

Steel Thermal Sprayed Coatings: Superficial Hardening by Nitrogen Ion Implantation

Experiments were conducted to optimize process parameters for thermal spraying and coating modifications

BY M. BELOTSEKOVSKY, A. YELISTRATOV, A. BYELI, AND V. KUKAREKO

ABSTRACT

Modification of steel thermal sprayed coatings by nitrogen ion implantation can dramatically increase wear resistance of their surface layers. The only obstacle for successful modification is low strength and defects in regular wire-sprayed coatings. However, different methods are available to improve physical properties of coatings. In this study, two methods of wire spray process activation were investigated: gas-jet activation (high-velocity spraying) for reduction of sizes and increasing velocity of particles; and ultrasonic activation for reduction of sprayed particle sizes. Once coatings were modified by implantation of nitrogen ions, wear resistance tests were performed. As a result of the following experiments, process parameters for thermal spraying and coating modification were optimized.

Introduction

Wear of machine parts creates a demand for new materials, which have optimized surface properties without altering the bulk properties of the part. This new demand stimulates development of bimetallic parts with special coatings to enhance their service performance.

Thermal spray coatings are a cost-effective way for manufacturing bi-metallic parts, and resurfacing and hardfacing worn machine components. There are two

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groups of thermal spray methods in relation to what kind of material is in use: powder-spray methods and wire-spray methods. The latter group includes oxyfuel wire spraying (OFWS) and dual-wire arc spraying (DWAS) that have some advantages in terms of lower cost of materials (wire vs. powder), and highest productivity (DWAS) in the group of thermal spray coatings. In the present study, only wire-spray methods were investigated.

For general engineering, wire-spray methods are extremely efficient in cases where there is no need to deposit special high-alloy coatings (ceramic, heat-resistant alloys), but single metals (aluminum, copper) or alloy steels. Since for most friction-wearing parts dimensional allowances are limited, just a thin surface coating with improved hardness/wear resistance can dramatically increase the whole machine's performance.

Ion implantation is a process of inserting ionized atoms into a substrate with energy sufficient for their "penetration" into the crystalline lattice of a solid metal. The amount of ions of alloying elements implanted into a layer of substrate is determined not by the physical properties of the alloys and the diffusion coefficients, but by implantation process parameters. As a result, a superficial layer with a gradient structure and high wear resistance can be formed (Refs. 1–3). Radiation-stimulated diffusion of nitrogen ions, developed in a superficial layer of metal with a thickness of 10–200 μm (0.0004–0.008 in.), enhances its hardness. This method is applied in industry (Refs. 1, 2) for superficial harden-

ing of metals. Preliminary experiments revealed steel thermal sprayed coatings could be successfully hardened by ion implantation.

In the present research, both wire-spray methods were modernized to improve their coating's properties and steel coatings were superficially hardened by vacuum implantation of nitrogen ions.

Background

When metal wire(s) feeds inside the active zone of a thermal spray gun, it melts by gas flame (OFWS) or by electric arc [dual wire arc spray (DWAS)] and then is atomized by the gas jet. Existing atomizing units can't provide even or uniform particle sizes. For general OFWS and DWAS thermal spray methods, the range of atomized particle sizes varies from 10 to 300 μm (0.0004–0.012 in.). As a rule, particles are separated in the gas jet with the finest particles located in the middle of the gas jet along the axis and heavier particles are located in the outer layers of the gas jet (Ref. 4). Since the finest particles can be accelerated by the gas jet faster, they have higher dynamic characteristics and, upon striking the substrate, provide higher adhesion strength of the coating, compared to larger, slower moving particles. In other words, a coating formed from only fine axial particles can have a much higher adhesion strength than one formed from heavier outer particles. Furthermore, when the spray gun or workpiece is moving during spraying, the first layer on the surface, which is responsible for the adhesion strength of the whole coating, is formed from the outer, heavier particles (Ref. 5). As a result, the total adhesion strength of the coating is not high enough.

