

Morphology Replication Effects in Hybrid-treated MIM 17-4PH Stainless Steel and Their Influence on Friction and Wear Behavior

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Abstract

This study investigated the morphology replication and resulting structural and tribological effects of different surface treatment methods on metal injection-molded (MIM) 17-4PH martensitic precipitation-hardenable stainless steel. Three conditions were evaluated: untreated, TiAlN + TiCN PVD-coated, and hybrid-treated samples combining low-temperature plasma nitriding (LTPN) with PVD coating. The specimens were characterized for surface roughness and morphology, phase composition, hardness, adhesion, friction behavior, and wear resistance. The results showed that LTPN significantly modified the substrate surface through crack formation, grain boundary shear offsets, and grain surface uplifts, with the latter two morphological features being subsequently replicated by the deposited coating, leading to increased roughness parameters (R_{pk} , R_{vk} , and R_k). The nitrided layer improved coating support and increased the hardness of the PVD-coated system. In addition, the altered surface morphology was associated with changes in the coating's preferred crystalline orientation, including the appearance of the (220) and strengthening of the (222) TiAlN/TiCN reflections. Tribological testing demonstrated that the hybrid-treated samples exhibited improved tribological performance, including lower steady-state friction and enhanced wear resistance, attributed to the combined effects of the modified surface morphology and enhanced load-bearing capacity of the nitrided substrate. The findings highlight the role of substrate grain morphology in governing LTPN-induced surface modifications and their subsequent replication by the PVD coating, suggesting that further optimization of MIM processing parameters could enable control of the surface morphology and, consequently, the tribological properties of hybrid surface-treated MIM components.

Keywords

metal injection molding, PVD, LTPN, morphology, stainless steel

1 Introduction

17-4PH stainless steel is widely used in engineering applications due to its favorable combination of high strength, corrosion resistance, and good manufacturability [1–3]. In recent years, metal injection-molded (MIM) 17-4PH steel has gained increasing attention for the cost-effective production of complex-shaped components [4, 5]. However, their surface hardness and wear resistance are often insufficient for demanding tribological applications, making additional surface engineering treatments necessary [6–9]. Physical vapor deposition (PVD) coatings and low-temperature plasma nitriding (LTPN) are commonly used to improve the tribological performance of stainless steels [10, 11]. LTPN enhances the load-bearing capacity of the substrate through nitrogen supersaturation and the formation of expanded martensite [12], while PVD coatings provide high

hardness and wear resistance [13]. Besides improving hardness, LTPN may significantly modify the surface morphology through lattice expansion [14], stress-induced deformation [15], and localized crack formation [16, 17]. Since PVD coatings generally replicate the substrate topography, such nitriding-induced surface features may directly influence the morphology and structural characteristics of the deposited coating [18]. Korsós and Kovács [19] conducted on MIM 17-4PH stainless steel, this characteristic nitriding-induced surface morphology and its formation were already observed and described. These findings also suggested that the altered surface topography may also affect the behavior and structure of subsequently deposited PVD coatings.

Das et al. [20] investigated the influence of surface pre-treatment on plasma nitrided and TiN PVD-coated AISI P20

tool steel. They observed that the PVD coating deposition reduced the roughness of the plasma-nitrided surface from Ra 0.40 to 0.15 μm by filling surface voids and cavities formed during the plasma nitriding step, while preserving the characteristic morphology created by the diffusion treatment. Naeem et al. [10] reported that the increase in surface roughness caused by plasma nitriding of AISI 304 steel enhanced the adhesion of subsequently deposited PVD coatings by increasing the effective contact area and promoting mechanical interlocking. They also noted that the roughened substrate altered the coating's preferred crystalline orientation, shifting from the dominant (311) and (400) peaks to (111), (200), and (220). Similarly, Liu et al. [21] demonstrated that microwave plasma etching could increase the surface roughness of AISI 304 from Ra 0.0015 to 0.0160 μm without altering its phase composition, thereby improving coating adhesion and wettability. Chen et al. [22] further highlighted the beneficial role of substrate roughness in additively manufactured Ti6Al4V alloy coated with MoS_2 PVD films, showing that rougher surfaces retained more solid lubricant and trapped wear debris more effectively, thereby reducing friction and improving tribological performance.

Although previous studies have highlighted the influence of substrate surface morphology and pretreatment on the performance of PVD coatings, the morphological relationship between nitriding-induced surface features and subsequently deposited coatings remains poorly understood, particularly for low-temperature plasma nitriding of powder metallurgy materials.

The present study, therefore, focuses on the morphology replication occurring between the LTPN-treated MIM 17-4PH substrate and the subsequently deposited PVD coating, and on how this replicated morphology is associated with the resulting structural and tribological characteristics. To distinguish the effects of the individual and combined treatments, the untreated, PVD-coated, and LTPN + PVD-treated conditions were compared.

Particular attention is devoted to changes in surface morphology and roughness; the replication of substrate features by the PVD coating; the resulting friction and wear behavior, evaluated by ball-on-disc tests; and differences in coating crystallographic orientation, as observed by X-ray diffraction. The primary contribution of this work is therefore to clarify how LTPN-induced surface morphology is transferred to the subsequent PVD coating

and how this morphology replication is associated with the resulting coating structure and tribological behavior, rather than solely demonstrating the well-established performance benefits of hybrid surface treatments.

2 Experimental methods

2.1 Material

Martensitic precipitation-hardenable stainless steel – 17-4PH (AISI 630) – was used for this research. The specimens were manufactured specifically for the investigation by AFT-Hungary Ltd. (Rétság, Hungary). The production of the samples followed the standard MIM procedure, using alloyed metallic powder and polymer binder. The material received no additional heat treatment after production. The chemical composition of the material is reported in Table 1, according to ASTM A564/A564M-13 standard [23].

