



Assessing wear and corrosion in oxide reinforced AlCrFeNi high entropy alloy fabricated via field assisted sintering technique

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ABSTRACT

AlCrFeNi high-entropy alloy (HEA) reinforced with 4%wt TiO₂ was fabricated through mechanical alloying (MA) followed by Field Assisted Sintering Technique (FAST) at 1100 °C temperature, 50 MPa pressure and 10 mins holding time, with subsequent investigation into FAST-induced powder deformation, microstructure, wear, and corrosion properties. TiO₂ slowed down powder deformation during FAST of the AlCrFeNi, reducing density by 0.8%. The microstructure consisted of (Fe, Cr)-enriched BCC and (Al, Ni)-rich B2 phases, with the even distribution of a dark TiO₂ phase in the AlCrFeNi HEA. The composite exhibited enhanced properties, including a high indentation resistance of 679 HV. The resistance to corrosion of the AlCrFeNi-TiO₂ composite exhibited a substantial enhancement, as evidenced by a significant reduction in the corrosion current density (*I*_{corr}). Additionally, the wear tests unveiled improved tribological behavior in the composite, characterized by reduced friction coefficients (CoF) and wear rates. The wear mechanism was abrasive in the alloy and adhesive in the composite.

1. Introduction

High Entropy Alloys (HEAs) are gaining prominence due to their remarkable properties, including exceptional strength, hardness, corrosion resistance, wear resistance, and thermal stability. HEAs feature a complex atomic structure resulting from multiple element combinations, enhancing their mechanical performance. They offer robust solutions for applications requiring resistance to deformation, chemical corrosion, abrasive wear, and elevated temperatures, making them valuable materials across diverse industries [1,2]. To further enhance the properties of HEAs, the incorporation of oxide particles like Al₂O₃, TiO₂, GO, and Y₂O₃ into the HEA matrix has been explored and reported to enhance the properties of HEAs by creating more interfaces, grain boundaries, and dislocation loops [3]. For instance, the introduction of 5 wt% Y₂O₃ into FeCrCoNi-based HEA demonstrated a stable FCC phase and increased strength due to nano-sized yttria particle dispersion [4]. Fe₂₂Co₂₄Cr₂₀Ni₂₃Al₁₁-based HEA composites reinforced with graphene oxide (GO) were fabricated via FAST in a related study. The research revealed increased hardness and wear resistance with GO addition, attributed to GO's grain refinement strengthening and lubrication effect [5].

Among oxide-based reinforcements, TiO₂ particles have emerged as a promising enhancer for augmenting the structural compactness of HEAs. Functioning as a filler within the HEA matrix, TiO₂ effectively enhances the alloy's hardness and strength [6]. Studies conducted by Zhu et al. highlight the positive impact of adding the TiO₂ ceramic phase to CoCrFeMnNi HEA, demonstrating improvements in wear resistance, plastic deformation resistance, and reduced elastic recovery resistance [7]. Similarly, in a work conducted by Qi et al., the strength of AlCoCrFeNi HEA peaked at 489 MPa when reinforced with 2.5 vol% of TiO₂ ceramic phase, marking an 11.1% increase compared to the matrix HEA [6]. Chang et al.'s research, involving the addition of varying amounts of TiO₂ particles, revealed a prevalence of grain refinement and improved strength without significant elasticity loss [8]. These studies position TiO₂ as an attractive candidate for fabricating HEA composites. Despite the identified benefits, limited reports on TiO₂ reinforced HEAs underscore the necessity for further comprehensive investigation.

Strategically eliminating elements from High Entropy Alloys (HEAs) has proven highly impactful on their microstructural characteristics and properties. For example, the exclusion of copper (Cu) from the AlCoCrCuFeNi system, attributed to Cu's magnetic and toxic attributes, has created AlCrFeNi HEAs. This elemental modification has led to

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noteworthy alterations in the alloy's composition and behavior, expanding the possibilities for tailoring HEAs for diverse applications [9, 10]. Munitz et al. studied the consequence of removing Co from AlCoCrFeNi and found that AlCrFeNi HEAs exhibited exceptional strength and elasticity [11]. The AlCrFeNi alloy represents a notable example of a quaternary HEA. Despite its formulation with only four elements, this alloy exhibits unique properties characteristic of HEAs. The exceptional attributes of the AlCrFeNi alloy underscore the nuanced nature of defining alloys as high entropy based on their specific composition and ensuing characteristics. This exemplifies that a quaternary combination of elements can indeed be regarded as a HEA, challenging conventional perceptions and broadening the scope of HEA formulations [12].

