

Article

Ultrasonic Surface Modification of WC-Co-Cr Thermal Spray Coatings: Microstructure, Mechanical Properties and Performance

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Abstract

Thermal-sprayed WC–Co–Cr coatings are widely used to improve the wear and corrosion resistance of steel components operating under severe service conditions. This study investigates WC–Co–Cr coatings deposited by detonation spraying (DS), high-velocity oxy-fuel (HVOF), and atmospheric plasma spraying (APS) before and after ultrasonic nanocrystal surface modification (UNSM). The coatings were characterized using scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM/EDS), together with microhardness, tribological, abrasive, erosive, impact-abrasive, cavitation, corrosion, oxidation, and valve endurance tests. The results demonstrate that the deposition technology strongly affects the coating structure and performance. UNSM produced a pronounced increase in microhardness, reaching 1468 HV for DS + UNSM, 1596 HV for HVOF + UNSM, and 1561 HV for APS + UNSM. Ultrasonic treatment also reduced material loss under abrasive, erosive, impact-abrasive, and cavitation conditions. APS + UNSM demonstrated the best performance in the principal laboratory wear tests, with the lowest abrasive mass loss (0.42–0.45 mg), erosive mass loss (0.55–0.60 mg), impact-abrasive mass loss (0.75–0.85 mg), and cavitation mass-loss rate (0.33–0.38 mg/h). In tribological testing, APS + UNSM exhibited the lowest coefficient of friction (0.28), compared with 0.32 for HVOF + UNSM and 0.36 for DS + UNSM. In valve endurance testing, HVOF + UNSM showed the lowest plunger displacement growth rate (0.050 mm/h) and the longest calculated time to reach the 2 mm displacement criterion (22 h). Overall, the combination of thermal spraying and UNSM improves the mechanical and service performance of WC–Co–Cr coatings, while the optimal coating condition depends on the specific degradation mechanism and operating conditions.



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Keywords: WC–Co–Cr; thermal spraying; HVOF; atmospheric plasma spraying; detonation spraying; ultrasonic post-treatment; UNSM; abrasive wear; erosion; corrosion; tribology

1. Introduction

Surface engineering makes it possible to combine a relatively inexpensive structural substrate with a surface layer designed for high hardness, wear resistance or environmental protection [1]. Thermal spraying is particularly suitable for this purpose because hardmetal

coatings can be deposited on components whose geometry or size would make bulk hardmetal construction impractical. WC-based coatings are therefore widely used where abrasive and sliding wear are dominant degradation mechanisms [2,3].

Among thermal-spray technologies, High-Velocity Oxygen Fuel (HVOF) generally provides high particle velocities and dense carbide-containing coatings, while Atmospheric Plasma Spraying (APS) and Detonation Spraying (DS) offer different thermal and kinetic conditions during particle deposition. The resulting porosity, carbide dissolution, oxidation and residual-stress state can strongly affect wear and corrosion behavior [3–5]. Thus, the deposition process establishes the initial condition from which subsequent surface treatment must be evaluated (Figure 1).

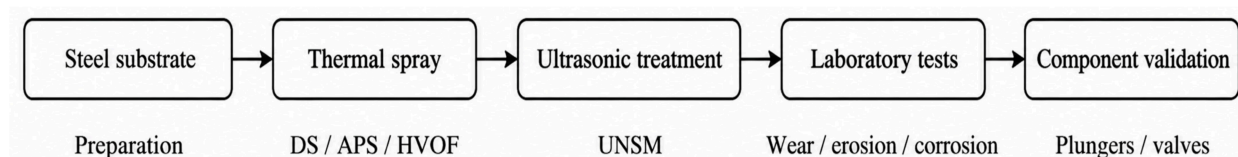


Figure 1. Experimental workflow used to compare WC–Co–Cr thermal-spray coatings before and after ultrasonic post-treatment.

Ultrasonic nanocrystal surface modification (UNSM) and related ultrasonic surface-treatment methods introduce repeated high-frequency mechanical impacts under a static force. Depending on the material and processing conditions, such treatment has been reported to modify the near-surface microstructure, reduce surface roughness, increase hardness, and generate compressive residual stress [6,7]. Previous work on thermally sprayed coatings has shown that such treatment can alter sliding-wear and scratch behavior, but the response is not necessarily universal and depends on the initial coating state and treatment parameters [8–10].

The objective of this study is to compare WC–Co–Cr coatings produced by DS, APS, and HVOF before and after ultrasonic post-treatment and to determine whether the treatment provides a consistent performance benefit across different degradation mechanisms. Particular attention is paid to distinguishing laboratory-scale surface tests from component-level performance. The reported results include ranges, individual experimental values, and engineering estimates; therefore, the data are interpreted according to their original form and are not combined as statistically equivalent measurements. Statistical parameters are not introduced where they are not directly supported by the available results.

2. Materials and Methods

Structural alloy steel 20Kh was used as the substrate material. According to GOST 4543-2016, its chemical composition includes 0.17–0.23 wt.% C, 0.17–0.37 wt.% Si, 0.50–0.80 wt.% Mn, and 0.70–1.00 wt.% Cr. The Ni and Cu contents do not exceed 0.30 wt.%, while the S and P contents are limited to 0.035 wt.%. Iron constitutes the remainder of the composition. Prior to coating deposition, the substrate surface was mechanically cleaned and abrasive-blasted using white electrocorundum. The blasting pressure was 0.7 MPa, the nozzle-to-surface distance was maintained at 100–120 mm, and the impact angle was kept within the range of 75–90°.

WC–Co–Cr powder was used as the coating material. Its nominal composition was 5.3 wt.% C, 10 wt.% Co, and 4 wt.% Cr, with the remainder being W.

WC–Co–Cr powder with a particle size of 20–40 µm was used for coating deposition. SEM morphological analysis revealed that the powder particles were predominantly spherical in shape, with a pronounced rough surface morphology. Small satellite particles and porous regions were observed on the surfaces of some particles (Figure 2).

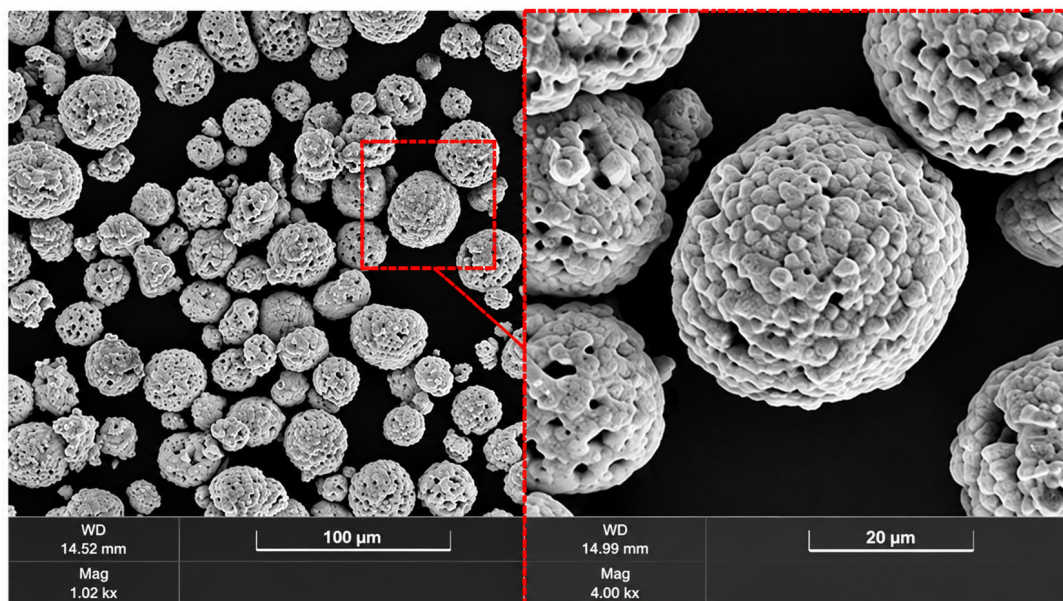


Figure 2. Morphology of WC–Co–Cr powder obtained by scanning electron microscopy (SEM).