In order to increase adhesion of the coatings, modernized thermal spray guns with improved atomizing of the liquid metal at the tip of the wires were used in this study as follows:

- **Dual-wire arc spray gun** (HVAF-ARC, Fig. 1) has an activator (Refs. 8, 9) that provides atomizing of liquid metal

KEYWORDS

Steel Wires
Thermal Spray Coating
Particle Diameters
Particle Velocity
Nitrogen Ion Implantation
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Wear Resistance

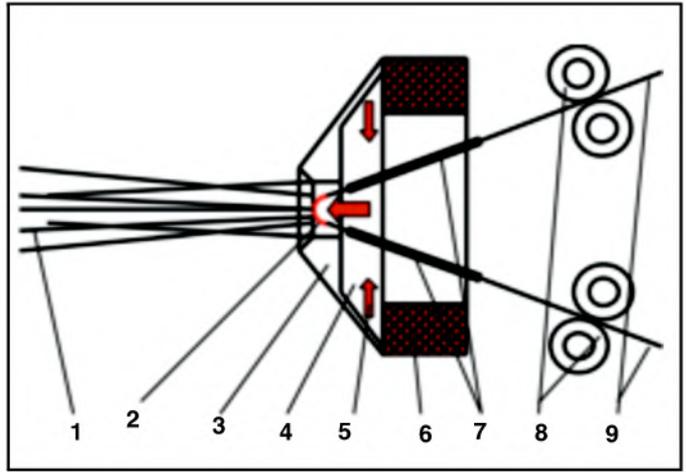
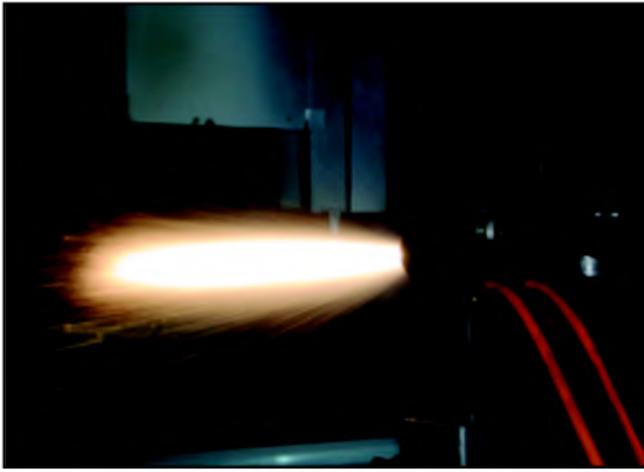


Fig. 1 — Dual-wire thermal spray gun with activated combustion HVOF-ARC. (Photo courtesy of Uniquocoat Technologies, LLC.)

Fig. 2 — Schematic of high-velocity wire-atomizing unit: 1) Metal particles jet; 2) electric arc; 3) nozzle; 4) exhaust gas flow; 5) air-fuel combustion chamber; 6) wire tips; 7) wire feeders; 8) wire feeders; 9) consumable wires.

and acceleration of particles. Two consumable wires are fused by an electric arc and atomized by a supersonic jet of air-fuel combustion products, generated in a combustion chamber — Fig. 2. The rear wall of the chamber is made of ceramic containing a high-temperature catalyst. After flame ignition with a spark, the rear

wall is heated and provides continuous air-fuel combustion during the spray process. Wire tips are located inside the chamber with the flow of exhaust gases directed along them. Wire material atomization occurs in a short expanding nozzle. Since fine metal particles are prone to rapid in-flight oxidation, reduced condition in the

arc zone is a critical factor. This is provided by an excess of the fuel gas (propane) in the air-fuel combustion mixture. The activator generates a supersonic gas jet with a total dynamic action three times that of regular plasma spraying. It has a velocity of up to 900 m/s (2950 ft/s) while for general DWAS guns, the atomizing air has a velocity of around 300 m/s (984 ft/s). The jet temperature was up to 1000 K (1340°F), while for general DWAS, the atomizing air is at room temperature. This provides an acceleration of particles up to 300 m/s (984 ft/s) with particle sizes ranging from 2 to 20 μm (8–80 μin.) while for general DWAS guns, the velocity of particles is up to 150 m/s (492 ft/s), with their size at 10–300 μm (0.0004–0.012 in.).