Therefore, this study aims to extend the design concept of HEAs by dispersing TiO₂ ceramic particles into the AlCrFeNi HEA matrix. The research investigates the impact of varying TiO₂ reinforcement on the microstructure, wear resistance, and corrosion properties of the AlCrFeNi. Field Assisted Sintering Technique (FAST), an advanced and highly efficient powder consolidation method, is utilized to fabricate the AlCrFeNi-TiO₂ composite. This process involves subjecting the mixed powders to extremely rapid heating by pulsed direct current, enabling the simultaneous application of high pressure for sintering [13].

The benefits of this research lie in the potential enhancement of the already impressive properties of HEAs by incorporating TiO₂ ceramics, which could lead to improved mechanical and corrosion resistance characteristics. The novelty of this work stems from the exploration of a relatively uncharted territory—examining the wear and corrosion properties of AlCrFeNi HEA composites with TiO₂—an area with limited existing research. This research has the potential to expand the application range of HEAs and advance our understanding of their composite variants.

2. Materials and methods

High-purity metallic powders comprising Al, Cr, Fe, and Ni, each contributing 25 at%, were combined with TiO₂ ceramic powder to form a homogeneous composite with a TiO₂ content of 4 wt%. The fabrication process involved several key steps. Mechanical alloying in a QM-3SP4 Planetary Ball Mill was initially performed to achieve a uniform AlCrFeNi-4 wt% TiO₂ powder blend through thorough mixing. The process involved vacuum milling for 24 h at 150 rpm, with a 10 min relaxation time for every 20 min of milling, maintaining a 10:1 ball-to-powder weight ratio. Subsequently, the blended powders were cold-pressed at 10 MPa using a hydraulic press to enhance powder density, improve homogeneity, facilitate sintering, reduce porosity, and enable shape and size control in the final compacted powder precursor. The sintering process was then carried out using a FAST machine (HHPD-25 FCT model, Germany). Sintering conditions included heating to 1100 °C under a pressure of 50 MPa, holding for 10 min, and a heating rate of 100 °C/min, resulting in the final TiO₂-reinforced AlCrFeNi composite.

Microstructural analysis and elemental composition were conducted using Scanning Electron Microscopy (SEM) with an energy-dispersive detector. Crystal structure assessment employed X-ray diffractometry (XRD) with Cu K α radiation, providing insights into the phase composition of the material. FALCON 500 series Vickers hardness tester was used to evaluate the hardness. The samples were subjected to indents across different regions under a 100-gf load for 10 s, and data reliability was guaranteed through 10 repeated tests. Wear tests were conducted in accordance with ASTM G99-05 (2016) standards, utilizing a ball-on-disc setup at room temperature with a varying 5 and 10 N load, 20 Hz frequency, and a sliding distance of 200 m. Parameters such as wear rate, coefficient of friction (COF), and wear track morphology were evaluated and analyzed via SEM. Corrosion behavior assessment employed the potentiodynamic polarization electrochemical method, with the material immersed in a 3.5 wt% NaCl solution for 3600 s to stabilize the open-circuit potential. Potentiodynamic polarization

measurements were executed at a scan rate of 0.002 V/s within a potential range of -1.5 V to $+1.0$ V. The optical microscope (OM) was used to examine the morphology of the corroded surfaces, providing insights into the material's corrosion resistance and behavior. This comprehensive set of experimental procedures and analyses allowed for a thorough characterization of the AlCrFeNi-TiO₂ composite, encompassing its microstructure, crystal structure, mechanical properties, wear behavior, and corrosion resistance.

