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Understanding the role of Si alloying on the structural, mechanical, wear and high temperature oxidation behavior of CrFeNiTiX (X=Si) high entropy alloys

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ABSTRACT

The present study explores the structural, mechanical, wear, and high temperature oxidation behavior of CrFeNiTiX(X = Si) high entropy alloys (HEAs) processed by powder metallurgy. The results revealed the formation of single-phase body centered cubic (BCC) structure in both alloys after 30 h of milling. After sintering, the Si added HEA exhibited an increased fraction of the BCC phase and promotes the formation of Cr₃Si phases. The addition of Si enhances the microhardness (1200–1330 HV), ultimate compressive strength (1400 \pm 80 MPa to 1700 \pm 50 MPa), nanohardness (12–15.7 GPa) and elastic modulus (12–15.7 GPa) attributed to the solid solution strengthening resulting from the lattice distortion. The Si added HEA showed reduced specific wear rates under all the applied loads, attributed to increased hardness and surface oxidation, which resists plastic deformation. Moreover, the oxidation resistance was enhanced by the addition of Si up to 800 °C, attributed to the formation of a protective oxide layer on the surface. However, at 900 °C, the resistance decreased due to spallation of the oxide layer. Therefore, the present study demonstrates the addition of Si improves the hardness, wear resistance, and oxidation resistance, making HEAs are suitable for high-temperature and wear-resistant applications.

1. Introduction

The revolutionary alloy design called high entropy alloys (HEAs) have recently drawn global attention in materials engineering due to their unique microstructure and remarkable properties such as high strength-ductility, good thermal stability, excellent wear, and corrosion resistance [1,2]. Unlike conventional alloys, HEAs are composed of five or more principal elements with a mixing entropy >1.61 R, which tends to form thermodynamically stable solid solutions such as face-centered cubic (FCC), body-centered cubic (BCC), and hexagonal close-packed (HCP) phases [3]. In addition, most of the HEAs are prepared by conventional melting routes; however, these methods show certain disadvantages, including elemental segregation and compositional inhomogeneity [4]. To overcome these challenges, mechanical alloying

(MA) has been considered as a promising route for producing HEAs with compositional homogeneity and fine-grained microstructures [5,6]. Additionally, spark plasma sintering (SPS) is a potential method for compacting milled powders into fully dense materials in short time intervals while maintaining the initial grain sizes by controlling the pressure and temperature [7]. Therefore, the combination of MA and SPS has been widely used to produce high-performance HEAs with potential mechanical properties for various structural applications [8].

Indeed, developing materials with superior wear resistance is crucial for structural applications such as automotive, aerospace, and manufacturing sectors because wear resistance plays a significant role in material lifespan, environmental sustainability, and reliability of structural components [9]. In recent years, numerous efforts such as alloying, surface coatings, heat treatments, surface modifications, composites,

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and lubrications have been developed to enhance the wear resistance of HEAs [10]. For example, Wu et al. [11] observed the transition in the wear mechanism from delamination wear to oxidative wear whereas increasing Al content in AlxCoCrCuFeNi HEA. Hsu et al. [12] found the major wear mechanism to be abrasive wear in AlCoCrFeMo_{0.5}Ni HEA, while Chuang et al. [13] observed it to be delamination wear in Alx-Co1.5CrFeNi1.5Tix HEAs. It has been observed that the addition of nonmetallic Si into HEA significantly affects strength, hardness, and resistance to abrasion and wear [14]. Recently, Lee et al. [15] found that an improved hardness and compressive strength with an increase in Si content in CoFeNiAlSix HEA. In addition, they found that the addition of Si increases the lattice distortion and transition in phase from FCC + BCC to BCC and BCC + B2. Chen et al. [16] found the formation of the BCC phase and grain refinement by the addition of Si in Al_{0.4}CoCu_{0.6}Ni HEA, which improved the hardness and ultimate fracture strength. Kumar et al. [17] observed that increasing Si content in CoCrCuFeNiSix HEAs led to the enhancement of hardness and wear resistance, which could be attributed to the solid solution strengthening induced by atomic radii mismatch between Si and constituent elements. Besides, the addition of Si can form a protective layer on the worn surface due to their high affinity toward oxygen, showing lubricant behavior, thereby reducing the friction between contacting surfaces, and inhibiting the plastic deformation or delamination near the surface layers of wear tracks. Considering the several advantages of Si element, the present study investigated the friction and wear behavior of CrFeNiTiX (X = Si) HEAs under dry-sliding conditions by varying the normal loads.

On the other hand, exploring the oxidation resistance of novel HEAs is crucial for high-temperature applications, particularly in oxidizing conditions, because surface oxidation can deteriorate mechanical properties and cause premature failure [18]. Thus far, AlxCoCrFeNi HEA has been proven a promising candidate for superior oxidation resistance in the range of temperature from 900°C to 1100 °C. It was noted that the oxidation resistance increased with an increase in Al content due to the formation of an Al₂O₃-rich oxide layer rather than that of a Cr₂O₃-rich layer [19,20]. Shaburova et al. [21] found that Al_{0.25}CoCrFeNiCu has the best high-temperature oxidation stability due to a protective Al₂O₃-rich film, whereas Ti- and V-doped alloys perform poorly because they form loose transition metal oxide layers instead of a continuous alumina film. Hou et al. [22] found that increasing Cr concentration in HEA led to the formation of a dense Cr₂O₃ layer at 600 °C, which improved oxidation resistance by preventing oxygen penetration along grain boundaries. Chang et al. [23] discovered that increasing Cr content in FeCrxCoNiB coatings improves high-temperature oxidation resistance by transitioning the oxidation mechanism from Fe to Cr, ultimately forming a continuous Cr₂O₃ layer. On the other hand, Kai et al. [24] observed an increase in oxidation resistance in FeCoNiCrSi_x HEAs with the addition of Si due to the formation of intermixed scale-layer of Cr₂O₃ and SiO₂. Li et al. [25] found that enhanced oxidation resistance in AlCoCrFeNiSi_{0.2} HEA by forming a dense Al₂O₃ scale. However, excessive Si content reduced the oxidation resistance. Shaik et al. [26] found that adding Si to CoCrCuFeNiSix HEA enhances the oxidation resistance up to 0.6% Si, but further increases in Si content reduced the oxidation resistance. Chen et al. [27] reported the addition of Si enhanced the oxidation resistance in $Al_{0.6} CrFeCoNiSi_{0.3}$ HEA at 800 $^\circ C$ by forming a dense Al₂O₃ layer; however, it did not show a positive effect on the oxidation resistance of the HEA at 900 °C and 1000 °C. Al_{0.25}CoCrFeNiMn HEA oxidized at 900 °C. Moghaddam et al. [28] found that the $Al_{0.45}$ CoCrFeNiSi_{0.45} HEA has better oxidation resistance because it forms a protective Al2O3-SiO2-rich oxide layer. In contrast, the $Al_{0.25}$ CoCrFeNiMn HEA forms a non-compact oxide layer with transition elements, which does not offer sufficient protection against oxidation at high temperatures. Based on the literature, it has been demonstrated that the addition of Si, Cr and Al are active elements for oxidation, and thus form a dense oxide layer such as Al₂O₃, Cr₂O₃ and SiO₂ on the material's surface and thus suppresses the diffusion of oxygen into materials. Despite, Si element having positive effect on

enhancing the oxidation resistance, it's not always straight forward for all the alloy systems with temperature. Therefore, evaluating the oxidation resistance of novel CrFeNiTiX (X = Si) HEAs is noteworthy.

