

Article

Post-Wear Corrosion Evolution of Nanocrystalline Ni Coatings with Sliding-Induced Crystallographic Texture

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Abstract

Nanocrystalline Ni coatings have attracted attention as protective materials for tribological applications in chloride-containing environments, but their corrosion response after prior sliding damage remains unclear. Here, electrodeposited nanocrystalline Ni coatings were subjected to circular sliding wear at 0.30 and 0.03 m/s. The specimens were selectively sealed to define an exposure window in which the 0.30 m/s wear track, the 0.03 m/s wear track, and the as-deposited surface were simultaneously exposed to a 3.5 wt.% NaCl solution for up to 120 h. Corrosion evolution was followed quasi-in situ at the same locations after successive immersion intervals using SEM and surface topography analysis. The 0.30 m/s track showed faster degradation and more severe localized corrosion, whereas the 0.03 m/s track remained groove-dominated with weaker pitting. TEM and four-dimensional scanning transmission electron microscopy (4D-STEM) revealed grain growth in both wear tracks. Sliding at 0.30 m/s induced a $\langle 1\ 1\ 2 \rangle$ orientation along the normal direction, while the 0.03 m/s track largely retained the $\langle 1\ 1\ 1 \rangle$ and $\langle 2\ 0\ 0 \rangle$ texture components along the normal direction, similar to those of the as-deposited coating. The enhanced localized corrosion of the 0.30 m/s track is interpreted as the coupled consequence of wear-induced surface damage, debris-layer instability, grain coarsening, and crystallographic texture redistribution.

Keywords: nanocrystalline; sliding wear; post-wear corrosion; texture; 4D-STEM



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1. Introduction

Metallic coatings used in marine and other chloride-containing environments often experience mechanical contact before corrosion exposure. Sliding, vibration, or particle abrasion can leave grooves, compacted debris, locally ruptured surface films, and plastically deformed near-surface regions [1,2]. When such worn surfaces are subsequently exposed to NaCl solution, corrosion does not proceed on the original coating surface but on a mechanically preconditioned surface with strong morphological and structural heterogeneity [3,4]. For this reason, post-wear immersion corrosion is an important service condition for protective coatings, especially when localized attack rather than uniform dissolution controls durability.

Nickel-based coatings are widely used because of their processability, adhesion to engineering substrates, and generally favorable corrosion resistance. Nanocrystalline Ni is of particular interest because grain refinement can improve hardness and wear resistance [5], while the high density of grain boundaries can influence oxide formation, passivation, and corrosion morphology [6–10]. Previous studies have shown that the corrosion behavior of nanocrystalline Ni and Ni-based coatings is sensitive to grain size and deposition conditions [11–14], as well as to alloying and texture [15–17]. However, a coating that performs well in the as-deposited state may respond differently after sliding because wear can modify both the surface geometry and the near-surface microstructure before the coating encounters chloride solution.

Most existing studies on nanocrystalline Ni coatings emphasize either wear behavior, corrosion behavior, or simultaneous tribocorrosion. Wear-focused studies commonly discuss friction coefficient, wear track geometry, debris formation, and subsurface deformation, whereas corrosion-focused studies often examine the response of unworn surfaces in aggressive media [18,19]. Tribocorrosion studies on Ni and Ni-based coatings further demonstrate that mechanical damage and corrosion can interact strongly during contact in chloride-containing media [20–26]. Additional studies on nanocrystalline Ni-Co, electroless Ni-P, and electrodeposited Ni-B coatings show that grain refinement, coating architecture, subsurface deformation, and tribological layers can change corrosion and tribocorrosion performance [24,26]. However, comparatively few studies follow the subsequent immersion corrosion of exactly the same pre-worn regions and then connect the time-dependent corrosion morphology with the nanoscale as-worn crystallographic state. This distinction is important because the post-wear surface contains grooves, adherent debris, local defects, and deformation-induced microstructural gradients before chloride exposure begins. Similar mechanically modified or gradient nanostructured surfaces show that the affected layer can influence pit development and passive-film stability [27–29]. Therefore, linking worn-surface morphology, near-surface microstructure, and subsequent immersion-corrosion morphology is necessary for understanding the durability of nanocrystalline Ni coatings after frictional service.

However, in a post-wear immersion sequence, the important question is not only how much material is removed during sliding but also how the same worn region subsequently evolves during chloride exposure. A quasi-in situ strategy is therefore useful because it allows the same exposed area to be examined after successive immersion intervals, reducing ambiguity caused by spatial variability among nominally identical wear tracks. Without same-region tracking, debris detachment, corrosion-product redistribution, and pit formation could be confused with differences among separate wear tracks or separately electrodeposited specimens. The need for spatially resolved or same-area characterization is consistent with localized corrosion studies showing that macroscopic electrochemical methods can obscure small active sites and heterogeneous local reactions [30,31]. This approach is relevant to worn or tribocorrosion-affected surfaces where debris loosening, corrosion-product accumulation, and pit development can occur concurrently [32–34].

To link the corrosion behavior of worn nanocrystalline materials with their friction-induced state, the as-worn microstructure should be characterized directly. Generally, plastic deformation can increase the density of dislocations, grain boundaries, and stored strain energy, which may promote local dissolution and passive-film instability. At the same time, severe deformation may generate preferred crystallographic orientations that alter surface reactivity and passivation behavior. For nanocrystalline Ni-W alloys, sliding and abrasive wear have been shown to drive grain growth, grain-boundary relaxation, and near-surface hardening rather than simply removing material [35,36]. Panzarino and Rupert further identified a gradient nanograined subsurface structure during wear, demonstrating that wear

rate and both surface and subsurface microstructures can evolve concurrently, although that study did not provide crystallographic orientation information [37]. Deformation-induced texture and orientation-dependent surface reactivity may influence passivation and localized corrosion [38]. Recent developments in four-dimensional scanning transmission electron microscopy (4D-STEM) enable nanoscale crystallographic information to be extracted from scanning nanodiffraction datasets [39]. In particular, py4DSTEM-based data analysis and sparse-correlation orientation mapping [40,41] provide a practical route to connect the as-worn subsurface structure with subsequent immersion-corrosion morphology.

For tribologically modified surfaces, both applied load and sliding speed can strongly influence the resulting wear morphology and subsurface microstructure. The applied load mainly determines the contact stress and the overall severity of plastic deformation, whereas sliding speed directly affects the local shear rate, strain-rate conditions, frictional heating, and oxidation behavior at the sliding interface [42–45]. Therefore, under a fixed normal load, varying the sliding speed provides an effective approach to generate different deformation conditions in the near-surface layer before corrosion testing [46]. This consideration is particularly important for nanocrystalline metals. Due to their ultrafine grain size, nanocrystalline materials may deform through mechanisms distinct from those in coarse-grained counterparts, including grain-boundary-mediated processes, partial-dislocation activity, and deformation twinning. These deformation mechanisms are sensitive to both grain size and strain rate [47,48]. Therefore, in the present study, sliding speed was selected as the primary variable while maintaining a constant applied load, with the aim of producing different wear-induced surface microstructures and elucidating how the microstructural evolution induced by wear influences the subsequent corrosion behavior of nanocrystalline Ni coatings.

In this work, electrodeposited nanocrystalline Ni coatings were subjected to circular sliding at 0.30 and 0.03 m/s and then exposed to a 3.5 wt.% NaCl solution using a selectively sealed quasi-in situ immersion configuration. The exposed window contained the two wear-track regions and the as-deposited surface, enabling the same specimen to be followed during immersion. SEM, three-dimensional surface topography, TEM, 4D-STEM orientation mapping, and ODF analysis were combined to connect the time-dependent corrosion morphology with the as-worn subsurface microstructure and sliding-induced texture. The objective was to compare the localized post-wear corrosion responses generated under the two sliding speed conditions and to relate these responses to debris-covered surface morphology, surface-height redistribution, grain growth, and crystallographic texture redistribution.

2. Materials and Methods

2.1. Preparation of Nanocrystalline Nickel Coatings

Nanocrystalline nickel coatings were prepared by direct-current electrodeposition on pure copper substrates. A high-purity electrolytic nickel plate with a purity of 99.997% was used as the anode. The anode dimensions were 5.0 cm × 5.0 cm × 2.0 cm. Before electrodeposition, the nickel anode was mechanically ground using 400, 800, and 1000 grit SiC papers to remove surface oxides. It was then ultrasonically cleaned successively in acetone and deionized water, with each cleaning step repeated three times. The cleaned nickel plate was placed in an anode bag before use. Pure copper plates with a purity of 99.97% and dimensions of 10 cm × 6 cm × 0.3 cm were used as cathode substrates. The copper substrates were wet-ground sequentially with 400, 1000, 2000, and 5000 grit SiC papers until no visible scratches remained. To further improve the surface finish, the

substrates were electropolished at 7 V for approximately 30 s in an electrolyte composed of phosphoric acid, ethanol, and distilled water at a volume ratio of 1:1:2.