3. Results and discussion

The utilization of Hume-Rothery rules has significantly contributed to our understanding of the behavior and phase formation within the AlCrFeNi High Entropy Alloy (HEA). These rules rely on empirical factors such as atomic radius difference (δ), mixing enthalpy (ΔH_{mix}), and valence electron concentration (VEC) to predict the HEA's structural characteristics. In the case of AlCrFeNi HEA, the values of ΔH_{mix} (-21.0173 kJ/mol) and VEC (6.75) were instrumental in identifying the Body-Centered Cubic (BCC) crystal structure as the predominant phase. The Thermophysical properties of designed AlCrFeNi HEA is presented in Table 1. The schematic depiction in Fig. 1a illustrates the Mechanical Alloying (MA) process for blending the Al, Cr, Fe, Ni, and TiO₂ powders. The degree of powder dispersion post-MA is evaluated, and Fig. 1b showcases the resulting TiO₂-reinforced AlCrFeNi powder morphology. The micrograph reveals a homogeneous dispersion of individual starting metallic powders with well-integrated TiO₂ reinforcements. The XRD investigation of the MAed powders, as depicted in Fig. 1c, confirms the presence of distinct Bragg peaks corresponding to a BCC crystal structure, consistent with the predicted BCC phase based on the VEC value of 6.75. This experimental validation underscores the accuracy of empirical calculations in predicting HEA structures. The subsequent step involves the consolidation of MAed powders using FAST, as schematically represented in Fig. 1d. The sintering profile, shown in Fig. 1e, indicates the completion of the consolidation process within a concise duration of 30 min, highlighting the efficiency of the FAST technique [14]. The densification behavior of the powder during sintering, as illustrated in Fig. 1f, reveals a noteworthy effect of adding 4 wt% TiO₂ particles to the HEA. This addition leads to a substantial decrease in the shrinkage rate, reducing it from 0.74 to 0.44. This phenomenon is credited to the variance in thermal conductivity between the AlCrFeNi matrix and particles of TiO₂. The lower thermal conductivity of TiO₂ particles hinders electron discharge and local heating, consequently slowing down the melting rate of particle surfaces. Moreover, Fig. 1g demonstrates that while the addition of TiO₂ reduces the overall density of the AlCrFeNi HEA, it significantly increases its hardness. The increase can be linked to the TiO₂ particles acting as impediments to the movement of dislocations within the HEA composites during micro-indentation [15]. The incorporation of ceramic TiO₂ to the AlCrFeNi HEA has a multifaceted effect. It decreases the shrinkage rate during sintering due to thermal conductivity differences, resulting in a slower melting rate. Simultaneously, this addition enhances the HEA composite's hardness by impeding the dislocations' mobility. These findings emphasize the

Table 1
Thermophysical properties of designed AlCrFeNi HEAs.

Parameters	Formula	Reference	Calculated Values for AlCrFeNi
Mixing Entropy (ΔS_{mix})	$-R \sum_{i=1}^n (c_i \ln c_i)$	[16]	11.5257 J/k.mol
Mixing enthalpy (ΔH_{mix})	$\sum_{i=1, j \neq i}^n \Omega_{ij} c_i c_j$	[17]	-21.0173 kJ/mol
Valence electronic configuration (VEC)	$\sum_{i=1}^n c_i (VEC)_i$	[18]	6.75 VEC < 6.87 for BCC
Atomic size mismatch (δ)	$100 \sqrt{\sum_{i=1}^n c_i \left(1 - \frac{r_i}{r_a}\right)^2}$	[17]	2.84