Table 1 — Chemical Composition of Base Metal and Welding Wires, wt-%

	C	Cr	Ni	Ti	Mn	Fe
Base Metal	0.18	0.3	—	—	0.42	Balance
Wire 40H13	0.45	12.0	0.5	0.05	—	Balance
Wire H18N10T	0.06	18.4	9.25	0.62	0.20	Balance

Table 2 — Modified Layer Parameters for 40H13 Coatings

Modification process	Modified layer parameters			
	Thickness, mkm	Hardness, HV	Phase composition	
OFS	620 K (656°F)	5–10 (0.0002–0.0004 in.)	1020	α-Fe, γ-Fe, γ _N , Fe ₃ O ₄ , ε-(Fe, Cr) ₃ N
	670 K (746°F)	10–20 (0.0004–0.0008 in.)	1530	α-Fe, γ-Fe, γ _N , Fe ₃ O ₄ , ε-(Fe, Cr) ₃ N, α''-(Fe, Cr) ₈ N, and γ'-Fe ₄ N
	720 K (836°F)	15–25 (0.0006–0.001 in.)	1530	α-Fe, Fe ₃ O ₄ , ε-(Fe, Cr) ₃ N, γ'-Fe ₄ N, CrN
	770 K (926°F)	30–40 (0.0012–0.0016 in.)	1123	α-Fe, Fe ₃ O ₄ , γ'-Fe ₄ N, CrN
	620 K (656°F)	5–10 (0.0002–0.0004 in.)	918	α-Fe, γ-Fe, γ _N , Fe ₃ O ₄ , ε-(Fe, Cr) ₃ N
HVOF-ARC	670 K (746°F)	10–15 (0.0004–0.0006 in.)	1428	α-Fe, Fe ₃ O ₄ , ε-(Fe, Cr) ₃ N, γ'-Fe ₄ N
	720 K (836°F)	15–20 (0.0006–0.0008 in.)	1377	α-Fe, Fe ₃ O ₄ , ε-(Fe, Cr) ₃ N, γ'-Fe ₄ N, CrN
	770 K (926°F)	25–30 (0.001–0.0012 in.)	1020	α-Fe, Fe ₃ O ₄ , γ'-Fe ₄ N, CrN



Fig. 3 — Unit for ion implantation of nitrogen: 1) Vacuum chamber; 2) mechanism for controlling position of parts during processing.

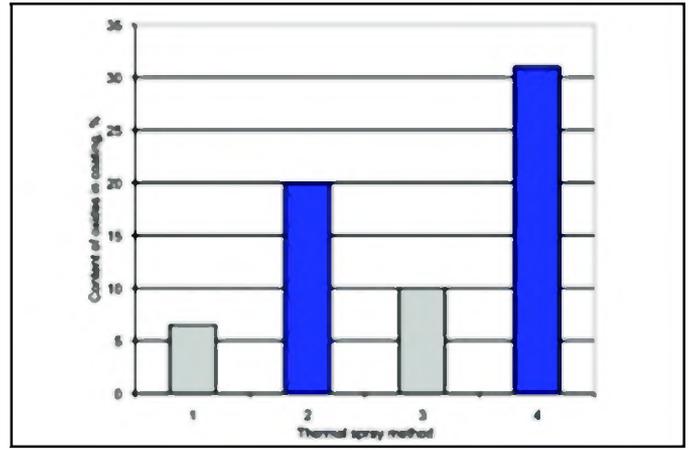


Fig. 4 — Oxide content of thermal spray coatings: 1, 2) 40H13 wire; 3, 4) X18H10T wire. 1, 3) OFWS; 2, 4) HVAF-ARC.

This results in higher coating density with porosity <1%, compared to 5–10% for regular DWAS (Ref. 10).

• **Oxyfuel wire spray gun (OFWS-TERKO)** had an ultrasonic activator (Refs. 6, 7) that generates high-frequency acoustic waves inside the overlapping stream of air. Dynamic action of high-frequency oscillation of the air stream helps to form fine droplets of metal by breaking large liquid metal drops near the tip of the wire into small pieces by the gas jet. Also, turbulent pulses of air pressure helped to refine the particles during their movement to the base metal. In the activated mode, the velocity of the particles was 120–150 m/s (394–492 ft/s) and particle size was 5–120 μm (0.0002–0.0047 in.).