The specimens were manufactured in a ring geometry with dimensions of $\varnothing 40 \times \varnothing 30 \times 8$ mm. After the sample production, the specimens were mechanically ground with SiC grinding papers ranging from P80 to P4000. Thereafter, the samples were fine-polished using 3 and 1 μm diamond suspensions to create a clean, uniform surface for the subsequent surface treatments. After the polishing steps, the samples were cleaned in an ultrasonic bath of ethanol.

2.2 Surface treatments and characterization

Three samples in total were prepared for this investigation. A sample, serving as the reference piece, underwent no subsequent surface treatment and was referred to as "R". A specimen received an arc-based TiAlN + TiCN bilayer PVD coating in 2 h at 450 $^{\circ}\text{C}$, using titanium and aluminum targets. The procedure used N_2 gas to form the TiAlN layer and a mixture of N_2 and CH_4 to form the TiCN layer. The sample bias voltage was kept between 80 and 120 V for the coating process. This fine-polished and PVD-coated sample is referred to as "RA". The third sample was low-temperature plasma nitrided for 2 h at 450 $^{\circ}\text{C}$ in a 1:3 N_2 – H_2 plasma, with the process pressure held between 10^{-2} and 10^{-3} mbar. After the LTPN treatment, the sample received the PVD coating as sample RA. The LTPN and PVD processes were carried out consecutively, maintaining a vacuum between steps. This hybrid surface-treated piece was referred to as "HA".

The surface treatment technologies were performed with the assistance of Voestalpine High Performance Metals

Table 1 The standard chemical composition of 17-4PH (m/m%)

Fe (%)	C (%)	Cr (%)	Ni (%)	Cu (%)	Mn (%)	Si (%)	Nb + Ta (%)
Rest	< 0.07	15.0–17.5	3.00–5.00	3.00–5.00	< 1.00	< 1.00	0.15–0.45

Hungary Ltd. (Dunaharaszti, Hungary), using an Eifeler Vacotec alpha400P device. The created samples and surface treatment parameters are summarized in Table 2.

After the deposition process, the samples were assessed for surface roughness using a Mitutoyo Surftest SJ-401 device, according to ISO 21920-2:2021 standard [24], 10 times per specimen. The R_k , R_{pk} , and R_{vk} values were automatically calculated by the device from the recorded Abbott-Firestone curves. The stylus tip was a 60° diamond cone with a 2 μm tip radius; the cut-off wavelength was 0.08 mm with a Gaussian filter; and the sampling length was 0.40 mm. The functional surface roughness parameters were used to characterize the load-bearing surface (R_k), the protruding peaks (R_{pk}), and the surface valley (R_{vk}) [25]. The adhesion properties of the PVD-coated specimens were assessed using the Rockwell indentation test, according to ISO 26443:2023 standard [26]. Each sample was tested 10 times, using an indentation load of 981 N (Rockwell D scale). The characteristic indentation marks were further evaluated based on the work of Fu et al. [27] by electron microscopy.

Afterward, the friction and wear properties were tested using an Anton Paar TRB³ pin-on-disc tribometer in ball-on-disc configuration in accordance with ASTM G99-17 standard [28]. The wear tests were performed twice per sample using a 6 mm-radius SiC ball bearing as the counterpart, given the material's excellent hardness, chemical and thermal stability, and high thermal conductivity. The mechanical and physical properties of the SiC counterpart were the following: 2800 HV10 hardness, 400 GPa elastic modulus, and 3.1 g/cm³ density. The ball-on-disc test parameters are reported in Table 3.

The tribometer's built-in software recorded the sliding distance and the coefficient of friction (COF). The maximum running-in COF values were defined as the maximum value before 30 m of sliding, while the steady-state COF is defined as the average value after 600 m of sliding. The wear scars were evaluated using the Mitutoyo device, performing profilometric measurements perpendicular to

Table 3 Wear test parameters

Parameter	Value
Normal force (N)	20
Sliding speed (m/s)	0.1
Sliding distance (m)	1000
Counterpart diameter (mm)	6
Temperature, nominal (°C)	25–28
Humidity (%RH)	30–40
Wear track diameter (mm)	36

the wear scar, 5 times per sample, evenly spaced along the circular wear track. The measurement length was 1.8 mm, with no filter or cut-off wavelength, using a 60° diamond cone stylus, with a 2 μm radius tip.

The surface phases of the samples were analyzed using a Bruker X-ray diffractometer equipped with a θ – 2θ vertical goniometer and Cu K α radiation ($\lambda = 1.54184$ Å), generated from a copper target at 40 kV and 30 mA. The scan parameters were set to 20°–120° in 2θ with 0.01° increments and an exposure time of 0.2 s per step. The surface hardness was tested with a Bühler Micromet 3 microhardness tester using a Vickers indenter at an indentation force of 50 g. Surface hardness was measured 10 times per sample. After the surface tests, cross-sectional cuts were prepared using a water-cooled abrasive-cutting method. The cross-sections were mounted in high-hardness hot-mounting resin, mechanically ground using SiC abrasive discs, polished with 3 and 1 μm diamond suspensions, and finally polished with 0.04 μm colloidal silica. The surface morphology, wear characteristics, wear-scar chemical analysis, adhesion test evaluation, cross-sectional layer thickness measurements, and microstructural characterization were carried out using an EVO MA 10 scanning electron microscope (Carl Zeiss AG, Oberkochen, Germany) equipped with an Octane Elect EDS detector (EDAX Inc., Mahwah, NJ, USA). The general beam parameters were 15 kV voltage and 5 nA probe current for all measurement types. The nitrided layer and bulk microstructures were characterized using electron channeling contrast imaging (ECCI) [29], thereby eliminating the need for chemical etching.