Three coating deposition technologies were considered: detonation spraying (DS), high-velocity oxy-fuel (HVOF) spraying, and atmospheric plasma spraying (APS). Detonation spraying was performed using a CCDS200 system (Figure 3) at a nominal stand-off distance of 200 mm. The system consisted of a gas distribution system, an ignition chamber, a detonation barrel, a powder feeder, and a controlled fuel and oxidizer supply system. A combination of $C_2H_2/C_3H_8/N_2/O_2$ gases was used during the process.

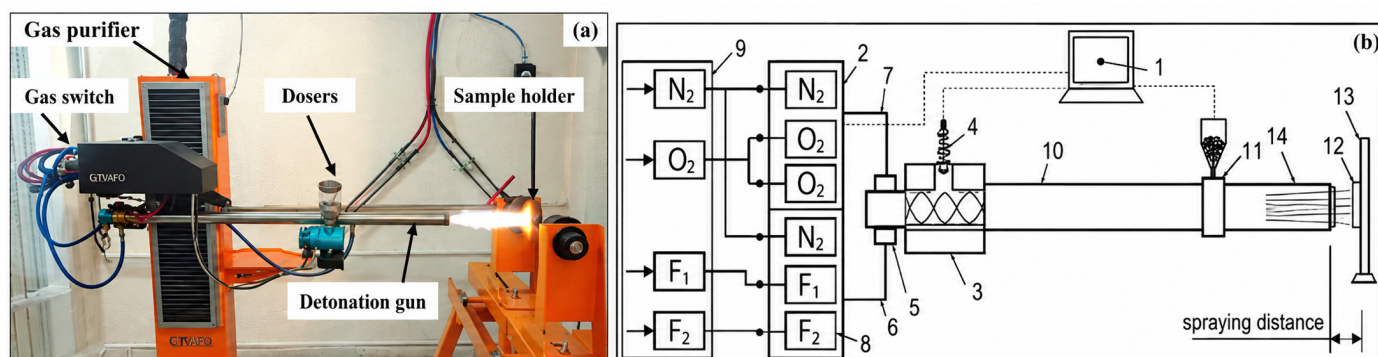


Figure 3. General view of the CCDS200 system (a) and its process schematic (b): 1—control panel; 2—gas distributor; 3—ignition chamber; 4—spark plug; 5—barrel valve; 6—fuel line; 7—oxygen line; 8—gas valves; 9—gas supply unit; 10—breach section of the barrel; 11—powder feeder (dosing unit); 12—workpiece; 13—manipulator; 14—muzzle section of the barrel. Reprinted from Ref. [11].

The HVOF coating was deposited at a nominal stand-off distance of 200 mm. The pressures of propane, oxygen, and compressed air were 2.9, 5.0, and 3.2 bar, respectively. The oxygen flow rate was 170 L/min, the powder feed rate was 8 g/min, and the coating was deposited using four passes of the spray gun over the substrate surface.

Atmospheric plasma spraying was performed using a laboratory plasma spraying system equipped with a plasma torch, power supply, powder feeder, and closed-loop water-cooling system. The spraying was carried out at a stand-off distance of 100 mm and an arc current of 200 A. Argon was used as the plasma-forming gas, while nitrogen was used as the powder carrier gas. The effect of substrate preheating on the coating adhesion strength was evaluated at temperatures of 150, 300, 400, and 700 °C. The corresponding adhesion

strength values were 33.4, 39.5, 40.2, and 23.0 MPa, respectively. The maximum adhesion strength of 40.2 MPa was obtained at a substrate preheating temperature of 400 °C.

After thermal spraying, the selected WC–Co–Cr coatings were subjected to ultrasonic nanocrystal surface modification (UNSM). The treatment was performed through repeated mechanical impacts under a controlled static load of 20 N. The ultrasonic frequency was 20 kHz, and the amplitude was 20 µm. The tool feed rate was 2000 mm/min, and the step between passes was 70 µm. The effect of UNSM was evaluated by comparing the as-sprayed coating with the treated samples [12].

Microstructure and surface morphology were examined using a TESCAN VEGA 4 scanning electron microscope (TESCAN, Brno, Czech Republic). The elemental composition of the coatings was determined by energy-dispersive X-ray spectroscopy (EDS) using an EDS Xplore 30 system (Oxford Instruments, Oxford, UK).

Hardness measurements were performed using a FISCHERSCOPE HM2000 system (Helmut Fischer GmbH, Sindelfingen, Germany) in accordance with the DIN EN.

ISO 14577-1 standard [13]. A diamond Vickers indenter with a square pyramidal geometry and an angle of 136° between opposite faces was used. In all tests, the holding time was 10 s at a load of 300 mN. For each coating condition, 10 indentations were performed, with a spacing of 50–100 µm between adjacent indents to minimize mutual interaction. The hardness values were directly determined by the instrument as Vickers hardness (HV). The primary processing of the indentation data was performed using the WIN-HCU 7.1 software. The reported hardness values represent the arithmetic mean of the 10 individual measurements, and the standard deviation was calculated accordingly. According to the instrument specifications, the hardness measurement range is 0.001–120,000 N/mm², the load-setting accuracy is 4 mg, the displacement measurement accuracy is 0.1 nm, and the uncertainty of microhardness determination is approximately 2% of the measured value.

To evaluate the operational performance of the coatings, tests were conducted for abrasive, erosive, and impact-abrasive wear, cavitation erosion, corrosion resistance under salt spray conditions, and dry sliding. Abrasive wear tests were performed on an experimental test rig in accordance with GOST 23.208-79 [14]. The tests were conducted at a normal load of 20 N and a relative motion speed of 0.2 m/s. The duration of each test was 30 min. The tests were carried out under dry conditions at an ambient temperature of (27 ± 1) °C. The main parameter used to evaluate abrasive wear was the mass loss of the specimen after testing. Erosion resistance was evaluated using an experimental gas-jet erosion setup based primarily on ASTM G76 [15]. The specimens had dimensions of 20 × 20 × 5 mm. Electrocorundum was used as the erosive medium. The tests were performed under dry conditions at 27 ± 1 °C. The abrasive particle stream was directed normally to the specimen surface at an impact angle of 90°. The nozzle diameter was 3 mm, the nozzle-to-specimen distance was 10 mm, and the exposure time was 180 s (3 min). Prior to testing, the specimens were ground, degreased and weighed. After testing, the specimens were cleaned and reweighed, and the mass loss was determined from the difference between the initial and final masses.

Salt-spray corrosion testing was performed in accordance with ASTM B117 [16] and ISO 9227 [17]. A 5 wt.% aqueous NaCl solution was used as the corrosive medium. The chamber temperature was maintained at 35 ± 2 °C, and the pH of the salt solution was maintained within the range of 6.5–7.2. The exposure duration was 72 h. After testing, the specimens were removed from the chamber, rinsed with distilled water to remove residual salt and loose corrosion products, dried, and visually inspected. The specimens were then weighed, and the corrosion rate was calculated from the mass loss per unit exposed area and exposure time.

Impact-abrasive wear tests were performed on an experimental test rig in accordance with GOST 23.207-79 [18]. Erosion resistance was evaluated using an experimental test rig in accordance with ASTM G76 [15] and ASTM G211-14 [19]. Cavitation-erosion tests were performed using a hydrodynamic test rig equipped with a high-pressure pump.

Tribological tests were performed using a TRB3 universal tribometer (Anton Paar GmbH, Graz, Austria) in a ball-on-disk configuration in accordance with the ASTM G99 standard [20]. The tests were conducted at room temperature under dry conditions without lubricants. A 100Cr6 steel ball with a radius of 6 mm was used as the counterbody. The tribological tests were performed under a 6 N load at a linear sliding speed of 3 cm/s, with a wear track radius of 3 mm and a total sliding distance of 60 m.

