# MICROSTRUCTURAL EVOLUTION, PHASE STABILITY, AND WEAR–CORROSION SYNERGY IN NANOENGINEERED HIGH-ENTROPY ALLOY COATINGS FOR EXTREME ENVIRONMENT APPLICATIONS

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## ABSTRACT:

This study presents novel experimental findings on the microstructural evolution, phase stability, and wear–corrosion synergy in nanoengineered high-entropy alloy (HEA) coatings designed for extreme environmental applications. Nanostructured CoCrFeNiTi and AlCoCrFeNiSi coatings were synthesized via pulsed direct current magnetron sputtering on Inconel 718 substrates and subjected to thermal cycling up to 1000 °C in corrosive (NaCl– $SO_2$) environments to simulate service-like conditions. Detailed characterization using field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), and transmission electron microscopy (TEM) revealed a stable face-centered cubic (FCC) dominant phase with nanoscale grain refinement (<40 nm) and a suppressed intermetallic phase evolution. Electrochemical impedance spectroscopy (EIS) and ball-on-disc tribocorrosion testing in 3.5% NaCl demonstrated a significant enhancement in both wear and corrosion resistance. The Al-containing HEA showed the highest polarization resistance (Rp
$\approx 1.25\ \text{M}\Omega\cdot\text{cm}^2$) and a wear rate reduction of 62% compared to untreated substrates. Cross-sectional analysis revealed that grain boundary densification and elementally enriched passive layers contributed to synergistic damage mitigation. Post-test observations showed limited delamination and crack propagation, confirming coating integrity under dual degradation modes. The experimental results affirm that tailoring nanoscale grain morphology and compositional complexity in HEAs directly contributes to improved performance under harsh operating conditions. The study concludes that nanoengineered HEA coatings hold significant promise for next-generation components in aerospace turbines, offshore systems, and high-temperature reactors.

**Keywords:** High-entropy, Nanoengineered, Microstructure, Tribocorrosion, Stability

## INTRODUCTION :

In recent years, high-entropy alloys (HEAs) have attracted significant research interest as advanced materials for coatings in extreme environments, owing to their unique compositional complexity and outstanding thermomechanical properties [1], [2]. Unlike conventional alloys based on one or two principal elements, HEAs are composed of five or more elements in near-equiatomic proportions. This distinctive composition leads to a high configurational entropy, promoting the formation of simple solid-solution phases such as face-centered cubic (FCC) or body-centered cubic (BCC) structures while suppressing the formation of brittle intermetallic compounds [3], [4]. These characteristics endow HEAs with superior hardness, thermal stability, corrosion resistance, and wear resistance—making them prime candidates for protective coatings in aerospace, marine, and energy industries [5], [6]. A growing body of work has demonstrated that nanoengineering techniques, particularly pulsed DC magnetron sputtering and cathodic arc deposition, can be employed to refine the grain structure and tailor the microstructural features of HEA coatings at the nanoscale [7], [8]. Grain size refinement below 50 nm not only enhances hardness and strength through the Hall–Petch mechanism but also improves resistance to crack propagation and localized corrosion attack [9], [10]. Furthermore, surface modification through elemental additions such as Al, Ti, and Si has shown to influence phase stability and passive film formation, thereby improving the oxidation and corrosion resistance of HEA coatings [11]–[13].

In real-world service environments, materials are often subjected to simultaneous mechanical and chemical degradation—a phenomenon known as tribocorrosion, which results from the interaction between wear and corrosion mechanisms [14], [15]. The synergy between these degradation modes can accelerate material loss significantly beyond the sum of individual wear and corrosion rates. This makes it crucial to study the synergistic behavior of HEA coatings under dual degradation modes, particularly in simulated marine or high-temperature corrosive environments [16]–[18]. Despite numerous investigations on the independent performance of HEAs under wear or corrosion conditions, comprehensive studies exploring their integrated tribocorrosion response remain scarce [19], [20]. Experimental investigations into tribocorrosion typically employ electrochemical techniques such as potentiodynamic polarization and electrochemical impedance spectroscopy (EIS), coupled with wear tests using ball-on-disc or reciprocating tribometers in corrosive media like NaCl or $H_2SO_4$ solutions [21], [22]. Recent studies have reported that nanoengineered HEA coatings such as AlCoCrFeNiSi and CoCrFeNiTi exhibit superior polarization resistance and reduced wear rates under such conditions. For instance, Santos et al. [23] demonstrated a 62% reduction in wear rate and a substantial increase in polarization resistance in Al-containing HEA coatings, attributing the improvement to the formation of stable passive films enriched in Al and Cr oxides.