3 Results and discussion

3.1 Microstructural characterization

The microstructure of 17-4PH martensitic, precipitation-hardenable stainless steel exhibits a characteristic metal injection-molded microstructure, composed of uniformly distributed grains with residual porosity.

Well-developed necking regions between powder particles were evident, indicating effective grain bonding formed

Table 2 Surface treatments of the specimens

Name	Substrate	LTPN	PVD
R	MIM 17-4PH	–	–
RA	MIM 17-4PH	–	TiAlN + TiCN, 2 h, 450 °C, Ti–Al targets, N ₂ –CH ₄ gases, 80–120 V bias
HA	MIM 17-4PH	2 h, 450 °C, 1:3 N ₂ –H ₂ , 10 ^{–2} –10 ^{–3} mbar	TiAlN + TiCN, 2 h, 450 °C, Ti–Al targets, N ₂ –CH ₄ gases, 80–120 V bias

during sintering. Dispersed niobium-rich [19] precipitates were visible on the grain boundaries, contributing to the heterogeneous microstructural appearance of the alloy. Fig. 1 illustrates the microstructure of the 17-4PH material.

The cross-sections of Sample RA (Fig. 2 (a)) exhibited a uniform, fine-polished surface across the whole sample. In contrast, due to the LTPN technology, sample HA (Fig. 2 (b)) exhibited a relatively thin, highly inhomogeneous nitrided layer, with significant roughening of the previously polished substrate surface.

The thickness of the formed, plasma nitrided layer was $15.2 \pm 0.9 \mu\text{m}$. Fig. 3 illustrates the coated cross-sections of Samples RA and HA. Both samples showed a continuous PVD layer, with uniform thickness across the surface. The PVD coating thickness was $5.8 \pm 0.2 \mu\text{m}$ for sample RA and $4.6 \pm 0.1 \mu\text{m}$ for sample HA. Although the HA coating was approximately 21% thinner than the RA coating, the relatively uniform thickness within each sample suggests that this difference is more likely attributable to variations in the deposition process and/or the underlying

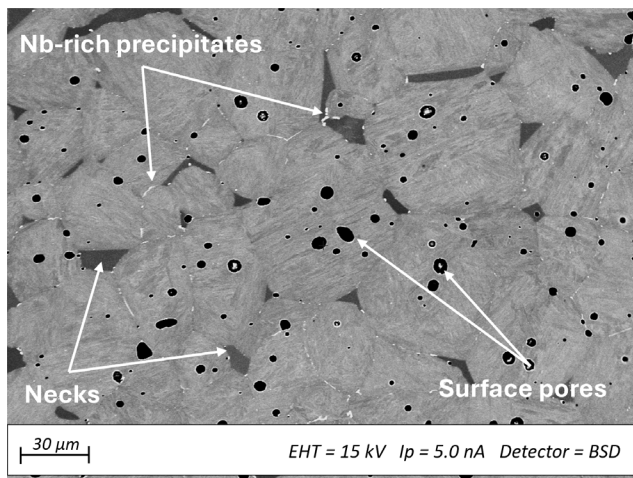


Fig. 1 The microstructure of the samples

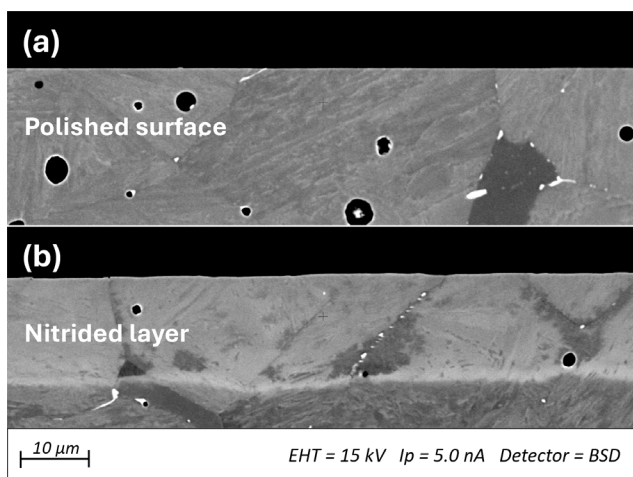


Fig. 2 The cross-sections of samples: (a) RA; (b) HA

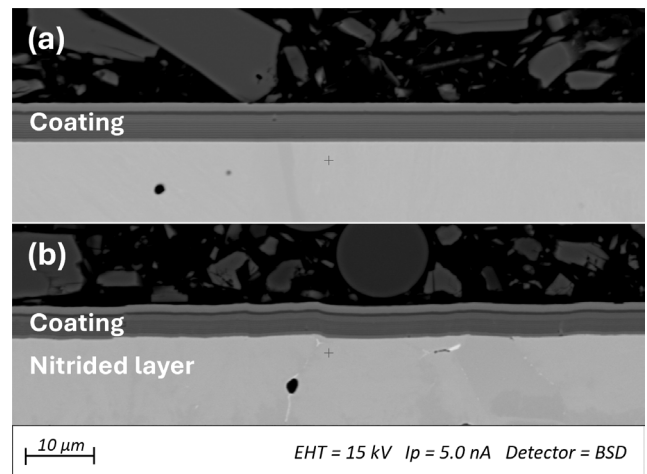


Fig. 3 The cross-section of the coatings deposited on: (a) RA; (b) HA

substrate morphology than to local coating irregularities. The potential influence of this thickness difference on the tribological performance is discussed below.

