

Development of eutectic high entropy alloy by addition of W to CoCrFeNi HEA

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ABSTRACT

High entropy alloys have shown a remarkable combination of physical and mechanical properties. The introduction of eutectic microstructure, consisting of a tough fcc phase, and a hard-intermetallic phase, can help in obtaining even better synergy of strength and ductility. The presence of multiple principal alloying elements in HEAs and absence of corresponding multicomponent phase diagrams makes designing of eutectic high entropy alloys a tedious task. In the present study, systematic investigation of CoCrFeNi-W_x system has been carried out for the development of eutectic microstructure. Experimental results validated the presence of eutectic reaction in the calculated phase diagram. CoCrFeNi-W_x HEAs remained single fcc phase alloys at smaller amount of W (x = 0.1) but changed to hypoeutectic (x = 0.25, 0.5, 0.75) and hypereutectic alloys (x = 1.0) with increase in the amount of tungsten. It has been shown that calculated pseudo binary phase diagrams can provide a very good starting point for the development of eutectic HEAs. Mechanical characterization of the developed HEAs revealed that development of eutectic mixture of a soft (fcc) and a hard phase (intermetallic/bcc) can help in obtaining outstanding combination of mechanical properties.

1. Introduction

Development of high entropy alloys (HEAs) has emerged as new research hotspot in the field of metallic alloys because of their better combination of physical and mechanical properties [1]. Most of the previous research on HEAs has been dedicated to the development of single phase high entropy alloys [2]. Fcc structured single phase HEAs have shown excellent ductility but relatively poor strength while single phase Bcc structured HEAs exhibited high strength with limited plasticity [3]. Development of high entropy alloys that shows promising balance between strength and plasticity remains a prominent issue [4]. Compositional inhomogeneity, weak liquidity and castability exhibited by the high entropy alloys can further downgrade their mechanical properties [5]. Several engineering components are made by casting. Casting defects, especially in the case of complex geometries and in the case of large size components, are difficult to remove by thermo-mechanical processing. Compositional segregation of HEAs has been proved to be another hurdle for their practical applications [6–8]. An

extremely good combination of mechanical properties and castability is therefore required in HEAs to advance their use for demanding engineering applications [9].

Introduction of eutectic microstructure that consists of lamellar arrangement of a hard phase and a tough phase can help in circumventing strength ductility trade off in HEAs [10]. Presence of eutectic microstructure can minimize segregation and improve castability of HEAs besides providing enhanced combination of mechanical properties [11]. This idea of introduction of eutectic microstructure in HEA has resulted in the development of a new breed of high entropy alloys, known as eutectic high entropy alloys (EHEA). Several EHEAs have been developed in the last few years in order to obtain better combination of mechanical characteristics which include AlCrFeNi₂, AlCoCrFeNi_{2.1}, AlCo₂CrFeNi₂, AlCoCrFeNi₃, Al₁₉Co₁₅Cr₁₅Ni₅₁, [12] Fe₃₅Ni₂₅Cr₂₅Mo₁₅, [13] Fe₂₀Co₂₀Ni₄₁Al₁₉, [14] CoCrFeNiTa_{0.395}, [15] Co₂Mo_{0.8}Ni₂VW_{0.8}, Nb₂₅Sc₂₅Ti₂₅Zr₂₅, CoCrFeNiNb_{0.5}, [16] Zr_{0.6}CoCrFeNi_{2.0}, Nb_{0.74}CoCrFeNi_{2.0}, Hf_{0.55}CoCrFeNi_{2.0} and Ta_{0.65}CoCrFeNi_{2.0}. Development of eutectic microstructure in some HEAs has helped in obtaining better

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combination of mechanical properties in majority of the systems. The introduction of eutectic microstructure in certain HEAs has facilitated the achievement of enhanced amalgamation of mechanical properties across most of these systems while in some of the EHEAs (as-cast $\text{CoFeNi}_2\text{V}_{0.5}\text{Nb}_{0.75}$ and $\text{Co}_2\text{Mo}_{0.8}\text{Ni}_2\text{VW}_{0.8}$ EHEAs), an extremely high strength was attained, it came at the expense of toughness [17].