The nickel plating bath contained approximately 280 g/L NiSO₄, 15 g/L NiCl₂, and 35 g/L H₃BO₃ (Chongqing Chuandong Chemical (Group) Co., Ltd., Chongqing, China). Saccharin, sodium dodecyl sulfate, and 1,4-butyne diol (Shanghai Aladdin Biochemical Technology Co., Ltd., Shanghai, China) were added at concentrations of approximately 1.0, 0.2, and 0.7 g/L, respectively, to promote grain refinement and improve coating quality. Electrodeposition was then carried out in a 1 L glass beaker placed in a thermostatic water bath. Before deposition, the bath pH was adjusted to approximately 5 using H₂SO₄ and NaOH solutions. The cleaned nickel plate was placed in an anode bag to reduce the transfer of anode sludge or loose particles into the electrolyte before use. The distance between the facing anode and cathode surfaces was kept at approximately 8 cm, the bath temperature was maintained at 50 °C, and a magnetic stirrer (Gongyi Yuhua Instrument Co., Ltd., Gongyi, China) was operated at approximately 400 rpm to maintain homogeneous mass transport. A direct-current power supply (Shenzhen LODESTAR Precision Tools Co., Ltd., Shenzhen, China) was operated under galvanostatic conditions at a current density of 2.5 A/dm² for approximately 4 h. The resulting nickel coatings had a thickness of approximately 70 µm.

2.2. Sliding Wear and Post-Wear Corrosion Tests

Sliding wear tests were performed using an Rtec Instruments MFT-5000 tribometer (Rtec Instruments, San Jose, CA, USA). The counter body was an E52100 alloy steel ball, corresponding to GCr15 bearing steel, with a hardness of 63 HRC and a diameter of 9.524 mm. The tests were conducted under a normal load of 5 N in a circular sliding configuration, with a total sliding distance of 300 m. Sliding at 0.30 m/s was performed at a radius of 10 mm, whereas sliding at 0.03 m/s was performed at a radius of 5 mm. The normal load, counter body, ambient environment, and total sliding distance were kept constant, while two sliding speeds were selected to create high-speed and low-speed as-worn conditions for post-wear corrosion comparison. Sliding speed was used as a comparison variable since previous studies on nanocrystalline Ni and related Ni alloys have also used selected discrete speeds or speed-contact conditions to reveal differences in friction, wear, and near-surface structural evolution [36,49,50]. The two speeds of 0.30 and 0.03 m/s were therefore used as contrasting high- and low-speed conditions rather than as a complete velocity series. This choice was also made because the quasi-in situ immersion experiments, SEM-based statistical analysis, TEM characterization, and 4D-STEM measurements require substantial experimental time and sample preparation, making a systematic velocity series beyond the scope of the present study. The total sliding distance was kept constant to ensure that the two wear tracks experienced the same cumulative sliding-path exposure before immersion. This choice provides a consistent basis for comparing post-wear corrosion because cumulative sliding distance is directly related to the extent of repeated interfacial contact, material removal, debris generation, and subsurface deformation developed during wear. If the number of cycles had instead been kept constant, the different track radii would have produced different total sliding distances according to $L = 2\pi rN$. In that case, the 10 mm-radius track would have experienced twice the sliding distance of the 5 mm-radius track at the same cycle number, resulting in greater cumulative wear exposure. The resulting differences in material removal, frictional work, debris accumulation, and near-surface deformation would then have been introduced by track geometry rather than by the intended sliding condition, making the subsequent corrosion comparison less well controlled. Therefore, maintaining a constant total sliding distance was used to avoid an additional twofold difference in sliding exposure caused by the different track radii. Under

this condition, the corresponding cycle numbers were approximately 4770 revolutions for the 0.30 m/s track and 9550 revolutions for the 0.03 m/s track. We acknowledge that the effects of track radius and cycle number cannot be completely separated from sliding speed in this configuration; therefore, the two tracks are treated as two defined as-worn conditions generated under the same cumulative sliding distance, rather than as conditions differing solely in sliding speed.

After sliding wear, the specimens were selectively sealed so that a defined exposure window containing the 0.30 m/s wear track, the 0.03 m/s wear track, and the as-deposited coating surface was exposed to the electrolyte, as shown in Figure 1. To ensure the validity of the selective sealing strategy, the non-working regions of the sample were encapsulated with an electrolyte-resistant insulating coating, leaving only the predefined testing area exposed to the electrolyte. Before sealing, the sample surface was cleaned and thoroughly dried. Subsequently, the photoresist was uniformly applied to the non-testing regions of the sample, while a testing window was reserved at the center of the sample. After the application of the photoresist coating, the insulating layer was fully dried and cured to improve its adhesion and barrier performance. Through this process, only the central testing window was exposed to the electrolyte, while the remaining regions were covered by the coating. In addition, the exposed window was designed away from the physical edges of the sample, and the regions close to the boundary were not used for interpreting the corrosion morphology of the wear tracks. Post-wear corrosion tests were carried out in 3.5 wt.% NaCl aqueous solution. The same specimen was sequentially immersed and repeatedly removed after 6, 12, 18, 24, 36, 48, 72, and 120 h, immediately rinsed with deionized water to remove residual electrolyte, air-dried at room temperature, and examined by SEM before being returned to the solution for the next interval. This procedure provided a quasi-in situ observation sequence for tracking the time-dependent morphological evolution of the same worn regions and avoided comparing different specimens or different wear tracks after separate immersion periods. To keep the main figures concise, representative states, namely the as-worn surface and the surfaces after 12, 48, and 120 h of immersion, are shown in the Section 3. Fresh samples prepared under the same sliding conditions were used for cross-sectional TEM and 4D-STEM characterization of the as-worn microstructure.

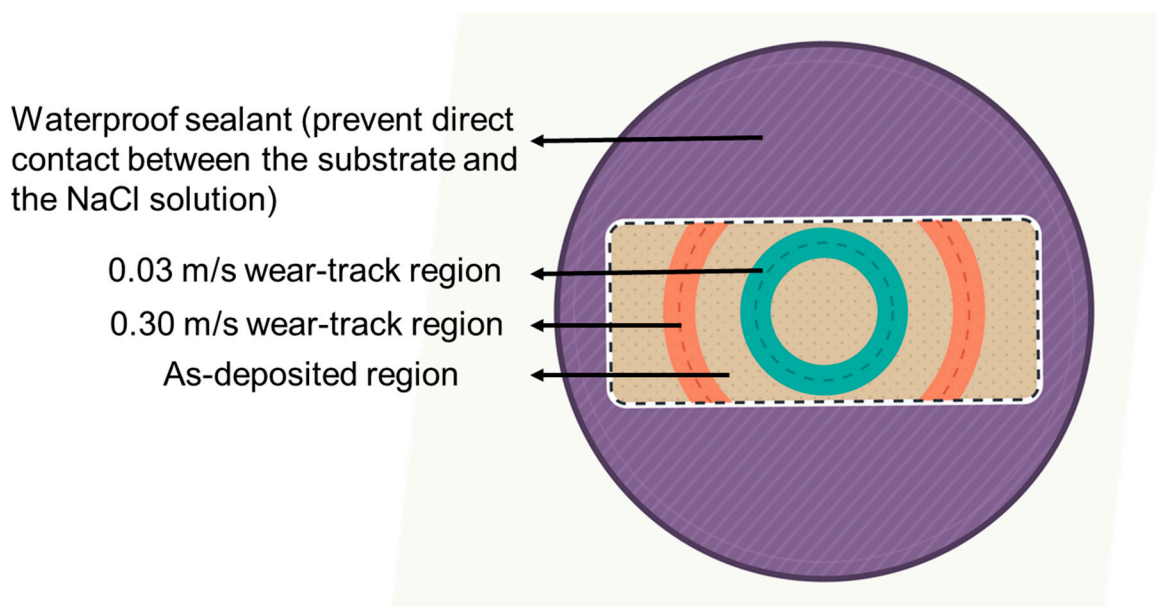


Figure 1. Schematic illustration of the selectively sealed specimen with a defined exposure window for quasi-in situ immersion corrosion testing.

2.3. Microstructure Characterization

SEM observations were conducted in secondary-electron imaging mode at an accelerating voltage of 5 kV using a Thermo Fisher Helios 5 CX focused ion beam field-emission scanning electron microscope (Thermo Fisher Scientific, Waltham, MA, USA). Three-dimensional surface morphology of the wear tracks was measured using a Zeiss LSM 900 laser scanning confocal microscope (Carl Zeiss Microscopy GmbH, Jena, Germany). The confocal measurements were performed using a 405 nm wavelength laser, with a spatial pinhole diameter of 230 μm and a Z-direction slicing step of 0.1 μm . For quantitative image analysis, corrosion micro-pits or holes in the high-magnification SEM images were identified from manually checked masks. Pit density was calculated as the number of identified features divided by the analyzed area, and the equivalent circular diameter was calculated from the segmented area. Areal roughness S_a and S_q values were extracted from the confocal topography datasets for the corresponding wear-track regions at each immersion time.

Transmission electron microscopy specimens were prepared using the same Zeiss Auriga FIB-SEM system (Carl Zeiss Microscopy GmbH, Oberkochen, Germany) by a conventional lift-out procedure. The lamellae were extracted with the observation plane parallel to the sliding direction–normal direction plane of the specimen. For 4D-STEM analysis, the lamellae were taken from the as-worn wear-track regions before NaCl immersion, so the orientation maps represent the friction-induced surface and subsurface state rather than the corrosion-modified state. Bright-field imaging, dark-field imaging, and selected-area electron diffraction were performed using a JEOL JEM-2100 transmission electron microscope (JEOL Ltd., Akishima, Tokyo, Japan).

Quantitative crystallographic orientation mapping was performed by four-dimensional scanning transmission electron microscopy using a Thermo Fisher Spectra 200 transmission electron microscope (Thermo Fisher Scientific, Waltham, MA, USA) equipped with a probe spherical-aberration corrector and an EMPAD direct electron detector (Thermo Fisher Scientific, Waltham, MA, USA). Four-dimensional STEM is well suited for nanoscale orientation mapping because local diffraction information can be linked to the scan position [39]. The accelerating voltage was 200 kV. The EMPAD detector had a pixel array of 128×128 , and the exposure time for each diffraction pattern was set to 5 ms. The electron probe size was approximately 1.5 nm, and the convergence semi-angle was 12 mrad. For each specimen, a 512×200 -pixel scan region was acquired with a scan step of approximately 2.1 nm. The 4D-STEM diffraction datasets were processed using the open-source py4DSTEM (<https://github.com/py4dstem/py4DSTEM>, accessed on 11 May 2026) package to determine crystallographic orientations [40,41].