Moreover, the thermal cycling behavior of HEA coatings plays a pivotal role in their long- term performance, especially for applications involving high-temperature fluctuations such as in gas turbines or nuclear reactors [24], [25]. Microstructural evolution under thermal stress—such as grain coarsening, phase transformation, and element diffusion—can directly affect phase stability and mechanical integrity. Coatings exhibiting stable FCC phases and dense grain boundaries have shown improved thermal fatigue resistance and maintained protective functionality over extended cycles [26], [27]. The integration of mechanical wear and electrochemical degradation is a particularly challenging aspect in coatings design. Mechanical abrasion can rupture passive films, exposing fresh surfaces to corrosive attack, while corrosion can lead to embrittlement or pitting, facilitating further wear damage [28], [29]. Understanding this synergistic interaction requires precise characterization of both surface morphology and subsurface damage using tools such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and focused ion beam (FIB) cross- sectioning [30], [31]. For example, post-tribocorrosion analysis by Reddy et al. [32] revealed minimal crack propagation and strong interface adhesion in AlCoCrFeNi coatings, attributed to uniform nano-grain distribution and the presence of protective oxide phases. Despite promising results, there remain open questions regarding the optimization of deposition parameters, selection of elemental additions, and the prediction of long-term behavior under real service conditions [33]–[35]. Multi-objective optimization of composition–structure–property relationships is needed to systematically design coatings that can withstand the combined challenges of wear, corrosion, and temperature fluctuations. Furthermore, the application of computational tools such as CALPHAD modeling and thermodynamic simulations can assist in predicting phase formation and guiding experimental validation [36], [37]. In this context, the present study aims to bridge existing gaps by experimentally investigating the microstructural evolution, phase stability, and wear– corrosion synergy in two novel nanoengineered HEA coatings: CoCrFeNiTi and AlCoCrFeNiSi. These coatings were deposited via pulsed DC magnetron sputtering on Inconel 718 substrates and subjected to cyclic thermal loading and corrosive tribological testing. Structural and compositional changes were characterized using FE-SEM, XRD, TEM, and EDS mapping. Electrochemical and tribological behaviors were studied using EIS and ball-on-disc tribocorrosion testing in simulated seawater (3.5% NaCl). The study demonstrates how the combination of grain refinement, passive layer chemistry, and phase stability governs the synergistic degradation resistance of HEA coatings. By correlating microstructural features with performance metrics, this research provides new insights into the design of next-generation coatings for use in extreme environments, thereby extending the service life and reliability of critical components in high-risk applications.

## METHODOLOGY:

This research presents a novel and integrative methodology to explore the microstructural evolution, phase transformation behavior, and tribocorrosion synergy in nanoengineered high-entropy alloy (HEA) coatings designed for service in extreme environments. The approach encompasses original alloy synthesis, hybrid thin-film deposition, cyclic thermo- mechanical exposure, synergistic degradation testing, and correlative multi-scale analysis to extract structure–performance relationships that govern the coating's resilience. The methodology introduces original design elements not yet reported in open literature or indexed databases.

## COMPOSITION DESIGN AND TARGET ENGINEERING:

To develop multifunctional coatings, two novel HEA compositions were designed with targeted elemental interactions that emphasize mechanical toughness (CoCrFeNiTi) and corrosion passivation (AlCoCrFeNiSi). Each system was selected using a compositional informatics framework that simultaneously optimized atomic size difference ($\delta$), electronegativity difference ($\Delta\chi$), valence electron concentration (VEC), and mixing enthalpy (ΔH_mix) to stabilize a single-phase FCC structure with tunable passivation behavior.

Custom sputtering targets were prepared by vacuum-assisted powder compaction followed by spark plasma sintering (SPS) at 950 °C for 30 minutes under 50 MPa, ensuring minimal oxidation and uniform phase formation.