While the coating present on RA reflected the smooth, polished surface of the substrate, due to its excellent conformity, the HA sample coating replicated the LTPN-roughened surface morphology.

3.2 Surface characterization

Fig. 4 shows the surface morphologies of samples R, RA, and HA.

The surface micrograph of R (Fig. 4 (a)) showed a generally smooth surface, with significant surface porosity. Due to the high-quality sample preparation, orientation contrast is also present on this specimen, highlighting grains, grain boundaries, necks, and precipitates. In contrast, the RA (Fig. 4 (b)) sample showed droplets and pinholes, which were generally associated with arc-based PVD technology [30, 31]. In the HA sample, a characteristic

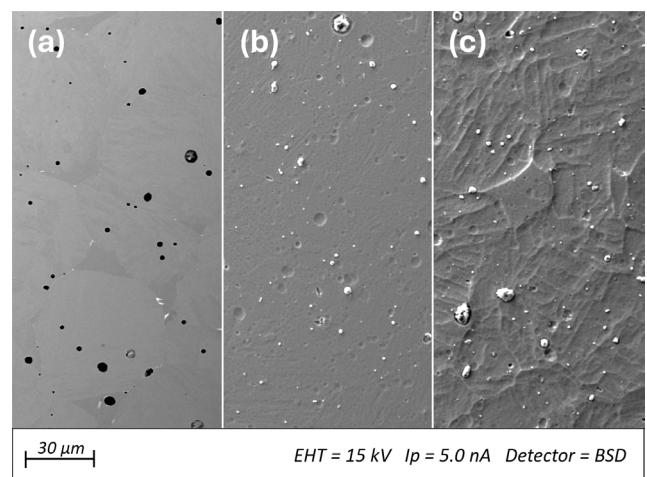


Fig. 4 The surface morphologies of samples: (a) R; (b) RA; (c) HA

surface morphology was observed, revealing changes in the underlying LTPN-treated microstructure. Similar to RA, HA also showed droplets and pinholes on its surface, and both coated sample surfaces exhibited dimples with sizes similar to those of the pores observable on the reference piece. Fig. 5 illustrates the raw surface roughness measurement data for all samples.

Similar to the findings illustrated on the morphology micrographs, the R specimen showed minimal to no surface faults with low R_k , R_{pk} , and R_{vk} values. The differences between the RA and HA conditions were evaluated by comparing the magnitudes of the mean differences relative to the corresponding standard deviations. The HA condition exhibited a less uniform surface morphology than RA, which can be attributed to the morphological modifications induced by the LTPN treatment and the subsequent replication of this morphology by the PVD coating. The R_k parameter increased from $0.213 \pm 0.054 \mu\text{m}$ for RA to $0.581 \pm 0.028 \mu\text{m}$ for HA, with the difference between the mean values substantially exceeding the experimental scatter. A similar trend was observed for R_{vk} , which increased from $0.147 \pm 0.031 \mu\text{m}$ to $0.212 \pm 0.006 \mu\text{m}$. In contrast, the R_{pk} values increased from $0.353 \pm 0.087 \mu\text{m}$ to $0.580 \pm 0.148 \mu\text{m}$, but the relatively large standard deviation of the HA condition indicates greater variability. This larger scatter may be related to the substantial population of droplets characteristic of the arc-PVD deposition process, which can contribute significantly to the surface peak structure represented by R_{pk} , in addition to the underlying morphology inherited from the LTPN-treated substrate. Thus, the increase in R_{pk} is attributed to the combined contributions of the LTPN-induced surface morphology and the PVD droplet population.

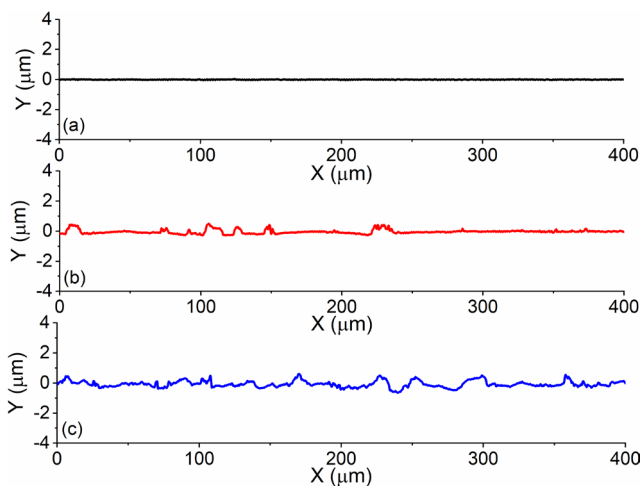


Fig. 5 The surface roughness data of: (a) R; (b) RA; (c) HA

The functional surface roughness parameters and the measured surface hardnesses are collected in Table 4.

The hardness increased from $3005 \pm 67 \text{HV}0.05$ for RA to $3227 \pm 282 \text{HV}0.05$ for HA. However, the comparatively large deviation in the HA condition indicates substantial variability, which may be attributed to the heterogeneous MIM microstructure and the resulting locally inhomogeneous nitriding response [32]. Consequently, although the mean hardness increased following the hybrid treatment, the relatively large scatter limits the strength of the interpretation based solely on the difference between the mean values. Fig. 6 illustrates the adhesion test marks of samples RA (Fig. 6 (a)) and HA (Fig. 6 (b)).

