 150–300 mg. Measurements were performed at a traverse speed of 1 mm/s using a Gaussian filter, with a cut-off length of 0.8 mm and an evaluation length of 4.0 mm, in accordance with ISO 4287 and ISO 4288. The arithmetic mean roughness, R_a , and maximum profile height, R_z , were determined.

Tribological tests were conducted using a TRB3 tribometer (Anton Paar, Austria) according to the pin-on-disk configuration specified in ASTM G99 [4]. During relative sliding of the disk against the pin, a tangential friction force F_t was generated in the direction opposite to the relative motion of the contacting surfaces. The pin was mounted on a precision elastic lever connected to a force sensor. Under the action of the tangential friction force, the lever underwent a small elastic deflection, which was detected by the sensor and converted into the corresponding friction force F_t .

The coefficient of friction was determined from the measured tangential force and the applied normal load F_n according to:

$$\mu = \frac{F_n}{F_t}, \quad (1)$$

where F_t is the tangential friction force and F is the applied normal load.

The pin was made of cylindrical 12X18H10T stainless steel with a diameter of 8 mm, while 40X steel was used as the disk material. The chemical compositions of the investigated steels are presented below. A normal load of 5 N was applied to the contacting surfaces. The disk rotation corresponded to a sliding speed of 3 cm/s, and the total sliding distance was 60 m (Fig. 1).

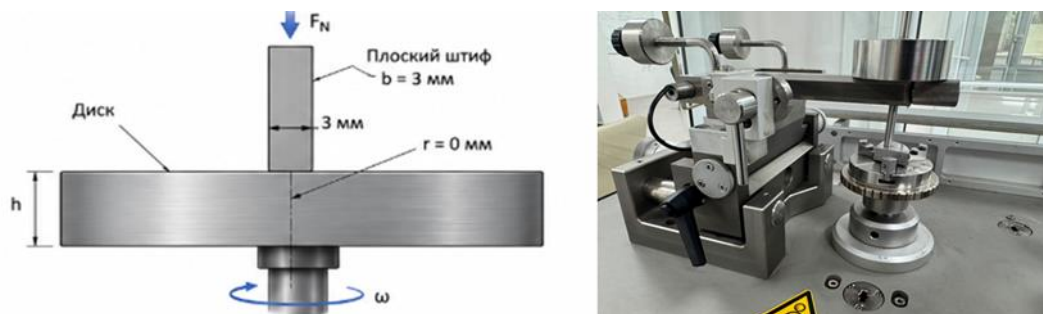


Fig. 1. Schematic representation of the pin-on-disk friction model.

Tensile tests were performed using a Shimadzu AGS-X universal testing machine at room temperature in accordance with ISO 6892-1. The specimens were subjected to uniaxial tensile loading until fracture, with continuous recording of the applied load and deformation. The yield strength and ultimate tensile strength (σ) were determined from the obtained stress–strain data to characterize the mechanical properties of the investigated materials. The ultimate tensile strength was determined from the maximum tensile load recorded during the test.

Table 1 presents the initial experimental data characterizing the properties of the investigated materials after testing.

Table 1 presents the initial experimental data characterizing the properties of the investigated materials after testing.

Parameter	Value
Arithmetic mean deviation, R_a	0.06578 μm
Profile peak height, R_p	0.2219 μm
Maximum profile height, R_{max}	1.082 μm
Total cutoff length, $\sum \Delta l$	0.25 mm
Sampling length, l	0.8 mm
Hardness of 40X steel, HB	270 kgf/mm ²
Elastic modulus of 40X steel, E	2×10^6 kgf/cm ²
Poisson's ratio of 40X steel, μ	0.30
Mean normal stress, σ_0	1400 kgf/cm ²
Frictional interaction parameter, β_f	0.004
Yield strength, σ_T	780 kgf/cm ²
Parameter of the unit process, t	2
Fracture strain, ε_0	0.16
Waviness height, W_f	4 μm
Waviness radius, R_w	50 mm
Waviness exponent, ν_w	2
Relative elongation, δ	$\approx 40\%$
Hardness of 12X18H10T steel, HB	$\approx 170\text{--}179$ HB
Temperature	20–25 $^{\circ}\text{C}$