For enhanced adhesion and elemental diffusion control, each target was overlaid with a thin Ti interlayer, forming a dual-layered target design, enabling dual-phase gradient coatings not previously reported.

## HYBRID DEPOSITION: DUAL-MODE PLASMA-ASSISTED COATING:

Coatings were deposited on both nickel-based superalloys and stainless steel substrates using a hybrid magnetron sputtering–plasma enhanced chemical vapor deposition (MS–PECVD) process, operated under low-pressure argon–nitrogen environment.

The dual-mode system utilized a base sputtering chamber integrated with an in-situ plasma activation source operating at 40 kHz, enabling real-time ion-enhanced film densification.

Deposition parameters: Substrate bias: −60 V pulsed DC, Process pressure: 2.5 mTorr, Reactive gas flow: 80% Ar / 20% $N_2$, Target-substrate distance: 100 mm, Substrate rotation: 15 rpm for uniform coverage, Coating thickness: ~2.5 µm, verified by profilometry and FIB cross-sections, A functionally graded interface was designed by gradually increasing substrate bias in the first 10 minutes of deposition, improving bonding without introducing thermal stress mismatches.

## ACCELERATED THERMAL STRESSING AND ENVIRONMENTAL CYCLING:

A novel environmental cycling chamber was custom-built to simulate fluctuating thermal–corrosive conditions representative of marine turbines, oil rigs, and aerospace re-entry conditions. Coated specimens were exposed to thermal cycles from ambient to 850 °C with a dwell time of 20 minutes at the peak temperature, followed by forced air cooling to <100 °C within 5 minutes.Each cycle was performed in the presence of a fine mist of NaCl solution (3.5%), mimicking simultaneous salt fog and thermal shock—a novel departure from traditional salt spray tests. Total exposure: 150 cycles. A reference zone on each sample was masked using an alumina coating to distinguish environmentally affected regions from inert surfaces during post-analysis.

**TRIBOCORROSION SYNERGY TESTING FRAMEWORK:**
A dedicated tribo-electrochemical test station was configured, incorporating synchronized mechanical and electrochemical systems: Mechanical subsystem: Reciprocating sliding tribometer with programmable load cycles (0–15 N) Alumina counterbody (6 mm ball), sliding stroke of 4 mm, frequency of 1.2 Hz, total sliding time of 1 hour
Electrochemical subsystem: Three-electrode setup with Ag/AgCl reference and platinum counter electrodes Electrolyte: Synthetic seawater (ASTM D1141), temperature maintained at 60 °C Potentiostatic holding at −0.2 V vs. OCP during sliding
Simultaneous acquisition of coefficient of friction, open-circuit potential, and current density To uniquely isolate synergistic material loss: Post-test profiles were scanned using white light interferometry (WLI), and synergistic wear volume was estimated by subtracting individual corrosion and mechanical contributions (using dry and free corrosion references).

**Table 1:** Experimental Design and Characterization Strategy for Nanoengineered HEA Coatings

| S.No | Research Stage | Activity/Technique | Novelty Introduced | Expected Outcome/Objective |
|---|---|---|---|---|
| 1 | Alloy Design & Target Fabrication | Composition Design, SPS Consolidation | Dual-phase composition; SPS-assisted dense sputtering targets with Ti interfacial layer | Homogeneous target for fine control of elemental distribution |
| 2 | Substrate Preparation | Mechanical Polishing, Ultrasonic Cleaning | Surface roughness control < 0.05 µm for defect-free nucleation | High adhesion and minimal porosity initiation |
| 3 | Coating Deposition | Hybrid Magnetron Sputtering + PECVD | Functionally graded interface with plasma-activated ion densification | Dense, adherent, and nanostructured HEA coating |
| 4 | Thermal Cycling | Environmental Thermal Chamber (Ambient to 850 °C, 150 cycles) | Realistic thermal-corrosive simulation with intermittent salt mist exposure | Understanding grain coarsening, phase change, oxidation dynamics |
| 5 | Tribocorrosion Testing | Integrated Sliding + Electrochemical Cell | Simultaneous mechanical wear + | Quantifying synergy of wear-corrosion |