Valve endurance tests were performed using a specially developed test rig for specimen testing. The test system comprised a dynamometric and loading device, an electronic control unit, and SCADA MasterSCADA v3/3.x software for test control and data acquisition. During the tests, plunger displacement was recorded as a function of test time. For component-level assessment, plunger displacement was evaluated as a function of test time using the reported linear relationships $s(t)$ for the uncoated and coated conditions. The displacement growth rate was taken as the slope of the corresponding linear relationship, and the time required to reach the 2 mm displacement criterion was calculated by direct substitution into the corresponding equation. Because these service-life values were calculated from linear relationships, they were treated as extrapolated estimates rather than direct failure times.

A total of 7 plungers were tested, corresponding to three specimens for each investigated condition. The endurance tests were performed under dry-contact conditions using a 20 mm diameter counterbody made of 40 Kh steel. The applied load was 56 N, and the counterbody rotational speed was 150 rpm (2.5 Hz). Temperature was monitored using a thermocouple and a pyrometer, while the experimental parameters were recorded using MasterSCADA. The actual endurance test duration was up to 800 h.

High-temperature oxidation testing. Cyclic oxidation testing was performed in a VMF4-17PX muffle furnace at 1000 °C under atmospheric pressure. The specimens were heated for 1 h, followed by cooling in air for 20 min; this sequence was considered one complete cycle. A total of 50 cycles were performed for each sample. The specimen mass was measured before testing and after each cycle, and the mass change was recorded.

3. Research Results

3.1. Results of SEM (Scanning Electron Microscopy)

Figure 4 presents the cross-sectional microstructures of the WC–Co–Cr coatings deposited by APS, HVOF, and DS methods. The coating thickness was determined from the SEM images of the cross-sections. All investigated coatings exhibit a structure characteristic of thermal spraying. For the APS coating, the thickness in the investigated areas ranges from 62.9 to 74.1 µm. The HVOF coating has a thickness of 62.7–75.3 µm and exhibits a denser and more compact structure. For the coating deposited by the detonation spraying method, the measured thickness ranges from 47.2 to 58.2 µm, with greater thickness non-uniformity observed.

Thus, the APS and HVOF coatings have greater thicknesses compared with the DS coating, while the HVOF coating exhibits a higher degree of density and structural uniformity.

Figure 5 presents the EDS elemental mapping results for the WC–Co–Cr coatings deposited using three methods: APS (a), HVOF (b), and DS (c). In all three images, the interface between the steel substrate and the deposited coating is clearly visible. Fe is predominantly concentrated in the substrate region, whereas W, Co, and Cr are distributed

within the coating layer. This confirms the formation of a tungsten carbide-based coating with a Co–Cr metallic binder phase.

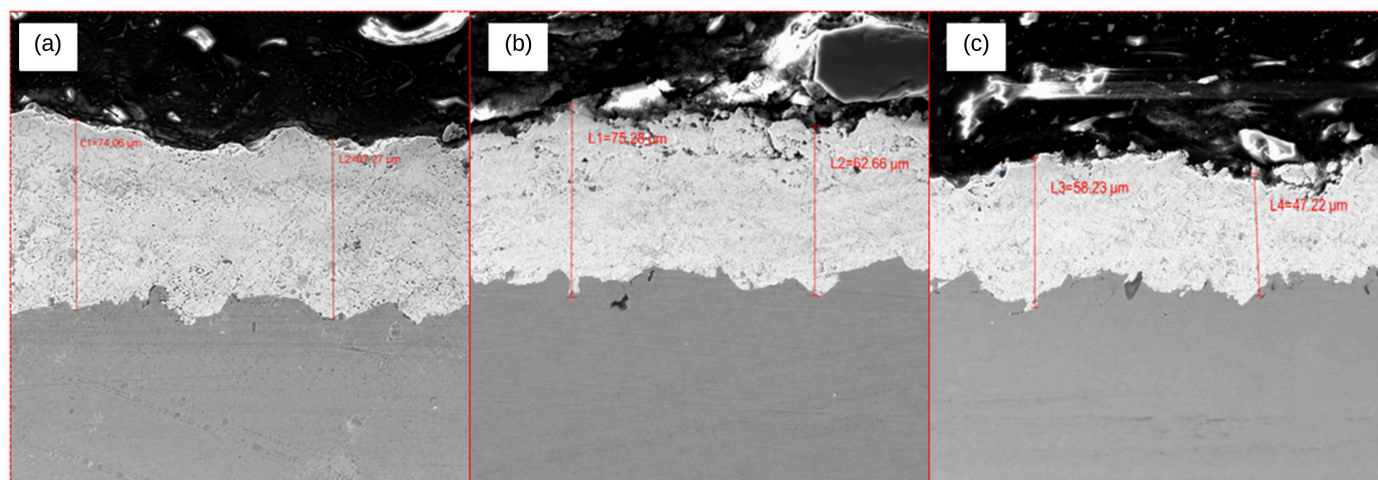


Figure 4. Cross-sectional microstructure of WC–Co–Cr coatings deposited by: (a) APS; (b) HVOF; (c) DS.

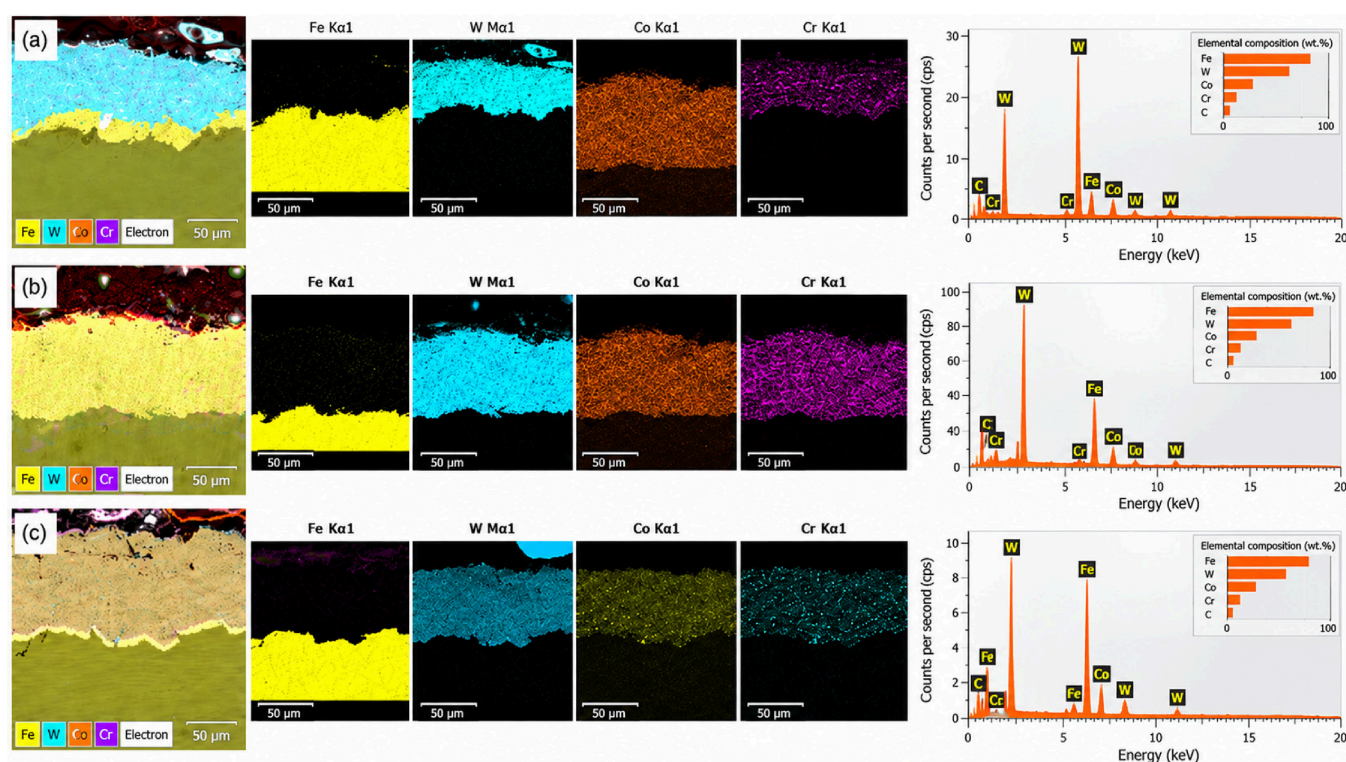


Figure 5. EDS elemental mapping of the cross-sections of WC–Co–Cr coatings deposited by: (a) APS; (b) HVOF; (c) DS.