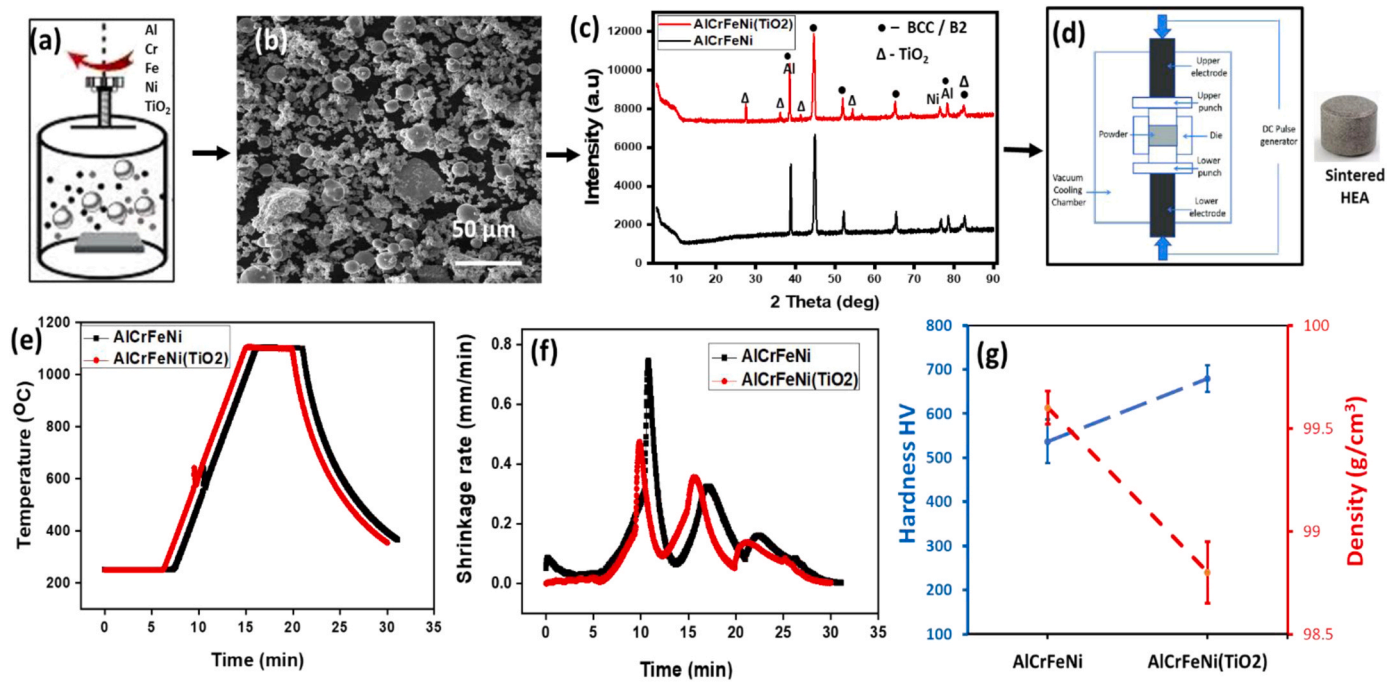


Fig. 1. (a) MA of starting individual powders; (b) SEM of MAed AlCrFeNi(TiO₂); (c) XRD of mechanical alloyed AlCrFeNi(TiO₂) powders; (d) consolidation of MA powders using FAST; (e) Sintering profile; (f) Shrinkage/deformation of AlCrFeNi(TiO₂) powers with time during FAST (g) density and hardness variation.

intricate interplay between ceramic reinforcement and HEA matrix properties, opening avenues for tailoring HEA composites for specific applications with enhanced mechanical characteristics.

Fig. 2(a & b) provides valuable insights into the microstructures of sintered AlCrFeNi and composite, as observed through SEM in secondary electron (SE) and backscattered electron (BSE) modes, respectively. The microstructure of AlCrFeNi (Fig. 2a) reveals a distinct presence of both Body-Centered Cubic (BCC) and B2 phases. Further insights into the elemental compositions, obtained through Energy-Dispersive X-ray Spectroscopy (EDS) analysis in Fig. 2(c) and (d), shed light on the composition of these phases. Precisely, the B2 phase demonstrates an enrichment in Al and Ni (Fig. 2c), whereas the Bcc phase prominently contains Fe and Cr (Fig. 2d). Fig. 2b showcases the microstructure of the AlCrFeNi composite. Notably, an interstitial dark phase rich in Ti is evident between the B2 and BCC phases of the AlCrFeNi matrix. EDS analysis (Fig. 2e) provides a comprehensive elemental distribution, highlighting the presence of Al, Cr, Fe, Ni, and Ti within the composite. This structural identification is consistent with the microstructural observations. The complex processes associated with FAST, such as densification, creep, deformation, and mass diffusion, play a crucial role in the observed microstructure. In particular, the diffusion of titanium and oxygen atoms into interstitial positions within the BCC and B2 crystal lattices results from these intricate thermal and mechanical interactions [19]. The addition of TiO₂ ceramics to the AlCrFeNi HEA matrix profoundly influences the microstructure. The interstitial dark Ti-rich phase observed in the composite signifies the successful incorporation of TiO₂ particles. This phase acts as a reinforcement within the HEA matrix, introducing unique interfacial interactions. Additionally, the presence of TiO₂ is likely responsible for the observed changes in elemental composition.

The XRD examination of the HEA and composite (Fig. 3) further confirms the existence of a TiO₂ phase in the AlCrFeNi matrix. The presence of elemental Ni and Al in the milled HEA powders was acknowledged in Fig. 1c, but it's crucial to note that these elements were absent in the HEA after sintering. The primary objective is to achieve complete alloying after sintering, ensuring the final product is devoid of elemental components and possesses the desired homogeneous alloy

composition. The XRD peak indexing, including Ni and Al in the milled powder, reflects an intermediate stage in the alloying process, and the absence of these elements after sintering affirms the successful attainment of the targeted homogeneous alloy structure.

Fig. 4(a) provides insights into the coefficient of friction (CoF) of both the AlCrFeNi and TiO₂ reinforced composite samples when subjected to loads of 5 and 10 N. Notably, the inclusion of TiO₂ leads to a reduction in CoF regardless of the applied load. Additionally, the decrease in CoF is more pronounced at lower wear loads. To provide a comprehensive comparison, the average CoF of the samples is calculated and presented in Fig. 4(b). It is evident that the TiO₂-reinforced AlCrFeNi composite exhibits a slightly lower CoF compared to AlCrFeNi. This phenomenon can be attributed to the higher hardness of the composite in comparison to AlCrFeNi. The increase in hardness plays a pivotal role in diminishing the contact area during sliding, which enhances wear resistance. The requirement for more energy for surface degradation during sliding results from the reduced contact area, contributing to the improved wear resistance observed in the composite [20]. Fig. 4(c) illustrates the wear rate of the HEA samples, which is noticeably reduced with the addition of TiO₂. This reduction indicates an enhancement in wear resistance when TiO₂ is incorporated into the HEA matrix. The microstructure of the worn surfaces for both the AlCrFeNi and composite samples is presented in Fig. 4(d-e). In the case of AlCrFeNi, the wear tracks display smooth grooves with no delamination, indicating an abrasive wear mode. Conversely, adhesive wear is the dominant mechanism observed in the TiO₂ composite. Adhesive wear leads to surface plastic deformation, resulting in work hardening and increased wear resistance. This is attributed to the improved properties of the strengthened composite, which can better withstand the forces associated with adhesive wear [21].