3. Results

3.1. Quasi-In Situ SEM Characterization of Wear Tracks

The quasi-in situ SEM observations reveal that the two wear tracks evolved differently during immersion in a 3.5 wt.% NaCl solution. For the wear track produced at 0.30 m/s (Figure 2), the as-worn surface is characterized by a continuous damaged band, grooves parallel to the sliding direction, and numerous granular adherents. These adherents are consistent with compacted wear debris, transferred material, and locally oxidized fragments formed during sliding. After 12 h of immersion, the edges of these adherent features become less sharp, and the local contrast around debris-covered regions becomes more heterogeneous, indicating early corrosion modification of the mechanically damaged surface. After 48 h, interfacial gaps and locally loosened debris are more evident, suggesting progressive corrosion consumption and partial detachment of the adherent layer. By 120 h, the wear track contains fewer compacted adherents and is increasingly dominated by

grooves, exposed depressions, and discontinuous corrosion-product-covered areas. This sequence indicates that the 0.30 m/s track underwent a rapid transition from a debris-covered wear surface to a corrosion-modified heterogeneous surface.

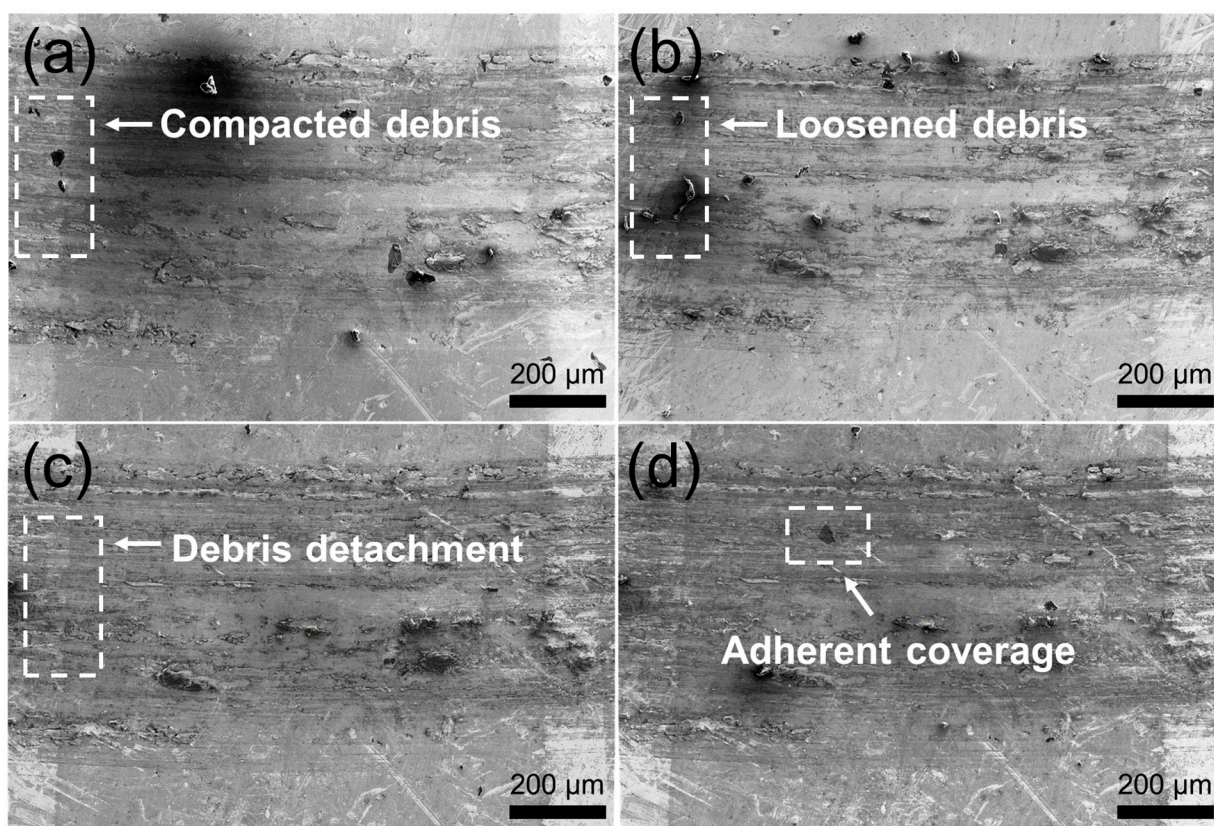


Figure 2. Quasi-in situ SEM observations (secondary electron images) of the 0.30 m/s wear-track region after immersion in a 3.5 wt.% NaCl solution for different durations: (a) as-worn state; (b) 12 h; (c) 48 h; and (d) 120 h.

The wear track produced at 0.03 m/s shows a more stable morphology over the same immersion sequence (Figure 3). In the as-worn condition, it also contains sliding-direction grooves and local granular adherents, but the damaged band is more continuous and less disrupted than that produced at 0.30 m/s. After 12 h of immersion, the groove morphology remains clearly visible, and the amount of adherent debris changes only slightly. At 48 h, some adherent particles appear loosened or partly removed, but the overall banded wear-track morphology is still retained. Even after 120 h of immersion, the surface remains mainly groove-dominated with limited residual adherents and without the same degree of debris degradation and surface discontinuity observed on the 0.30 m/s track. The slower morphological evolution suggests that sliding at 0.03 m/s produced a less defective and less corrosion-sensitive surface state.

Additional high-magnification SEM observations further reveal the different local corrosion responses of the two wear tracks (Figures 4 and 5). For the 0.30 m/s wear track (Figure 4), the surface exhibits a relatively unstable corrosion morphology. Small dark pit-like features appear after 12 h of immersion, indicating pit initiation within mechanically disturbed surface regions. With increasing immersion time, these features become more numerous and locally clustered, showing pit growth after 48 h and further pit enlargement after 120 h. The progressive development from isolated pit initiation to clustered pit growth and enlargement suggests that the high-speed wear track contains more persistent local corrosion-active sites. In contrast, the 0.03 m/s wear track remains mainly dominated by

continuous sliding grooves throughout the immersion sequence (Figure 5). In the as-worn state and after immersion, only scattered adherent particles or corrosion products are observed on the worn surface, and no dense population of dark pit-like features develops. Even after prolonged immersion, the groove morphology is largely retained, indicating that the low-speed wear track preserves a relatively continuous worn surface and undergoes only limited localized corrosion.

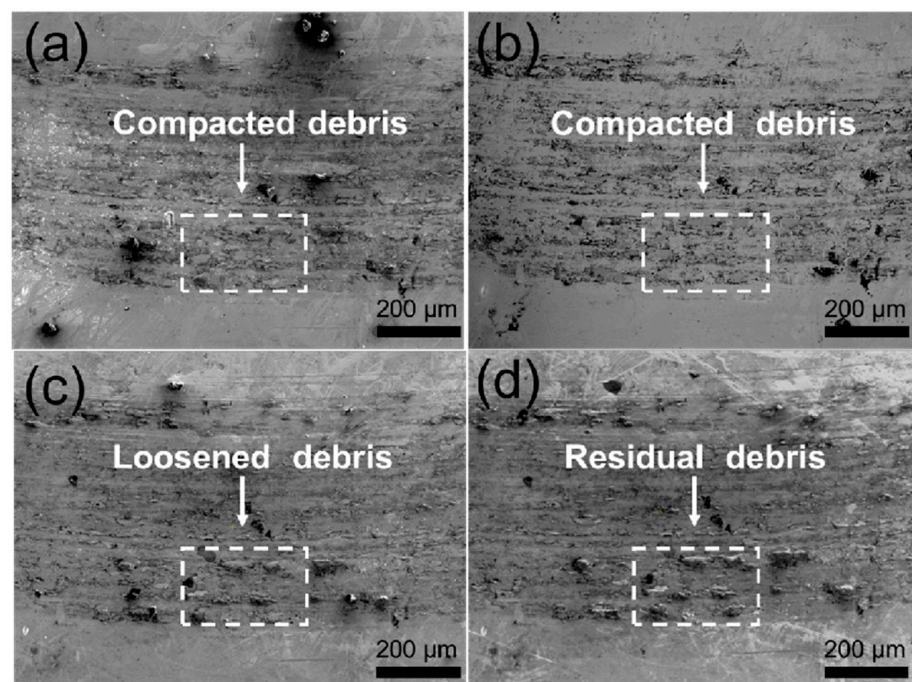


Figure 3. Quasi-in situ SEM observations (secondary electron images) of the 0.03 m/s wear-track region after immersion in a 3.5 wt.% NaCl solution for different durations: (a) as-worn state; (b) 12 h; (c) 48 h; and (d) 120 h.