2.1 Method for Determining the Contour Contact Pressure

In technical literature, four main types of deviations from the nominal surface geometry are traditionally distinguished: macrodeviations, waviness, roughness, and sub-roughness. Figure 2 shows the surface waviness profile and its main geometric parameters, including the maximum profile height R_{max} , maximum waviness height W_{max} , waviness peak height W_p and waviness spacing S_w .

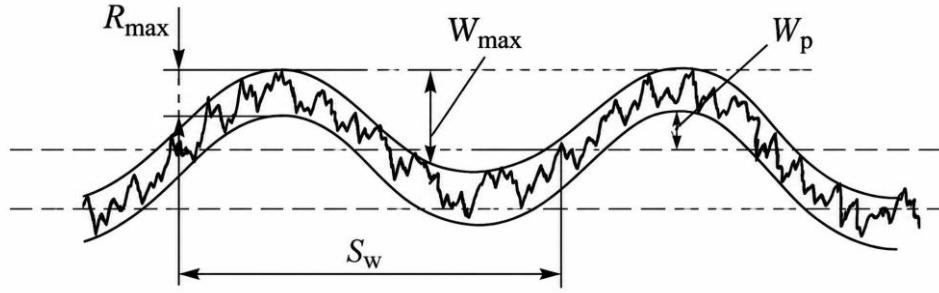


Fig. 2. Surface waviness profile and its main geometric parameters.

During contact interaction between the specimen and the counterbody, the real contact area differs substantially from the nominal contact area (Fig. 3) [5].

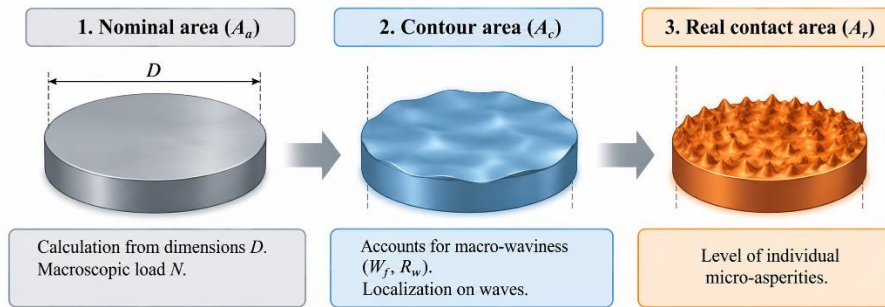


Fig. 3. Surface microrelief parameters and the regularities governing contact area formation.

A single parameter, R_a , representing the arithmetic mean deviation of the surface profile, is insufficient for a comprehensive description of the real contact surface. Surfaces characterized by identical R_a values may differ substantially in their microrelief characteristics and load-bearing capacity. This difference is primarily associated with the distribution of material along the height of surface irregularities, as well as with the shape and density of asperities, which directly determine the conditions under which real microcontacts are formed.

Therefore, a more comprehensive description of the contact properties of a surface requires consideration not only of the average height of surface irregularities but also of the distribution of material along the height of the surface microrelief [6]. One approach to such a description is the use of the bearing area curve (Abbott–Firestone curve), which relates the degree of approach of the mating surfaces to the change in the area of material brought into contact.

In the initial stage of contact, the variation of the bearing area curve parameters with the profile section depth can be approximated by a power-law function:

$$t_p = b\varepsilon^\nu, \quad (2)$$

$\varepsilon = a/R_{\max}$ is the relative approach of the contacting surfaces.