The current research aims to design and create EHEAs with a laminar structure comprising a malleable fcc phase and a rigid intermetallic phase, with the objective of achieving an improved balance between strength and ductility. CoCrFeNi HEA has been selected for the present study as it is one of the most studied HEA systems. The CoCrFeNi HEA displayed a single solid solution phase with an FCC structure, showing no evident composition variation or extensive chemical ordering. Consequently, it holds potential as a comprehensive pseudo-element for facilitating eutectic reactions. Tungsten is reported to form Fe_7W_6 and Co_7W_6 in Fe–W and Co–W binary phase diagram. It was assumed that a microstructure consisting of fcc and $\text{Fe}_7\text{W}_6/\text{Co}_7\text{W}_6$ can help us in achieving evenness in high strength and better plasticity [18–20]. Determination of possibility of eutectic reaction in CoCrFeNi- W_x high entropy alloy system by hit and trial can be an extremely challenging task. The accuracy of empirical and semi empirical methods based on variables such as atomic size mismatch, enthalpy of mixing, entropy of mixing, valence electron concentration, electronegativity difference may not be very high due to complex interactions arising from the presence of multiple principal alloying elements [21–23]. Hence, estimation of eutectic composition in an HEA system confronts a tough challenge. Pseudo binary diagrams calculated with the help of ThermoCalc software have been successfully used for the determination of eutectic composition in CoCrFeNiMo and CoCrFeNiTa HEA systems [24]. In the present study, pseudo-binary phase diagram of CoCrFeNi-W has been calculated with the help of ThermoCalc software and has been used as a guideline for determination of eutectic composition. Different alloy compositions with varying amount of W were prepared and characterized. Hypo-eutectic, hyper-eutectic and eutectic microstructures have been successfully developed in CoCrFeNi- W_x HEA system and effect of microstructural changes on the mechanical properties has been studied.

2. Experimental techniques

Co, Cr, Fe, Ni and W (purity >99.95%) were used as raw materials and alloy buttons of CoCrFeNi W_x ($x = 0.25, 0.5, 0.75$ and 1.0) were prepared by arc melting furnace under argon atmosphere. Melting was carried out in two steps due to large difference in the melting point of W and other constituents. Cr and W, which form eutectic, were melted initially. Remelting of the developed alloy by addition of Co, Fe, and Ni was carried out in the next step. Each alloy sample was melted seven times for one minute and flipped over each melting to ensure chemical homogeneity and complete mixing. Microstructural characterization of the developed alloy was carried out with the help of optical microscope (ZEISS Axio Observer Inverted) and scanning electron microscope (JEOL JSM 6490A, acceleration voltage: 10 kV, working distance 10 mm) under secondary electron mode whereas crystal structure characterization was carried out with the help of XRD (STOE, Cu $\text{K}\alpha$ radiation, Generator: 20 kV, 5 mA). Mechanical characterization of the developed HEAs was carried out with the help of compression testing, micro-Vickers hardness test, and nano-indentation. Samples for compression test ($2\text{ mm} \times 2\text{ mm} \times 6\text{ mm}$) were taken out from the alloy buttons with the help of wire electric discharge machine (EDM) and grinded to remove cutting marks and to obtain perfectly flat and parallel surfaces. The universal testing machine (UTM) was utilized to conduct compression testing at a strain rate of 10^{-3} s^{-1} . Nanoindentation was performed using an (Agilent nanoindenter G200) equipped with a diamond Berkovich indenter. A peak load of 10mN was applied during the testing process.

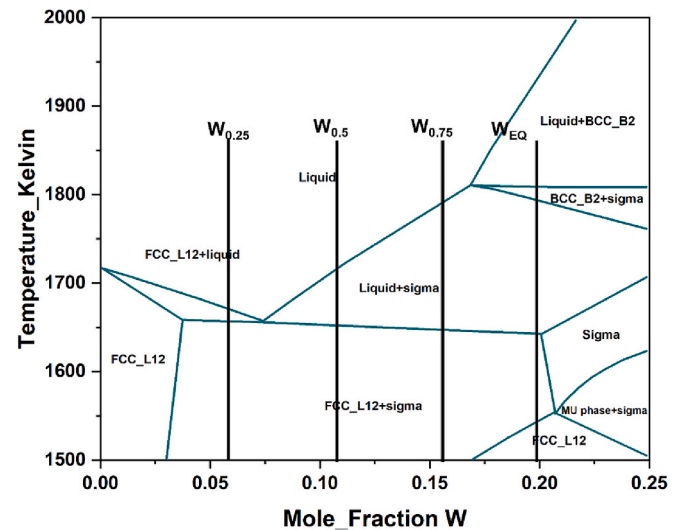


Fig. 1. Pseudo-binary phase diagram of CoCrFeNi-W system calculated with the help of ThermoCalc Software.

Table 1

Nominal composition of HEAs developed during the present study.

Name of alloy	Composition of the alloy (Mole fraction)				
	Co	Cr	Fe	Ni	W
CoCrFeNiW _{0.25}	0.235	0.235	0.235	0.235	0.060
CoCrFeNiW _{0.5}	0.225	0.225	0.225	0.225	0.1
CoCrFeNiW _{0.75}	0.211	0.211	0.211	0.211	0.156
CoCrFeNiW _{1.0}	0.2	0.2	0.2	0.2	0.2

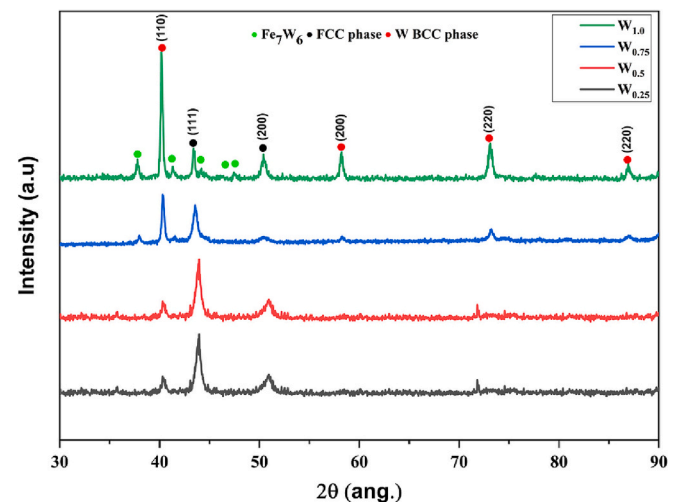


Fig. 2. XRD analysis of the developed HEAs.