Experimental Procedure

Mild steel plates with dimensions of 150 × 70 × 10 mm (6 × 3 × ¾ in.) were sandblasted to an average surface roughness of 30–50 μm (0.0012–0.002 in.). Coatings were made by spraying of steel wires Ø 2 mm (¾ in.), from two different alloy groups: martensitic 40H13 (U.S. analog: 420SS) and austenitic X18N10T (U.S. analog: 321SS). The coating thickness was 0.8–1.2 mm (0.03–0.047 in.). The chemical composition of the wires and base metal is presented in Table 1.

After spraying, test blocks were cut from the plates, ground, cleaned, and subjected to nitrogen ion implantation. An implanter (Fig. 3) with an ion beam source based on close-looped drift of electrons

(Ref. 3) was used for ion beam processing. The implanter generated a strip-type (120 × 2.5 mm or 4.72 × 0.098 in.) beam of nitrogen ions. The beam contained approximately 70% molecular nitrogen ions and approximately 30% atomic nitrogen ions.

The implantation parameters are as follows:

- Ion current density 2 mA/cm² (12 mA/in.²);
- Energy of ions 3000 eV;
- Fluency of ions (amount of ions per area at 1 cm²) 3 × 10¹⁹ cm⁻² (18.5 × 10¹⁹ in.⁻²);
- Temperature during implantation: 620–670–720–770 K (656°–746°–836°–926°F).

Metallographic investigations were conducted using an optical microscope “Unimet.” To obtain durometric measure-

Table 3 — Modified Layer Parameters for H18N10T Coatings

Modification process	Modified layer parameters		
	Thickness, mkm	Hardness, HV	Phase composition
OFS	620 K (656°F)	3–5 (0.00012–0.0002 in.)	663 α -Fe, γ -Fe, γ _N , Fe ₃ O ₄
	670 K (746°F)	5–10 (0.0002–0.0004 in.)	969 α -Fe, γ -Fe, $\gamma'N, Fe3O4$
	720 K (836°F)	10–15 (0.0004–0.0006 in.)	1275 α -Fe, γ -Fe, Fe ₃ O ₄ , γ' -Fe ₄ N, CrN, γ' N
	770 K (926°F)	15–20 (0.0006–0.0008 in.)	1224 α -Fe, γ -Fe, Fe ₃ O ₄ , γ' -Fe ₄ N, CrN
	620 K (656°F)	3–5 (0.00012–0.0002 in.)	663 α -Fe, γ -Fe, γ _N , Fe ₃ O ₄
HVAF-ARC	670 K (746°F)	3–8 (0.00012–0.0003 in.)	918 α -Fe, γ -Fe, $\gamma'N, Fe3O4$
	720 K (836°F)	10–15 (0.0004–0.0006 in.)	1224 α -Fe, γ -Fe, Fe ₃ O ₄ , $\gamma'N, \gamma'-Fe4N, CrN$
	770 K (926°F)	15 (0.0006 in.)	1173 α -Fe, γ -Fe, Fe ₃ O ₄ , γ' -Fe ₄ N, CrN