The exponent ν in this relationship characterizes the manner and rate at which the material area changes with increasing depth into the surface microrelief. Within the considered calculation approach, its value is determined from the parameters obtained from the surface profilogram:

$$\nu = 2t_p \frac{R_p}{R_a} - 1 \quad (3)$$

$$b = t_p \left(\frac{R_{\{\max\}}}{R_p} \right)^\nu \quad (4)$$

The surface characteristics used as input parameters are R_p , R_a , and $R_{\max}$. From a physical standpoint, the exponent ν reflects the geometric characteristics and distribution of surface asperity peaks. Variations in ν characterize differences in the rate at which the real contact area develops as the mating surfaces approach each other. Therefore, the combined consideration of R_a and R_p provides a more comprehensive

characterization of the surface microgeometry and its contact behavior than the use of either parameter individually [7].

For an integrated description of the height and geometric characteristics of the surface microrelief, a dimensionless parameter Δ is introduced, combining the corresponding surface parameters:

$$\Delta = \frac{R_{\{max\}}}{r_{eq} b^{\frac{1}{\nu}}} \quad (5)$$

Consequently, a decrease in Δ corresponds to a reduction in the concentration of local geometric interaction. The nominal contact pressure is determined using the classical equation:

$$p_a = N/Aa, \quad (6)$$

where N is the normal load and Aa is the nominal contact area.

After converting the applied load to the required system of units, the nominal contact pressure is determined as the ratio of the applied load to the nominal contact area. However, this quantity represents an averaged characteristic of the contact and does not reflect the actual pressure arising directly within the zones of asperity-to-asperity interaction.

The next level of contact is represented by the contour contact area A_c , whose formation is governed by the deformation of macro-irregularities and surface waviness under the applied load. For its calculation, the N. B. Demkin approach is employed [8].

$$A_c = A_a k_w k_v \left[\frac{2R_w}{HB} * \theta p_a \right]^{\nu_w/\nu_w+1.95} \quad (7)$$

where k_v and k_w are tabulated coefficients; R_w is the radius of waviness, ν_w is the waviness exponent, p_a is the nominal contact pressure, HB is the material hardness, and E_r is the reduced elastic modulus of the contact pair.

The contour contact pressure is then calculated as follows:

$$p_c = N/Ac, \quad (8)$$

Thus, the transition from nominal contact pressure to contour contact pressure reflects the redistribution of the applied load over a reduced contact area. At this stage, an increase in the average pressure acting on the surface becomes evident as a consequence of the reduction in the effective contact area (Fig. 4).

The mean real contact pressure in the zones of direct interaction between elastically deformed asperities is determined by the following expression [8]:

$$p_r = 0.4 \cdot E_r \cdot \Delta^{(2\nu+1)} \frac{W_t^{0.1}}{R_w} \quad (9)$$

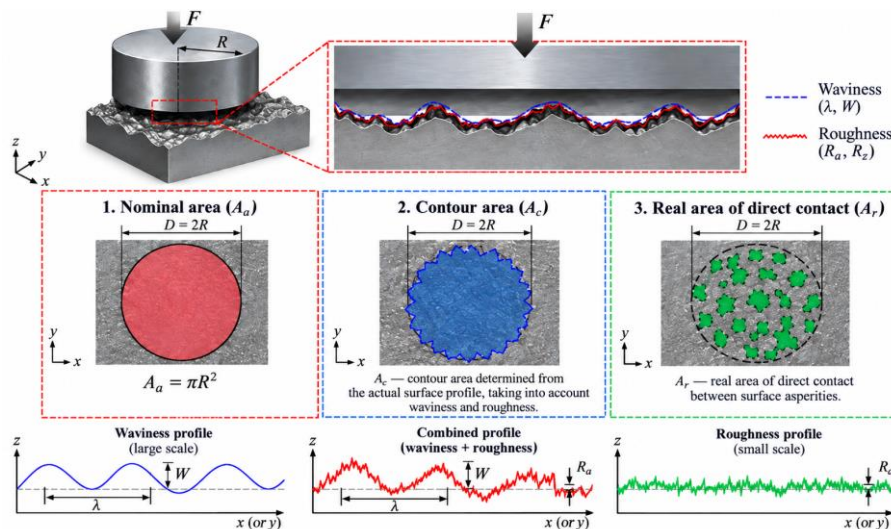


Fig. 4. Schematic representation of nominal, contour, and real contact areas of a rough surface.