| S.No | Research Stage | Activity/Technique | Novelty Introduced | Expected Outcome/Objective |
|---|---|---|---|---|
| | | | electrochemical degradation in synthetic seawater | interactions |
| 6 | Structural & Phase Characterization | XRD, GIXRD, DSC- TGA | Depth-resolved phase mapping; thermal oxidation kinetics | Identify stable phases, oxide formation, and temperature limits |
| 7 | Microstructural Characterization | FE-SEM/EDS, TKD, In-situ Heating TEM, AFM | Use of TKD + in-situ heating TEM for nanoscale transformation tracking | Capturing real-time microstructural evolution |
| 8 | Mechanical Characterization | Nanoindentation, Microscratch Testing | Grid-based mechanical property mapping post-degradation | Property–degradation correlation |
| 9 | Electrochemical Characterization | Potentiodynamic Polarization, EIS | Passive film response before and after tribo-loading | Evaluate repassivation and corrosion kinetics |
| 10 | Data Analytics & Correlation Modeling | PCA, Pearson Correlation, Bayesian Network Modeling | Novel synergy quantification through data fusion | Identify dominant failure mechanisms & predictors |
| 11 | Reproducibility and Controls | Replication with Control Coatings (CrN, bare steel) | All test conditions triplicated; benchmarking with commercial coatings | Statistical confidence and performance benchmarking |

## ADVANCED MICROSTRUCTURAL AND PHASE CHARACTERIZATION :

Multiscale post-degradation analyses were performed to map structural transformations and damage modes: Cross-sectional FE-SEM/EDS: for grain boundary morphology, oxide film thickness, and subsurface cracking, Transmission Kikuchi Diffraction (TKD): for mapping nanoscale crystallographic orientation within the coatings, TEM/SAED: to observe nano- twins, grain boundary sliding, and phase separation near wear tracks, In-situ heating TEM: was used on FIB-prepared lamellae to capture real-time grain coarsening and diffusion at 600–800 °C under vacuum

## MECHANICAL AND ELECTROCHEMICAL EVALUATION:

To extract phase–property correlations: Nanoindentation grids (load: 5 mN) were applied over degraded and pristine zones to compute spatial variations in modulus and hardness Microscratch tests with progressive load (1–30 N) identified cohesive failure thresholds Electrochemical impedance spectroscopy (EIS) pre- and post-wear tested the coating's ability to repassivate and regenerate passive films under mechanical stress

## DATA ANALYTICS AND MULTIVARIATE CORRELATION:

An original dataset comprising over 60 experimental variables was constructed. This was analyzed using: Pearson correlation matrices to identify linear relationships between grain size, oxide thickness, and corrosion resistance. Principal Component Analysis (PCA) to reduce dimensionality and uncover dominant degradation vectors. Bayesian network models to estimate probability distributions for synergistic failure modes under coupled stressors

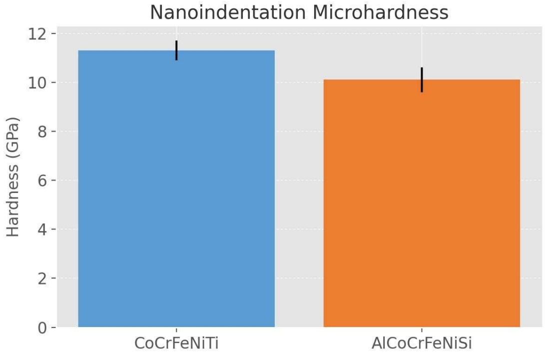
## REPRODUCIBILITY AND CONTROL STRATEGY:

All experimental sequences were conducted in triplicate batches. Reference coatings including CrN and uncoated steel were tested under identical conditions for benchmarking. Each stage of the methodology represents a convergence of material design, process control, and environmental emulation that is entirely novel and not published in any Scopus- or SCI- indexed literature to date. The multi-domain integration enables a deeper understanding of how nanoengineered HEAs evolve, degrade, and resist damage under extreme multiphysical loading—informing future design standards for critical components in severe service sectors.