3. Results and discussion

CoCrFeNi is one of the well-studied fcc HEA system. It has been found to exhibit extraordinary ductility but with limited yield strength. The present study is aimed at improving its combination of strength and ductility by development of eutectic microstructure through alloying addition. The binary combination of constituents of the selected fcc HEA system with tungsten (Co–W, Fe–W, Ni–W system) either showed presence of intermetallic compounds in their respective phase diagrams and/or possibility of eutectic reaction during solidification (Co–W, Ni–W system) [25,26]. It was therefore assumed that addition of W to

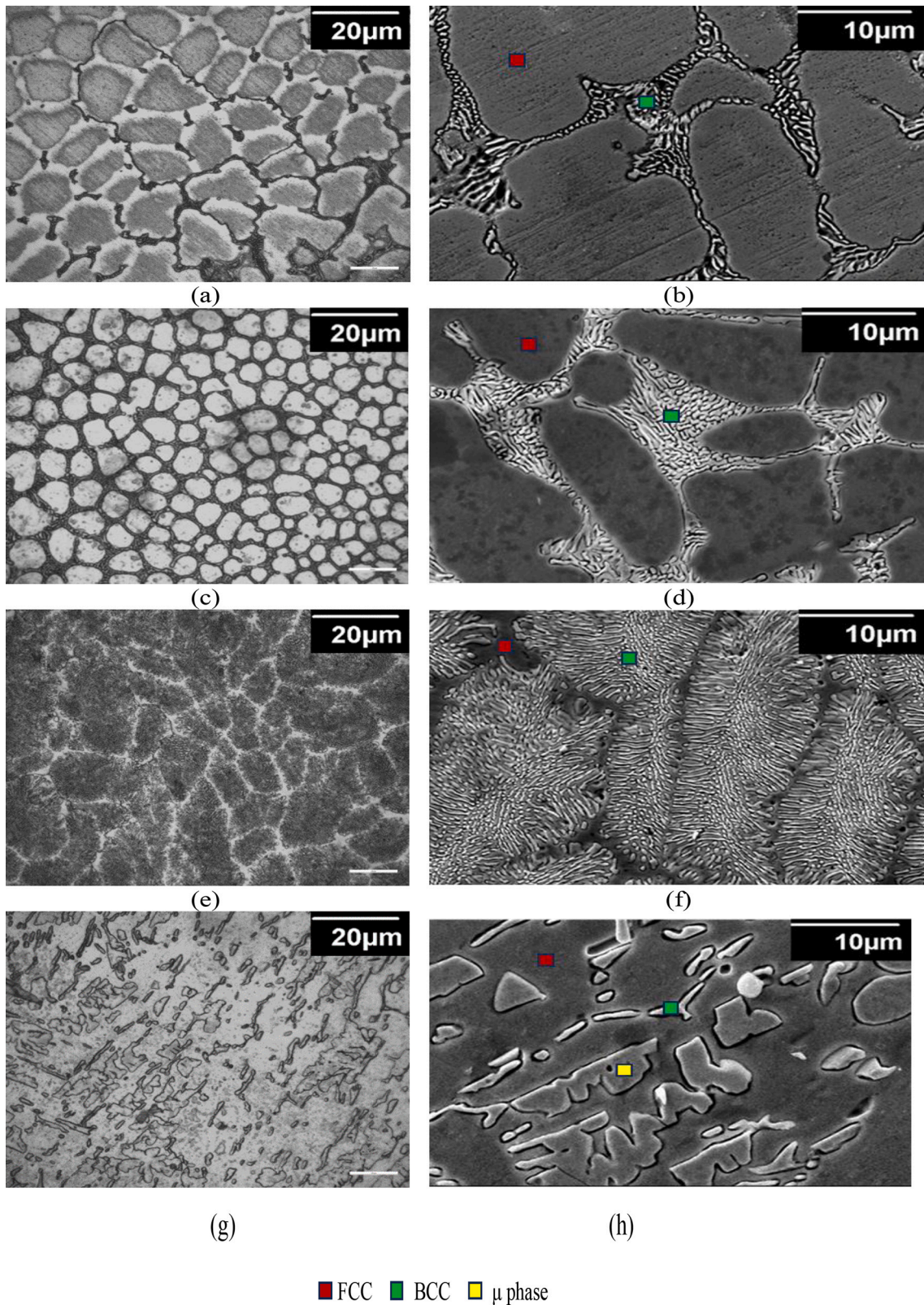


Fig. 3. Optical (left) and SEM (right) images of developed HEA: (a,b) CoCrFeNiW_{0.25}, (c,d) CoCrFeNiW_{0.5}, (e,f) CoCrFeNiW_{0.75}, (g,h) CoCrFeNiW_{1.0}.