3. Results and discussion

For the investigated surface condition, the calculated values were $p_c = 2.02 \cdot 10^7$ kgf/cm² for the contour contact pressure and $p_r = 1.18 \cdot 10^5$ kgf/cm² for the real contact pressure. Comparison of the obtained value of p_r with the material hardness, $HB = 270$ kgf/mm², indicates that the contact interaction within the adopted calculation model is predominantly plastic [9].

This result is of particular importance for the subsequent evaluation of the coefficient of friction, since under predominantly plastic contact conditions the resistance to relative motion is determined by the combined contribution of the adhesive component arising in the zones of real contact and the deformation component associated with the penetration and displacement of asperities on the mating surfaces. Based on the established nature of the contact interaction, the coefficient of friction is subsequently determined using the following relationship [5]:

$$f = \left(\frac{\tau_0}{H_B} \right) + \beta_f + 0,52 \sqrt{\Delta \left(\frac{p_c}{H_B} \right)^{\frac{1}{4}}} \quad (10)$$

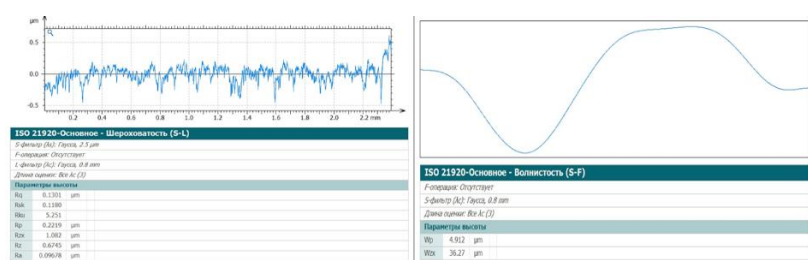


Fig. 5. Roughness and waviness profiles of the investigated surface obtained by profilometric analysis.

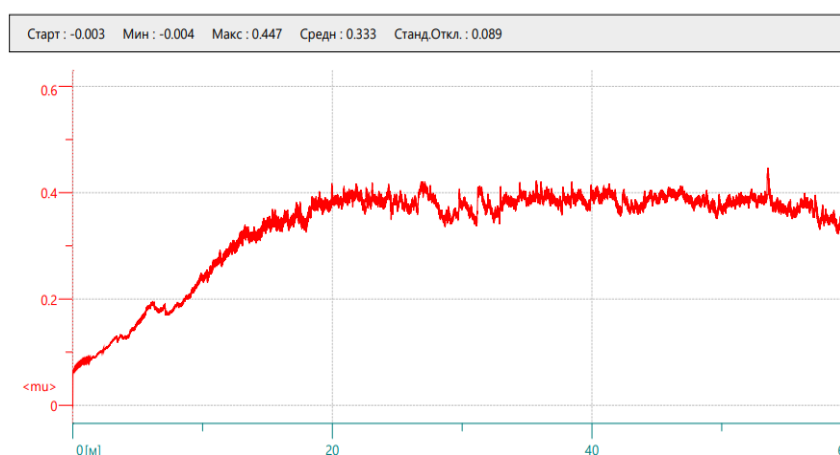


Fig. 6. Variation of the coefficient of friction as a function of sliding distance.

For 40X steel, the calculated coefficient of friction was 0.3527, whereas the experimental value obtained using the TRB3 tribometer was 0.3330. The close agreement between these values indicates that the proposed calculation procedure accounts for the main factors governing the frictional interaction of the investigated surface (Fig. 5-6). At the same time, the calculated value is slightly higher than the experimental one, which can be attributed to the difference between the idealized representation of individual microcontacts in the calculation model and the actual friction process [10].