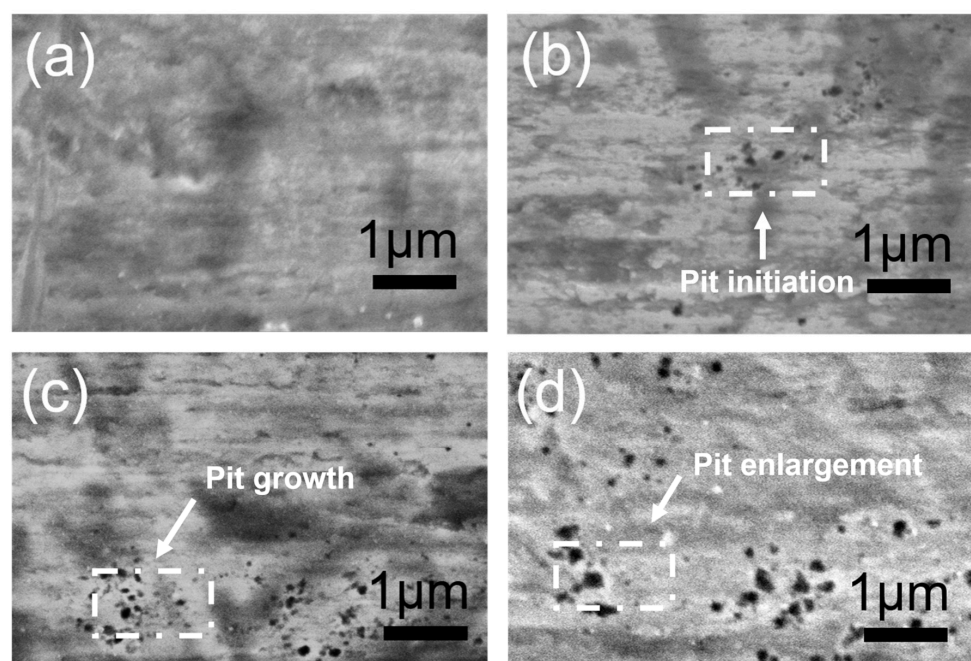


Figure 4. High-magnification SEM observations (secondary electron images) of the 0.30 m/s wear-track region after immersion in a 3.5 wt.% NaCl solution for different durations: (a) as-worn state; (b) 12 h; (c) 48 h; and (d) 120 h.

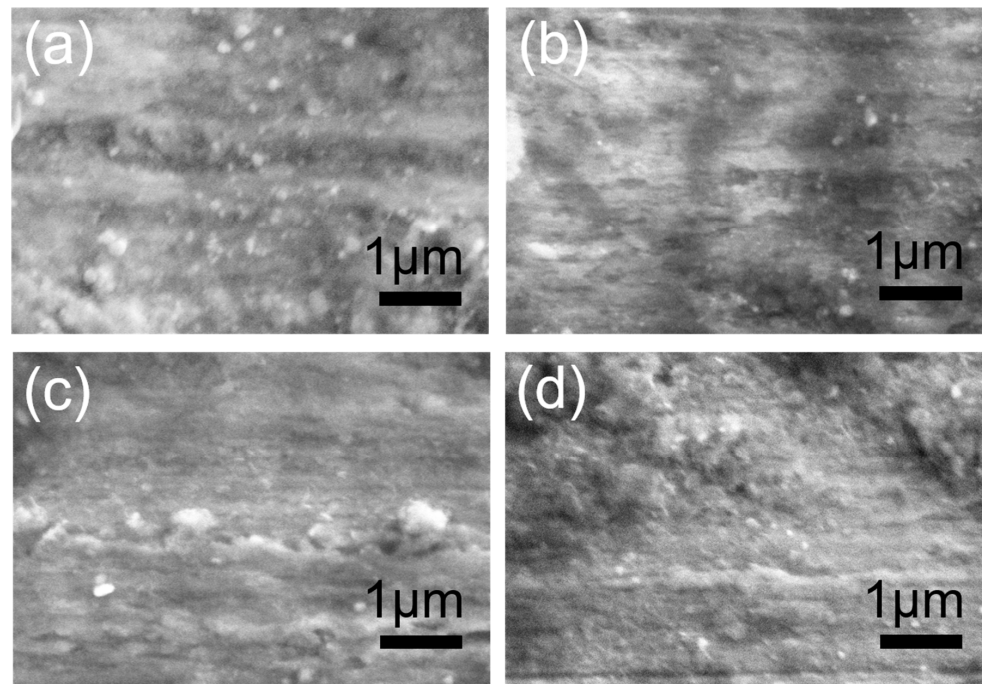


Figure 5. High-magnification SEM observations (secondary electron images) of the 0.03 m/s wear-track region after immersion in a 3.5 wt.% NaCl solution for different durations: (a) as-worn state; (b) 12 h; (c) 48 h; and (d) 120 h.

The quantitative pit statistics further confirm the progressive localized corrosion of the 0.30 m/s wear track (Figure 6). At 12, 48, and 120 h, the analyzed areas were 97.25, 94.94, and 95.28 μm^2 , respectively, and the corresponding numbers of identified pits were 178, 203, and 245. The calculated pit density therefore increased from 1.83 μm^{-2} at 12 h to 2.14 μm^{-2} at 48 h and 2.57 μm^{-2} at 120 h (Figure 6a). Meanwhile, the mean pit diameter increased from 0.07 μm to 0.12 μm and 0.15 μm over the same immersion sequence (Figure 6b). The pit-diameter distributions also shift toward larger sizes with increasing immersion time (Figure 6c): the 12 h condition is dominated by smaller pits, whereas the 48 and 120 h conditions show broader distributions and a more pronounced tail toward larger diameters. These results indicate that localized corrosion on the 0.30 m/s wear track proceeds through both pit initiation and pit enlargement during immersion.

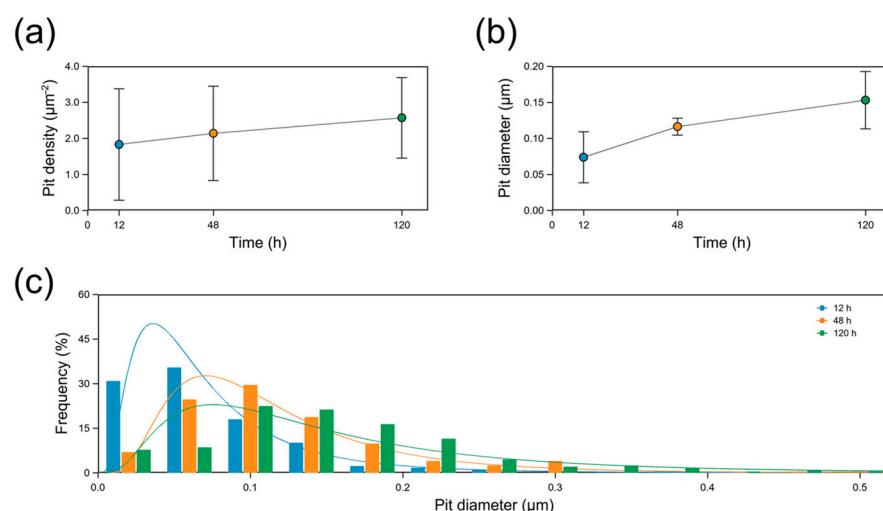


Figure 6. Quantitative micro-pit statistics for the 0.30 m/s wear track after immersion in a 3.5 wt.% NaCl solution for 12, 48, and 120 h. (a) Pit density, calculated from the number of identified pits divided

by the analyzed area. (b) Pit diameter, determined from the equivalent diameters of individual pits. Error bars in (a) and (b) represent standard deviations. (c) Pit-diameter frequency distributions, with bars representing the measured distributions and fitted curves showing the corresponding trends.

Overall, the quasi-in situ SEM results show that both wear tracks evolve from mechanically damaged surfaces toward corrosion-modified surfaces, but the 0.30 m/s track exhibits earlier debris degradation, more obvious interfacial separation, and stronger pit development.

3.2. Quasi-In Situ Surface Topography Evolution of the Wear Tracks

The surface topography maps provide complementary height information for the same post-wear immersion process. For the 0.30 m/s wear track (Figure 7), the as-worn surface contains a banded height profile along the sliding direction, with localized protrusions associated with compacted adherents and debris-rich regions. After 12 h of immersion, the height distribution becomes more heterogeneous, and local high-value regions are more distinct within the wear track. This agrees with the SEM evidence that early chloride exposure modifies the debris-covered surface through local corrosion-product accumulation, adherent loosening, and the formation of incipient depressions.

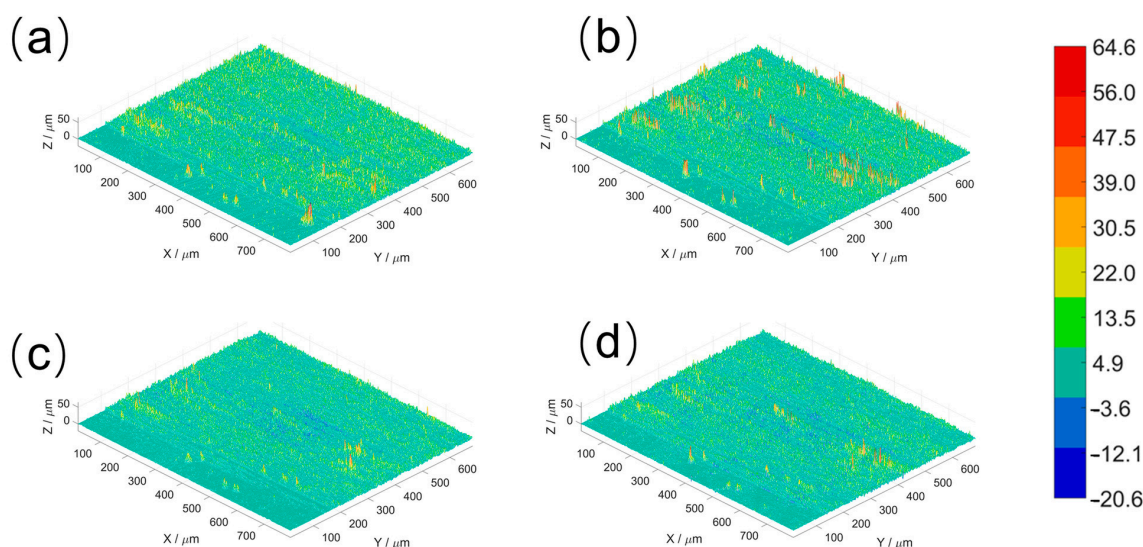


Figure 7. Quasi-in situ surface topography maps of the 0.30 m/s wear-track region after immersion in a 3.5 wt.% NaCl solution: (a) as-worn state; (b) 12 h; (c) 48 h; and (d) 120 h.