Based on the adhesion test marks, the hybrid-treated specimen exhibited a significantly lower density of radial cracks and no substantial coating spallation compared with the PVD-only specimen. This behavior indicates that the nitrided diffusion zone provides enhanced load-bearing capacity, reducing substrate deformation and the associated tensile stresses within the coating [27]. Furthermore, the absence of extensive delamination suggests improved resistance to interfacial failure, likely resulting from the combined effects of increased substrate support [33] and the enhanced mechanical interlocking provided by the nitrided surface morphology. Based on the adhesion tests, samples RA and HA can be classified as adhesion classes 2 and 1, respectively. Fig. 7 shows the identified LTPN-driven surface modification modes.

By further analyzing the LTPN layer of Sample HA, four specific surface phenomena can be identified, which cause characteristic changes in surface morphology. Fig. 6 (a) shows surface and subsurface crack formation, presumably caused by the relaxation of the high compressive stresses generated during the nitrogen uptake-induced surface grain expansions. In contrast, Fig. 6 (b) shows plastic deformation of the surface, which accommodated the generated compressive stresses *via* grain-boundary shear offset. This phenomenon can be observed as clusters on the surface plane. Fig. 6 (c) shows the phenomenon of surface plain shift by grain surface uplifting, creating smooth surface plateaus, caused by the expansion of neighboring grains. As described, the deposited PVD coating inherited the morphological features generated by grain boundary shear offsets and grain uplifts; however, no surface crack propagation was observed from the nitrided substrate to the coating. This finding indicated a characteristic change in surface topography due to plasma nitriding, which was inherited by the PVD coating and was governed by the

Table 4 Surface characterization results

Sample	R_k (μm)	R_{pk} (μm)	R_{vk} (μm)	Hardness (HV0.05)
R	0.039 ± 0.001	0.010 ± 0.000	0.010 ± 0.000	410 ± 14
RA	0.213 ± 0.054	0.353 ± 0.087	0.147 ± 0.031	3005 ± 67
HA	0.581 ± 0.028	0.580 ± 0.148	0.212 ± 0.006	3227 ± 282

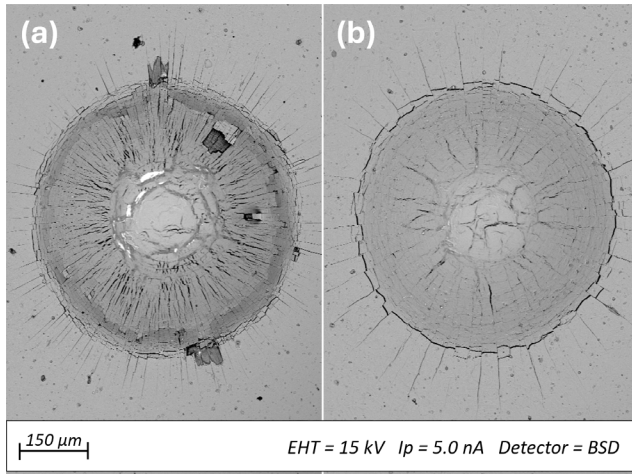


Fig. 6 The characteristic adhesion marks of samples: (a) RA; (b) HA

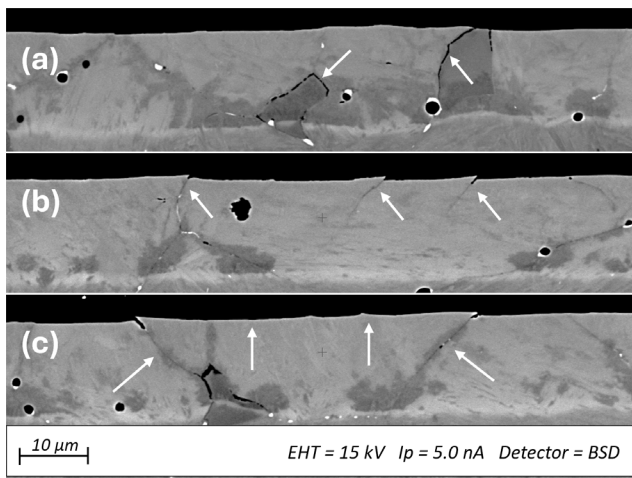


Fig. 7 The surface plane modifications formed by LTPN: (a) surface and subsurface cracks; (b) grain shear offset; (c) grain surface uplift

substrate material's inherent microstructure. It can be proposed that by the careful modification of the metal injection molding production parameters, namely the powder grain size, sintering time, temperature, and pressure, the post-hybrid treatment surface roughness properties can be optimized for enhanced tribological characteristics. The XRD spectra of all samples are illustrated in Fig. 8.

Sample R showed a well-defined peak at 44.02° (110), and four minor peaks at 64.35° (200), 81.68° (211), 98.28° (220), and 115.41° (310), indicating BCC martensitic (PDF card No. 00-006-0696) phase peaks shifted to lower degrees [34, 35]. The spectrum of sample RA showed the signal of the martensite (110), (211), (220), and (311)

peaks, while also introducing a strong peak at 35.78° (111), and two minor peaks at 76.48° (311) and 77.22° (222) indicating a NaCl-like, cubic metal nitride structure (PDF card No. 38-1420 and 65-7994) for both the TiAlN and the TiCN layers [34, 36, 37]. Sample HA exhibits XRD spectra similar to those of sample RA, with two significant differences: only the (110) martensite peak was detectable, presumably as the LTPN treatment broadened and weakened the substrate reflections [14], and an additional TiAlN/TiCN peak was observed at 61.32° (220), while the (222) peak exhibits a stronger signal than in sample RA. The differences in the relative intensities of the PVD coating reflections suggest a change in the coating's preferred crystallographic orientation in sample HA. The observed texture variation may be related to the modified surface morphology induced by LTPN, although other factors associated with the PVD deposition process and coating growth may also contribute to the resulting crystallographic orientation. A similar phenomenon was also suggested by Naeem et al. [10] in the case of cathodic cage plasma nitrided and TiN PVD-coated, wrought AISI 304 steel. Fig. 9 illustrates the different coating – surface pore interactions detectable on samples RA and HA.