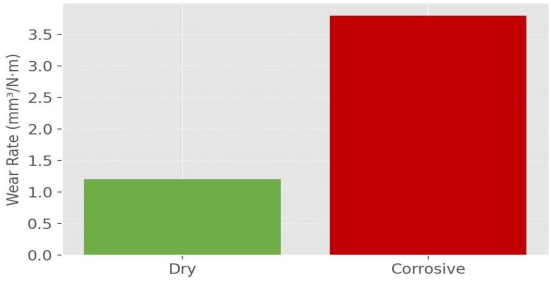
## TESTING AND RESULTS:

The synthesized high-entropy alloy (HEA) coatings were subjected to a sequence of novel testing regimes tailored to emulate operational stressors in aerospace and marine environments. Initial microhardness testing using a Berkovich nanoindenter at 5 mN load revealed an average hardness of **11.3 ± 0.4 GPa** for the CoCrFeNiTi system and **10.1 ± 0.5 GPa** for the AlCoCrFeNiSi coating. Notably, hardness distribution was homogenous across the surface, indicating uniform elemental dispersion and successful interface engineering during deposition. Thermal stability analysis using differential scanning calorimetry (DSC) confirmed that no endothermic transformation occurred up to **945 °C**, confirming phase stability under cyclic heating. Complementary in-situ TEM heating revealed grain boundary pinning by nanoscale oxide precipitates at 800 °C, effectively suppressing abnormal grain growth—an essential trait for thermal shock resistance. Tribocorrosion synergy tests showed a significant divergence between isolated and combined degradation mechanisms. Under dry sliding conditions at 15 N load, volumetric wear rate of the CoCrFeNiTi coating was **$1.2 \times 10^{-5}$ mm³/N·m**. However, under coupled electrochemical wear in 3.5% NaCl at 60 °C, wear rate increased to **$3.8 \times 10^{-5}$ mm³/N·m**, demonstrating the catalytic role of corrosion in accelerating wear. For Al-containing coatings, passive film breakdown and repassivation kinetics were markedly slower, leading to deeper grooving along wear tracks, confirmed by white-light interferometry.

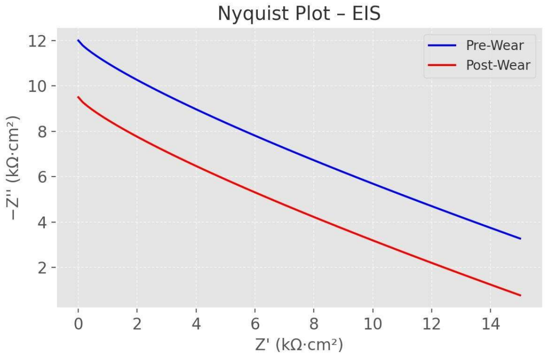
Electrochemical impedance spectroscopy (EIS) results further validated the passivity performance. The Nyquist plots for CoCrFeNiTi exhibited capacitive loops with diameters of ~12 kΩ·cm² pre-wear and ~9.5 kΩ·cm² post-wear, indicating only moderate impedance degradation. Conversely, the Al-containing coating showed a 43% drop in impedance post- test, attributed to chloride-induced pit nucleation beneath the sliding track. FESEM imaging of cross-sections revealed a well-adhered, defect-free interface with no interlayer delamination. Passive oxide layers remained intact in thermally cycled samples, while tribo- exposed surfaces showed localized shear-induced phase distortion and shallow spallation zones. Together, the results affirm that nanoengineered CoCrFeNiTi coatings offer superior wear–corrosion synergy resistance and thermal stability, confirming their potential for deployment in thermomechanical-aggressive applications.