Fig. 4(f & g) presents SEM images of the wear debris from the AlCrFeNi and composite samples. The wear debris observed in the base HEA (Fig. 4 f) is characterized by larger particles, suggesting substantial plastic deformation within the base alloy during wear. In contrast, the wear debris in the composites (Fig. 4 g) exhibits a finer morphology than the base HEA. This finer particulate structure indicates milder wear in the composite samples. The presence of smaller particles can be

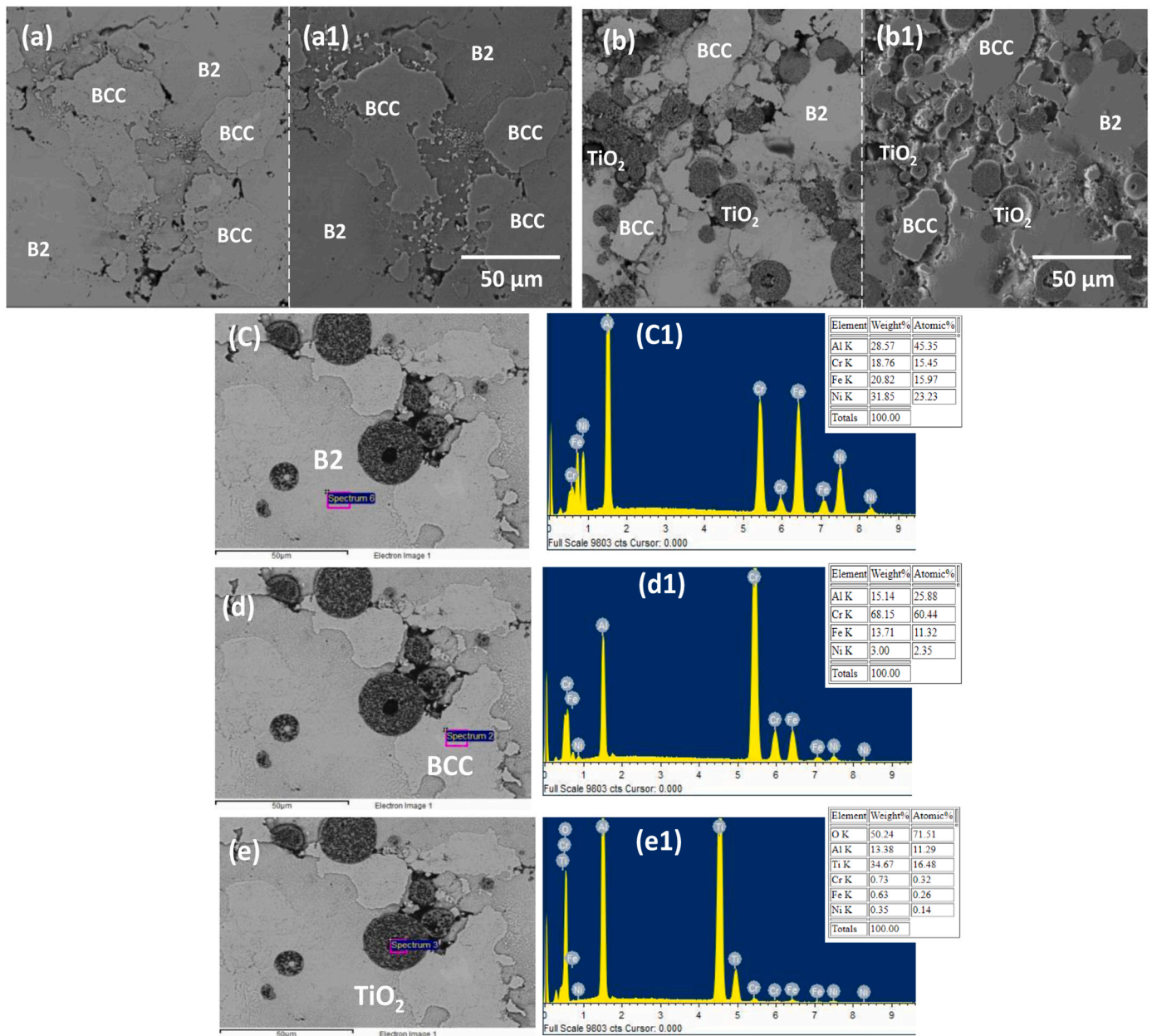


Fig. 2. (a) SE and BSE of sintered AlCrFeNi; (b) SE and BSE of sintered AlCrFeNi(TiO₂); (c), (d) and (e) EDS analysis of B2, BCC, and TiO₂ phases respectively.

attributed to the reinforcing TiO₂ dispersed in the matrix, effectively scattering and minimizing the size of the wear debris, thereby mitigating the severity of wear in the composite.