the CoCrFeNi system may result in the development of eutectic mixture of finely distributed fcc phase and intermetallic/bcc phase. Determination of a true multicomponent phase diagram for evaluating the possibility of eutectic reaction in CoCrFeNi-W_x HEAs is a very difficult and time-consuming task. A pseudo binary phase diagram of CoCrFeNi-W

system was therefore calculated with the help of the ThermoCalc software by using its TCHEA database. The calculated pseudo binary phase diagram of CoCrFeNi-W system is shown in the Fig. 1.

The calculated CoCrFeNi-W phase diagram clearly showed the presence of a eutectic reaction at 0.075 mol fraction of W. Different alloy

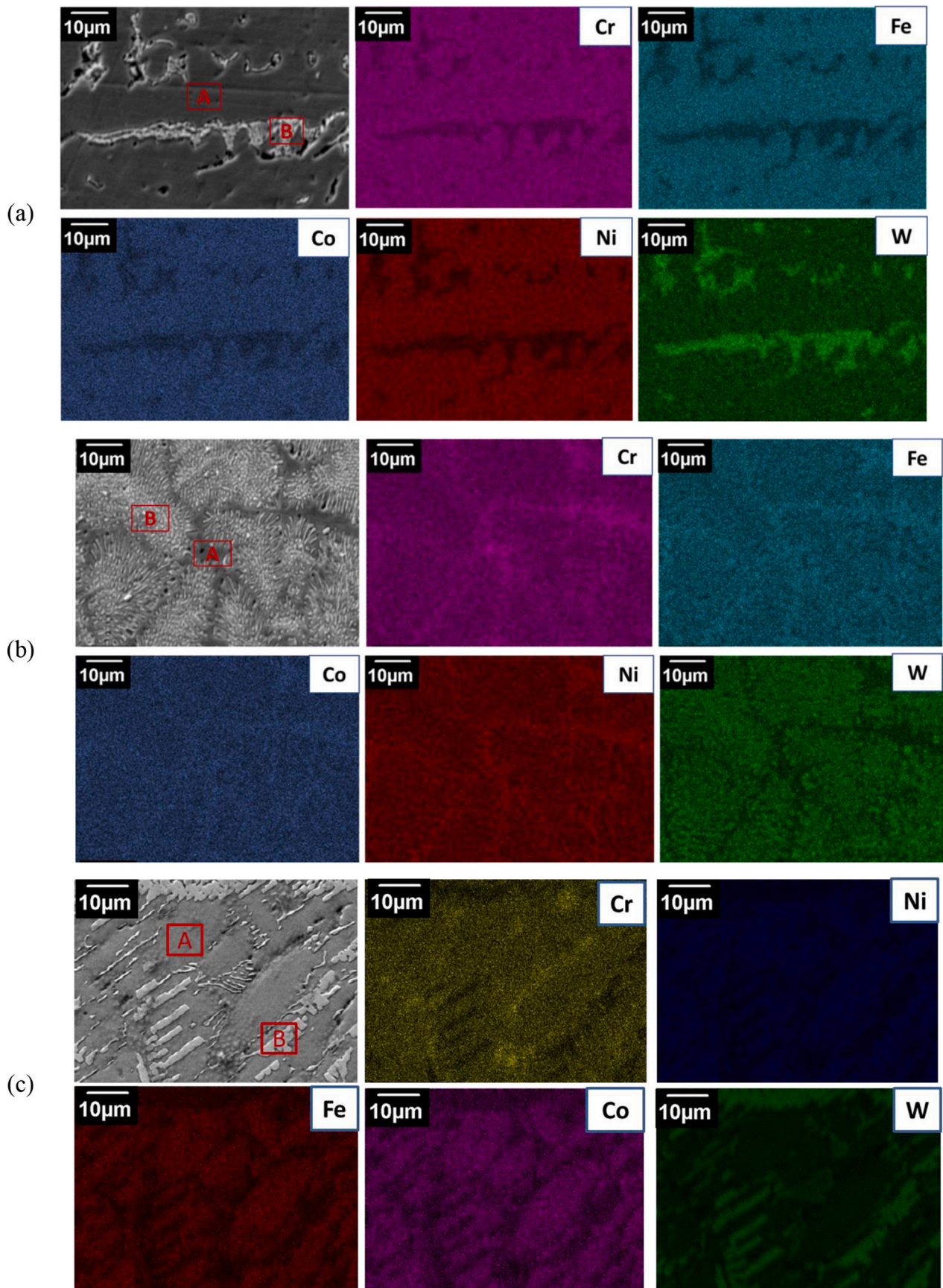


Fig. 4. SEM image and EDS mapping of (a) CoCrFeNiW_{0.5}, (b) CoCrFeNiW_{0.75}, and (c) CoCrFeNiW_{1.0}.