For the APS coating, the EDS analysis revealed approximately 54 wt.% Fe, 28 wt.% W, 17 wt.% C, and 1 wt.% Cr. The high Fe content is associated with the steel substrate.

For the HVOF coating, the EDS analysis showed approximately 37.5 wt.% W, 33 wt.% C, 19 wt.% Fe, 4.5 wt.% Co, 3.5 wt.% O, and 1.5 wt.% Cr. The elevated W and C contents indicate a well-defined WC carbide phase, while the Fe content is associated with the substrate.

For the DS coating, the elemental composition was approximately 45 wt.% Fe, 39 wt.% W, 16 wt.% C, 5 wt.% Co, and 1 wt.% Cr. The high W and C contents confirm the

presence of the WC phase, while Co and Cr constitute the metallic binder component of the coating.

It should be noted that the EDS Map Sum Spectrum represents the integrated X-ray signal collected from the entire selected mapping area. Since the analyzed cross-sectional region includes the coating/substrate interface and may partially contain material from the steel substrate within the electron interaction volume, the measured Fe content cannot be considered representative of the intrinsic Fe content of the coating itself. Therefore, the relatively high Fe contents measured for the APS (54 wt.%) and DS (45 wt.%) samples are primarily attributed to the contribution from the Fe-based steel substrate.

Figure 6 presents SEM images of the WC–Co–Cr coatings deposited by APS (a), HVOF (b), and DS (c). For all samples, a well-formed coating layer with a continuous interface between the coating and the substrate is observed.

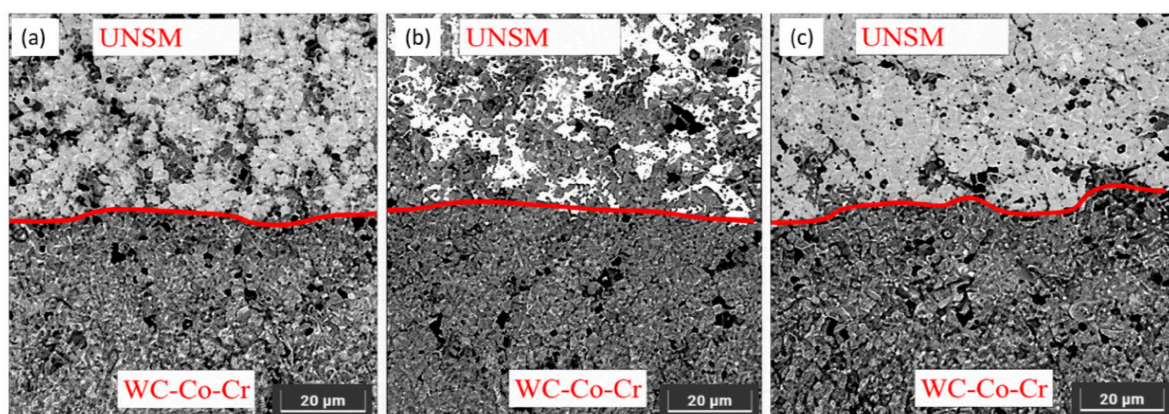


Figure 6. Surface microstructure of WC–Co–Cr coatings deposited by: (a) APS; (b) HVOF; (c) DS.

3.2. Wear and Corrosion Resistance

The abrasive wear test results presented in Table 1 and Figure 7 demonstrate a pronounced influence of both the coating deposition technology and subsequent ultrasonic nanocrystal surface modification (UNSM) on the mass loss of WC–Co–Cr-coated specimens. The uncoated specimen exhibited the highest mass loss, ranging from 2.30 to 2.40 mg. After coating deposition, the mass loss decreased to 1.35–1.45 mg for DS, 0.95–1.05 mg for HVOF, and 0.80–0.88 mg for APS, corresponding to reductions of approximately 37%–44%, 54%–60%, and 62%–67%, respectively.

Subsequent ultrasonic treatment provided a further reduction in mass loss, to 0.68–0.75 mg for DS + UNSM, 0.55–0.60 mg for HVOF + UNSM, and 0.42–0.45 mg for APS + UNSM. The lowest mass loss was observed for the APS + UNSM condition, corresponding to an approximately 80% reduction compared with the uncoated specimen.

Table 1. Abrasive wear performance of WC–Co–Cr coatings before and after UNSM treatment.

Condition	Reported Mass-Loss Range, mg	Reduction vs. Uncoated Range
Uncoated	2.30–2.40	–
DS	1.35–1.45	37%–44%
HVOF	0.95–1.05	54%–60%
APS	0.80–0.88	62%–67%
DS + UNSM	0.68–0.75	67%–72%
HVOF + UNSM	0.55–0.60	74%–77%
APS + UNSM	0.42–0.45	80%–82%

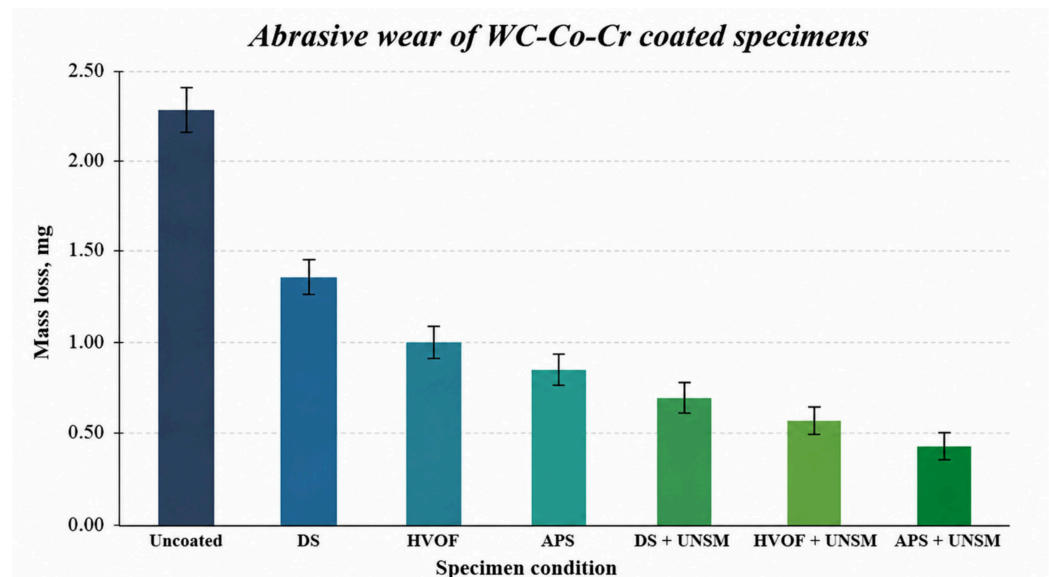


Figure 7. Reported range of abrasive-wear mass loss. Each condition represents three tested specimens; the source report presents the results as ranges.

These results indicate that both the coating deposition technology and subsequent ultrasonic treatment contribute to improving the abrasive wear resistance of the coatings. Among all investigated conditions, the APS + UNSM condition demonstrated the highest abrasive wear resistance.

The test results presented in Tables 2 and 3 and Figures 8 and 9 show that the deposition of WC–Co–Cr coatings substantially reduces mass loss compared with the uncoated specimen. For the uncoated steel, the mass loss under erosive conditions was 3.40–3.60 mg, while under impact-abrasive wear it was 3.90–4.10 mg. After coating deposition, these values decreased to 2.00–2.20 mg and 2.70–2.90 mg for DS, 1.30–1.50 mg and 2.00–2.20 mg for HVOF, and 1.00–1.20 mg and 1.60–1.80 mg for APS, respectively.