After immersion for 48 h, the 0.30 m/s topography shows fewer concentrated protrusions and a more discontinuous height distribution, which is consistent with partial consumption or removal of initially protruding adherent debris. At 120 h, the surface becomes comparatively more uniform in the areal height map, although groove-like features and local depressions remain. This does not imply the absence of localized corrosion; rather, it indicates that the average height contrast becomes less dominated by large debris-related protrusions as immersion proceeds.

The wear track produced at 0.03 m/s shows a similar but weaker topographic evolution (Figure 8). The as-worn surface is dominated by continuous sliding grooves and a relatively stable wear-track profile. After 12 h of immersion, scattered high-value regions appear, indicating that residual adherents and early corrosion products introduce additional local unevenness. However, compared with the 0.30 m/s track, the banded morphology remains more continuous, and the topographic reconstruction is less severe. At 48 and 120 h, localized height extremes gradually decrease, and the surface height distribution

becomes more uniform along the sliding direction. This behavior is consistent with limited loosening or removal of residual debris rather than extensive pit-related surface breakup.

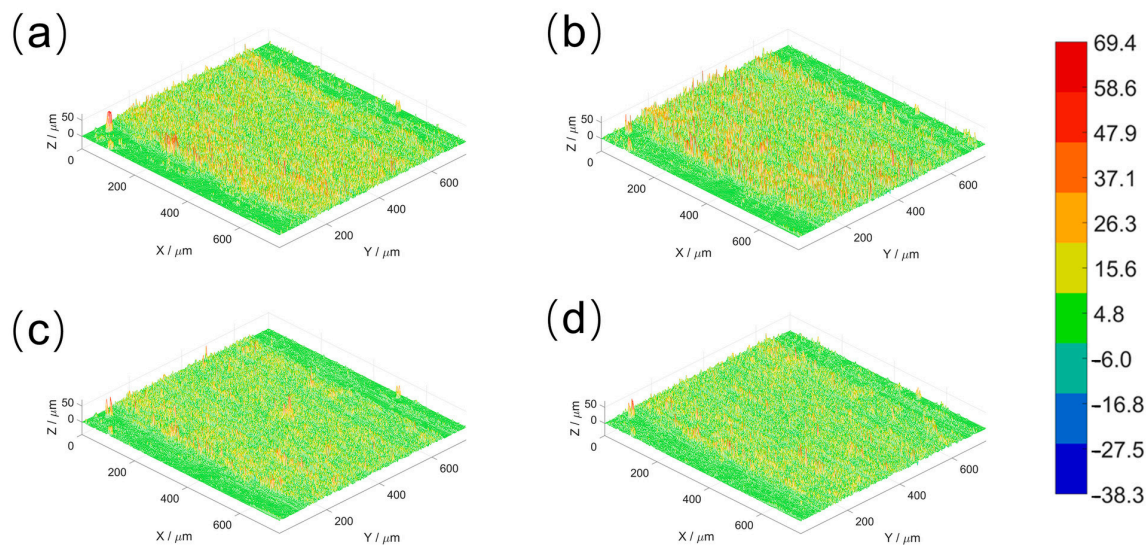


Figure 8. Quasi-in situ surface topography maps of the 0.03 m/s wear-track region after immersion in a 3.5 wt.% NaCl solution: (a) as-worn state; (b) 12 h; (c) 48 h; and (d) 120 h.

The S_a and S_q roughness evolution provides quantitative descriptors of surface-height redistribution during post-wear immersion (Figure 9). For the 0.30 m/s track, S_a changed from 3.02 μm in the as-worn state to 3.17 μm after 12 h and decreased to 2.57 μm after 120 h, whereas S_q changed from 4.04 to 4.46 μm and then decreased to 3.40 μm . For the 0.03 m/s track, S_a changed from 4.65 μm in the as-worn state to 4.57 μm after 12 h and decreased to 3.50 μm after 120 h, while S_q changed from 6.03 to 6.35 μm and then decreased to 4.58 μm . Both parameters showed short-term fluctuations followed by an overall decrease during prolonged immersion. The lower-speed track consistently exhibited higher S_a and S_q values, whereas the higher-speed track showed more pronounced localized pitting. The same confocal datasets were used to quantify the initial wear damage before immersion. The total wear volumes were $1.23 \times 10^{-3} \text{ mm}^3$ for the 0.30 m/s track and $1.86 \times 10^{-3} \text{ mm}^3$ for the 0.03 m/s track, while the corresponding mean depths within the below-baseline areas were 1.42 and 1.46 μm , respectively.

The decrease in S_a and S_q at long immersion times should therefore not be interpreted as evidence of weaker corrosion. Surface topography maps are strongly influenced by the loss of protruding adherents and corrosion-product accumulations, whereas pit growth is localized and may occupy only a small fraction of the measured area. The S_a and S_q data should therefore be interpreted together with the SEM observations and pit statistics. Average roughness alone does not rank corrosion severity, since the 0.30 m/s track develops more evident localized corrosion even though its average S_a and S_q values are lower than those of the 0.03 m/s track.

3.3. TEM and 4D-STEM Characterization of As-Worn Microstructure

TEM characterization was first used to define the initial microstructure of the electrodeposited nanocrystalline Ni coating before analyzing the worn regions. The as-deposited coating consists of fine nanocrystalline grains (Figure 10a). Microstructural size statistics confirm that sliding caused measurable coarsening of the nanocrystalline coating (Figure 10d–f). After sliding at 0.30 m/s, a lamellar structure developed in the near-surface layer, whereas the grains remained equiaxed after sliding at 0.03 m/s; therefore, lamellar thickness was measured for the 0.30 m/s track, while grain size was measured for the

0.03 m/s track. The broader distribution after 0.30 m/s sliding supports the combined TEM and 4D-STEM observations that the high-speed track developed more extensive microstructural coarsening in the friction-affected subsurface layer.

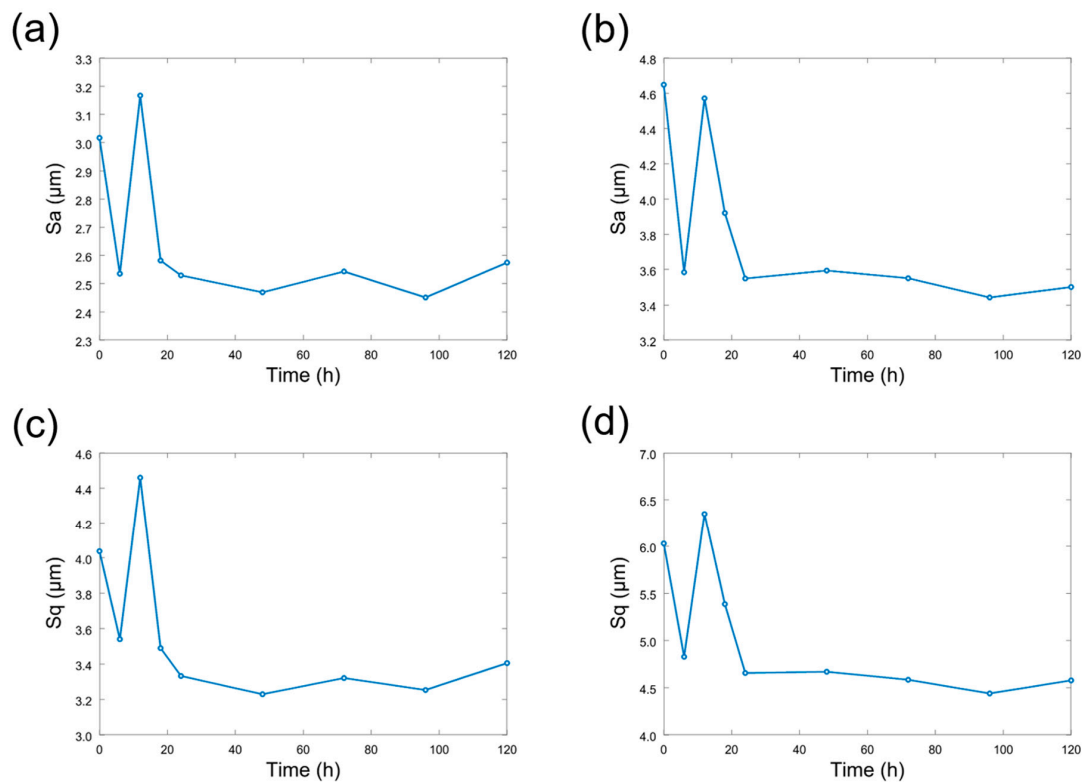


Figure 9. Evolution of the areal roughness parameters S_a and S_q of the wear tracks during post-wear immersion: (a,c) 0.30 m/s and (b,d) 0.03 m/s.

