

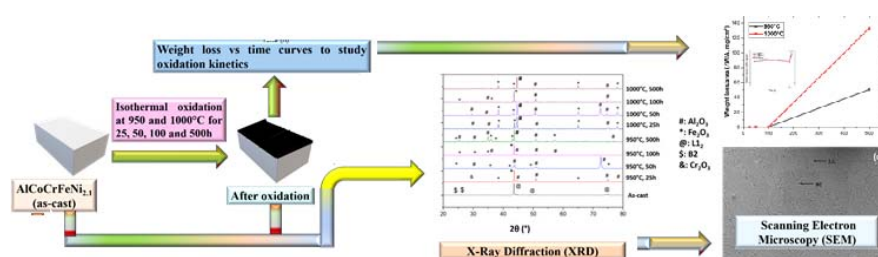
Deciphering the role of Al_2O_3 formed during isothermal oxidation in a dual-phase $\text{AlCoCrFeNi}_{2.1}$ Eutectic High-Entropy Alloy

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ABSTRACT



In recent times, there has been a significant volume of work on Eutectic High Entropy Alloys (EHEAs) owing to their remarkable castability combined with excellent mechanical properties, which aids in clearing obstacles for their technological applications. One of the most common EHEAs, which has been of enormous interest at present, primarily owing to its solidification and tensile behavior, is $\text{AlCoCrFeNi}_{2.1}$. However, for its high-temperature applications, oxidation behaviour of this material is one of the major aspects that needs to be extensively investigated. To this end, the present work aims to study the phases evolved during oxidation at elevated temperatures as high as 950 and 1000°C in $\text{AlCoCrFeNi}_{2.1}$ using XRD and also to determine the rate law followed for isothermal oxidation of this alloy at 950°C and 1000°C, in order to understand the role of Al_2O_3 phase formed during isothermal oxidation at 950°C and 1000°C.

Keywords: Oxidation, Eutectic High-Entropy Alloys (EHEAs), Optical microscopy, X-Ray Diffraction (XRD), SEM

INTRODUCTION

High-entropy alloys (HEAs), or multi-principal-element alloys, differentiate from conventional alloys as these have at least four principal elements, instead of one or at most, two in the latter. This has led to a paradigm shift in alloy design strategies and opened a new path for the exploration of new materials.

A significant volume of work has been done on understanding solidification behaviour of Eutectic HEAs (EHEAs), mainly, due to the excellent castability of these alloys,¹⁻⁴ arising due to meagre chances of segregation and shrinkage cavities during solidification.

Accordingly, if eutectic HEAs with the dual-phase FCC + BCC microstructure may be synthesised, they may be expected to possess the combined advantages of excellent mechanical properties and castability of eutectic alloys coupled with a remarkable strength-ductility optimization arising out of a dual-phase FCC+BCC microstructure, especially when both FCC and BCC phases are ordered.¹⁻⁹ This has primarily been the ideology behind designing EHEAs, as reported by a number of recent studies.¹⁻⁹ In this context, $\text{AlCoCrFeNi}_{2.1}$ is a popular EHEA, in which quite a significant volume of work has been done on correlating the dual-phase microstructure comprising of ordered FCC (L_{12}) and ordered BCC (B_2) phases with the overall solidification behaviour and mechanical response of the alloy, synthesized using different techniques.¹⁻⁸ With regard to oxidation, there have been a number of publications that have reported the oxidation behaviour of HEAs. For instance, Daoud et al.¹⁰ examined the oxidation behaviour of $\text{Al}_{18}\text{Co}_{17}\text{Cr}_{17}\text{Cu}_8\text{Fe}_{17}\text{Ni}_{33}$, $\text{Al}_{23}\text{Co}_{15}\text{Cr}_{23}\text{Cu}_8\text{Fe}_{15}\text{Ni}_{15}$, and $\text{Al}_{17}\text{Co}_{17}\text{Cr}_{17}\text{Cu}_{17}\text{Fe}_{17}\text{Ni}_{17}$ HEAs at 800°C and 1000°C in air and reported that at 800°C, that the alloy