In this study, equiatomic CrFeNiTiX (X = Si) HEAs were prepared by the powder metallurgy route. The primary objective of this study is to examine the effect of Si addition on the phase composition, microstructure, and mechanical properties of HEAs. Ball-on-disk sliding tests were performed at room temperature (RT) to explore the friction and wear behavior of CrFeNiTi (Si) HEAs at different loads. In addition, the oxidation resistance of HEAs was investigated in the temperature range of 700 °C-900 °C. Furthermore, the correlation among the microstructural characteristics of HEAs after wear and oxidation tests was determined to understand the corresponding mechanisms.

2. Experimental process

2.1. Alloy design and fabrication

Commercially purchased high-purity (99.99%) raw elemental powders of Cr, Fe, Ni, Ti, and Si with a particle size of $<45 \,\mu m$ were used as starting materials. The weighing was performed according to the stoichiometric ratio to achieve equiatomic compositions, namely, CrFeNiTi and CrFeNiTiSi alloys. The constituent elemental powders were placed into stainless steel vials along with balls at a ball-to-powder weight ratio of 10:1 inside a glove box filled with argon gas. The powder processing was conducted under an argon atmosphere to prevent contamination. The milling process was conducted for 30 h using a planetary miller (Retsch PM-400 MA, Germany) at a rotational speed of 350 rpm. To reduce the cold-welding of powder during milling, stearic acid (0.3 wt %) was used as a process control agent. The crystal structure and powder morphology of HEA were observed every 10 h of milling. Subsequently, the milled powders were sintered using SPS (DR. SINTER LAN Jr, SPS-332Lx) at 950 °C for 8 min under a pressure of 40 MPa with a heating rate of 100 $^{\circ}$ C/min in vacuum condition (-0.1 MPa). The detailed experimental steps in the fabrication of CrFeNiTiX (X = Si) HEA powders by MA and subsequent SPS can be schematically represented in Fig. 1.

2.2. Materials characterization

The crystal structure of the powder and sintered HEAs were examined by X-ray diffraction (XRD, Bruker D8 Advance X-ray diffractometer, $\lambda = 1.540598$ Å). The microstructures of the powder and sintered HEAs were observed using scanning electron microscopy (SEM, JSM- 7500F JEOL, Japan). Additionally, high-resolution transmission electron microscopy (HRTEM, JEM-2100 F instrument by JEOL, Japan) was utilized to analyze the phase and microstructural properties of both the powder and sintered HEAs. The hardness of the sintered HEAs were assessed using a micro-Vickers hardness tester (HM-200, Mitutoyo, Japan) with an applied load of 100 gf for a duration of 10 s. Moreover, nanoindentation measurements were conducted employing a Berkovich indenter, applying a load of 100 mN, and maintaining a loading and unloading rate of 600 mN/min for a duration of 20 s. Cylindrical specimens (3 mm diameter \times 4.5 mm height) were prepared using wire electrical discharge machine (WEDM) to measure the compressive properties of bulk HEAs. The compressive stress-strain curves of HEAs were measured using the MTDI MINOS-100S equipment at a strain rate of 1×10^{-3} s⁻¹. To ensure experimental accuracy, three measurements were conducted, and average results are presented.

2.3. Wear and oxidation characterization of HEAs

The coefficient of friction and wear behavior of HEAs were investigated using the ball-on-disk wear tester under dry-sliding conditions by varying the normal load from 5 to 10 and 15 N at a constant sliding time of 10 min and a velocity of 6 cm/s. The detailed wear test conditions are given in Table 1. For the dry-sliding tests, the sintered HEAs were



Fig. 1. The schematic diagram shows the fabrication of alloy powder and following sintering process.

Table 1

Operating parameters of the ball-on-disk wear tester performed in dry-sliding conditions.

Wear tester	Counterpart	Variable load (fixed conditions)	Temperature
Reciprocating ball-	WC (12.7	5, 10, and 15 N (10 min	298 K
on-disk	mm)	and 0.06 m/s)	

prepared with a diameter and height of 20 and 4 mm, respectively. For the wear tests, tungsten carbide (WC) ball was utilized as a counter material to slide against the substrate of the HEA specimen. For all sliding tests, the stroke length was fixed at 6 mm. After the wear tests, the loss of wear volume was estimated by analyzing the wear track geometry using a surface profilometer. The debris morphology, wear tracks, and compositional behavior were investigated by SEM-energy dispersive X-ray spectroscopy (SEM-EDS) analysis. Further, the oxidation behaviors of CrFeNiTi and CrFeNiTiSi specimens were assessed using a thermogravimetric analyzer (TGA-6000, PerkinElmer). For the oxidation tests, the specimens were prepared using the wire electrical discharge machine to make the sample with a precise dimension of about 3 mm diameter and 1 mm thickness. Specimens were precisely prepared with dimensions of approximately 3 mm in diameter and 1 mm in thickness using a wire electro discharge machine. The oxidation tests were performed at different temperatures such as 700 °C, 800 °C, and 900 °C for 30 h. To ensure repeatability, we performed oxidation tests twice for each condition. For the oxidation tests, nitrogen gas was used as a protective atmosphere at a flow rate of 20 ml/min to shield the specimens from environmental exposure. After the oxidation tests, the surface morphology and compositional behavior of the specimens was examined using SEM-EDS analysis.

3. Results and discussion

3.1. Calculation of thermodynamic parameters

The formation of solid solution phases in HEAs is influenced by various thermodynamic parameters [29,30]. From the literature, it has been mentioned that the criteria for the formation of solid solution phases in HEAs, the thermodynamic parameters would be in the following ranges. For instance, mixing entropy (ΔS_{mix} , 12.47 $\leq \Delta S_{mix} \leq$ 19.5 J/(K·mol), mixing enthalpy (ΔH_{mix} , -15 kJ/mol $< \Delta H_{mix} < 5$ kJ/mol), atomic size difference (δ , 0 $< \delta <$ 6.6%), valence electron concentration (VEC; if VEC \leq 6.87; BCC, 6.87 \leq VEC \leq 8; FCC + BCC, VEC \geq 8; FCC) and the combination of mixing entropy and mixing

enthalpy is termed as Ω ($\Omega \ge 1.1$). The corresponding thermodynamic parameters can be estimated using the following equations:

$$\Delta S_{mix} = -R \sum_{i=1}^{n} C_i \ln C_i \tag{1}$$

$$\Delta H_{mix} = \sum_{i=1, i\neq j}^{n} 4\Delta H_{AB}^{mix} x_i x_j$$
⁽²⁾

$$\delta = 100 \sqrt{\sum_{i=1}^{n} c_i \left(1 - \frac{r_i}{\overline{r}}\right)^2} \tag{3}$$

$$VEC = \sum_{i=1}^{n} C_i (VEC)_i \tag{4}$$

$$\Omega = \frac{T_m \Delta S_{mix}}{|\Delta H_{mix}|} \tag{5}$$

Table 2 lists the estimated thermodynamic parameters suggesting that ΔS_{mix} and ΔH_{mix} increased by Si addition, while the VEC parameter decreases by Si alloying owing to the lower VEC of Si compared to other elements. In addition, the δ between the constituent elements increased significantly, suggesting that severe lattice distortion occurred by Si addition. Based on the estimated ΔS_{mix} values, the CrFeNiTi alloy fall under medium entropy, while the CrFeNiTiSi alloy found to be HEA. With Si addition, increases the negative mixing enthalpy, and atomic size difference, leading to form number of phases when compared to the CrFeNiTi MEA. Also, the Ω doesn't meet the criteria for the alloy formation in the present study.