The corrosion properties of the AlCrFeNi and AlCrFeNi-TiO₂ samples involved recording Open Circuit Potential (OCP) curves, as depicted in Fig. 5(a), before conducting potentiodynamic polarization measurements. These preliminary OCP curves were essential to confirm the attainment of a steady-state potential. Fig. 5(b) presents the alloy and composite's potentiodynamic polarization behaviors in a 3.5 wt% NaCl electrolyte. Electrochemical kinetic parameters were subsequently determined through the Tafel extrapolation technique, with results provided in Table 2. Upon careful analysis of the polarization curves, it becomes evident that the TiO₂-reinforced AlCrFeNi exhibits a notably enhanced corrosion resistance compared to the AlCrFeNi alloy. This improvement is particularly noteworthy in the order of magnitude reduction in the corrosion current density (I_{corr}), which decreases from $2.108 \times 10^{-6} \text{ A/cm}^2$ for AlCrFeNi to $1.347 \times 10^{-6} \text{ A/cm}^2$ for AlCrFeNi-TiO₂. The incorporation of TiO₂ ceramics into the AlCrFeNi

HEA matrix has a profound impact on its corrosion properties. The reduced corrosion current density (I_{corr}) suggests a significant enhancement in corrosion resistance. This improvement can be attributed to the role of TiO₂ in creating a protective barrier against corrosion, thus reducing the susceptibility of the composite to localized corrosion regions. Fig. 5(c) and (d) offer visual insights into the alloy and composite corroded surfaces, respectively, when exposed to the 3.5 wt% NaCl solution. In the instance of AlCrFeNi, the manifestation of dark corrosion regions signifies the alloy's inherent susceptibility to corrosion. AlCrFeNi-TiO₂ exhibits diminished areas of corrosion, a phenomenon attributable to the shielding influence exerted by the titanium-rich phase embedded within the alloy, effectively mitigating the corrosive impact. The observation of dark corrosion byproducts in the AlCrFeNi contrasts the identification of a white protective film on AlCrFeNi reinforced with TiO₂, indicative of titanium-rich phases. This discrepancy in visual characteristics underscores the pivotal role played by the Ti-rich phases in impeding corrosion mechanisms within the HEA.

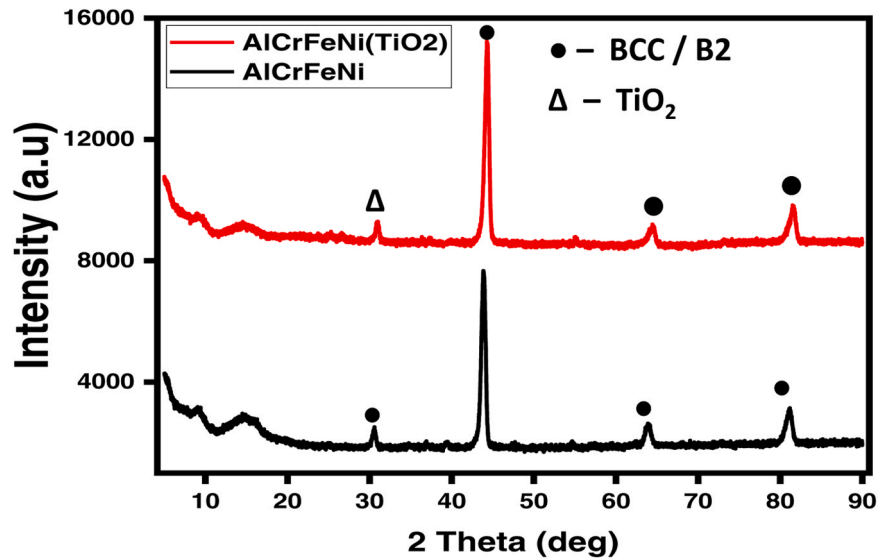


Fig. 3. XRD of sintered AlCrFeNi and AlCrFeNi(TiO₂).