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with lower Al content forms NiO along with other oxides, namely, Fe_2O_3 , Cr_2O_3 , and Al_2O_3 , unlike at 1000°C , where the same alloy preferentially forms Cr_2O_3 over Al_2O_3 , in contrast to the oxidation behaviour of HEAs with higher Al content, forming Al_2O_3 scales at both temperatures (800°C and 1000°C). Holcomb et al.¹¹ investigated the oxidation behaviour of 8 CoCrFeMnNi-based HEAs, along with different conventional alloys, and reported that the Cr and Mn containing HEAs preferentially form both Cr_2O_3 and Mn oxides. Senkov et al.¹² examined the isothermal oxidation behaviour of an arc-melted NbCrMo_{0.5}Ta_{0.5}TiZr refractory HEA (RHEA) at 1000°C and reported a superior oxidation resistance of the aforementioned alloy as compared to that of similar Nb-based refractory alloys. Similarly, Gorr et al.^{13,14} investigated the influence of Si on the high-temperature oxidation behaviour of a 20Nb-20Mo-20Cr-20Ti-20Al HEA and reported that, in general, for HEAs with no Si content, a linear oxide growth rate law is followed with the formation of porous and non-protective oxides whereas, the addition of even 1 at % Si promotes parabolic oxide growth kinetics, to a certain extent, and leads to the formation of a thin, continuous and protective Al and Cr rich oxide scale. Moreover, as reported by Lu et al.,¹⁵ EHEAs are excellent candidates for high-temperature applications, owing to: (i) near-equilibrium microstructures which resist change at elevated temperatures; (ii) the presence of low-energy interphase boundaries; (iii) excellent rupture strength and (iv) creep resistance at high-temperature.¹⁶

In Al-Co-Cr-Fe-Ni system, Al and Cr have been reported by Chen et al.¹⁷ to promote the formation of dense, continuous oxidation scales to inhibit further oxidation of the alloys. However, the presence of Fe limits the oxidation resistance of these alloys.^{17–20} Unfortunately, for most HEAs, understanding the oxidation behaviour of HEAs based on Al-Co-Cr-Fe-Ni system is rather complicated owing to the equiatomic and/or near-equiatomic composition in these alloys.^{21–25} Besides, the possibility of (i) inhibition of non-protective transient oxides due to sluggish diffusion kinetics¹⁶ and (ii) delay in the formation of protective oxides scales in these alloys¹¹ have also been reported. In other words, the oxidation behaviour of HEAs is influenced by the mutual interaction of a large number of: (i) microstructure-related and (ii) processing-related factors.¹⁷ This necessitates a systematic research on the oxidation behaviour of HEAs since the oxidation behaviour of a material system plays a key role in determining the performance of the material component at high temperatures.^{18,26,27}

However, in view of limited understanding on the oxidation behaviour of AlCoCrFeNi_{2.1} EHEA upto 1200°C , present work is aimed at understanding the nature of Al_2O_3 phase formed during isothermal oxidation of AlCoCrFeNi_{2.1} EHEA at 950°C and 1000°C .

EXPERIMENT

Initially, a single sample (in the shape of a button) of AlCoCrFeNi_{2.1} was prepared from Suction Casting of pure components (purity > 99.9%) (on a water-cooled Cu hearth, under Ar inert atmosphere and using Ti as getter). The bar with a rectangular cross-section, so obtained was flipped and remelted multiple times to promote homogeneity and then cut into eight samples of average dimensions ~ 10 by 6 mm² (approximate thickness ~ 1.2 mm) using EDM (Electro Discharge Machining).