Table 2. Individual and statistical results of solid-particle erosion tests.

Condition	Specimen 1, mg	Specimen 2, mg	Specimen 3, mg	Mean $\pm$ SD, mg
Uncoated	3.60	3.50	3.40	3.50 $\pm$ 0.10
DS	2.20	2.10	2.00	2.10 $\pm$ 0.10
HVOF	1.50	1.40	1.30	1.40 $\pm$ 0.10
APS	1.20	1.10	1.00	1.10 $\pm$ 0.10
DS + UNSM	0.95	0.90	0.88	0.91 $\pm$ 0.10
HVOF + UNSM	0.75	0.70	0.68	0.71 $\pm$ 0.10
APS + UNSM	0.60	0.58	0.55	0.58 $\pm$ 0.10

Table 3. Individual and statistical results of impact-abrasive wear tests.

Condition	Specimen 1, mg	Specimen 2, mg	Specimen 3, mg	Mean $\pm$ SD, mg
Uncoated	4.10	4.00	3.90	4.00 $\pm$ 0.10
DS	2.90	2.80	2.70	2.80 $\pm$ 0.10
HVOF	2.20	2.10	2.00	2.10 $\pm$ 0.10
APS	1.80	1.70	1.60	1.70 $\pm$ 0.10
DS + UNSM	1.35	1.30	1.25	1.30 $\pm$ 0.05
HVOF + UNSM	1.10	1.05	1.00	1.05 $\pm$ 0.05
APS + UNSM	0.85	0.80	0.75	0.80 $\pm$ 0.05

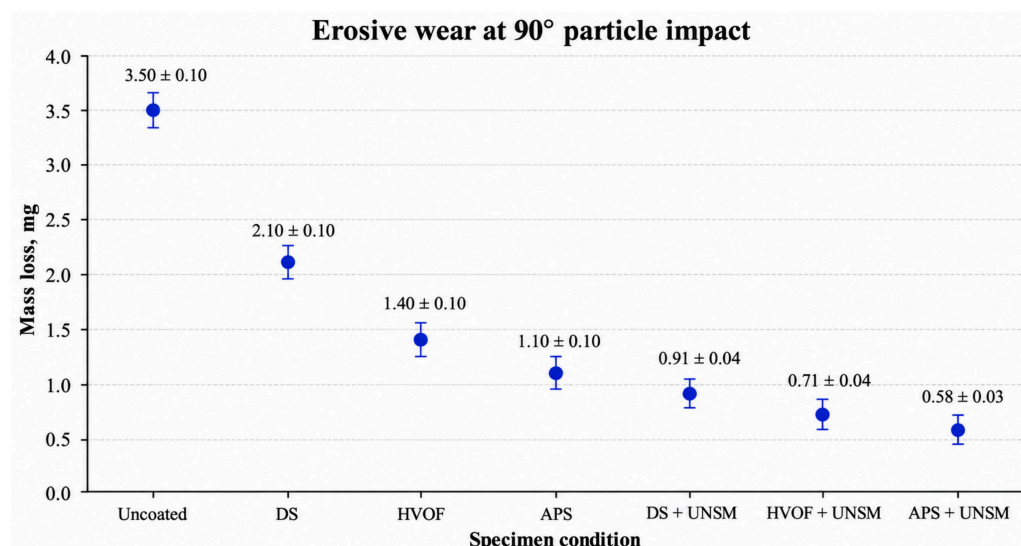


Figure 8. Reported range of erosive mass loss at 90° particle impact.

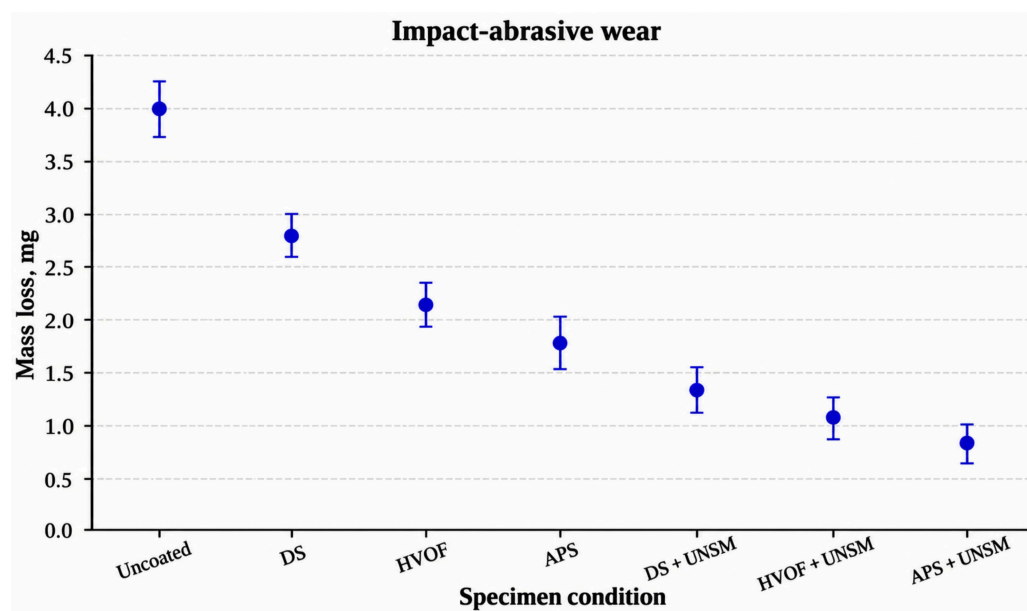


Figure 9. Reported range of impact-abrasive mass loss.

Subsequent ultrasonic treatment resulted in a further reduction in mass loss. For DS + UNSM, the values were 0.88–0.95 mg under erosive conditions and 1.25–1.35 mg under impact-abrasive conditions. For HVOF + UNSM, the mass loss decreased to 0.68–0.75 mg and 1.00–1.10 mg, respectively. The lowest values were obtained for APS + UNSM, with 0.55–0.60 mg under erosive conditions and 0.75–0.85 mg under impact-abrasive wear.

Thus, both types of testing demonstrate the same sequence of increasing wear resistance of the coatings: DS–HVOF–APS, while ultrasonic treatment provides an additional improvement in wear resistance. The best performance was obtained for APS + UNSM, indicating that the combination of atmospheric plasma spraying and subsequent ultrasonic treatment is effective in improving the resistance of WC–Co–Cr coatings to erosive and impact-abrasive wear.

The results of the cavitation erosion and salt-spray corrosion tests are presented in Table 4 and Figures 10 and 11. For the uncoated specimen, the mass loss rate under cavitation exposure was 1.80–1.95 mg/h, whereas a progressive decrease in this parameter was observed after coating deposition. For the DS, HVOF, and APS coatings, the cavitation

wear rates were 1.10–1.15, 0.80–0.85, and 0.70–0.75 mg/h, respectively. Subsequent ultrasonic treatment resulted in a further reduction in the mass loss rate to 0.60–0.65 mg/h for DS + UNSM, 0.45–0.50 mg/h for HVOF + UNSM, and 0.33–0.38 mg/h for APS + UNSM. Thus, the lowest cavitation wear rate was obtained for the APS + UNSM condition, indicating the highest resistance of this condition to cavitation exposure.

A similar trend was observed in the salt-spray corrosion tests. For the uncoated specimen, the corrosion rate was 0.0343–0.0350 mg/(cm²·h). After coating deposition, this parameter decreased to 0.0150–0.0157 mg/(cm²·h) for DS, 0.0114–0.0121 mg/(cm²·h) for HVOF, and 0.0089–0.0093 mg/(cm²·h) for APS. For the HVOF coating after ultrasonic treatment, the corrosion rate was further reduced to 0.0071 mg/(cm²·h), while for APS + UNSM it decreased to 0.0066–0.0070 mg/(cm²·h). The value for the DS + UNSM condition is not presented in the table.