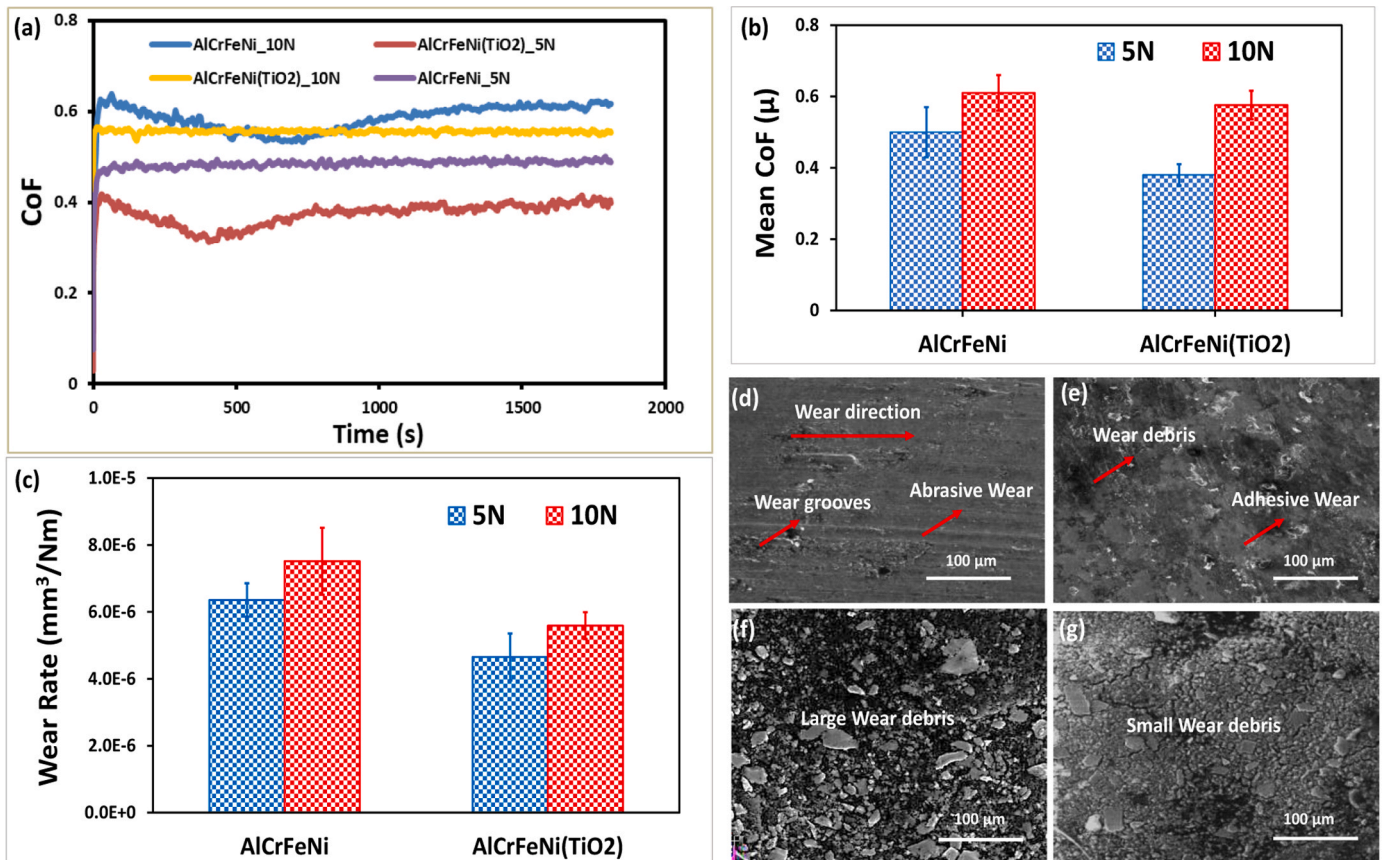


Fig. 4. (a) and (b) Variation in CoF and mean CoF at 5 and 10 N loads, respectively; (c) calculated wear rates at 5 and 10 N loads; (d-e) SEM images of wear tracks at 10 N load; (f-g) SEM of wear debris of the AlCrFeNi and AlCrFeNi(TiO₂).