Based on the observations, in cases of deeper surface porosity, the coating may bridge and completely cover the pores (Fig. 9 (a)). In contrast, in the case of shallow surface porosity or subsurface porosity with collapsed surface region, the coating may follow the surface plane geometry (Fig. 9 (b) and (c)). In the latter cases the pore – coating interaction creates the characteristic surface dimples, discussed on Fig. 4 (b) and (c). This finding indicated that, based on pore geometry, PVD coatings may reduce or eliminate surface porosity in metal injection-molded specimens [19] regardless of LTPN pretreatment.

3.3 Tribological characterization

Fig. 10 shows the wear distance–coefficient of friction diagrams for all tested specimens.

As shown in the wear distance – friction diagrams, all samples exhibited stable friction throughout the test. The application of surface treatments reduced the running-in and steady-state friction coefficients in both cases, despite an increase in the functional surface roughness parameters.

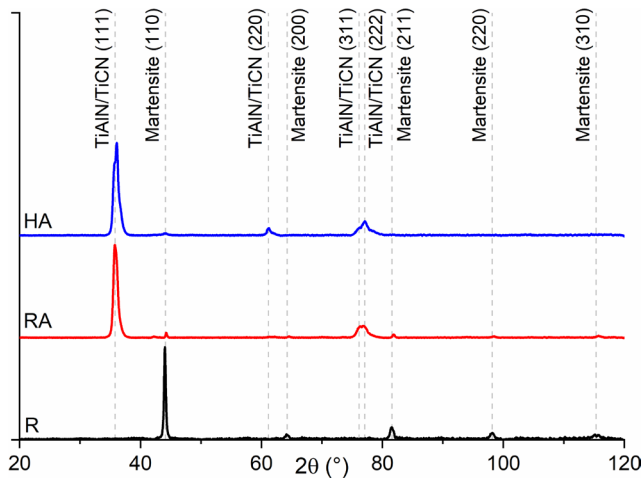


Fig. 8 The XRD spectra of samples R, RA, and HA

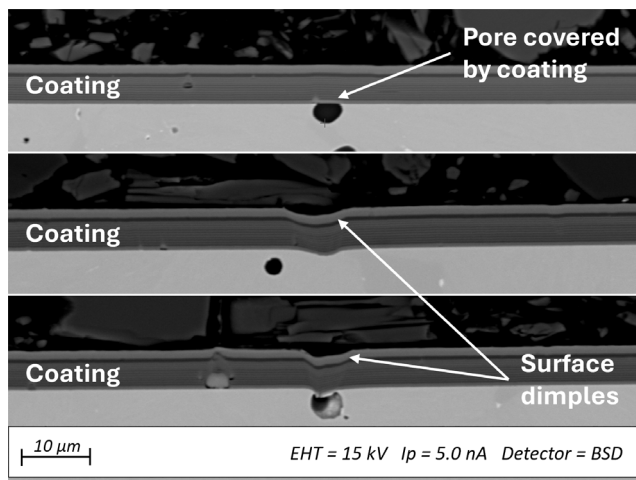


Fig. 9 The ability of the coatings to reduce surface porosity

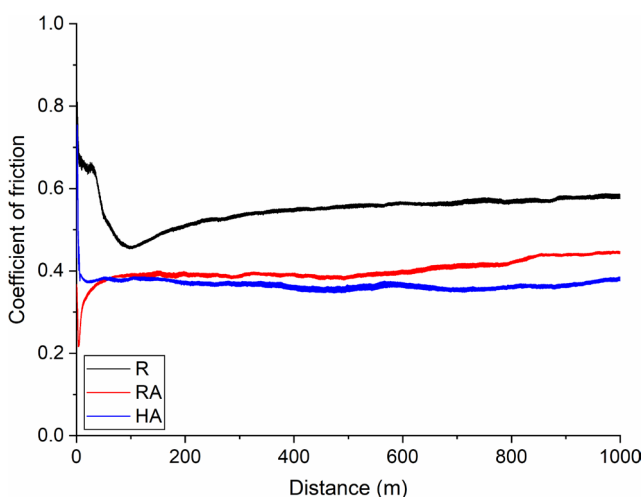


Fig. 10 Wear distance – friction coefficient diagrams of R, RA, and HA

Comparing the coated specimens, RA showed a lower running-in friction coefficient, presumably due to its lower R_{vk} surface roughness parameter [20]. However, the HA exhibited a lower stabilized steady-state friction, presumably

due to its higher R_k and R_{vk} properties. Fig. 11 illustrates the worn region and oxygen EDS map of R.

The wear scar of sample R showed signs of severe abrasive and oxidative wear, as evidenced by wear-directional grooves [38] in the worn region and oxygen-rich patches on the worn surface [27].

Fig. 12 shows the worn region and oxygen EDS map of sample RA.

In the case of sample RA, applying PVD coatings reduced surface oxide formation while maintaining the dominant wear mode as a combination of severe abrasive wear and minor oxidative wear.

Fig. 13 illustrates the wear scar and oxygen EDS map of sample HA.