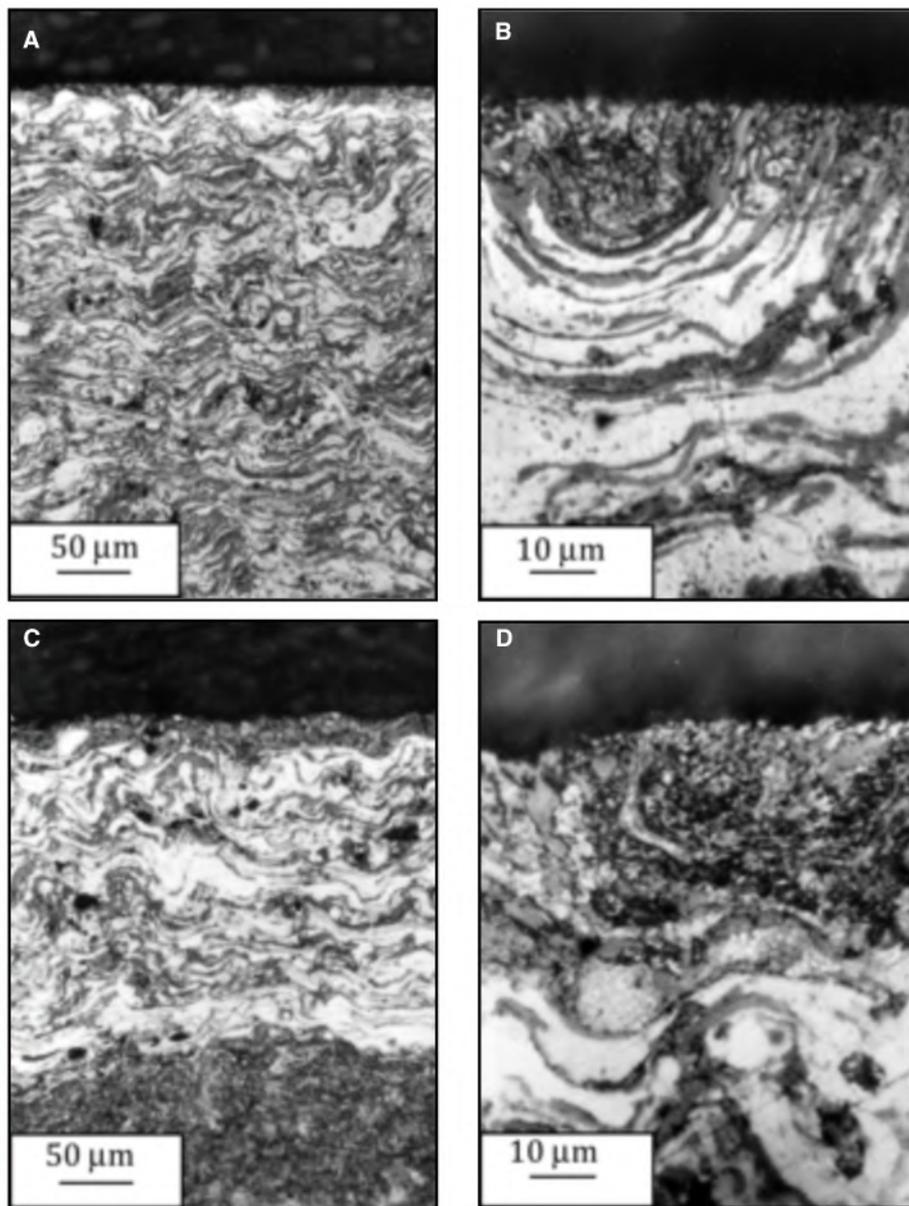


Fig. 5 — Microstructure of modified (at 770 K or 926°F) coatings: A, B) X18N10T steel; C, D) 40H13 steel.

ments, a microhardness tester “Micromet-11” with loads 0.19 and 0.49 N (0.042 and 0.11 lb-ft) was used. Phases and structure transformations inside the modified layer of coatings were studied with an X-ray diffractometer (DRON-2.0) in $\text{CoK}\alpha$ irradiation. For phase analysis, a standard PDF library was utilized.

Dry Friction Test

Tribotest of samples with superficial hardening by ion implantation was conducted with ATVP tester, with provision to monitor the coefficient of friction.

The tester was used in the reciprocating motion mode, with an average velocity

of contacting surfaces of ~ 0.1 m/s (3.93 in./s) and a specific load of 1.5 MPa (217.5 lb/in.²). A hardened (795–815 HV) counterbody with dimensions $2 \times 40 \times 90$ mm ($0.078 \times 1.57 \times 3.54$ in.) was used to evaluate wear. The test block was prism-shaped and had dimensions of $5 \times 6 \times 8$ mm ($0.196 \times 0.236 \times 0.314$ in.). Contacting surfaces of the block and the counterbody were ground, polished, cleaned with alcohol, and dried. Measurement errors of the coefficient of friction was $<5\%$ of the measured value.