After standard metallographic polishing, the 1st and 2nd samples were subjected to isothermal oxidation at 1000°C , 500h in a muffle furnace (CWF-1300). The 3rd sample was subjected to isothermal oxidation at 1000°C , 100h in Al_2O_3 crucible, using Kanthal wire as heating element and a static oxidation environment. The 4th and 8th sample were similarly put in the same furnace at 1000°C and 950°C , respectively, and each of them was taken out after 4, 18, 25, 50, and 100h. Similarly, samples 5 and 6 were oxidized at 950°C , 500h, and sample 7 at 950°C , 100h. Phase analysis of the base and oxidized samples was carried out using XRD (X-Ray Diffraction, Bruker) using $\text{Cu}_{\text{K}\alpha}$ radiation at 45 mV and 30 mA and, at a scanning rate of $0.02^\circ/\text{min}$ between the scanning range of 20 to 80° and peak fitting was done using Xpert Hi-score analysis software, in order to determine the phases present in the oxidised layer, at different temperatures and time of oxidation. For microstructural characterization, the base sample was etched with aqua regia (90% $\text{C}_2\text{H}_5\text{OH} + 10\% \text{HNO}_3$). The cross-sectional area and difference in weight (before and after oxidation) of every sample were measured to determine the oxidation rate law at different oxidation temperatures.

RESULTS AND DISCUSSIONS

Phase analysis during oxidation using X-Ray Diffraction (XRD)

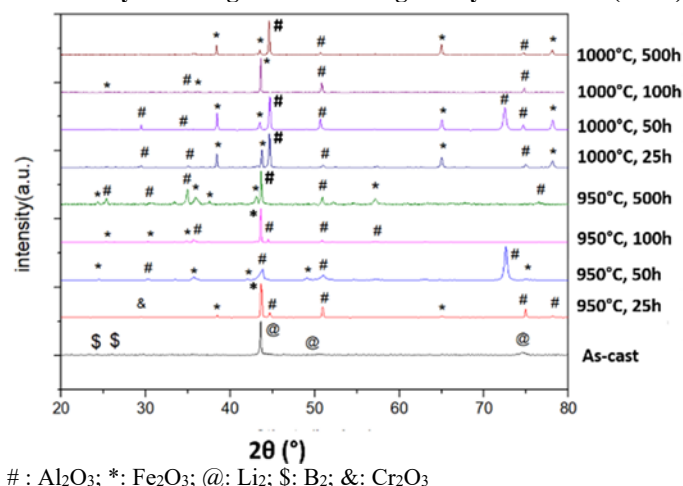


Figure 1. Stacked XRD plots of as-cast (at the bottom, marked as black) to oxidized samples at different temperatures and time of isothermal oxidation (marked as red, blue, pink, green, deep blue, violet indigo, and maroon).

Figure 1 shows the X-Ray diffractograms of as-cast along with oxidised samples. The variation in terms of angular Wavenumber (k), with the exception of 1000°C , 100h sample, is observed from 1 to 5. Whereas the variation in the same quantity is observed between 2 and 5 in 1000°C , 500h sample. Moreover, It is evident (from Figure 1) that in as-cast sample, there is a dual-phase microstructure comprising of B2 (ordered BCC) and L_{12} (ordered FCC) phases (lattice parameters: $3.6011\text{\AA}$ and $5.9375\text{\AA}$, respectively), with the most intense (111) superlattice peak at $2\theta = 43.5^\circ$, corresponding to L_{12} phase. Besides, it is also observed that in the as-cast sample, there are more peaks of L_{12} phase as compared to that of B2. After isothermal oxidation at varying temperatures and oxidation times till 1000°C , 500h, there are three