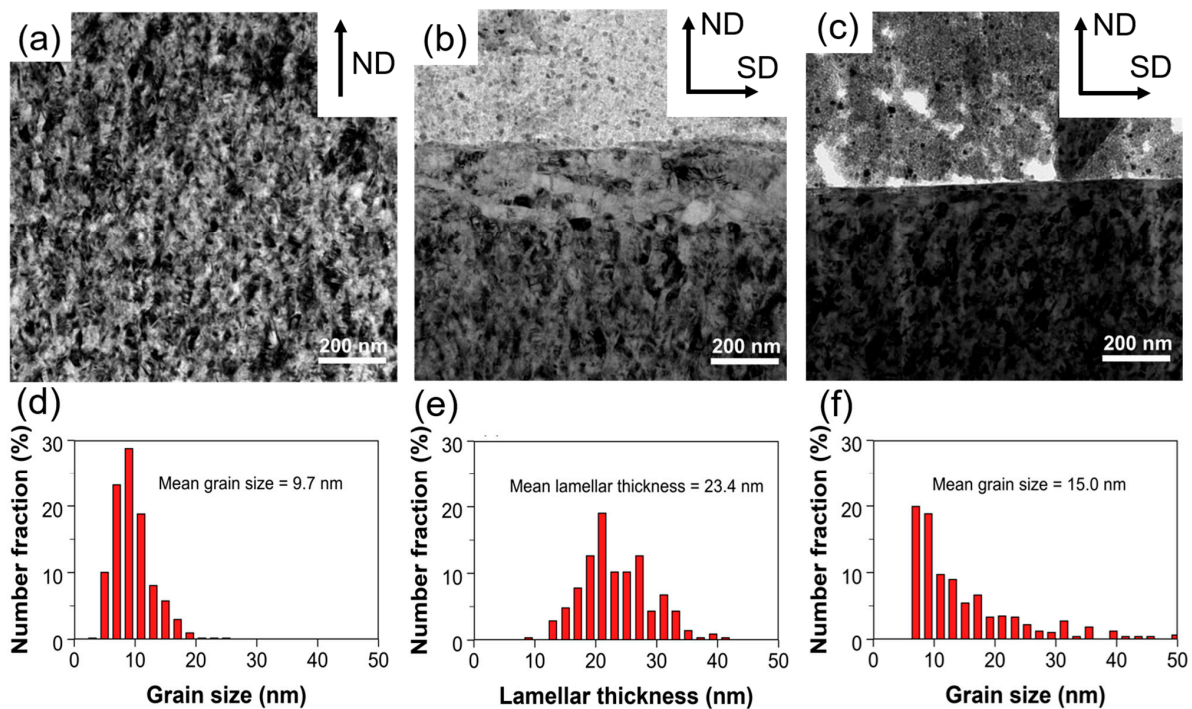


Figure 10. TEM images of the nanocrystalline Ni coating: (a) as-deposited state, (b) 0.30 m/s, (c) 0.03 m/s; grain size and lamellar thickness distributions of the nanocrystalline Ni coating: (d) as-deposited state, (e) 0.30 m/s, and (f) 0.03 m/s.

The 4D-STEM orientation maps reveal how sliding modified this nanocrystalline baseline in the as-worn state (Figure 11). The friction-affected subsurface layer is clearly dependent on sliding speed. The wear track produced at 0.30 m/s exhibits a more pronounced near-surface structural gradient, whereas the 0.03 m/s track retains a larger fraction of fine orientation domains. This contrast indicates that the two wear tracks entered the subsequent immersion stage with different crystallographic and microstructural states.

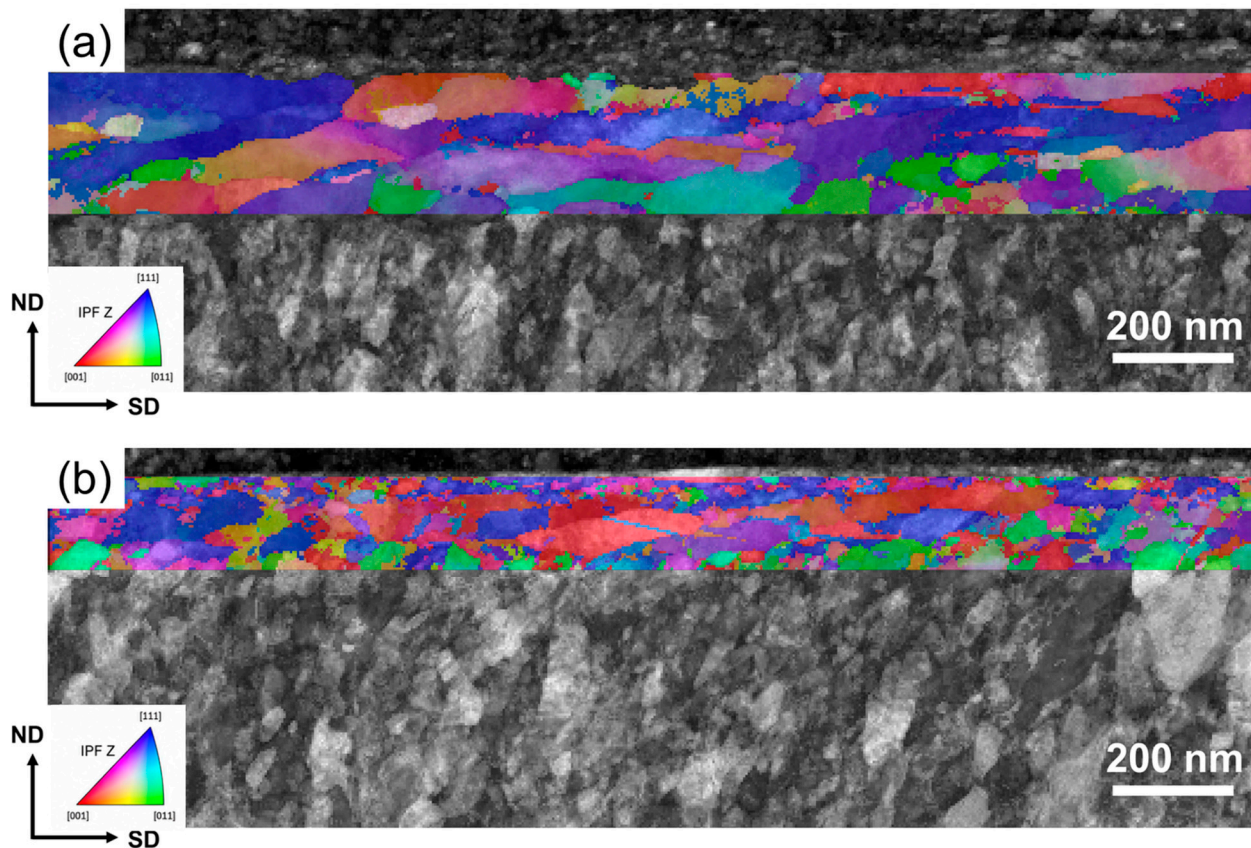


Figure 11. Orientation maps derived from 4D-STEM datasets of the subsurface regions in specimens tested at sliding speeds of (a) 0.30 m/s and (b) 0.03 m/s. The colors indicate the crystallographic direction parallel to the surface normal.

For the 0.30 m/s track, the orientation map shows a stratified friction-affected structure composed of a compacted near-surface layer, an underlying grain-coarsened layer, and the nanocrystalline matrix beneath it. Enlarged orientation domains are evident below the compacted surface layer, demonstrating substantial grain growth in the subsurface region. Many coarsened grains are elongated mainly along the sliding direction, suggesting stronger shear-driven microstructural reconstruction at 0.30 m/s. By comparison, the 0.03 m/s track shows a less distinctly layered structure. Local grain growth is still present near the surface, but the affected region contains a mixed structure of coarsened grains and retained nanocrystalline domains rather than a continuous combined compacted-layer and grain-coarsened-layer architecture.

The corresponding ODF sections at $\varphi_2 = 45^\circ$ provide further evidence of sliding-induced texture redistribution in the lamellar regions (Figure 12). Compared with the ideal FCC shear texture components in Figure 12c, the 0.30 m/s specimen shows a more continuous intensity distribution along the FCC shear-texture skeleton, together with additional intensity near the B ($\{1 \ -1 \ 2\} \langle 1 \ 1 \ 0 \rangle$) and ($\{-1 \ 1 \ -2\} \langle -1 \ -1 \ 0 \rangle$) components (Figure 12a). This result indicates that sliding at 0.30 m/s promotes a stronger shear-induced texture transition and produces a more heterogeneous combination of $\langle 1 \ 1 \ 1 \rangle$, $\langle 1 \ 0 \ 0 \rangle$, and

$\langle 1\ 1\ 2 \rangle$ orientation components along the ND. In contrast, the 0.03 m/s specimen mainly shows intensity maxima close to the $A1^*$ ($\{1\ 1\ -1\} \langle 1\ 1\ 2 \rangle$), $A2^*$ ($\{1\ 1\ -1\} \langle 2\ -1\ 1 \rangle$), and C ($\{0\ 0\ 1\} \langle 1\ 1\ 0 \rangle$) components (Figure 12b). These components correspond to preferred $\langle 1\ 1\ 1 \rangle$ and $\langle 1\ 0\ 0 \rangle$ directions along the ND and are broadly consistent with the texture inherited from the as-deposited coating.