Overall, the results demonstrate that the deposition of WC–Co–Cr coatings substantially improves resistance to both cavitation erosion and salt-spray corrosion. Subsequent ultrasonic treatment further enhances this effect. The lowest mass loss rate under cavitation exposure and the lowest corrosion rate under salt-spray conditions were observed for APS + UNSM, indicating the highest protective performance of this treatment combination among the investigated conditions.

Table 4. Cavitation and Salt-Spray Corrosion Resistance of WC–Co–Cr Coatings.

Condition	Cavitation Rate, mg/h	Salt-Spray Corrosion, mg/(cm ² ·h)
Uncoated	1.80–1.95	0.0343–0.0350
DS	1.10–1.15	0.0150–0.0157
HVOF	0.80–0.85	0.0114–0.0121
APS	0.70–0.75	0.0089–0.0093
DS + UNSM	0.60–0.65	-
HVOF + UNSM	0.45–0.50	0.0071
APS + UNSM	0.33–0.38	0.0066–0.0070

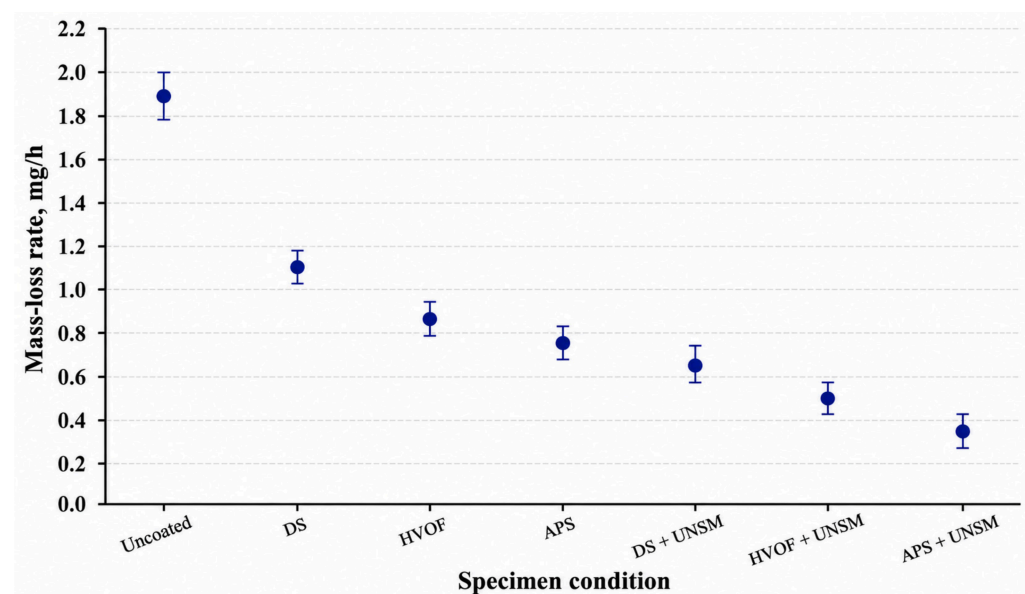


Figure 10. Reported range of cavitation mass-loss rate after 20 h exposure.

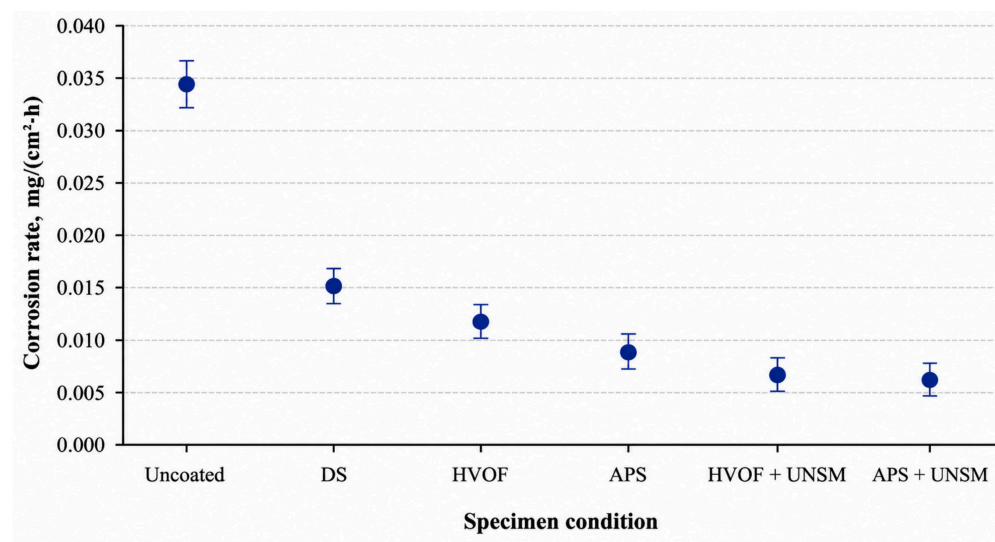


Figure 11. Reported range of salt-spray corrosion rate after 72 h. DS + UNSM was not reported numerically in the available results and is therefore omitted.

3.3. Tribological Performance

Table 5 and Figure 12 summarize the coefficient of friction (COF) of the WC–Co–Cr-coated specimens before and after UNSM. Before surface modification, the COF values were 0.515, 0.500, and 0.410 for the DS, HVOF, and APS coatings, respectively. After UNSM, the COF decreased to 0.36, 0.32, and 0.28 for DS + UNSM, HVOF + UNSM, and APS + UNSM, respectively. Thus, UNSM resulted in a noticeable reduction in the coefficient of friction for all investigated coating systems. The lowest COF was obtained for APS + UNSM (0.28), followed by HVOF + UNSM (0.32) and DS + UNSM (0.36).

Table 5. Tribological behavior of WC-Co-Cr-coated specimens before and after UNSM.

Condition	Coefficient of Friction
DS	0.515
HVOF	0.500
APS	0.410
DS + UNSM	0.36
HVOF + UNSM	0.32
APS + UNSM	0.28

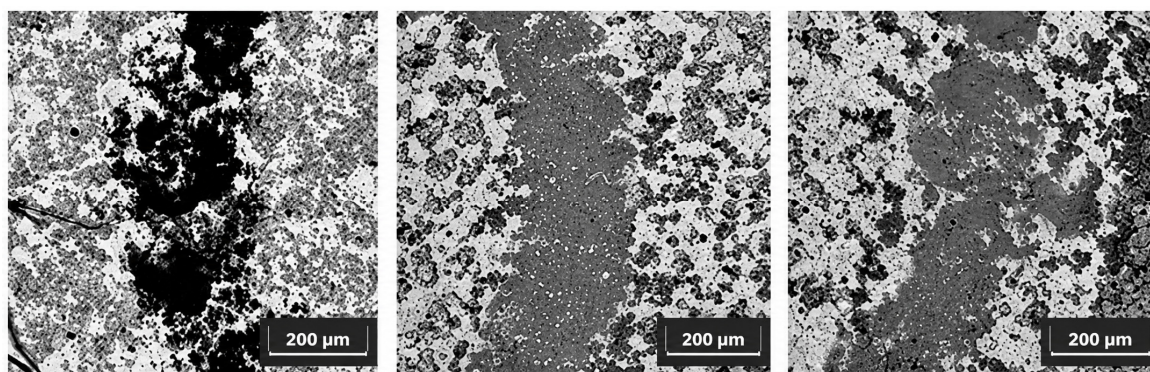


Figure 12. SEM images of the worn surfaces of WC-Co-Cr-coated specimens after tribological testing.

The tribological behavior of WC–Co–Cr coatings is strongly dependent on both coating condition and testing environment [21].