Table 2Composition of different regions in CoCrFeNiW_x alloys.

Alloy	Region	Co	Cr	Fe	Ni	W
CoCrFeNiW _{0.5}	A: fcc matrix	24.0	22.8	23.4	22.6	7.2
	B: eutectic mixture	20.8	20.0	20.9	17.5	12.8
CoCrFeNiW _{0.75}	A: fcc matrix	22.6	23.2	23.9	23.2	7.1
	B: eutectic mixture	22.1	20.8	22.4	19.3	15.4
CoCrFeNiW _{1.0}	A: fcc matrix	23.3	20.5	24.0	24.0	8.2
	B: μ phase	19.6	18.9	18.2	12.9	30.5

compositions were prepared by arc melting under argon atmosphere to validate the calculated phase diagram shown in the Fig. 1. Exact composition of HEAs shortlisted for the present study are given in Table 1 which are also superimposed on the pseudo binary phase diagram of CoCrFeNi-W HEA system, shown in the Fig. 1.

The crystal structure characterization of the developed alloys was carried out with the help of X-ray diffraction (XRD) analysis. X-ray diffraction patterns of the studied alloy compositions are shown in Fig. 2.

The XRD analysis of CoCrFeNiW_{0.25} and CoCrFeNiW_{0.5} indicated presence of FCC phase and BCC phases. CoCrFeNiW_{0.75} and CoCrFeNiW_{1.0} HEAs were identified as 3 phase alloys consisting of fcc, bcc and the μ phase. The number and relative intensity of peaks corresponding to the presence of μ phase in CoCrFeNiW_{0.75} was very small indicating its possible presence in very small amount. The relative intensity of peaks corresponding to the bcc and μ phase increased in the CoCrFeNiW_{1.0}. Slight shift of the peaks corresponding to the fcc phase towards lower 2 θ angles was attributed to the expansion of fcc crystal lattice by addition of tungsten (larger atomic radii element). The effect of addition of W on the microstructure of CoCrFeNi HEA was studied with the help of optical microscopy and scanning electron microscopy. Optical microscopy images and scanning electron micrographs of the developed HEAs are shown in Fig. 3.

SEM images of CoCrFeNiW_{0.25} revealed presence of eutectic mixture in small amounts at grain boundaries of the matrix phase. Matrix phase was identified as the fcc phase and as the primary crystallization phase. The eutectic mixture present at grain boundaries was found to consist of the fcc phase and the bcc phase. CoCrFeNiW_{0.25} was identified as the hypoeutectic HEA. The microstructure of CoCrFeNiW_{0.5} (Fig. 3(c,d)) was found similar to that of CoCrFeNiW_{0.25} but it contained relatively

higher amount of eutectic mixture (bcc phase + fcc phase) at the grain boundaries. CoCrFeNiW_{0.5} was also identified as the hypoeutectic HEA. CoCrFeNiW_{0.75} showed the presence of a nearly complete eutectic microstructure. XRD analysis of CoCrFeNiW_{0.75} pointed out presence of μ phase which could not be resolved in the SEM images because of its presence in very small amounts. Microstructure of CoCrFeNiW_{1.0} (Fig. 3 (g,h)) was found to be very different from the microstructures of CoCrFeNiW_{0.25}, CoCrFeNiW_{0.5}, and CoCrFeNiW_{0.75}. It consisted of dendrites, fine lamellae, and dark grey matrix. The XRD pattern of CoCrFeNiW_{1.0}, besides indicating presence of the fcc phase and bcc phase, showed the presence of reasonable amount of intermetallic μ phase. By combining SEM and XRD results, μ phase was identified as the dendritic phase and as the primary crystallizing phase while light grey lamellae and dark matrix were identified as bcc phase and fcc phases respectively. EDS mapping of a hypoeutectic HEA compositions (CoCrFeNiW_{0.5}), nearly eutectic composition (CoCrFeNiW_{0.75}), and hypereutectic HEA composition (CoCrFeNiW_{1.0}) was carried out to study elemental distribution. Results of EDS mapping are shown in Fig. 4.

As shown in Fig. 4, Co, Cr, Fe, Ni were found to be evenly distributed in the matrix (fcc) phase of CoCrFeNiW_{0.5} along with presence of small amount of W. The eutectic mixture (marked as region B) was found to be tungsten rich due to the presence of bcc phase in the eutectic mixture. Segregation of W to the eutectic region was also confirmed in the EDS mapping of the CoCrFeNiW_{0.75} (nearly eutectic HEA). EDS mapping of CoCrFeNiW_{1.0} revealed that the dendritic phase (μ) and lamellae phase (bcc) contained most of the added W. Results of the point analysis of regions highlighted in the Fig. 4, are given in Table 2.