3.2. Powder morphology of HEAs prepared by MA

The powder morphologies of CrFeNiTi and CrFeNiTiSi HEAs prepared by MA at different durations were examined using SEM analysis as shown in Fig. 2. The morphology of CrFeNiTi alloy powder before the milling appeared irregular as shown in Fig. 2(a). During the initial stages of MA after 10 h, the particle morphology showed a plate-like morphology or flattening behavior due to compressive forces arising

Table 2

Calculated thermodynamic parameters for the design of CrFeNiTi MEA and CrFeNiTiSi HEAs.

Alloy	ΔS_{mix} (KJ/mol)	ΔH_{mix} (KJ/mol)	VEC	Ω	δ (%)
CrFeNiTi	11.52	$-17.25 \\ -39.52$	7.0	1.09	6.52
CrFeNiTiSi	13.38		6.4	0.54	14.68



Fig. 2. Powder morphology of (a-d) CrFeNiTi MEA and (e-h) CrFeNiTiSi HEAs fabricated by MA at 0, 10, 20, and 30 h, respectively.

from the milling balls. Consequently, the morphology of powder particles significantly increased compared to the raw powders as shown in Fig. 2(b). Upon further increasing the milling time to 20 h, some of the flake-shaped particles were refined and their thickness also decreased as shown in Fig. 2(c). Finally, after milling at 30 h, the deformation of the particles was more pronounced, resulting in the agglomerated and flattened particles being refined, and homogeneous morphology was observed as shown in Fig. 2(d). This refinement of powder particles was mainly attributed to the extent of work hardening and increased brittleness of powders activating the fracture mechanism at longer milling times. Similarly, Fig. 2(e)–(h) show the powder morphology of CrFe-NiTiSi HEA fabricated under the same conditions. Similar morphological changes were observed in the CrFeNiTi alloy because of common elements, except Si. After 30 h of milling, the particle size significantly

decreased compared to that in CrFeNiTi alloy owing to the increased refinement caused by the brittle nature of Si.

3.3. Crystal structure analysis of powder and sintered HEAs

The XRD patterns of CrFeNiTi MEA and CrFeNiTiSi HEAs were investigated with respect to milling duration and the subsequent SPS route as shown in Fig. 3(a) and (b), respectively. All the elemental peaks were observed before milling (0 h). After milling for 10 h, peaks related to Cr and Ti were found to appear in both alloy systems owing to their higher atomic size and melting points. When the milling time was increased to 20 h, most of the element peaks vanished except Ti related peaks, because of its larger atomic radius takes longer milling time for the alloying. Further increase in milling time up to 30 h revealed a



Fig. 3. XRD patterns of (a) CrFeNiTi MEA and (b) CrFeNiTiSi HEAs fabricated by MA and following the SPS process, estimated crystallite size and lattice strain of (c) CrFeNiTi MEA and (d) CrFeNiTiSi HEA powders as a function of milling time.

formation of single-phase BCC solid solutions in both alloys. The formation of single phase through milling suggesting the MA is an effective technique to produce homogeneous composition. In addition, the peak broadening was observed in both the alloys after 30 h of milling indicates a reduction in crystallite size, increased lattice strain, and decreased crystallinity.

To observe the crystal structure evolution in both the CrFeNiTi MEA and CrFeNiTiSi HEAs after sintering, we plotted the XRD patterns of bulk samples along with the milled powders, as shown in Fig. 3(a) and (b), respectively. It was found that the CrFeNiTi MEA comprised multiple phases after SPS, including FCC phase (CrFeNi-rich), BCC phase (CrFerich), Ni₃Ti phase TiC phases, as shown in Fig. 3(a). The formation of FCC, BCC and Ni₃Ti phases were consistent with the Gao et al. report [31]. In addition, the Si-added HEA exhibits the phases with similar composition including FCC (CrFeNi-rich), BCC (CrFe-rich) and TiC phases (Fig. 3(b)). The addition of Si reduces the fraction of FCC phase and increase the fraction of BCC phase, also promote the Cr₃Si cubic phase. The formation of Cr₃Si phase with Si addition was consistent with Tan et al. report [32]. The precipitation of the TiC phase is due to the decomposition of stearic acid, which was used as a process control agent during milling, and the use of a graphite die during SPS. Among the constituent elements, Ti typically has the highest affinity for C, thus reacting with C obtained from the decomposed stearic acid and the graphite die, leading to the formation of the TiC phase after sintering. A similar formation of TiC phase without C content was also observed in CrFeNiTiV HEA [33]. To further confirm the phase formation in both alloys, TEM analyses was performed and discussed in the later sections.

Further, the estimated crystallite size and lattice strain of CrFeNiTi MEA and CrFeNiTiSi HEAs through the Williamson–Hall equation with milling time was presented in Fig. 3(c and d), respectively. The CrFeNiTi MEA showed a significant decrease in crystallite size from 34.1 to 17.6 and 9.59 nm and increased lattice strain from 0.26 to 0.56 % and 0.71%, when the milling time was increased from 10 to 20 and 30 h, respectively. Similarly, CrFeNiTiSi HEA showed a decrease in crystallite size from 33.2 to 14.12 and 7.2 nm, while the lattice strain increased from 0.27 % to 0.58 % and 0.74%, upon increasing the milling time from 10 to

20 and 30 h, respectively. The significant decrease in crystallite size with increasing milling time was mainly attributed to the increased plastic deformation and a fraction of grain boundaries caused by grain refinement. Furthermore, after 30 h of milling, the crystallite size of the CrFeNiTi MEA decreased by approximately 72% and that of the CrFeNiTiSi HEA decreased by around 78%. Thus, the presence of Si can facilitate particle fragmentation because of its brittle nature.

Further, the lattice parameters were calculated using X-ray diffraction data by determining the d-spacing values from high-intensity peaks in the XRD patterns. These d-spacing values were then used with Bragg's law and relevant crystal structure equations to derive the lattice parameters, as shown in Table 3. The lattice parameters of FCC and BCC phases are slightly reduced by Si addition into CrFeNiTi alloy. The Similar reduction in lattice parameter with Si addition was observed in previous literature [15]. Meanwhile, the estimated phase fractions were presented in Table 3 for both the alloys. It has been observed that the addition of Si increases the fraction of BCC phase and promote the Cr₃Si phase with a fraction of 32.58 %, which are effective for enhancing the strength and hardness of HEAs.

To further confirm the crystal structure in both the CrFeNiTi MEA and CrFeNiTiSi HEA powders after MA at 30 h, TEM analysis was performed, corresponding results are illustrated in Fig. 4 (a) and 4(d), respectively. It is evident the formation of the nanocrystalline structure

Table 3

Calculated lattice parameters of different phases in sintered CrFeNiTiX (X = Si) HEAs.