The coefficients of friction of the WC–Co–Cr coatings after UNSM as a function of sliding distance are shown in Figure 12. At the initial stage of the test, the coatings exhibit different trends in the evolution of the coefficient of friction. For DS + UNSM, the coefficient of friction increases most rapidly, followed by a transition to a relatively stable level at an early stage of the test. HVOF + UNSM is characterized by a more gradual increase in the coefficient of friction, followed by a pronounced increase at approximately 30–40 m. APS + UNSM demonstrates a smoother and more gradual increase in the coefficient of friction compared with DS + UNSM and HVOF + UNSM. As the sliding distance increases, the coefficients of friction of all three coatings gradually approach a relatively stable level. The differences observed at the initial stage may be associated with differences in the surface condition and baseline microstructural characteristics of the coatings formed by different spraying technologies. The SEM images presented below the curves correspond to individual areas of the worn surfaces and show localized surface damage during sliding.

As the sliding distance increases, the coefficients of friction of all three coatings gradually approach a relatively stable level. The differences observed at the initial stage may be associated with differences in the surface condition and microstructural characteristics of the coatings formed by different spraying technologies. The SEM images presented below the curves correspond to individual areas of the worn surfaces and show the development of localized surface damage during sliding.

3.4. Valve Endurance Testing

The results of the valve endurance tests demonstrate a pronounced influence of the coating deposition technology and subsequent ultrasonic treatment on the rate of increase in plunger displacement, as presented in Table 6 and Figure 13. For the uncoated specimen, the highest slope of the dependence was observed, at 0.122 mm/h, corresponding to the highest wear rate. The calculated time required to reach a displacement of 2 mm was approximately 9 h.

The application of the coatings resulted in a reduction in the rate of plunger displacement. For the DS, APS, and HVOF conditions, the slope values were 0.100, 0.085, and 0.075 mm/h, respectively. The calculated time required to reach a displacement of 2 mm increased to 11, 13, and 15 h, respectively. Thus, coating deposition improves the wear resistance of the plunger, with HVOF exhibiting the lowest displacement rate among the coatings without additional treatment.

Subsequent ultrasonic treatment provided a further reduction in the displacement rate. For DS + UNSM, the slope decreased to 0.060 mm/h, for APS + UNSM to 0.055 mm/h, and for HVOF + UNSM to 0.050 mm/h. Accordingly, the calculated time required to reach a displacement of 2 mm increased to 18, 20, and 22 h, respectively. The lowest displacement rate and the longest calculated service time were obtained for HVOF + UNSM.

Table 6. Valve Endurance Testing.

Condition	Reported Relationship $s(t)$, mm	Slope, mm/h	Time to 2 mm, h *
Uncoated	$s = 0.122t + 0.858$	0.122	9
DS	$s = 0.100t + 0.860$	0.100	11
APS	$s = 0.085t + 0.870$	0.085	13
HVOF	$s = 0.075t + 0.880$	0.075	15
DS + UNSM	$s = 0.060t + 0.900$	0.060	18
APS + UNSM	$s = 0.055t + 0.905$	0.055	20
HVOF + UNSM	$s = 0.050t + 0.910$	0.050	22

* The time to 2 mm was calculated by direct substitution into the reported linear relationship; therefore, these values are extrapolated estimates rather than direct failure times. The coefficient of determination for the linear relationships was $R^2 = 1.000$ for all investigated conditions.

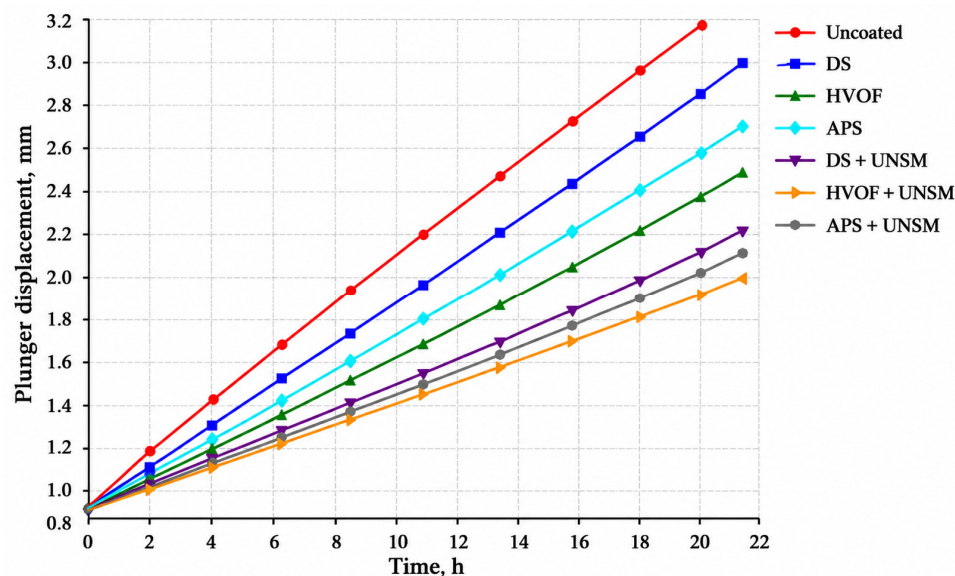


Figure 13. Calculated plunger displacement as a function of time based on the reported valve wear relationships. The dashed line indicates the 2 mm displacement criterion.

3.5. Hardness

The microhardness measurements presented in Table 7 demonstrate a pronounced dependence of the mechanical properties of the WC–Co–Cr coatings on the deposition technology and subsequent UNSM treatment. In the initial state, the microhardness was 724 HV for the coating deposited by detonation spraying, 877 HV for HVOF, and 833 HV for APS. Thus, among the investigated coatings without additional treatment, the highest microhardness was obtained for the HVOF coating.

Table 7. Microhardness of WC–Co–Cr coatings.

Condition	Microhardness, HV $\pm$ SD
Detonation	724
HVOF	877
APS	833
Detonation + UNSM (20 μ m, 20 N)	1468
HVOF + UNSM (20 μ m, 20 N)	1596
APS + UNSM (20 μ m, 20 N)	1561

After UNSM treatment, a substantial increase in microhardness was observed for all investigated coatings. For Detonation + UNSM, the microhardness increased to 1468 HV, for HVOF + UNSM to 1596 HV, and for APS + UNSM to 1561 HV. The maximum microhardness value (1596 HV) was obtained for HVOF + UNSM, indicating the most pronounced surface-hardening effect for this treatment combination.

Overall, the results demonstrate that UNSM substantially increases the microhardness of WC–Co–Cr coatings regardless of the applied spraying technology [22–24]. The observed hardening indicates increased resistance of the surface layer to mechanical deformation, which is consistent with the improved wear resistance after ultrasonic treatment.

3.6. High-Temperature Oxidation Cycles

The samples were heated in a furnace for 1 h and then cooled in air for 20 min. This process was considered one complete cycle. A total of 50 test cycles were performed for each sample. The mass of each sample was measured before the test and after each cycle, and the mass changes caused by oxidation were recorded.

Figure 14 shows distinct mass-change trends during cyclic oxidation of the uncoated and coated specimens. The uncoated specimen exhibited the highest mass increase throughout the test. The mass gain increased progressively from approximately 0.005 g at the 10th cycle to 0.0097 g at the 20th cycle and reached about 0.0188 g by the 50th cycle. No pronounced mass decrease was observed during the 50-cycle test, indicating continuous accumulation of oxidation products under the applied cyclic conditions.