The discrepancy between the calculated and experimental results may be associated with differences in the representation of the contacting surface. In the calculation model, the actual surface topography is described by a set of averaged parameters; therefore, the individual characteristics of particular asperities are not fully accounted for. Under experimental conditions, real contact is formed by a large number of asperities differing in height, geometrical shape, and local mechanical state. During relative sliding, individual contact regions continuously form, deform, and break down, accompanied by continuous redistribution of the contact load. Consequently, the experimentally recorded parameters may exhibit fluctuations that are not reproduced by the stationary calculation model based on an averaged representation of the surface.

4. Conclusion

The conducted study demonstrated that the assessment of the tribological interaction of contacting surfaces requires consideration not only of the nominal geometry of the mating surfaces but also of the actual state of their surface microrelief. It was established that the real contact area is formed by a set of individual asperities, resulting in real contact pressure that can differ substantially from both nominal and contour contact pressure.

The calculation analysis showed that consideration of surface roughness and waviness parameters provides a more physically justified assessment of contact-load distribution and asperity deformation. With increasing approach of the contacting surfaces, new regions of the surface microrelief are successively brought into contact, accompanied by changes in the real contact area and the corresponding contact pressure.

Comparison of the calculated and experimental results confirmed a relationship between the state of real contact and the frictional behavior of the surfaces. The observed discrepancies are associated with the fact that the calculation model employs an averaged description of the surface microgeometry, whereas under actual sliding conditions the composition and mechanical state of individual microcontacts continuously change.

Thus, real contact pressure is one of the key parameters governing the stress state of the contact zone and the frictional interaction of contacting surfaces. Its combined consideration with surface roughness parameters, real contact area, and coefficient of friction provides a more comprehensive description of the mechanisms governing tribological behavior and can be used as a basis for the calculation-based assessment of surface wear intensity.

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Conflict of interest

The authors declare that they have no competing interests.

References

1. Kombalov V.S. (1983) *Otsenka tribotekhnicheskikh svoystv kontaktiruyushchikh poverkhnostey* [Assessment of tribotechnical properties of contacting surfaces]. Moscow: Nauka.
2. Kragelsky I.V., Dobychin M.N., Kombalov V.S. (1977) *Osnovy raschetov na trenie i iznos* [Fundamentals of calculations for friction and wear]. Moscow: Mashinostroenie.
3. Kragelsky I.V., Demkin N.B. (1960) Opredelenie fakticheskoy ploshchadi kasaniya sherokhovatykh poverkhnostey [Determination of the real contact area of rough surfaces]. In: *Trenie i iznos v mashinakh*. Moscow: Publishing House of the Academy of Sciences of the USSR, Issue 14, 37–62.
4. ASTM International (2017) *ASTM G99: Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus*. ASTM International, 6.
5. Kragelsky I.V., Shchedrov V.S. (1956) *Razvitie nauki o trenii. Sukhoe trenie* [Development of the science of friction. Dry friction]. Moscow: Publishing House of the Academy of Sciences of the USSR.
6. Braun E.D., Bushe N.A., Buyanovsky I.A. et al. (1995) *Osnovy tribologii (trenie, iznos, smazka)* [Fundamentals of Tribology (Friction, Wear, Lubrication)]. Ed. by A.V. Chichinadze. Moscow: Nauka i Tekhnika, 777 p.
7. Kragelsky I.V. (1968) *Trenie i iznos* [Friction and wear]. Moscow: Mashinostroenie.
8. Fleischer G. (1973) Energetische Methode zur Verschleißbeurteilung [Energetic method for wear assessment]. *Schmierungstechnik*, 4(9), 269–274.
9. Fleischer G. (1990) Zur Energetik der Reibung und des Verschleißes [On the energetics of friction and wear]. *Wissenschaftliche Zeitschrift der Technischen Universität Magdeburg*, 8, 55–66.
10. Zakharov S.M., Zharov I.A. (2012) Betki qabaty beriktendirilgen bolattardyn tozuga tozimdiligin energetikalyk tasil negizinde bagalau [Assessment of the wear resistance of surface-hardened steels based on the energetic approach]. 33(4), 385–392.

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