Alloy	Phase and fraction (%)	Lattice parameter (Å)
CrFeNiTi	FCC (~32.7)	a = 3.635
	BCC (~ 20.69)	a = 2.89
	$N_{13}T_1 (\sim 28.24)$	a = 5.11 c = 8.304
	TiC (~18.37)	a = 4.322
CrFeNiTiSi	FCC (~21.42)	a = 3.608
	BCC (~28.32)	a = 2.88
	Cr ₃ Si (~32.58)	a = 0.388
	TiC (~17.68)	a = 4.321



Fig. 4. TEM image of (a) CrFeNiTi and (d) CrFeNiTiSi HEA powders prepared by MA at 30 h. (b, e) SAED patterns obtained from the white dotted circles. (c, f) HRTEM images obtained from the marked regions in (a) and (b) showing the estimated d-spacing.

after 30 h of milling in both alloys. The selected area electron diffraction (SAED) patterns in Fig. 4(b) obtained from the dotted circle in Fig. 4(a) indicate the formation of a single-phase BCC solid solution after milling. The HRTEM image in Fig. 4(c) shows the formation of nanocrystalline structures with a crystallite size of approximately 9 nm and measured d-spacing of around 1.447 Å corresponding to the (200) plane. Meanwhile, Fig. 4(d) shows the formation of nanocrystalline structures with the BCC solid solution even after adding Si into the CrFeNiTi MEA. It is further confirmed by the SAED patterns in Fig. 4(e) obtained from the dotted circle in Fig. 4(d). Moreover, Fig. 4(f) shows the HRTEM image confirms the formation of nanocrystalline structures with a crystallite size of ~7 nm and a measured d-spacing of approximately 1.183 Å corresponding to the (211) plane.

3.4. Microstructural characteristics of sintered HEAs

The microstructural characteristics of both the CrFeNiTi MEA and

CrFeNiTiSi HEA bulks were examined by field emission SEM (FESEM) backscattered electron (BSE) mode. Fig. 5(a) shows the cross-sectional microstructure of CrFeNiTi MEA reveals a fully dense microstructure without any porosity. It is evident that the formation of multiple phases with different contrasts and uniformly distributed throughout the matrix. These phases were examined using the EDS point analysis and presented in Table 4. Based on the EDS results, it is confirmed that light grey regions are corresponds to the FCC phase, the dark grey regions are associated with BCC phase, the white regions belong to the Ni₃Ti phase, and the dark particles are related to the TiC phases. These distinct phases are uniformly distributed throughout the microstructure. Meanwhile, Fig. 5(b) illustrates the microstructure of the CrFeNiTiSi HEA consisting of four different phases. It seems that the addition of Si showed a significant impact on phases formation. To reveal the phases formed in the CrFeNiTiSi HEA, EDS analysis was performed and presented in Table 4. It is evident that light-grey region belongs to FCC phase, the dark grey region corresponds to the BCC phase, white regions are associated with



Fig. 5. Cross-sectional microstructures of sintered (a) CrFeNiTi MEA and (b) CrFeNiTiSi HEAs examined by FESEM, corresponding phases represented by different colors.

Table 4

Compositional behavior of CrFeNiTi MEA and CrFeNiTiSi HEAs examined by SEM-EDS analysis.

	Phase Elemental composition (at. %)						
Sample		Cr	Fe	Ni	Ti	Si	С
CrFeNiTi	FCC	32.12	29.26	26.14	12.32	-	0.16
	BCC	36.63	35.27	12.99	14.83	-	0.28
	Ni ₃ Ti	7.92	13.62	59.51	18.53	-	0.42
	TiC	1.71	2.02	0.77	69.47	-	26.03
CrFeNiTiSi	FCC	28.92	25.36	24.51	9.41	11.62	0.18
	BCC	38.30	32.11	9.08	9.14	12.06	0.31
	Cr ₃ Si	49.82	13.87	11.26	6.44	18.35	0.26
	TiC	3.65	2.41	1.47	61.82	1.01	29.64

Cr3Si phase, and the black spots are related to the TiC phases. It is worth noting that the addition of Si reduces the fraction FCC phase and increases the fraction of BCC phase and promotes the formation of Cr₃Si phase. The presence of BCC, Ni₃Ti, Cr₃Si and TiC phases show a promising role in enhancing the strength and hardness of materials.

Further, the phase and microstructural characteristics of CrFeNiTi MEA and CrFeNiTiSi HEA bulks were examined using TEM analysis. Fig. 6 (a) shows the TEM image of CrFeNiTi MEA, indicating the formation of multiple phases including FCC, BCC, Ni_3Ti , and TiC phases. The corresponding phases were represented with different colors and confirmed using SAED patterns, as shown in Fig. 6(b1-b4). The formation of the FCC phase was confirmed by SAED pattern in Figs. 6 (b-1),

suggesting the crystallization in the direction of $[0\ \overline{1}1]_{FCC}$ While Figs. 6 (b-2) shows the SAED pattern of BCC phase, showing the crystallized in the $[\overline{1}11]_{BCC}$ direction. In addition, Figs. 6(b-3) shows the precipitation of Ni₃Ti phase was confirmed by SAED pattern, showing the crystallization in the direction of $[0\overline{1}2]_{Ni_3Ti}$. The precipitation of Ni₃Ti phase is due to its large negative mixing enthalpy between Ni and Ti elements (-35 kJ/mol), which is a stable phase and effective for enhancing the strength of materials. Furthermore, the TiC phase was confirmed by SAED pattern in Figs. 6 (b-4), which crystallized along the $[\overline{1}01]_{TiC}$ direction. Meanwhile, the HRTEM image was taken from the red square region in Fig. 6(a), to provide structural insights into the different phase boundaries. The different phase boundaries showing strong interfacial bonding with different crystallographic orientations. The presence of FCC, BCC, and Ni₃Ti phases were further confirmed by the Fast Fourier Transform (FFT) patterns, as shown in the inset of Fig. 6(c), along with the measured d-spacing values. The measured d-spacing of the Ni₃Ti phase is about 2.202 Å along the (200) plane, but the d-spacing of remaining phases couldn't measure due to lattice distortion.

On the other hand, Fig. 6 (d) shows the TEM image of CrFeNiTiSi HEA with the formation of multiple phases represented by different color arrows. It is evident that the formation of FCC, BCC, Cr₃Si, and TiC phases, corresponding phases were confirmed using the SAED patterns in Fig. 6(e1-e4), respectively. The formation of the FCC phase was confirmed by the SAED pattern in Figs. 6 (e-1), suggesting the crystallization in the of $[1\ \overline{12}]_{FCC}$ direction. Figs. 6(e-2) displays the SAED



Fig. 6. (a) TEM image of CrFeNiTi MEA bulk sample, indicating the formation of different phases including FCC, BCC, Ni₃Ti and TiC phases, corresponding SAED patterns shown in Fig. (b1-b4), respectively, (c) shows the HRTEM image obtained from Fig. (a), indicating the phase boundaries along with measured d-spacing and FFT images. While Fig. (d) displays the TEM image of CrFeNiTiSi HEA bulk sample, indicating the formation of different phases including FCC, BCC, Cr₃Si and TiC phases, corresponding SAED patterns shown in Fig. (e1-e4), respectively, (f) shows the HRTEM image obtained from Fig. (d), indicating the phase boundaries along with measured d-spacing and with measured d-spacing and FFT images.

pattern of the BCC phase, indicating crystallization in the $[\overline{1}13]_{BCC}$ direction. In addition, Si addition promoted the Cr₃Si phase, which was confirmed by SAED pattern in Figs. 6(e-3), revealing the crystallization in the of $[2\overline{1}0]_{Cr_3Si}$ direction. Furthermore, the TiC phase was confirmed by SAED pattern in Figs. 6 (e-4), which crystallized along the [110]_{Tic} direction. The average size of TiC particles is about \sim 50 nm. The presence of such nanoscale TiC particles shows a positive effect on enhancing the strength and wear resistance of HEAs. In addition, Fig. 6 (f) shows the HRTEM image obtained from red colour square region in Fig. 6 (d), indicating the good interfacial bonding between different phases but different orientation. The FFT patterns were taken from the corresponding phases and included in the inset of Fig. 6(f). While the measured d-spacing of the FCC phase is about 1.761 Å along the (200) plane, while the d-spacing of the BCC phase was about 2.043 Å along the (110) plane. The formation of Cr₃Si phase by Si alloying can be attributed to its large negative mixing enthalpy between Cr and Si (-37 kJ/ mol). The formation of Cr₃Si phase can enhance strength and hardness of HEAs [32].