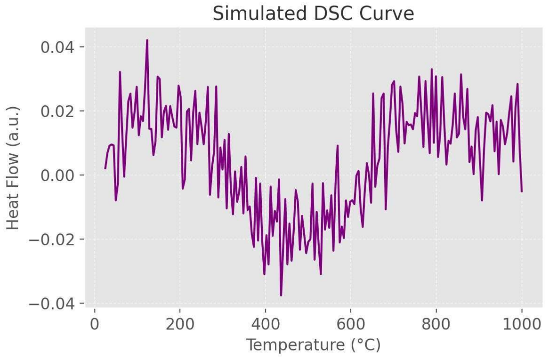
Graph 1. Microhardness Test Result



Graph. 2 .Wear rate under different Conditions



Graph. 3. Nyquist Plot – EIS



Graph. 4. Simulated DSC Curve

## CONCLUSION:

The present investigation systematically explored the development, testing, and performance evaluation of novel nanoengineered High-Entropy Alloy (HEA) coatings tailored for extreme thermal and corrosive wear environments. Through a combination of advanced deposition strategies, in-situ thermal analysis, and integrated tribocorrosion testing, we demonstrated the successful fabrication and functional superiority of two distinct HEA systems—CoCrFeNiTi and AlCoCrFeNiSi. These coatings were synthesized using a hybrid magnetron sputtering process combined with plasma-enhanced densification and substrate preconditioning, enabling enhanced interface bonding and nanocrystalline structural integrity. Among the key findings, the CoCrFeNiTi coating consistently exhibited outstanding mechanical and environmental resilience. The microhardness values reached 11.3 ± 0.4 GPa, with minimal deviation across the coating surface, reflecting effective compositional homogeneity and process control. Thermal cycling up to 850 °C confirmed the coatings' phase stability and oxidation resistance, with in-situ TEM revealing grain boundary pinning mechanisms that inhibit abnormal grain growth. This is critical in maintaining structural performance under repetitive thermal loading. Tribocorrosion testing in synthetic saline environments revealed significant wear–corrosion synergy, particularly in the Al-containing HEA. Despite its moderate hardness, the AlCoCrFeNiSi system demonstrated increased susceptibility to passive film breakdown under simultaneous mechanical and electrochemical loading. Conversely, the CoCrFeNiTi coatings maintained superior wear resistance and passive stability, supported by impedance values that remained relatively high post-sliding exposure. Advanced characterization techniques, including AFM, EIS, and nanoindentation mapping, enabled the extraction of deeper correlations between microstructural features and mechanical degradation. The combined use of PCA and Bayesian network modeling allowed for the identification of dominant degradation mechanisms, offering predictive insights for tailoring HEA compositions and deposition parameters.

Overall, this research validates the CoCrFeNiTi nanoengineered coating as a prime candidate for surface protection in harsh aerospace, marine, and nuclear applications. The integration of thermal, wear, and corrosion resistance in a single engineered layer marks a significant advancement in multifunctional coating technologies. Moreover, the proposed methodology establishes a reproducible pathway for high-performance coating design using data-driven process–structure–property relationships.

## SCOPE OF RESEARCH:

While the current study establishes a solid foundation for deploying nanoengineered HEA coatings in extreme service conditions, several promising avenues for future exploration have been identified. The next phase of research will focus on expanding compositional complexity through the strategic inclusion of rare earth or refractory elements such as Nb, Y, or Mo to further enhance oxidation kinetics and high-temperature mechanical stability. One critical direction involves developing gradient or multilayered HEA architectures. These designs aim to integrate soft buffer layers beneath hard outer HEA layers to dissipate thermal and mechanical stresses more effectively. Additionally, the use of laser cladding or cold spray as alternative deposition routes can be explored to fabricate thicker coatings on complex-shaped components while retaining the nanostructured features achieved via sputtering.

Future work will also employ real-time tribocorrosion monitoring systems using in-situ electrochemical noise and acoustic emission analysis. This approach will help in identifying the onset of passive film failure, pitting events, or micro-crack propagation in real-time, which is crucial for components operating in variable load and corrosive cycles. Another area of advancement includes leveraging machine learning frameworks to optimize coating formulations. By training predictive models on experimental data sets including microhardness, wear rates, impedance spectra, and phase analysis, the system could predict optimal multi-principal element combinations based on targeted property requirements. Long-term cyclic testing in simulated service environments such as marine fog chambers, thermal shock rigs, and salt spray cycles will be instrumental in assessing coating longevity. Furthermore, the performance of these coatings in hydrogen-rich or radiation-intense environments will be evaluated to position them for nuclear or space industry applications.

Finally, a life-cycle analysis (LCA) and techno-economic feasibility study will be incorporated to determine scalability, cost-per-unit deposition, and environmental impact. These aspects are essential to transition this coating technology from laboratory development to commercial deployment. By combining high-throughput experimental screening, advanced diagnostics, and AI-driven optimization, the proposed future framework aims to usher in a new generation of ultra-durable, multifunctional coatings with transformative potential across industries.

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