phases detected by XRD analysis: $L1_2$, Al_2O_3 , and Fe_2O_3 . After 25h of isothermal oxidation at $950^\circ C$, Cr_2O_3 phase is also observed. However, after 100h of oxidation, it is clearly observed that there are two oxides present (similar to that observed after isothermal oxidation at $950^\circ C$ for 25h) namely, Al_2O_3 and Fe_2O_3 phases, with the most intense (113) peak of Fe_2O_3 at $2\theta = 43.5^\circ$, indicating that Fe_2O_3 is the predominant oxide phase at $950^\circ C$, 100h sample. Unlike Al_2O_3 , Fe_2O_3 phase forms a non-protective oxide layer, but the exact information about oxides may be gained only by looking at cross-sections using SEM.¹⁵⁻¹⁷ After 500h of isothermal oxidation at $950^\circ C$, the most intense (113) peak corresponds to that of Al_2O_3 at exactly $2\theta = 43.5^\circ$ indicating that Al_2O_3 phase forms the matrix with 2nd phase as Fe_2O_3 , similar to the microstructure after

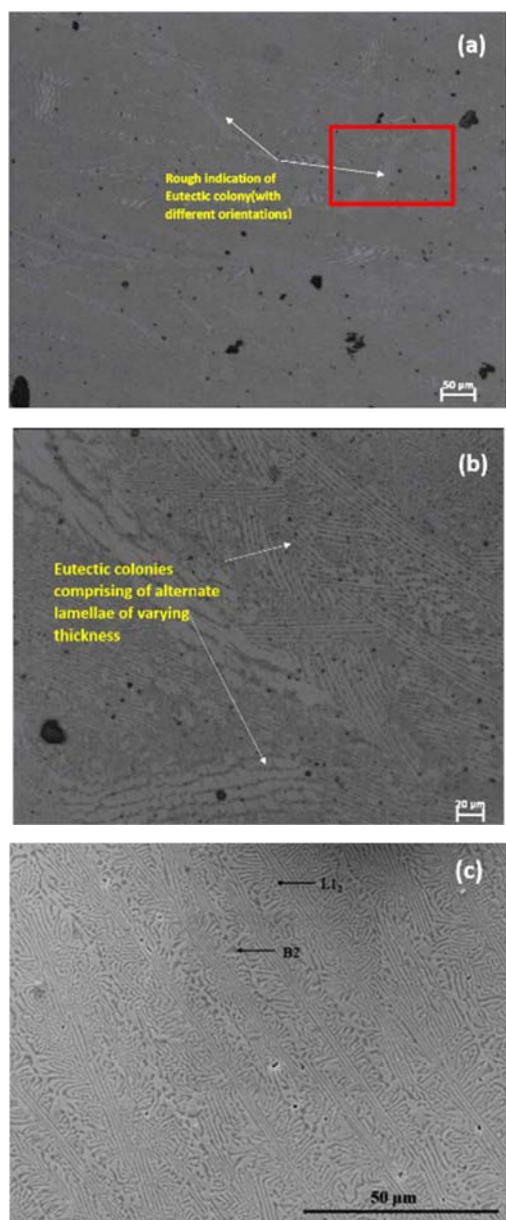


Figure. 2 As-cast sample: (a) Optical image (100x), (b) Optical image (500x) of the region highlighted with a red outlined enclosure in part (a); and (c) SEM image (3000x) showing eutectic colonies with alternate lamellae (of various thickness) of $L1_2$ and $B2$ phases (enclosed within orange outlined rectangular box in part (c))

50h of oxidation at $950^\circ C$. Besides, after $1000^\circ C$, 100h of isothermal oxidation, the volume fraction determination of individual phases based on the relative intensities of XRD peaks clearly indicates that Fe_2O_3 phase forms the matrix and Al_2O_3 forms the 2nd phase in a dual-phase $Al_2O_3 + Fe_2O_3$ microstructure, similar to that in samples oxidized at $950^\circ C$ for 25 and 100h. However, after 500h of isothermal oxidation at $1000^\circ C$, the most intense (113) peak of Al_2O_3 with its position (2θ) shifted slightly to the right, i.e., at $2\theta = 43.7^\circ$, indicates a dual-phase microstructure comprising of Al_2O_3 (matrix) and Fe_2O_3 phases, similar to that in samples after oxidation at $950^\circ C$ for 50 and 500h and that after oxidation at $1000^\circ C$ for 25 and 50h although the detailed phase analysis, is beyond the scope of discussion of the present work, as because XRD, being a bulk characterization technique, cannot detect phases with vol. fraction of less than 0.05.