3.5. Relative density, hardness and compressive properties of sintered HEAs

For advanced structural applications, developing materials with light weight and high strength are important factors. Accordingly, in the present study, Si was introduced into the CrFeNiTi MEA system to reduce the density and improve the strength of the alloy system. The densities of CrFeNiTi MEA and CrFeNiTiSi HEAs were measured by the Archimedes method at RT. The measured density of CrFeNiTi MEA was \sim 7 g/cm³ (theoretical density = 7.11 g/cm³, relative density = 98.45%) and that of CrFeNiTiSi HEA was \sim 6.1 g/cm³ (theoretical density 6.161 g/cm³, relative density 99%), respectively. The relative densities of both the HEAs were greater than 98%, suggesting that an almost fully dense microstructure was achieved without significant porosity and voids formed by the SPS process. Notably, the addition of Si into CrFeNiTi HEA reduces the density of the alloy system, which is advantageous

for structural applications, where weight reduction is a crucial factor.

The microhardness of the sintered CrFeNiTi MEA and CrFeNiTiSi HEAs was displayed in Fig. 7 (a) along with other HEAs for comparison. The average hardness of CrFeNiTi MEA was ~1200 HV, while the addition of Si into the CrFeNiTi MEA, the hardness significantly increased to 1330 HV. The typical improvement in hardness with Si addition was attributed to the increased fraction of BCC phase and solid solution strengthening effect resulting from the lattice distortion. A similar behavior of hardness improvement by Si addition has been observed in other HEAs [15,16,34]. The presence of hard phases such as BCC, Ni₃Ti and Cr₃Si phases could enhance the strength and hardness of HEAs. For instance, it is noted that the presence of TiC phase in both the alloys show a significant impact on the hardness behavior due to its superior hardness (~3200 HV). The TiC particles can act as obstacles to dislocation movement, which increases resistance to plastic deformation, leading to improved hardness [35]. Moreover, the obtained hardness values are significantly higher than those of other HEAs reported in the literature [15.36-47].

Fig. 7 (b) shows the typical load (*P*)–displacement curves of CrFe-NiTiX (X = Si) HEAs obtained from the nanoindentation measurements. From the average indent data, a final load–displacement curve was plotted. In the loading stage, the load–displacement curve appeared to be regular. Further, the creep curve was fitted using the following empirical equation [33]:

$$h(t) = h_0 + a(t - t_0)^b + kt$$
(6)

From equation (6), h_0 and t_0 denote the initial holding depth and time at the beginning of the holding period, respectively. In addition, *a*, *b*, and *k* are fitting constants. From the fitting curve, the change in the hardness *H* during the holding period could be estimated as follows:

$$H = \frac{P}{24.3h_c^2} \tag{7}$$

where *P* and h_c represent the holding load and contact displacement of the Berkovich indenter, respectively. The h_c measured by the Berkovich



Fig. 7. (a) Comparison of micro-Vickers hardness in the present study with those of other reported HEAs [15,36–47] and (b) nanoindentation measurement; load-displacement curves. (c) Creep displacement and (d) nano hardness and elastic modulus of the CrFeNiTi MEA and CrFeNiTiSi HEAs obtained from the nanoindentation tests.

indenter can be defined as

$$h_{\rm c} = h - 0.72 \times P / S \tag{8}$$

where h is the displacement of the indentation and contact displacement, while S denotes the stiffness derived from the unloading curve.

Fig. 7 (c) illustrates the penetration depth versus holding time of both samples under a constant load of 100 mN. The magnitude of penetration depth was strongly influenced by the alloy composition under a constant holding time of 20 s. The penetration depth of the CrFeNiTi MEA was higher than that of the CrFeNiTiSi MEA, suggesting that Si alloving could enhance the resistance to plastic deformation. Further, the creep behavior of samples was fitted using the second-order polynomial, which is represented by a dotted line. The polynomial fitting curves closely matched the experimental results. In addition, Fig. 7 (d) shows the improved nanohardness and elastic modulus by the addition of Si into the CrFeNiTi MEA. For instance, the nanohardness of the CrFeNiTi MEA was ~12.06 GPa and then increased to 15.77 GPa by Si addition, indicating an enhancement in the ability of the material to resist deformation at the nanoscale. Further, the elastic modulus improved from 267.2 to 280.47 GPa with Si addition, indicating improved stiffness and resistance to elastic deformation under stress. Furthermore, the measured compressive properties at room temperature were presented in Table 5, also compared with other HEAs reported in the literature [48-50]. The remarkable enhancement in mechanical properties through Si addition could be attributed to the increased fraction of BCC phase and solid solution strengthening resulting from lattice distortion.

3.6. Friction and wear behavior of HEAs under dry-sliding conditions

The coefficient of friction (COF) of both the CrFeNiTi MEA and CrFeNiTiSi HEAs were investigated under dry-sliding conditions at different normal loads (5, 10, and 15 N) to understand the effect of Si content. Fig. 8 (a) reveals that the COF of the CrFeNiTi MEA showed an increasing tendency at the beginning and stabilized over the sliding distance. Further, the COF slightly increased with an increase in the normal load from 5 to 10 and 15 N, suggesting increased contact pressure and surface deformation between the contacting surfaces. Unlike the CrFeNiTi MEA, the COF of the CrFeNiTiSi HEA showed a different tendency with an increasing normal load from 5 to 15 N as shown in Fig. 8 (b), where the COF increased and stabilized over the sliding distance regardless of the applied load. Notably, the COF gradually decreased with increasing load from 5 to 15 N, which could mainly be attributed to the compositional changes on the surface i.e., the formation of tribo-oxides during wear tests induces a lubricating effect and inhibits direct contact between the contacting surfaces. The formation of oxide layers on the wear tracks reduces the COF and wear rates. To elucidate the COF behavior with increasing load for both the HEAs, the average COF is presented in Fig. 8 (c). It was found that with an increasing load from 5 to 10 and 15 N, the average COF increased from 0.292 to 0.297 and 0.322 (~10.34 %) for the CrFeNiTi MEA, however, the Si-added HEA showed a significant reduction in COF from 0.304 to 0.292 and then 0.244, (\sim 20%). From these results, it can be concluded that the addition of Si can reduce the COF by forming a self-lubricating

Table 5

Room temperature compressive properties of CrFeNiTi MEA and CrFeNiTiSi HEAs compared with other HEAs.