The test block was weighed before and after the test; the weight loss can be used directly as the wear value or converted to volume loss. In the present study, weight

loss (wear) is reported. Accuracy of the analytical scale (ADV-200M) was ± 0.05 mg (1.1×10^{-7} lb). Before weighing, the block was cleaned, washed in alcohol, and dried at 370 K (206°F). Each block was weighed two or three times.

Dry friction tests were conducted on a basis of 16,000 cycles with intermediate weighing of the sample after 1000–2000 cycles. Friction track length was 800 m (874.89 yd). Tests were conducted until adhesive bonding occurred between the test block and the counterbody. At that moment, the coating should not lose its adhesion bond with the base metal.

Adhesion Wear Tests in Friction

The same ATVP tester in limited friction mode (resistance to adhesion) was used for friction wear tests of the blocks. Initial lubrication of the surface before the test was utilized. The specific load was 25 MPa (3625.9 lb/in.²), and the test base was 30,000 cycles with a track length of 1500 m (1640 yd) or until contacting surfaces were bonded by adhesion.

Results and Discussion

Structure and Phase Composition of Spray Coatings

For HVOF-ARC coatings (for both the 40H13 and X18N10T wires), porosity did not exceed 1–2 vol-%, and hardness was 357 and 331 HV, respectively.

The phase composition for HVOF-ARC (40H13) was as follows: α -phase (martensite), γ -phase (austenite), Fe_3O_4 , and FeO oxides. The phase composition for HVOF-ARC (X18N10T) was as follows: γ -phase (austenite), α -phase (martensite), and oxide Fe_3O_4 . Content of α -Fe in a superficial layer of the X18N10T coating did not exceed 20 vol-%. The total amount of oxides in HVOF-ARC coatings approached 20–30 vol-% — Fig. 4.

For OFWS coatings, porosity was 10–12 vol-%. Phase composition of the coatings from all types of wire was close to that of the HVOF-ARC coatings. According to X-ray analysis, OFWS coatings have a smaller concentration of oxides (5–10 vol-%). Hardness of the OFWS coatings was 204 and 153 HV for coatings from wires 40H13 and H18N10T, respectively. Lower hardness values for OFWS compared to HVOF-ARC coatings can be explained by the presence of larger particles in the coatings, more porosity, and limited amount of oxides.

Nitrogen Ion Modification of Coatings

The upper layer of the sprayed coating, modified by ion implantation, has a light gray color — Fig. 5. Structure parameters,

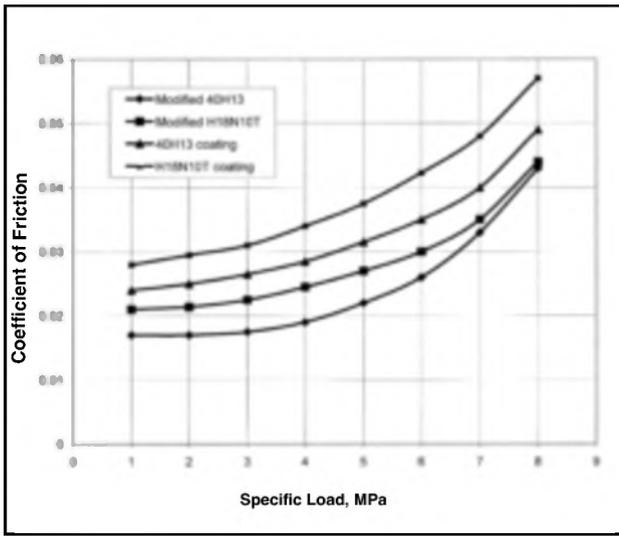


Fig. 6 — Coefficient of friction of initial and modified coatings vs. specific load.