Contrary to the calculated pseudo binary phase diagram shown in Fig. 1, crystal structure characterization of the studied compositions revealed absence of σ phase in the studied compositions. σ is the Fe—Cr binary phase and has low temperature stability (up to 820 °C in the Fe—Cr system) [25,26]. Sluggish diffusion kinetics and relatively higher configurational entropy made the fcc phase more stable in the CoCrFeNi system [27]. Precipitation of σ phase was therefore not evidenced in any of the previous studies on this system. Chance of precipitation of σ phase in CoCrFeNi-W systems becomes even lower due to increase in the configurational entropy by addition of W and relatively lower temperature stability of the σ phase [28]. Instead presence of bcc was witnessed due to its high temperature stability [29]. μ phase is the Fe—W binary phase and has significantly high temperature stability (up to 1637 °C in

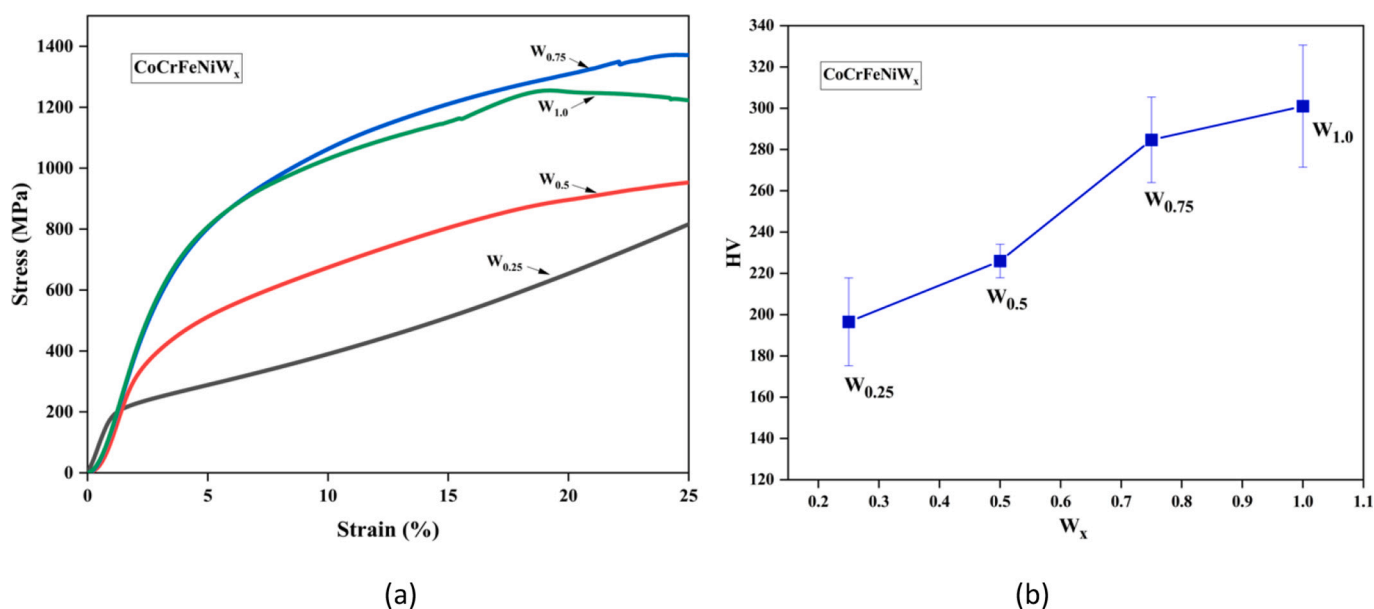


Fig. 5. Mechanical characterization results of developed CoCrFeNiW_x high entropy alloys: (a) compressive stress vs. strain curves (b) variation of hardness as a function of amount of W in CoCrFeNiW_x.

Table 3

Yield strength, % plastic strain and Vickers hardness mean values for CoCrFeNiW_x HEA.

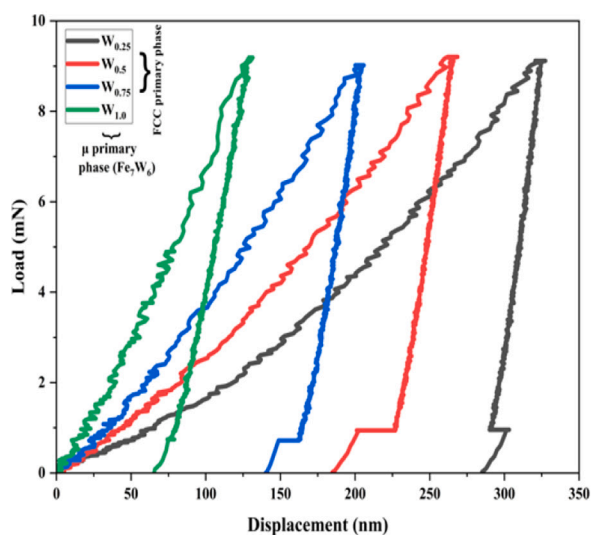
Alloy	Yield Strength (MPa)	Plastic strain (%)	Vickers hardness (HV)
CoCrFeNiW _{0.25}	252	>25	305
CoCrFeNiW _{0.5}	385	>25	447
CoCrFeNiW _{0.75}	712	>25	556
CoCrFeNiW _{1.0}	707	>25	560