Microstructural characterization of as-cast sample

Both optical and SEM images reveal eutectic colonies with alternate lamellae (of various thickness) of $L1_2$ and $B2$ phases. Based on image analysis using ImageJ software, the average thickness of $L1_2$ phase is found to be nearly $1\ \mu m$, whereas that of $B2$ lamella is found to be nearly $1.2\ \mu m$. Besides, the phase fraction of $L1_2$ and $B2$ phases (based on variation in contrast) in the as-cast microstructure is also found to be 0.89 and 0.11, respectively, from Figures. 2(a-c), similar to that inferred from the XRD of as-cast sample in Figure. 1.

Isothermal oxidation kinetics at $950^\circ C$ and $1000^\circ C$

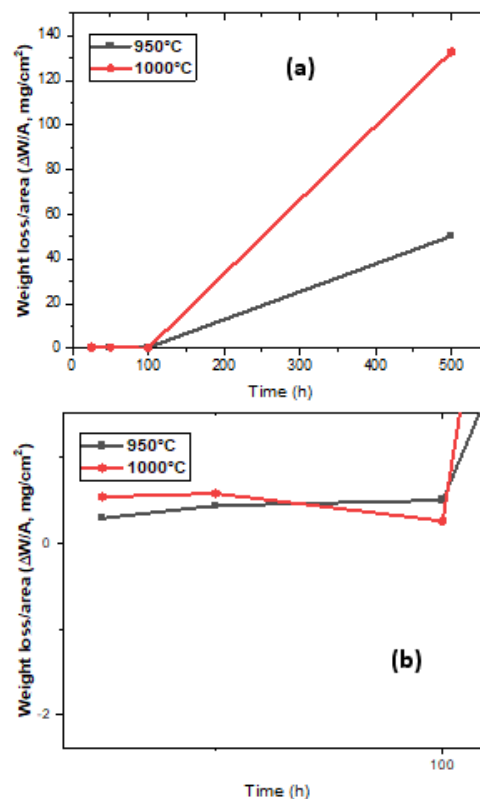


Figure. 3 (a) Comparative Isothermal oxidation plots of $AlCoCrFeNi_{2.1}$ EHEA at 950 and $1000^\circ C$ between 4h and 500h of oxidation, (b) Magnified view of part (a) between 25 to 100 h of oxidation.

From the previous studies of oxidation behaviour of AlCoCrFeNi_{2.1} EHEA,^{1–8} it has been reported that at elevated temperatures between 900°C and 1300°C, the weight loss curve for oxidation initially shows a parabolic nature, which saturates with higher periods of oxidation.

(Figure. 3(a)), its subplot (Figure. 3(b)) indicates that irrespective of considerable fluctuations in these weight loss curves due to reasonable amounts of spallation, a linear rate law is observed at both 950°C and 1000°C, with an increase in oxidation time at both these temperatures. Combining the observation on the phase fraction of Al₂O₃ and Fe₂O₃ phases (Figure. 1) with the weight loss curves (Figures. 3(a and b)), it may be inferred that a slightly higher volume fraction of Al₂O₃ phase (in a dual-phase Al₂O₃ + Fe₂O₃ microstructure) after isothermal oxidation at 1000°C for 500h (that after oxidation at 950°C for 500h) leads to a significantly higher amount of weight loss than that after isothermal oxidation at 950°C for 500h. This further indicates that Al₂O₃ scale formed during oxidation at elevated temperatures in AlCoCrFeNi_{2.1} EHEA aids in further oxidation, unlike the reported nature of Al₂O₃ scale.^{1–4}

Comparison with the oxidation behaviour of Al_{1.3}CrFeNi Eutectic High Entropy Alloy (EHEA)