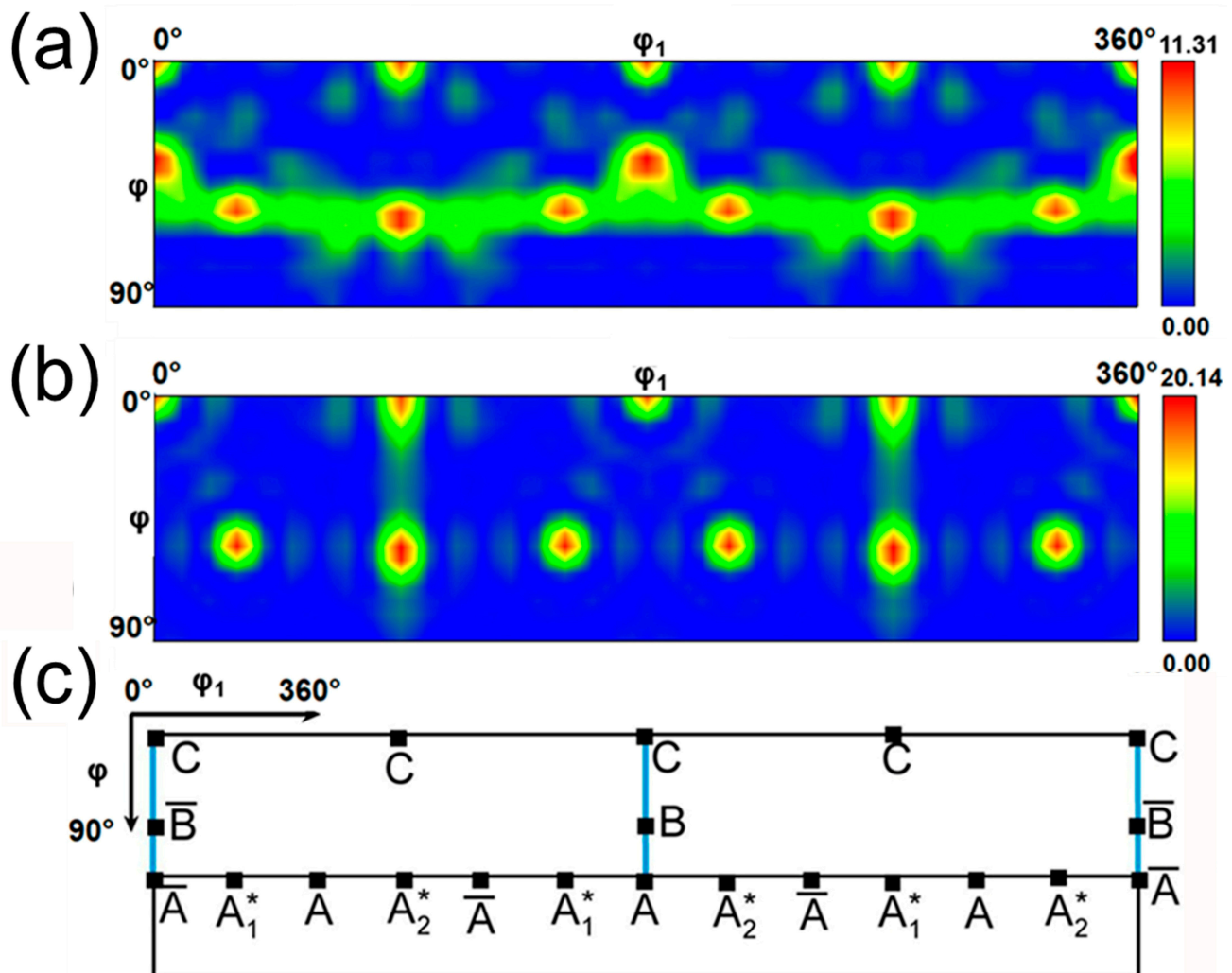


Figure 12. ODF sections at $\phi_2 = 45^\circ$ for the corresponding lamellar regions in the specimens tested at (a) 0.30 m/s and (b) 0.03 m/s; (c) ideal FCC shear texture components shown in the same ODF section [51]. The asterisk is part of the conventional notation for the A1 and A2 FCC shear-texture components.

Therefore, the TEM, 4D-STEM, and ODF results consistently show that the 0.30 m/s track developed a more extensively reconstructed as-worn subsurface state than the 0.03 m/s track. The coexistence of a compacted surface layer, grain coarsening, and redistributed texture provides a structural basis for the faster morphological degradation and stronger pit development observed during subsequent chloride immersion. In contrast, the 0.03 m/s track retained a finer and less stratified microstructure, which is consistent with its more stable groove-dominated corrosion morphology.

4. Discussion

The present results show that the corrosion behavior of worn nanocrystalline Ni coatings is governed by a sequential but coupled process: sliding first creates a mechanically and crystallographically modified surface, and subsequent chloride immersion selectively

amplifies the heterogeneity inherited from wear. The quasi-in situ design is central to this interpretation because the same exposed regions were repeatedly examined during immersion. The pit statistics, S_a and S_q roughness curves, and grain size distributions further show that the morphological sequence is accompanied by measurable pit growth, surface-height redistribution, and sliding-induced grain coarsening rather than by qualitative image contrast alone.

At the surface scale, sliding generated grooves, compacted adherent debris, local transferred material, and discontinuous corrosion-product-covered areas. These features may alter local electrochemical conditions after immersion because grooves and debris–substrate interfaces can behave as micro-crevice-like sites where electrolyte exchange and oxygen transport are restricted [1–4,20–23], while mechanically disrupted surface films may expose fresh Ni to chloride solution [20]. The 0.30 m/s track contained a more discontinuous debris layer and stronger interfacial separation than the 0.03 m/s track, which is expected to provide more locations for chloride retention and local film disruption. Localized corrosion studies also show that microstructural heterogeneity can produce spatially non-uniform corrosion activity [52]. Because direct electrochemical or surface-chemical measurements of film rupture and repair were not performed in this stage of the study, the discussion is limited to morphology-based evidence for localized corrosion susceptibility.

The topography evolution further supports this mechanism, but it should be interpreted as height redistribution rather than as a standalone metric of corrosion severity. Quantitatively, the 0.30 m/s track showed clearer clustered pitting and increasing pit density and equivalent diameter, whereas the 0.03 m/s track had higher S_a and S_q values than the 0.30 m/s track throughout immersion. This contrast shows that neither S_a nor S_q alone determines post-wear corrosion severity. S_a and S_q decreased at long immersion times for both tracks because protruding adherents and corrosion products can be loosened, consumed, or detached during immersion, whereas pit growth is spatially localized and may occupy only part of the measured area. This non-monotonic relation is consistent with studies showing that corrosion and pitting behavior do not necessarily vary monotonically with deformation level [53–56].

An important advantage of the quasi-in situ methodology used in this study is that the same pre-worn regions can be repeatedly relocated and examined after successive immersion intervals. This approach distinguishes genuine time-dependent corrosion evolution from morphological differences among independently prepared specimens or different positions within a heterogeneous wear track. It also enables pit initiation, growth, and enlargement to be related directly to the initial grooves, compacted debris, interfacial separation, and other wear-induced features within each track. Therefore, the unique contribution of the present methodology is the spatially resolved correlation between initial wear damage and time-dependent corrosion morphology within the same defined worn regions.

The grain size statistics further demonstrate the different degrees of subsurface reconstruction produced by the two sliding conditions. The mean lamellar thickness in the near-surface layer reached 23.4 nm after sliding at 0.30 m/s, and the mean grain size increased from approximately 9.7 nm in the as-deposited coating to 15.0 nm after sliding at 0.03 m/s. Together with the TEM observations described above, these results indicate that the 0.30 m/s condition produced more extensive microstructural reorganization, whereas the 0.03 m/s track retained more characteristics of the as-deposited coating. Such differences may influence the subsequent corrosion response because grain size, grain-boundary density, deformation-induced defects, and microstructural gradients can affect local dissolution and pitting tendency in Ni and its alloys [57–60]. However, grain coarsening alone cannot sufficiently explain the observed corrosion behavior because it may reduce the

overall grain-boundary density and, under some conditions, decrease the number of electrochemically active boundaries. In the present high-speed track, the spatially non-uniform formation of the lamellar near-surface structure, together with mechanically disrupted interfaces and surrounding finer grains, produced a heterogeneous friction-affected layer. This microstructural heterogeneity may contribute to spatially non-uniform local reactivity but cannot be separated experimentally from the accompanying surface damage and debris redistribution.

The subsurface characterization indicates that the two sliding speeds also produced different as-worn crystallographic states. The 0.30 m/s track developed a compacted near-surface layer, a grain-coarsened layer, and a stronger $\langle 1\ 1\ 2 \rangle$ orientation along the normal direction, whereas the 0.03 m/s track retained a less stratified microstructure with stronger remnants of the initial $\langle 1\ 1\ 1 \rangle$ and $\langle 2\ 0\ 0 \rangle$ texture components. The formation of the $\langle 1\ 1\ 2 \rangle$ // ND texture at the higher sliding speed can be understood from the viewpoint of shear-texture selection in FCC metals. Sliding wear imposes intense near-surface shear deformation, and previous studies have shown that the plastically deformed subsurface layer in FCC metals can develop typical shear texture components [51,61,62]. The relative stability of these components is closely related to the dominant mechanism by which shear strain is accommodated. In coarse-grained high-stacking-fault-energy FCC metals, such as Ni, plastic deformation is generally dominated by full dislocation slip, and simple shear deformation often favors the C $\{1\ 0\ 0\} \langle 1\ 1\ 0 \rangle$ component, corresponding to $\langle 1\ 0\ 0 \rangle$ // ND [63]. In contrast, the B $\{1\ 1\ 2\} \langle 1\ 1\ 0 \rangle$ component, corresponding to $\langle 1\ 1\ 2 \rangle$ // ND, is usually enhanced when shear deformation involves partial-dislocation slip, stacking-fault formation, and/or deformation twinning. Although Ni is generally considered a high-stacking-fault-energy metal, the deformation mechanism can change significantly when the grain size is reduced to the nanometer scale. In nanocrystalline Ni, conventional full-dislocation activity is suppressed, while partial-dislocation processes and twinning become more favorable [64,65]. Therefore, at the higher sliding speed of 0.30 m/s, the increased local shear rate and flow stress may promote partial-dislocation/twinning-assisted shear deformation, leading to the formation of the observed $\langle 1\ 1\ 2 \rangle$ // ND texture. By contrast, at 0.03 m/s, the lower shear rate produces a more gradual deformation condition and does not drive the subsurface toward the same B-type shear-texture state. As a result, the original electrodeposition-related $\langle 1\ 1\ 1 \rangle$ / $\langle 2\ 0\ 0 \rangle$ // ND texture is largely retained.