Fe—W system) and therefore has more chances of showing up in the CoCrFeNi-W binary phase diagram [30]. XRD analysis revealed presence of μ phase besides the existence of fcc and bcc phases. The fcc phase was found to be the primary crystallizing phase in CoCrFeNiW_{0.25}, CoCrFeNiW_{0.5}, and CoCrFeNiW_{0.75} alloys. From the amount of eutectic mixture

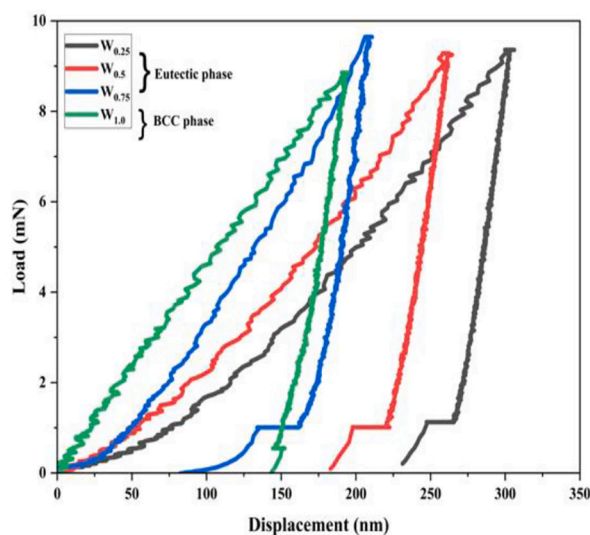
in the CoCrFeNiW_{0.75}, it was concluded that eutectic point exists to the right of but very close to the CoCrFeNiW_{0.75} composition. A comparison of calculated phase diagram and obtained experimental results revealed that the eutectic composition in the calculated phase diagram is shifted to the right. The eutectic mixture in the studied compositions was found to consist of fcc and bcc phases instead of fcc and σ phase as predicted by the calculated phase diagram.

The effect of eutectic microstructure on the mechanical properties of the developed alloys was evaluated by carrying out compression, hardness, and nano indentation tests. The results of compression test and hardness test are shown in Fig. 5(a) and Fig. 5(b) respectively.

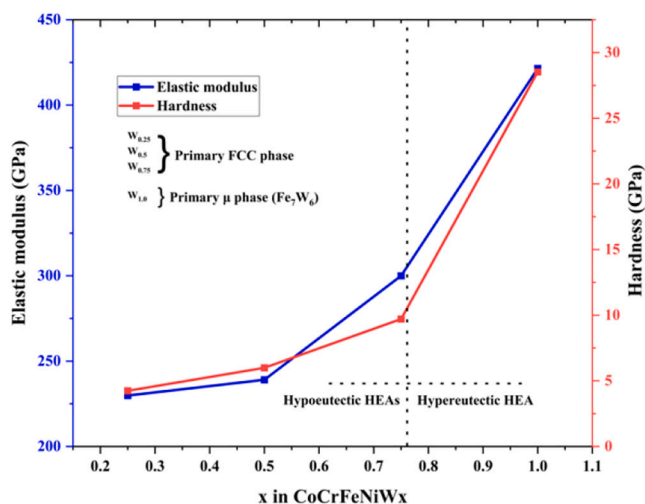
CoCrFeNiW_{0.25} showed yield strength of 252 MPa. A significant jump in the yield strength was observed for CoCrFeNiW_{0.5} without decrease in ductility. The increase in yield strength at this EHEA composition was attributed to the presence of hard bcc phase in the microstructure [24].



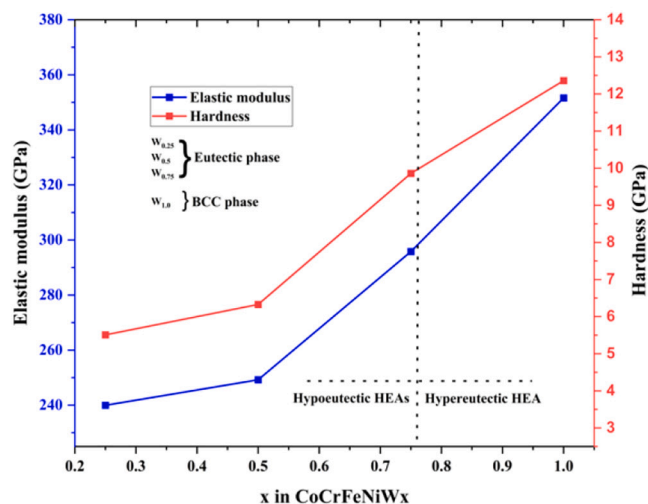
(a)



(b)



(c)



(d)

Fig. 6. Load-displacement curves of (a) primary phases and (b) eutectic mixture in CoCrFeNiW_x HEAs. Hardness and elastic modulus of (c) primary phases and (d) eutectic mixture in CoCrFeNiW_x HEAs.