Sample	UCS (MPa)	Strain (%)	Ref.
CrFeNiTi	1400 ± 80	5.3	This study
CrFeNiTiSi	1700 ± 50	5.1	This study
AlCoCrCuNiTi	1495	7.68	[48]
AlCoCrCuNiTiY	1192	3.54	[48]
AlCrCuFeTi	721	4.2	[49]
FeCrMoVTi	1450	5.1	[50]

oxide layer on the worn surface under dry-sliding conditions. Moreover, the surface roughness (Ra) at different loads is a crucial factor that influences the COF of HEAs. Therefore, the surface roughness of the wear tracks was measured by AFM analysis at different positions on surface of wear track at different loads. The average values were reported to maintain experimental accuracy. The obtained average Ra of both the samples at different loads as shown in Fig. 8 (d). The Ra of the CrFeNiTi MEA tended to increase from 28.84 to 35.08 and then 43.73 nm with an increase in the normal load from 5 to 10 and 15 N, respectively. By contrast, the Ra of CrFeNiTiSi HEA showed a decreasing tendency from 26.01 to 25.14 and 22.88 nm with an increase in the applied load from 5 to 10 and 15 N, respectively. COF is known to be directly related to the Ra of materials [47]. Accordingly, the COF of the CrFeNiTi MEA slightly increased with load owing to increased Ra, whereas the COF of the CrFeNiTiSi HEA decreased with load because of a reduction in Ra with increasing applied load.

3.6.1. Surface topography analysis of wear tracks examined by AFM

The surface topography of wear tracks was examined to understand the deformation behavior at different loads. Fig. 9(a)-(c) show the surface topography of the CrFeNiTi MEA under applied loads of 5, 10, and 15 N, respectively. As the load increased from 5 to 10 and 15 N, there was an apparent increase in the deformation of the wear track, which was mainly caused by an increase in the contact pressure between contacting surfaces, resulting in an increased Ra was observed (Fig. 8 (d)). Unlike CrFeNiTi MEA, the Si-added HEA showed a decrease in surface deformation with increasing load from 5 to 15 N, as shown in Fig. 9(d-f), respectively. This decrease in deformation with load could be attributed to the formation of a protective oxide layer on the worn surface during wear tests, which prevents or reduces the extent of plastic deformation on the material surface. Therefore, the Ra of the CrFeNiTiSi HEA showed a decreasing tendency with an increase in the applied load (Fig. 8 (d)). From these results, it can be concluded that increased plastic deformation and Ra could slightly enhance the COF for the CrFeNiTi MEA, while the reduction in plastic deformation and Ra could reduce the COF of the CrFeNiTiSi HEA with an increasing load.

Surface profilometry analysis was performed to examine the depth and width of wear tracks in the CrFeNiTi MEA and CrFeNiTiSi HEAs as shown in Fig. 10(a and b), respectively. It was found that the depth and width of wear tracks in both the HEAs tended to increase with an increase in the normal load from 5 to 10 and 15 N because of increased plastic deformation. However, the reduction in the depth and width of wear tracks by Si alloving suggests an improved resistance against plastic deformation, as shown in Fig. 10 (b). The resultant depth and width of wear tracks can be used to calculate the cross-sectional area of wear tracks at different loads. Then, the loss of wear loss volume can be assessed by multiplying the cross-sectional area and circumference length of the wear tracks. The estimated wear volume of both the HEAs at different loads is displayed in Fig. 10 (c). The estimated wear volume of the CrFeNiTi MEA was found to increase from 0.00763 mm³ to 0.02328 mm³ with an increase in the normal load from 5 to 15 N, respectively. Furthermore, the wear volume of CrFeNiTiSi HEA also showed an increasing tendency from 0.0064 to 0.01579 mm³ with the increase in normal load from 5 to 15 N, respectively. However, lower wear volumes were observed in the CrFeNiTiSi HEA compared to the CrFeNiTi MEA owing to their higher hardness resisting surface plastic deformation. Further, the specific wear rate of both samples at constituent loads was estimated by substituting the wear volume, applied load, and sliding distance in the following equation [9]:

 $\label{eq:specific wear rate (Ws) = \frac{Wear \ volume \ (Vw)}{Load \ (F) \times Sliding \ distance \ (S)}} \tag{9}$

The estimated specific wear rates of both the CrFeNiTi MEA and CrFeNiTiSi HEAs are displayed in Fig. 10 (d). An approximately stable wear rate was observed in the CrFeNiTi MEA with an increasing applied load. For instance, the specific wear rate of the CrFeNiTi MEA was



Fig. 8. Variation in the COF of (a) CrFeNiTi MEA and (b) CrFeNiTiSi HEAs, (c) average COF, and (d) surface roughness of both the HEAs obtained from AFM analysis after the dry-sliding test under different normal loads.



Fig. 9. Surface profiles of (a-c) CrFeNiTi MEA and (d-f) CrFeNiTiSi HEAs measured by AFM analysis after wear tests at 5, 10, and 15 N, respectively.

approximately 5.705 \times 10^{-5} mm³/Nm, 5.915 \times 10^{-5} mm³/Nm, and 5.805 \times 10^{-5} mm³/Nm for the corresponding loads of 5 N, 10 N, and 15 N, respectively. The specific wear rate of the CrFeNiTiSi HEA was lower than that of the CrFeNiTi MEA. Further, the specific wear rate slightly decreased from 4.452 \times 10^{-5} mm³/Nm to 4.305 \times 10^{-5} mm³/Nm and 4.015 \times 10^{-5} mm³/Nm with an increase in the load from 5 to 10 and 15 N, respectively. The obtained specific wear rate of both the alloys are significantly lower than that of other HEAs reported in the literature [9, 11,12]. This is mainly attributed to the formation of hard phases such as

 Ni_3Ti , Cr_3Si and TiC phases after sintering. The multiphase structures enhance the wear resistance and durability of material even at harsh conditions. For example, it is worth mentioning that the presence of TiC phase in both the alloys show a great impact on enhancing the wear resistance due to its superior hardness. During wear test, the presence of TiC particles resists plastic deformation, thereby reducing the material loss and prolonging the durability of materials under harsh conditions. A similar improvement in wear resistance through the addition of TiC contents was observed in other HEAs [51,52]. It is noted that Si addition



Fig. 10. Depth and width profile of wear tracks of (a) CrFeNiTi MEA and (b) CrFeNiTiSi HEAs measured using a surface profilometer after the wear tests, (c) calculated wear volume, and (d) specific wear rates of the HEAs under different normal loads (5, 10, and 15 N) at a constant sliding time of 10 min and velocity of 6 cm/s.

enhances the hardness and wear resistance than that of CrFeNiTi MEA caused by solid solution strengthening resulting from the lattice distortion effect. Similar behavior was observed in the CoCrCuFeNiSi_x HEA, where it was claimed that solid solution strengthening is the predominant mechanism that can be induced by atomic radii mismatch between Si and constituent elements [17].