The hybrid-treated sample's wear scar highlighted a different wear characteristic: minor abrasive wear and the wear-off of surface roughness peaks. It can also be proposed that the increased R_{vk} parameter enabled the entrapment of significantly more oxidized wear debris.

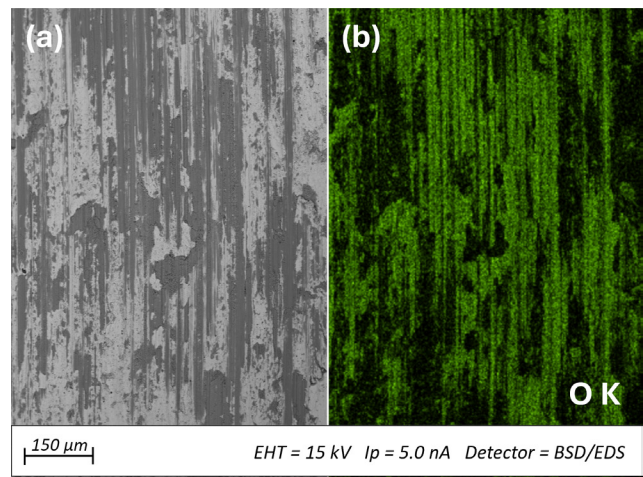


Fig. 11 The wear scar and oxygen EDS map of sample R

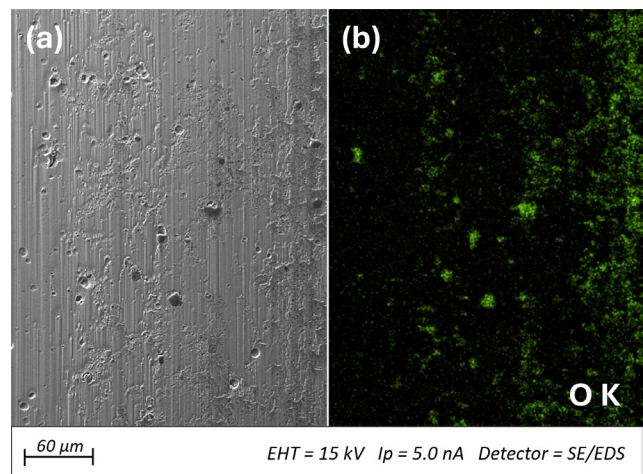


Fig. 12 The wear scar and oxygen EDS map of sample RA

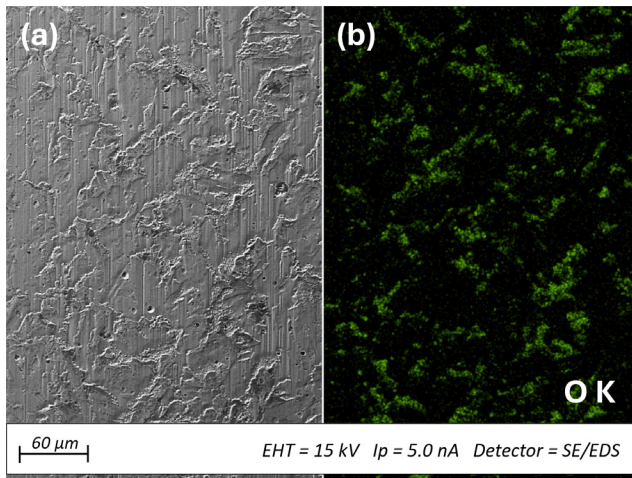


Fig. 13 The wear scar and oxygen EDS map of sample HA

Based on this finding, it can be suggested that the roughened surface of sample HA resulted in a higher friction coefficient during the early running-in stage of the wear test, due to mechanical interaction between the PVD and the SiC wear ball surface (R_{vk}) peaks. However, in the steady-state region, higher core (R_k) roughness reduced the real contact area. In contrast, higher valley (R_{vk}) roughness enabled the entrapment of wear debris, thereby limiting third-body wear [39]. It was suggested that the cleaner, smaller real contact area actually resulted in a lower steady-state friction coefficient for sample HA than for RA.

Similar to the wear scar evaluation, all samples showed deep grooves, indicating abrasive wear. In the case of RA, the worn region showed a minimal wear scar compared to the reference piece, and no signs of surface droplets or pinholes were observed. The worn region of HA showed a more uniform surface, with the surface valleys filled with oxidized wear debris [40], further strengthening the findings of the wear scar analysis. It can also be proposed that the higher core roughness (R_k) and better coating support enabled better wear resistance in the HA specimen.

Fig. 14 illustrates the characteristic wear profiles of all samples.

Table 5 collects the running-in and steady-state coefficient of friction values and the wear scar volume results of all samples.

Based on the experimental data, the application of PVD coating in both hybrid-treated and PVD-only states significantly reduced the running-in and steady-state coefficient of friction as well as the calculated worn volume. Comparing the coated pieces, the running-in coefficient of friction increased from 0.487 ± 0.031 for RA to 0.799 ± 0.002 for HA, while the steady-state coefficient of friction decreased from 0.424 ± 0.041 to 0.363 ± 0.015 . In both cases, the