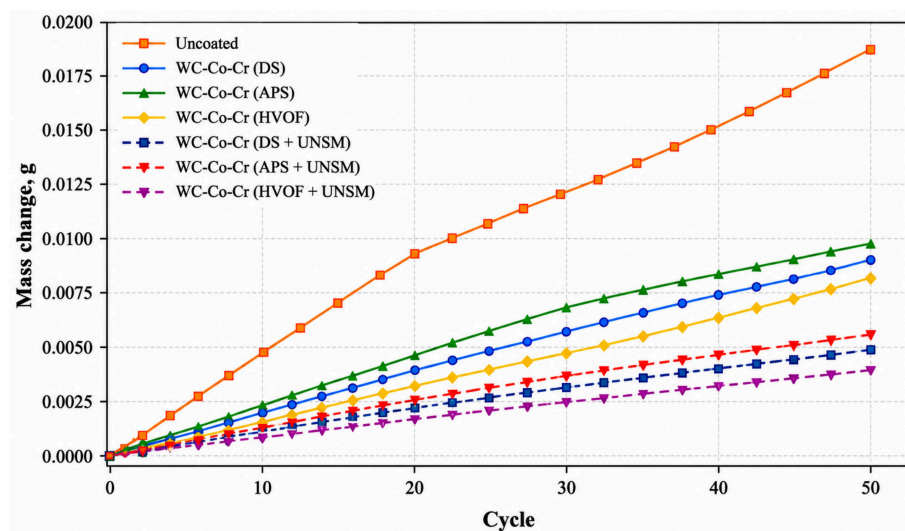


Figure 14. Dependence of the change in sample mass during cyclic heating and cooling simulating high-temperature oxidation conditions.

The coated specimens exhibited substantially lower mass gains than the uncoated specimen. Among the untreated coatings, the mass increase at the 50th cycle was approximately 0.0090 g for DS, 0.0098 g for APS, and 0.0084 g for HVOF. The relatively lower mass gain of the HVOF coating is consistent with its denser coating structure, which can limit oxygen transport through the coating. The APS and DS coatings showed slightly higher mass gains, indicating a greater extent of oxidation under the same cyclic conditions.

The coatings subjected to UNSM exhibited further reductions in mass gain compared with the corresponding untreated coatings. At the 50th cycle, the mass increase was approximately 0.0049 g for DS + UNSM, 0.0056 g for APS + UNSM, and 0.0040 g for HVOF + UNSM. All UNSM-treated specimens showed a relatively stable and nearly linear increase in mass throughout the test, without pronounced mass-loss events. Among the investigated conditions, HVOF + UNSM exhibited the lowest mass gain, indicating the highest resistance to oxidation under the applied cyclic conditions.

4. Discussion

The obtained results show that the properties of WC-Co-Cr coatings are strongly dependent on the spraying technology and subsequent ultrasonic nanocrystal surface modification (UNSM). The HVOF coating is characterized by a denser structure and higher initial microhardness, whereas after UNSM, the hardness of all coatings increases significantly. The increase in hardness after UNSM is accompanied by improved wear resistance. The lowest mass loss under abrasive, erosive, impact-abrasive, and cavitation loading was obtained for the APS + UNSM condition. A similar trend was observed in the tribological tests: after UNSM, the coefficient of friction decreases. The observed differences between APS, HVOF, and DS indicate the important role of the initial coating microstructure. In plunger tests, the HVOF + UNSM condition demonstrated the best performance, indicating that the optimal coating condition depends on the operating conditions. Thus, UNSM is

an effective approach for modifying the near-surface properties of WC–Co–Cr coatings; however, the selection of the spraying technology should be determined by the specific failure mechanism and loading conditions [25–27].

A limitation of the study is the absence of comprehensive post-test SEM characterization of worn surfaces and direct measurements of residual stress and surface roughness. Therefore, the wear mechanisms are interpreted on the basis of the available microstructure, microhardness, and quantitative wear indicators, and the contribution of individual near-surface modification mechanisms cannot be directly separated.

The observed differences in baseline coating microstructure provide a possible explanation for the different responses to UNSM. A dense WC-rich coating with a continuous Co–Cr-containing binder may better accommodate repeated ultrasonic impacts, whereas porous or weakly bonded regions may be more susceptible to cracking or delamination. The WC-rich constituent contributes to hardness and load-bearing capacity, while the Co–Cr-containing binder can accommodate mechanical loading. Because EDS provides elemental rather than phase-specific information and no comprehensive phase analysis was performed in the present study, these observations should be considered a microstructural interpretation rather than direct phase identification. This interpretation agrees with studies linking WC retention, carbide size, porosity, and binder chemistry with hardness, modulus, toughness, and wear resistance [28,29].

The combined results support a process–surface–performance relationship rather than a single universal ranking. Thermal spraying provides the WC–Co–Cr coating, while UNSM modifies the surface and near-surface region. The literature on ultrasonic surface processing indicates that repeated mechanical impacts can promote surface smoothing, work hardening, and compressive residual stress [30]. These changes provide plausible explanations for the improved sliding and particle-wear behavior observed here, but they were not directly measured in the present study. The available microstructural measurements are therefore insufficient to isolate the contribution of each mechanism.

APS + UNSM generally produced the best numerical results in the principal laboratory degradation tests, whereas HVOF + UNSM gave the lowest plunger displacement growth rate in the valve tests. This difference emphasizes that the optimum surface condition depends on the dominant failure mechanism and on component geometry and loading. HVOF is known for producing dense hardmetal coatings and has been widely investigated for wear-resistant WC-based systems [31]. The present results therefore do not support the conclusion that one deposition route is universally superior; instead, they indicate that the combination of deposition route and post-treatment should be selected for the intended service condition.

Previous studies emphasize that thermal-spray performance is governed by the feed-stock, deposition conditions, coating microstructure and oxidation state [32–34]. The present comparison is consistent with that framework because DS, APS and HVOF produced different baseline responses before UNSM. HVOF-related studies also show that spray parameters can influence porosity, residual stress and wear behavior [35].

The improvement observed after ultrasonic treatment can be associated with modification of the surface and near-surface structure induced by repeated mechanical impacts. Recent studies have demonstrated that ultrasonic impact treatment can reduce surface roughness, refine the near-surface microstructure, and increase surface hardness, resulting in improved wear resistance of coated and surface-engineered materials [36,37]. More recently, ultrasonic treatment of nickel-based laser-cladded coatings was shown to simultaneously improve hardness, wear resistance, and corrosion resistance, with the observed changes being associated with microstructural refinement and modification of residual stresses. These findings support the interpretation that the effect of ultrasonic treatment is not limited to surface smoothing, but involves a broader modification of the near-surface

state. However, surface roughness, residual stress, and post-treatment microstructural refinement were not directly quantified in the present study.

The main limitation is the form and completeness of the available results. Several laboratory results are reported as ranges rather than complete individual measurements, while valve service-life values are extrapolated from linear wear relationships. Accordingly, no artificial standard deviations or confidence intervals are introduced. In addition, the available material does not provide a complete set of coating microstructural observations before and after UNSM, so mechanistic explanations remain interpretative. The results should therefore be considered evidence of performance trends rather than a complete optimization study of UNSM parameters.

5. Conclusions

The comparative study demonstrated that the performance of WC–Co–Cr coatings is strongly influenced by the deposition technology and subsequent ultrasonic nanocrystal surface modification (UNSM). All investigated thermal-spray coatings provided substantially higher resistance to abrasive, erosive, impact-abrasive, cavitation and corrosion degradation compared with uncoated steel.

UNSM produced a pronounced surface-hardening effect. The microhardness increased from 724, 877, and 833 HV for DS, HVOF, and APS coatings to 1468, 1596, and 1561 HV, respectively. The maximum hardness was obtained for HVOF + UNSM. APS + UNSM demonstrated the most favorable performance in the principal laboratory degradation tests, whereas HVOF + UNSM exhibited the highest microhardness and the best component-level endurance performance.

Among the laboratory degradation tests, APS + UNSM demonstrated the most favorable overall performance, showing the lowest mass loss under abrasive, erosive, impact-abrasive and cavitation conditions, as well as the lowest coefficient of friction.

At the component level, HVOF + UNSM exhibited the lowest plunger displacement growth rate (0.050 mm/h) and the longest calculated time to the 2 mm criterion (22 h). These results confirm that the optimum coating condition depends on the specific degradation mechanism and service conditions.

Overall, the combination of thermal spraying and UNSM is an effective surface-engineering approach for improving the mechanical and service performance of WC–Co–Cr coatings. Further studies should focus on post-test SEM characterization, direct measurements of surface roughness and residual stress, phase characterization where required, and systematic optimization of UNSM parameters to establish the dominant degradation mechanisms.

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