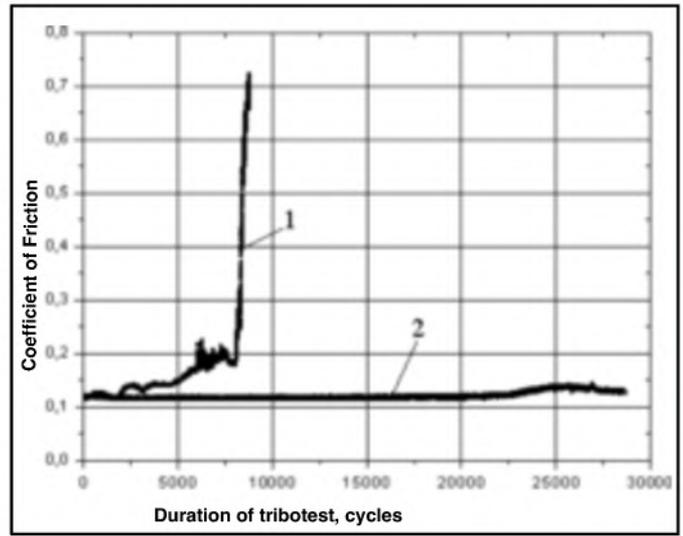


Fig. 7 — Coefficient of friction for HVAF-ARC 40H13 coating vs. tribotest duration: 1) Coating in initial state; 2) coating, modified by ion implantation at 770 K (926°F).

phase composition, and microhardness for modified OFWS coatings (40H13) are presented in Table 2. Thickness of the modified layer of the coating increased with processing temperature, up to 30–40 μm (0.0012–0.0015 in.). Hardness of the modified layer increased with processing temperature and reached a maximum value of 1480–1632 HV at $T_p = 670\text{--}720$ K (746°–836°F). Main phases in the modified superficial layer of coatings (40H13) are $\epsilon\text{-(Fe, Cr)}_3\text{N}$ and $\gamma\text{-Fe}_4\text{N}$ nitrides. After ion modification at $T_p = 720$ and 770 K (836° and 926°F), nitrogen-rich layers were observed to have sharply decreasing amounts of ϵ -nitrides (with high nitrogen content) and formation of CrN nitrides. Simultaneously, the hardness decreased up to 1020–1122 HV. In modified coatings the concentration of Fe_3O_4 oxides did not change, but concentration of

FeO oxides was reduced. For OFWS coatings, a relatively high hardness and larger thickness of the modified layer were observed. Also, an interesting feature of modified OFWS coatings was the presence of $\alpha\text{''-(Fe, Cr)}_8\text{N}$ phase, which have a lower amount of $\gamma\text{-Fe}_4\text{N}$ phase and higher amount of CrN nitrides.

The higher ability for diffusion for OFWS coatings can be explained by higher concentration of defects (vacancies, dislocations, pores), which were formed during rapid crystallization of molten particles. Thermodynamically stable Fe_3O_4 oxide alloyed by chromium was present inside the coating between particles. Those oxides impede the diffusion transfer of the nitrogen from the upper layer of the coating down. The maximum depth of the modified layer for OFWS coatings can be achieved at the lowest

level of oxide.

Ion modification of OFWS coatings (X18N10T) at 620–670–720–770 K (656°–746°–836°–926°F) processing leads to the forming of modified layers with thicknesses of 3–5, 5–10, 10–15, and 15–25 μm (0.00012–0.0002, 0.0002–0.0004, 0.0004–0.0006, and 0.0006–0.001 in.), respectively (Table 3).

The hardness values (vs. process temperature) of modified layers for HVAF-ARC and OFWS coatings were 663, 918–970, 1225–1275, and 1173–1224 HV, respectively. The hardness of the lower part of the coating, which was not modified by ionic implantation, did not change.