In the context of oxidation of another commonly investigated EHEA: Al_{1.3}CrFeNi with a dual-phase BCC + B2 microstructure at room temperature, Chen et al.¹⁷ have reported that on the basis of a few fundamental studies on the oxidation of HEAs, before steady-state conditions of oxidation is established, transient oxidation occurs for a few hours before attaining a steady-state (in terms of oxidation conditions).^{28,29} During this period, the fast uptake of oxygen by the alloy leads to the formation of nonuniform oxide phases including spinel.^{24,25} Moreover, during the initial stage of oxidation, interfacial reaction between the surface of the material and the surrounding atmosphere plays a key role due to the presence of high partial pressure of oxygen on the surface.¹⁷ The rapid uptake of oxygen converts the surface layer of the alloy to oxides. During transient oxidation, forming localized oxides (including spinel) because of a small amount of diffusion.¹⁷

As the oxidation proceeds, diffusional processes tend to dominate the oxidation process.¹⁷ Due to the presence of external oxide scale (for instance, spinel), the activity of oxygen in Cr and Al is not sufficient, leading to an inward diffusion of oxygen in the alloy.^{30–33} Besides, it has also been reported that the activity of oxygen required to oxidize Cr is much larger as compared to that for Al.^{34,35} Meanwhile, further diffusion of Fe has been reported to occur in the surface oxide scales.¹⁷ In the external mixture of Al₂O₃ and Cr₂O₃ scales, there is a possible substitution of Cr cations by Fe cations leading to the formation of FeCr₂O₄ (spinel).³⁴ As the oxidation proceeds further, Cr₂O₃ and Al₂O₃ in the middle oxide scale prevent the inward diffusion of oxygen from the outer scale into the base alloy.¹⁷ After several hours of oxidation, Al₂O₃ transforms into a continuous oxide scale and the transport of Al through the oxide scale of Al₂O₃ tends to control the oxidation process and the oxidation process tends to follow a parabolic rate law at 1000°C.^{17,31–35} On the contrary, Al₂O₃ scale formed during isothermal oxidation of AlCoCrFeNi_{2.1} EHEA (with a dual-phase

L1₂ + B2 microstructure at room temperature, investigated in the present work) for 25, 50, 100 and 500h, each at 950°C and 1000°C, promotes further oxidation of the base alloy. Besides, AlCoCrFeNi_{2.1} tends to follow a linear rate law, at both 950°C and 1000°C, irrespective of oxidation time (Figure. 3(a)). However, detailed microstructural investigations for the purpose of revealing the mechanism of further oxidation of the base alloy even in presence of Al₂O₃ scale in AlCoCrFeNi_{2.1} EHEA is beyond the scope of discussion of the present work.

CONCLUSIONS

During isothermal oxidation at 950°C and 1000°C, for oxidation periods ranging from 25 to 500h, AlCoCrFeNi_{2.1} EHEA (with a dual-phase L1₂ + B2 microstructure) shows a dual-phase Al₂O₃ + Fe₂O₃ microstructure. At 950°C, there is phase reversion from Al₂O₃ to Fe₂O₃ when oxidation is performed till 50h. However, Fe₂O₃ reverts back to Al₂O₃ when oxidation is performed at 950°C for 100 and 500h. Similarly, At 1000°C, there is phase reversion from Al₂O₃ to Fe₂O₃ when oxidation is performed till 100h followed by a re-reversion from Fe₂O₃ to Al₂O₃ phase for oxidation till 500h at 1000°C. Irrespective of considerable fluctuations in weight loss curves due to reasonable amounts of spallation, a linear rate law is observed at both 950°C and 1000°C, with increase in time of oxidation at both these temperatures from 4h till 500h. Al₂O₃ scale formed during oxidation at elevated temperatures in AlCoCrFeNi_{2.1} EHEA aids in further oxidation, unlike the reported nature of Al₂O₃ scale.

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