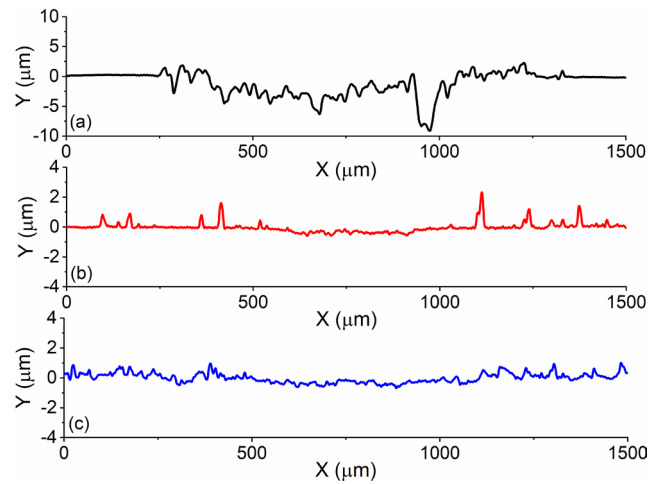


Fig. 14 The characteristic wear scars of: (a) R; (b) RA; (c) HA

Table 5 Friction and wear test results of all samples

Sample	Running-in COF (–)	Steady-state COF (–)	Worn volume (mm ³)
R	0.930 ± 0.005	0.571 ± 0.006	0.820 ± 0.345
RA	0.487 ± 0.031	0.424 ± 0.041	0.058 ± 0.007
HA	0.799 ± 0.002	0.363 ± 0.015	0.031 ± 0.007

differences between the mean values are considerably larger than the corresponding experimental scatter. Most notably, the worn volume decreased from 0.058 ± 0.007 mm³ for RA to 0.031 ± 0.007 mm³ for HA, corresponding to an approximately 47% reduction. The magnitude of this reduction is substantially larger than the experimental deviation, indicating a pronounced improvement in wear resistance following the hybrid treatment.

Although the lower coating thickness of HA could contribute to differences in tribological behavior, the improved wear resistance and lower steady-state coefficient of friction observed for HA, despite its thinner coating, indicate that coating thickness alone cannot account for the observed tribological improvement.

4 Conclusions

In this study, metal injection-molded 17-4PH, a martensitic precipitation-hardenable stainless steel, was subjected to untreated, TiAlN + TiCN PVD-coated, and hybrid LTPN + PVD surface treatments. The comparative approach was used not only to evaluate the individual and combined effects of the treatments on surface roughness and morphology, phase composition, hardness, friction, and wear resistance, but also to investigate how the surface morphology produced by LTPN is replicated by the subsequently deposited PVD coating. Particular emphasis was placed on relating the changes in substrate morphology to the resulting coating morphology, crystallographic

characteristics, and tribological behavior. The following conclusions can be drawn:

- Both the PVD coating and the hybrid surface treatment increased the overall surface roughness of the polished MIM 17-4PH specimens. Compared with RA, sample HA exhibited higher R_k (0.213 to 0.581 μm), R_{pk} (0.353 to 0.580 μm), and R_{vk} (0.147 to 0.212 μm) values. The observed changes are attributed to the combined effects of the surface morphology modifications induced by LTPN, including crack formation, grain-boundary shear offset, and grain surface uplift, and the subsequent replication of these features by the PVD coating. The R_{pk} increase may additionally be influenced by the substantial droplet population characteristic of the arc-PVD process.
- The mean coating hardness increased from 3005 to 3227 HV0.05 following LTPN treatment, indicating an enhanced supporting effect of the hardened nitrided substrate. However, the HA condition exhibited substantially higher hardness scatter, which may be associated with the heterogeneous MIM microstructure and the resulting locally varying nitriding response. The hardened substrate also provided improved mechanical support for the coating, contributing to the enhanced resistance to interfacial failure observed in the Rockwell adhesion tests.
- Differences in the relative intensities of the TiAlN/TiCN coating reflections were observed between RA and HA, including the appearance of the (220) reflection and an increased intensity of the (222) reflection in HA. These changes indicate a difference in the preferred crystallographic orientation of the deposited coating. The modified substrate morphology may contribute to this variation by influencing coating nucleation and growth; however, other deposition-related factors may also affect the resulting coating texture.
- Both surface treatment approaches reduced the running-in and steady-state friction compared with the untreated reference. Relative to RA, HA exhibited a higher running-in coefficient of friction (0.487 to 0.799) but a lower steady-state coefficient of friction (0.424 to 0.363). The higher running-in friction is considered to be associated with the increased population of surface peaks, reflected by the higher R_{pk} value, which may promote greater mechanical interaction with the counterpart during the initial stage of sliding. The subsequent decrease in steady-state friction may be related to the evolution of the surface

during wear, including the reduction of prominent surface peaks and the resulting contact conditions. The higher R_{vk} value may additionally contribute to wear-debris retention. These interpretations are supported by the observed morphology of the wear scars and profilometry results, but the individual contributions of these mechanisms cannot be isolated from the present experiments.

- The HA sample exhibited improved wear resistance, with the wear-induced volume loss decreasing from 0.058 to 0.031 mm^3 , corresponding to an approximately 47% reduction. This improvement occurred despite the HA coating being thinner than the RA coating ($4.6 \pm 0.1 \mu\text{m}$ compared with $5.8 \pm 0.2 \mu\text{m}$), indicating that the difference in coating thickness alone is unlikely to account for the improved wear performance. The enhanced wear resistance is instead attributed to the combined effect of the LTPN-hardened substrate and the modified surface morphology, which provides increased load-bearing support for the PVD coating.
- The results demonstrate that the primary significance of the hybrid treatment lies not solely in improved hardness, friction, or wear resistance, but in the interaction between the LTPN-modified substrate morphology and the subsequently deposited PVD coating. The LTPN-induced morphological features were replicated by the PVD coating and were associated with changes in surface roughness, coating texture, and tribological behavior. Since these morphological changes are governed by the underlying substrate microstructure, variations in surface grain morphology, size, and orientation may likewise influence the morphology and performance of hybrid-treated MIM materials.

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