Wear Resistance of Modified Coatings

Wear tests, with a load of 1.5 MPa (217.5 lb/in.²) for HVAF-ARC (40H13)

Table 4 — Wear Resistance of Modified HVAF-ARC Coatings in Dry Friction Mode

Coating	Temperature of implantation	Wear of layer, (10^{-3}) mg/m	Wear of counterbody, (10^{-3}) mg/m
40H13	Initial condition	4.5	16.8
	620 K (656°F)	3.5	16.0
	670 K (746°F)	0.7	15.9
	720 K (836°F)	1.1	16.2
	770 K (926°F)	1.3	18.5
H18N10T	Initial condition	12.5	23.7
	620 K (656°F)	10.3	28.5
	670 K (746°F)	4.1	26.2
	720 K (836°F)	1.6	29.1
	770 K (926°F)	1.4	16.9

Conversion: 1 mg/m is equal to 6.7×10^{-7} lb/ft

coatings at different levels of ion modification, showed that increasing the implantation temperature led to a sharp increase in wear resistance (Table 4). Coatings modified at a higher temperature ($T_p = 670\text{--}770\text{ K}$ or $746\text{--}926^\circ\text{F}$) have higher wear resistance. Ion modification at $T_p = 620\text{ K}$ (656°F) for X18N10T coatings resulted in a modified layer with a thickness of $3\text{--}6\ \mu\text{m}$ ($11\text{--}23\ \mu\text{in.}$); however, no increase in wear resistance occurred during dry friction tests (Table 4). The reason for fast destruction of the thin hard layer is, probably, intensive plastic deformation of bulk metal under the hard layer during the test (Ref. 11).

Wear resistance of coatings increased when the ion modification was carried out at temperatures above 700 K (800°F). Higher hardness of the modified layer and greater thickness are the main reasons for this.

Studying the coefficient of friction's variation (test with limited lubrication) revealed that modified coatings (Fig. 6) have a smaller value of coefficient of friction compared to the same coatings without ion modification. Also, ionic modification of the steel coatings leads to an increase of their resistance to adhesion during dry friction test.

Tribotests of HVOF-ARC (40H13) coatings that were modified at 770 K (926°F) revealed (Fig. 7) a four times greater amount of cycles before adhesion in friction occurred between the test block and counterbody at a test load of 25 MPa (3625 lb/in.^2) and its subsequent catastrophic destruction. Test conditions were as follows: lubrication, oil L-20A; load, 25 MPa (3625 lb/in.^2); and counterbody, hardened 0.6% C steel. Higher wear resistance (resistance to adhesion in friction) of coatings modified at 770 K (926°F) is connected to the higher strength of the modified layer, which contains dispersed particles of CrN.

During dry friction tests of OFWS coatings with a load of 1.5 MPa (217.5 lb/in.^2), there was local destruction of the coatings with formation of debris with a size of $100\text{--}300\ \mu\text{m}$ ($3.93\text{--}11.8 \times 10^{-3}\text{ in.}$). This can be explained by insufficient adhesive strength of the OFWS coatings and sizeable porosity due to low velocity of particles. Gases from pores caused local destruction of the coating during implantation or following wear tests. Because of this, OFWS coatings were eliminated from the dry friction test.

Test results demonstrated that maximum efficiency of ionic implantation could be achieved for high-velocity spray coatings with a test load of up to 25 MPa (3625 lb/in.^2) and a friction speed of up to 1.5 m/s (4.92 ft/s).

The technology of steel coating modification by ion implantation of nitrogen

described in this paper was applied for resurfacing and hardening of hydro pump shafts, crankshafts, valve rods, etc.

The possibility of controlling the composition and concentration of diffused ions on a localized area of the workpiece makes the process technologically flexible. Modified coatings do not require subsequent polishing.

Conclusions

- The HVOF-ARC atomizing unit for wire thermal spraying allows considerable improvement in coating formation. Coatings, formed with particles of $2\text{--}30\ \mu\text{m}$ ($8\text{--}110\ \mu\text{in.}$) size and a velocity up to 300 m/s (984 ft/s) demonstrated high adhesion strength and uniform structure with minimal porosity, less than $1\text{ vol.}\%$.
- High-density coatings can be effectively modified by implantation of nitrogen ions. They form a superficial layer with thicknesses of up to $40\ \mu\text{m}$ ($160\ \mu\text{in.}$) and hardnesses of up to 1530 HV .
- Ion implantation of steel coatings, sprayed by the high-velocity wire-spray process, provides an $8\times$ increase in wear resistance and up to $4\times$ increase in resistance to adhesion in friction.

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