4. Conclusion

The study uses FAST to incorporate TiO₂ ceramic particles into the AlCrFeNi High Entropy Alloy (HEA) matrix. This addition significantly impacts the microstructure and properties of the resulting alloy. The introduction of TiO₂ leads to a reduction in the rate of powder shrinkage during FAST, resulting in decreased density. The microstructure of the

AlCrFeNi HEA consists of a disordered Body-Centered Cubic (BCC) phase and an ordered B2 phase. With TiO₂ incorporation, a Ti-rich BCC phase is evident. This complex microstructure contributes to enhanced hardness, improved wear resistance, and better corrosion properties in the composite. The addition of TiO₂ introduces unique characteristics, allowing for tailored HEA composites suitable for various applications. This research underscores the potential for advanced materials

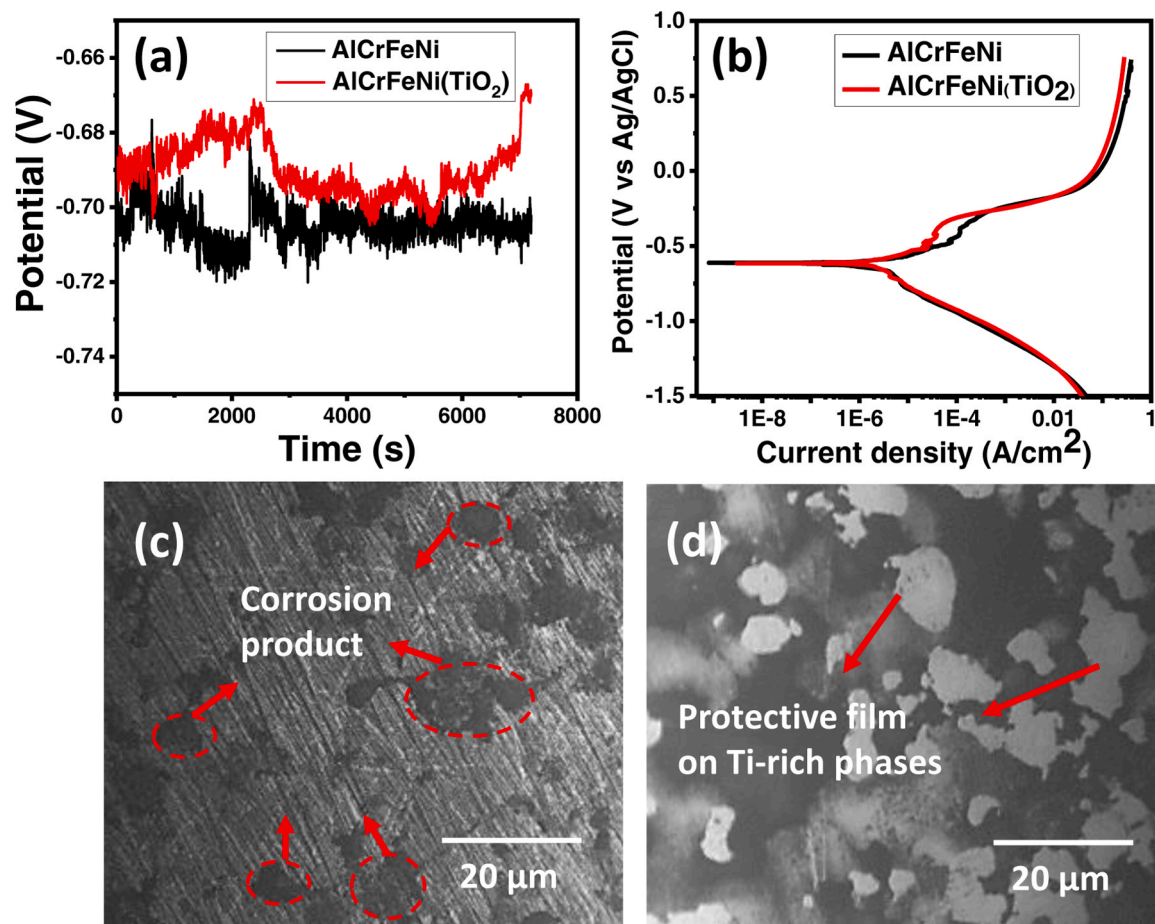


Fig. 5. (a) OCP curves; (b) Potentiodynamic polarization curves in 3.5 wt% NaCl solution (c) and (d) optical images of corroded alloy and composite surfaces after immersion in 3.5 wt% NaCl solution.

Table 2

Electrochemical data of sintered AlCrFeNi and AlCrFeNi-TiO₂ in 3.5 wt% NaCl solution.

samples	E _{corr} (mV)	I _{corr} (µA)	Cathodic beta (mV)	Anodic beta (mV)
AlCrFeNi	-639.163	2.108×10^{-6}	188.317	109.161
AlCrFeNi (TiO ₂)	-612.556	1.347×10^{-6}	159.807	137.853

engineering through ceramic additives in HEAs, opening new avenues for designing high-performance materials with superior mechanical and corrosion resistance properties.

CRediT authorship contribution statement

Falodun Oluwasegun Eso: Methodology, Writing – review & editing. **Bayode Abiodun:** Funding acquisition, Resources, Supervision. **Oke Samuel:** Conceptualization, Formal analysis, Investigation, Result analysis, Writing – original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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