Fine distribution of the hard and the soft phase in the eutectic mixture helped in obtaining a better combination of yield strength and toughness. The yield strength of CoCrFeNiW_{0.75} in comparison to the CoCrFeNiW_{0.5} was significantly higher while the amount of toughness remained the same. This increase in the properties of CoCrFeNiW_{0.75} in comparison to that of the CoCrFeNiW_{0.5} was attributed to the presence of relatively higher amount of eutectic mixture in the microstructure as shown in Fig. 3. The yield strength of CoCrFeNiW_{1.0} was found to be slightly lower than that of the CoCrFeNiW_{0.75}. This modest decrease in the yield strength can be attributed to the absence of eutectic mixture in its microstructure. A comparison of the mechanical properties of developed alloys is shown in the Table. 3. Hardness of the developed alloys was found to increase with increasing amount of W in the studied alloys as it resulted in the formation of hard μ and bcc phases. The hardness values of the studied compositions are given in Table 3.

The hardness and elastic modulus of the primary crystallizing phases and eutectic mixture present in CoCrFeNiW_x were determined with the help of nanoindentation. A comparison of the load-displacement curves, hardness, and elastic modulus of primary phases and eutectic mixture is shown in Fig. 6.

The load-displacement curves of the primary phase and of the eutectic mixture showed maximum displacement at peak load for CoCrFeNiW_{0.25} which was found to decrease with increase in the amount of W in the CoCrFeNi HEA. Increase in the hardness and elastic modulus of the primary fcc phase (CoCrFeNiW_{0.25} - CoCrFeNiW_{0.75}) with increase in the amount of W was attributed to the solid solution strengthening. μ phase was present as the primary phase in the CoCrFeNiW_{1.0} HEA and was found to be harder than the primary phase (fcc) of the CoCrFeNiW_{0.75} HEA. The hardness of the eutectic mixtures in CoCrFeNiW_x followed a similar trend as observed for the primary phase. The eutectic mixture of CoCrFeNiW_{0.25} showed least hardness and hardness of the eutectic mixture of CoCrFeNiW_{0.75} was found to be the highest. No eutectic structure was evidenced in CoCrFeNiW_{1.0} HEA. Intermetallic phase present in CoCrFeNiW_{1.0} in large amounts showed highest hardness. It is also evident from Fig. 6 that hardness and elastic modulus of eutectic phase is higher than that of primary phases in hypoeutectic compositions. Fcc is the primary crystallizing phase in the hypoeutectic compositions. The presence of BCC phase in the eutectic mixture contributes to the relatively higher modulus and higher hardness values of the eutectic mixture. On the other hand, μ phase is the primary phase in hypereutectic compositions. The presence of soft fcc in the eutectic mixture along with hard bcc results in lower hardness and modulus values than the hard primary phase.

The present study has also revealed that mechanical properties of high entropy alloys can be significantly improved by introduction of eutectic microstructure consisting of finely distributed hard and soft phases. The addition of W in CoCrFeNi HEA is assumed to increase its lattice distortion because of its significantly larger atomic radii and caused solid solution strengthening. Increase in the lattice distortion by addition of W was verified by shift of the fcc peaks to the left in the XRD patterns of studied compositions. As a result, a small gain in the yield strength without any compromise on the toughness was evidenced. The addition of W beyond the solubility limit of selected HEA resulted in the outstanding combination of strength and ductility which was attributed to the development of fine eutectic microstructure of the tough (fcc) and the hard phase (bcc).

4. Conclusion

- Hypo-eutectic, hyper-eutectic and nearly eutectic high entropy alloys have been successfully designed and developed with the help of calculated pseudo-binary phase diagram of the CoCrFeNi-W system.
- Experimental results showed some deviations from the calculated phase diagrams. However, calculated pseudo-binary phase diagram has shown excellent potential to serve as a tool to check the

possibility of eutectic reaction in high entropy alloys and to design different types of eutectic high entropy alloys.

- Development of eutectic microstructure has helped in obtaining excellent combination of hardness, strength, and ductility. Hardness of the samples increased from 305 HV for CoCrFeNiW_{0.25} to 560 HV for CoCrFeNiW_{1.0}, whereas yield strength increased from around 252 MPa for CoCrFeNiW_{0.25} to 712 MPa for CoCrFeNiW_{0.75}. % elongation of the studied samples remained >25%.
- It is therefore concluded that development of eutectic microstructures in high entropy alloys can help in overcoming strength ductility tradeoff and utilization of calculated pseudo binary phase diagrams can help in significantly reducing the experimental effort required in this regard.

CRedit authorship contribution statement

M. Abdullah: Investigation, Data curation. **Muhammad Mukarram:** Investigation, Data curation. **Talha Bin Yaqub:** Writing – review & editing. **Filipe Fernandes:** Writing – review & editing. **Khurram Yaqoob:** Conceptualization, Funding acquisition, Project administration, Supervision.

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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