3.6.2. Wear track morphology of HEAs examined by SEM analysis

The wear track morphology of the CrFeNiTi MEA after wear tests under different normal loads (5, 10, and 15 N) is shown in Fig. 11(a)-(c), respectively. It can be seen in Fig. 11 (a) that the formation of fine scratches along the sliding direction and surface layers is partially removed from the wear tracks at the load of 5 N. The surface of the wear tracks appears to partially change into a dark color, which is mainly attributed to the partial oxidation of the wear tracks during the wear test. This is further confirmed by the SEM-EDS color mapping presented in Fig. 11 (d). This type of surface oxidation can reduce the COF and wear rates [47]. Upon increasing the load to 10 N, the surface layers were delaminated, and fine debris adhered to the wear tracks, which were oxidized during continuous sliding (Fig. 11(b)). The oxidation of adhesive wear debris can be confirmed by the EDS analysis presented in Fig. 11 (e). Further, the passive layer was destroyed at higher normal loads (15 N) as shown in Fig. 11 (c). This is mainly due to the oxidation of wear tracks that are brittle in nature and easily undergo plastic deformation at higher normal loads. The oxidation of wear tracks can be further confirmed by the EDS analysis presented in Fig. 11 (f), indicating the removal of fine wear debris from the wear tracks. The removal of wear debris from the worn surface increases the mechanical vibrations and surface roughness of the HEA, thereby increasing the COF. Indeed, the extent of plastic deformation and surface oxidation was observed at higher applied loads. Deng et al. [53] also observed that the predominant wear mechanisms of CoCrFeNiMo_{0.2} HEA were oxidation and delamination at higher applied loads.

In addition, to understand the effect of Si addition on wear behavior,

the wear track morphology was examined by SEM analysis under the same applied loads. Fig. 12 (a) shows that the partial delamination of the wear track was observed at 5 N, suggesting the initiation of surface delamination at lower load levels. Upon increasing the applied load to 10 N (Fig. 12 (b)), the wear track morphology appeared to increase surface deformation owing to increased contact pressure between the contacting surfaces. Finally, a further increase in the load to 15 N caused a significant increase in plastic deformation and delamination of surface layers, particularly in the dark regions of wear tracks as shown in Fig. 12 (c). It was noticed that the dark contrasts on the worn surface increased, suggesting the increased surface oxidation by Si addition. With increasing load, the oxide regions of the wear tracks tended to fracture or delaminate from the surface because of the brittle nature of the oxides. To confirm the compositional behavior of corresponding loads of 5, 10 and 15 N, EDS color mapping was examined as shown in Fig. 12(d and e and f), respectively. Notably, an increased applied load could increase the surface oxidation of the wear tracks. This was because the increased frictional heat between the contacting surfaces could enhance the surface oxidation of the wear tracks, particularly under dry-sliding conditions. Consequently, the surface oxidation (dark regions) of the wear tracks increased with an increase in applied loads, as confirmed by the EDS analysis presented in Table 6. The surface oxidation of both alloys increased with the load; however, the oxygen concentration of the Si-added HEA was greater. Therefore, it can be concluded that Si tends to form protective oxide layers on the worn surface, which reduces the COF and wear rates by reducing the direct contact between contacting surfaces during sliding tests.

3.6.3. Morphology of wear debris under different loads

The COF and wear behavior of the HEAs were influenced by the type of wear debris delaminated from the worn surface after wear tests. Thus, the wear debris morphologies of the CrFeNiTi MEA and CrFeNiTiSi HEAs were investigated at different loads (5, 10, and 15 N) using SEM analysis as shown in Fig. 13(a-c) and 13 (e-g), respectively. The



Fig. 11. Wear track morphology of the CrFeNiTi MEA under different normal loads: (a) 5, (b) 10, and (c) 15 N. (d), (e), (f) Compositional behavior of corresponding wear tracks at different loads observed using the SEM-EDS analysis.

generation of large wear debris was observed in both the HEAs under an applied load of 5 N, as shown in Fig. 13 (a and e). This was because abrasive wear was the dominant wear mechanism, where the sliding surfaces were mechanically worn down by hard particles under this condition. As the load increased to 10 N, the size of wear debris particles decreased for both HEAs, which was due to the fracture of large debris particles at higher loads, as shown in Fig. 13(b and f). With a further increase in the applied load to 15 N, the size of wear debris was effectively reduced because of the increased fragmentation of debris particles between sliding surfaces, as shown in Fig. 13 (c and g). To further confirm the oxidation of wear debris after the wear test at 15 N for both the HEAs, SEM-EDS color mapping was performed as shown in Fig. 13 (d and h), respectively. The oxidation of wear debris after the wear tests is evident, suggesting that these fine oxidized wear debris particles act as a lubricant during the sliding tests. The EDS results revealed that the oxidation of wear debris increased with increasing load in Si-added HEA compared to the CrFeNiTi MEA. Accordingly, the COF decreased significantly with increasing load compared to the CrFeNiTi MEA. Therefore, the addition of Si enhances surface oxidation and increases the wear resistance of HEA by forming a protective layer on the worn surface under dry-sliding conditions.

3.7. Surface oxidation behavior of HEAs

The oxidation resistance of the CrFeNiTi MEA and CrFeNiTiSi HEAs

were investigated at different temperatures (700 °C, 800 °C, and 900 °C) for 30 h using TGA. The obtained results are presented in Fig. 14, showing the relationship between oxidation time and weight change during the oxidation test. Typically, the oxidation behavior followed a parabolic law at elevated temperatures. The parabolic rate law can be expressed as follows [54]:

$$K_p \times t = \left(\frac{\Delta W}{A}\right)^2 \tag{10}$$

where K_p represents the parabolic rate constant, A is the surface area of sample, ΔW is the change in mass per unit area and t is the oxidation time. It can be seen in Fig. 14, both the samples showed a negligible change in mass in the initial stage of oxidation time. However, CrFeNiTi MEA showed an increase in weight with an increasing oxidation time at 700 °C. Further increasing temperature to 800 and 900 °C, the weight gain significantly reduced, suggesting the formation of metal oxides on the surface and protect it from further oxidation. In contrast, Si added HEA showed a different tendency than that of CrFeNiTi MEA. For instance, the weight of the CrFeNiTiSi HEA considerably lower and saturated at 700 °C with an increasing oxidation time. As increasing oxidation temperature to 800 °C, the weight of sample significantly reduced compared to all samples. It means that the addition of Si can improve surface oxidation and prevent further degradation by forming a protective oxide layer on the material surface. A similar improvement in

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Fig. 12. Wear track morphology of the CrFeNiTiSi HEA at different normal loads: (a) 5, (b) 10, and (c) 15 N. (d), (e), (f) Compositional behavior of the corresponding wear tracks under different loads observed using SEM-EDS analysis.

Table 6
SEM-EDS analysis of the CrFeNiTi MEA and CrFeNiTiSi HEAs after wear tests at
loads of 5, 10, and 15 N.

Alloy	Load	Cr	Fe	Ni	Ti	Si	0
CrFeNiTi	5 N	20.27	22.25	17.77	18.05	-	21.66
	10 N	16.15	17.42	13.52	14.33	-	38.59
	15 N	13.33	14.26	11.31	11.75	-	49.34
CrFeNiTiSi	5 N	14.13	17.22	12.59	13.06	11.76	31.25
	10 N	10.95	13.32	9.55	10.33	9.23	46.62
	15 N	7.31	8.89	7.26	6.50	6.62	63.42

oxidation resistance has been observed in other HEAs with the addition of Si [24–26]. The presence of Si in HEA promotes the formation of protective oxide layers, such as SiO₂, which enhance passivation and reduce oxygen diffusion, thereby improving the overall oxidation resistance of the alloy. The formation of such oxide layers on the HEA surface tends to reduce surface oxidation, leading to drop in the oxidation curve observed between 17 and 20 h. However, after oxidation at 900 °C, the weight of the CrFeNiTiSi HEA substantially increased among others, suggesting the reduced oxidation resistance at higher temperatures. Similarly, Chen et al. [27] also observed the similar behavior of mass gain at higher temperatures including 900 and 1000 °C in the case of Si added HEA. It is worth mentioning that the possible reason for the reduction in oxidation resistance at 900 °C is due to development of cracks and delamination of protective oxide layers from the materials surface. It can be further discussed in the later section using the surface morphology after oxidation tests.