Crystallographic texture constitutes another possible contribution because crystallographic orientation can influence surface energy, plane-dependent dissolution, and passive-film properties [9,57,59,60,66]. This interpretation is consistent with studies of cold-rolled Ta-4W alloys, in which corrosion behavior was governed by competition between deformation-induced defect accumulation and the protective effect associated with strong deformation texture [38]. Related Ta-W studies have also shown that recrystallization and severe plastic deformation can modify corrosion resistance by altering defect structures and crystallographic texture [67,68]. Furthermore, orientation-dependent electrochemical behavior has been reported for ordered intermetallic single crystals [69], Ni-based single-crystal alloys [57,58], Ni-Fe-Cr alloys [59], and Cu-Ni alloys [66]. In the present nanocrystalline Ni coating, retention of the initial $\langle 1\ 1\ 1 \rangle$ and $\langle 2\ 0\ 0 \rangle$ components after sliding at 0.03 m/s may contribute to a more uniform corrosion response, whereas the enrichment of $\langle 1\ 1\ 2 \rangle$ // ND at 0.30 m/s reflects a different shear-deformation path. Nevertheless, the present results do not establish an intrinsic corrosion-resistance ranking among these orientations. At 0.30 m/s, texture redistribution occurred together with near-surface lamellar-structure formation, deformation gradients, surface disruption, debris redistribution, and possible residual-stress development. The resulting crystallographic, mechanical, and chemical heterogeneities may generate micro-galvanic-like differences

in local reactivity, particularly at groove-damaged regions and debris–coating interfaces where chloride access is facilitated.

Plastic deformation represents a separate contribution to the corrosion response. The compacted near-surface layer, grain-coarsened subsurface layer, and broader grain size distribution observed after sliding at 0.30 m/s demonstrate that this condition produced more extensive and spatially heterogeneous microstructural reconstruction. Sliding-induced deformation can alter dislocation structures, grain-boundary configurations, deformation twins, lattice defects, and stored strain energy. Previous studies of cold-worked and severely deformed alloys have shown that these deformation-induced changes can affect local dissolution, passivation, and pitting behavior [53–56,67,68]. Under some conditions, grain coarsening may reduce the overall grain-boundary density and thereby decrease the number of electrochemically active boundaries; therefore, grain coarsening alone does not sufficiently explain the observed corrosion behavior. In the present high-speed track, however, grain growth was spatially non-uniform. The coexistence of the compacted surface layer, coarsened subsurface grains, finer surrounding grains, and mechanically disrupted interfaces produced a deformation gradient and a heterogeneous microstructure. Regions with different defect densities, stored energies, and surface conditions may consequently exhibit different local dissolution and passivation behavior, favoring pit initiation at structurally discontinuous or mechanically damaged sites. Because the defect density and stored strain energy were not measured directly, these effects are considered literature-supported mechanistic possibilities rather than independently demonstrated causes.

Sliding-induced residual stress may constitute an additional influence. Non-uniform plastic flow, repeated contact, and frictional heating can generate heterogeneous residual-stress fields within and beneath a wear track. Such stresses may modify local electrochemical activity and passive-film stability, particularly where they are concentrated near grooves, debris–substrate interfaces, or microstructural gradients. However, the magnitude, spatial distribution, and tensile or compressive character of the residual stress were not measured in this study. Its contribution therefore cannot be separated quantitatively from the accompanying deformation and microstructural reconstruction and is discussed only as a possible component of the wear-induced state.

Overall, increasing sliding speed intensifies surface disruption and debris-layer instability under the present testing conditions. This produces greater topographic heterogeneity during the early immersion stage, promotes more pronounced localized corrosion, and leaves a grain-coarsened and texture-modified as-worn layer that may further contribute to non-uniform corrosion. Figure 13 schematically compares the as-worn and post-corrosion states produced at 0.30 and 0.03 m/s and summarizes the coupled roles of surface morphology, debris stability, grain coarsening, and texture redistribution. The schematic treats texture as a possible contributing factor within the broader wear-induced state rather than as an independent cause of pitting. The stronger localized corrosion observed on the 0.30 m/s track is attributed to the coupled effects of these wear-induced heterogeneities rather than to S_a , S_q , residual stress, or the $<1\ 1\ 2>/ND$ texture acting independently. The quasi-in situ observations therefore connect the time-dependent corrosion morphology directly with the wear-induced surface and subsurface state of the nanocrystalline Ni coating. It should also be noted that conventional macroscopic electrochemical measurements would average the 0.30 m/s wear track, the 0.03 m/s wear track, and adjacent coating areas within the exposed window, making it difficult to isolate the narrow worn regions. Local electrochemical micro-cell or scanning electrochemical techniques are more suitable for such small heterogeneous areas, but they require dedicated instrumentation and sealing configurations [30,31]. Therefore, the present work is positioned as a quasi-in situ mor-

phology and microstructure study, while local electrochemical quantification remains an important direction for future work.

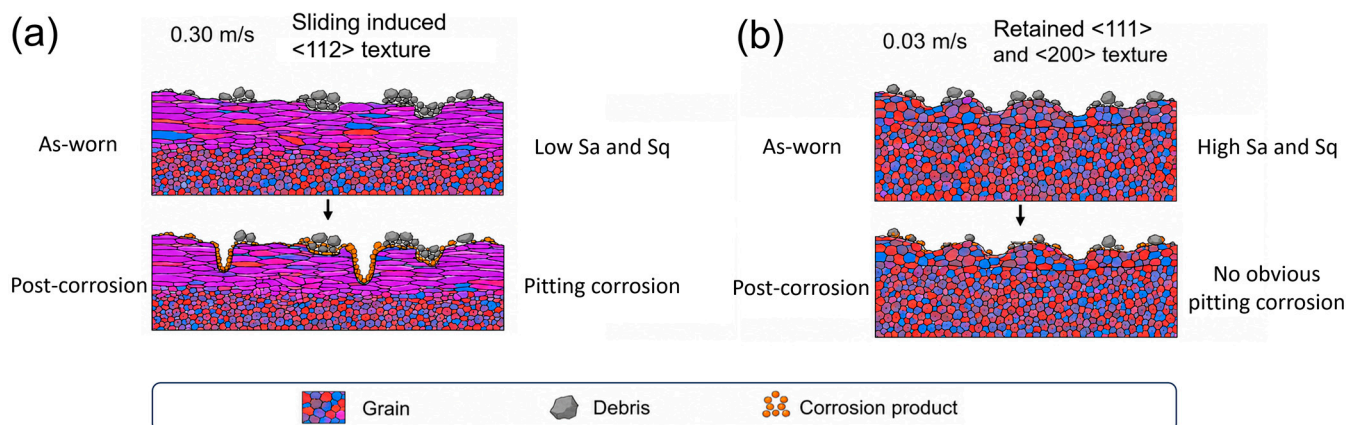


Figure 13. Schematic comparison of the proposed post-wear corrosion responses of nanocrystalline Ni coatings following sliding at (a) 0.30 and (b) 0.03 m/s.

5. Conclusions

In this study, the post-wear immersion corrosion behavior of electrodeposited nanocrystalline Ni coatings was investigated by combining quasi-in situ SEM observations with surface topography, pit statistics, TEM, 4D-STEM orientation mapping, and ODF analysis. The main conclusions are as follows:

- The quasi-in situ SEM sequence and high-magnification images show that the 0.30 m/s wear track undergoes faster debris degradation, stronger interfacial separation, and more evident pit development than the 0.03 m/s track during immersion in a 3.5 wt.% NaCl solution.
- For the 0.30 m/s track, the pit density increased from 1.83 to 2.57 μm^{-2} , while the mean equivalent pit diameter increased from 0.07 to 0.15 μm between 12 and 120 h. The Sa and Sq values were consistently higher for the 0.03 m/s track than for the 0.30 m/s track, indicating that average surface roughness alone does not determine localized post-wear corrosion severity.
- TEM, 4D-STEM, ODF, and grain size statistics show that sliding induced more extensive subsurface reconstruction at 0.30 m/s. The mean lamellar thickness in the near-surface layer reached 23.4 nm after sliding at 0.30 m/s, whereas the mean grain size increased from approximately 9.7 nm in the as-deposited coating to 15.0 nm after sliding at 0.03 m/s. The 0.30 m/s track developed a more stratified friction-affected layer and stronger shear-induced texture redistribution with enhanced $\langle 112 \rangle$ /ND components, whereas the 0.03 m/s track retained more of the initial $\langle 111 \rangle$ and $\langle 200 \rangle$ texture components.

Overall, the post-wear corrosion response is interpreted as the combined effect of wear-induced surface damage, debris-layer instability, grain coarsening, and sliding-induced texture redistribution. These factors generate local mechanical, chemical, and crystallographic heterogeneity that may favor localized corrosion after 0.30 m/s sliding.

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