3.7.1. Surface morphology and compositional behavior HEAs after oxidation tests

The surface morphology of both the specimens was observed using SEM analysis after oxidation tests for 30 h at different temperatures. Fig. 15 (a) shows the morphology of the CrFeNiTi MEA after oxidation at 800 °C, indicating the formation of spherical oxide particles. With a further increase in the oxidation temperature to 900 °C (Fig. 15 b), the oxide particles continued to grow with irregular shapes on the surface layers of the material and became denser compared to 800 °C. Thus, the formation of dense oxide particles on the surface enables the protection



Fig. 13. Wear debris morphology of CrFeNiTi MEA after wear test at different loads 5, 10, and 15 N, shown in Fig. (a, b and c), respectively. Fig (d) shows the SEM-EDS color mapping of wear debris obtained after wear test at 15 N, Fig. (e, f, and g) shows the wear debris morphology of CrFeNiTiSi HEA after wear test at 5, 10 and 15 N conditions, respectively, Fig. (h) shows the SEM-EDS color mapping of wear debris obtained from Fig. (g).



Fig. 14. Weight gain curves of CrFeNiTi MEA and CrFeNiTiSi HEAs after oxidation at different temperatures of 700, 800 and 900 $^\circ C$ for 30 h.

of the material surface from a further increase in oxidation resistance and reduced weight loss was observed in CrFeNiTi MEA. In contrast, with the addition of Si into the CrFeNiTi MEA, the surface morphology significantly changed compared to the CrFeNiTi MEA at 800 °C (Fig. 15 c). The surface of the CrFeNiTiSi HEA was more uniform with fine oxide particles on the material surface. These fine oxide particles suggest that the material was effectively protecting itself from further oxidation by forming a stable and adherent oxide layer. Consequently, the wight loss effectively decreased for the CrFeNiTiSi HEA up to 800 °C. Upon further increasing the oxidation temperature to 900 °C, spallation of oxide layers was observed from the material surface (Fig. 15 d). Accordingly, the weight loss increased significantly compared to all conditions. This spallation of the oxide layer from the material surface is denoted by yellow arrows and dotted lines. Thus, the present study suggests that the addition of Si into the CrFeNiTi MEA effectively improves oxidation resistance up to 800 °C and then reduces the oxidation resistance due to spallation of protective oxide layers from the material surface.

To further confirm the surface oxidation behavior of both the alloys after oxidation at 700 °C for 30 h, SEM-EDS analysis was performed, and results are presented in Fig. 16. The morphology of CrFeNiTi MEA after oxidation indicating the growth of oxides on the surface, as shown in Fig. 16 (a). It is confirmed by EDS mapping and point analysis (Fig. 16 c). From the EDS results, it is observed that surface being oxidized after oxidation at 700 °C, and the concentration of Cr and Ti are higher than that of Fe and Ni elements, i.e., Cr and Ti are actively participating in the



Fig. 15. Surface morphology of the CrFeNiTi HEA oxidized at (a) 800 °C and (b) 900 °C, and CrFeNiTiSi HEA oxidized at (c) 800 °C and (d) 900 °C.



Fig. 16. Surface morphology of (a) CrFeNiTi MEA and (b) CrFeNiTiSi HEAs after oxidation at 700 °C for 30 h, corresponding compositional behavior was examined using EDS color mapping and point analysis in Fig. (c and d), respectively.

oxidation process due to their higher affinity towards oxygen. Therefore, it is believed that the formation of metal oxides including Cr2O3 and TiO₂ on the oxidized surface. The formation of continuous oxides on the surface acts as protective layer and reduces the further oxidation. However, less protective oxides (NiO and Fe₂O₃) formed as well. As oxidation progresses, these oxide layers grown and cracks are initiated under thermal stress, leading to spallation and exposing fresh metal [55, 56]. In addition, Fig. 16 (b) shows the morphology of CrFeNiTiSi HEA after oxidation test at 700 °C, indicating the formation of uniform oxides on the surface. It is further confirmed by EDS mapping and point analysis (Fig. 16 d). Compared to CrFeNiTi MEA, the surface of Si added HEA found to be uniformly oxidized under same conditions. Based on EDS results, the concentration of Cr, Ti and Si elements are higher than that of Fe and Ni elements, suggesting the formation of metal oxides related to Cr₂O₃, TiO₂ and SiO₂ elements. These oxides effectively inhibit further oxidation, enhancing passivation and limiting oxygen diffusion. It is important to mention that the presence of TiC phase in the case of Ti containing HEAs, enhances the oxidation resistance by mitigating surface cracking. In general, the nanoscale TiC particles refine the grain size and strengthen the grain boundaries, which reduces the crack formation in the surface layers. Additionally, Ti is considered as a beneficial element for enhancing the oxidation resistance as it forms a protective TiO₂ layer on the surface that acts as a shield against further oxidation. Similarly, Chen et al. [57] observed the formation of Ti containing oxides (TiO₂) on the surface and prevents from further oxidation in WMoTaNbV alloy. From these results, it has been observed that the presence of Cr, Ti and Si in the present alloy system could improves the oxidation resistance by forming protective oxide layers on the surface, making them more suitable for high-temperature applications.

4. Conclusions

The present study reports the fabrication of CrFeNiTi MEA and CrFeNiTiSi HEAs by the powder metallurgy route. The phase composition and microstructural characteristics of powder and sintered HEAs were investigated by XRD, FESEM, and TEM analyses. The mechanical behavior of the sintered HEAs were examined by micro-Vickers hardness and nanoindentation measurements at RT. Ball-on-disk dry-sliding tests were performed by varying the normal load from 5 to 15 N. Furthermore, the oxidation behavior of both alloys was investigated at different temperatures. The main conclusions drawn from this study are as follows.

- (1) Based on the experimental results, both the alloys formed a single-phase BCC solid solution. After sintering, CrFeNiTi MEA exhibited the formation of FCC, BCC, Ni₃Ti and TiC phases, while the Si-added HEA showed the FCC, BCC, Cr₃Si and TiC phases.
- (2) The addition of Si enhances the microhardness (1200–1330 HV), nanohardness (12–15.7 GPa), elastic modulus (267.2–280.4 GPa) and UCS (1400 \pm 80–1700 \pm 50 MPa), which is due to increased fraction of BCC phase, solid solution strengthening effect.
- (3) With an increasing load from 5 to 15 N, the average COF increased ~10.34 % for the CrFeNiTi MEA, but the Si-added HEA showed a significant reduction in COF ~20%. The reduction in COF was mainly attributed to increased surface oxidation acts as protective layer and reduced Ra compared to that of CrFeNiTi MEA.
- (4) The specific wear rate decreased by Si alloying into the CrFeNiTi MEA regardless of the applied load, owing to improved hardness could resist further the plastic deformation of the wear tracks.
- (5) The Si addition improved oxidation resistance up to 800 °C however, the spallation of oxide layer at 900 °C caused to reduce the oxidation resistance. Additionally, Cr and Ti were found to participate more actively in surface oxidation compared to Fe and Ni.

(6) From the abovementioned results, it can be concluded that the addition of Si into the CrFeNiTi MEA is an effective way to enhance the mechanical properties, wear and oxidation resistance of HEAs.

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.

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