



Microstructure and wear resistance of an intermetallic-based $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ high entropy alloy

Bharat Gwalani ^{a, b}, Aditya V. Ayyagari ^b, Deep Choudhuri ^a, Thomas Scharf ^{a, b},
Sundeep Mukherjee ^{a, b}, Mark Gibson ^{c, d}, Rajarshi Banerjee ^{a, b, *}

^a Advanced Materials and Manufacturing Processes Institute, University of North Texas, Denton, TX, 76207, USA

^b Department of Materials Science and Engineering, University of North Texas, Denton, TX, 76207, USA

^c CSIRO Manufacturing, Private Bag 10, Clayton South, Clayton, Victoria, 3169, Australia

^d Department of Materials Engineering, Monash University, Victoria, 3800, Australia

HIGHLIGHTS

- $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ high entropy alloy (HEA) consists of a chi (χ) phase matrix with L_{21} ordered second phase.
- A third nano-twinned phase with fcc crystal structure also forms.
- Hardness of the matrix bcc chi (χ) phase is $1090 \text{ H}_v \pm 14$ and of the L_{21} phase is $570 \text{ H}_v \pm 9$.
- Ascast alloy offers low sliding coefficient of friction (~ 0.3) and low wear rate ($\sim 1.2 \times 10^{-5} \text{ mm}^3/\text{N}\cdot\text{m}$).

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ABSTRACT

An $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ high entropy alloy (HEA), consisting of multiple principal elements, forms the uncommonly observed chi-phase, which is a large lattice parameter intermetallic phase based on the body centered cubic crystal structure, as the matrix phase and a L_{21} phase (ordered Huesler phase, X_2YZ -type based on the face-centered cubic structure) as a major secondary phase. Additionally, a face centered cubic phase with a high density of nano-twins is also present in the microstructure as a third phase. The extremely high Vicker's hardness of the matrix chi phase ($1090\text{H}_v \pm 14$) and of the L_{21} phase ($570 \pm 9 \text{ H}_v$) along with low sliding coefficient of friction (~ 0.3) and low wear rate ($\sim 1.2 \times 10^{-5} \text{ mm}^3/\text{N m}$) makes this HEA a promising candidate for mechanical wear-resistant applications.

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1. Introduction

High entropy alloys (HEAs) are based on the strategy of mixing multiple principal elements (MPEs) in near equal proportions to obtain novel microstructures with enhanced properties [1–6]. The phases forming in such systems are a combined effect of thermodynamic factors such as higher configurational entropy and enthalpy of mixing, as well as kinetic factors such as diffusion coefficients of the constituent elements. The sequence of phase evolution in such HEAs is often a result of competing processes of

phase separation via spinodal decomposition or nucleation and growth and chemical ordering. Initial efforts on HEAs were largely focused on exploiting the high configurational entropy effect due to the presence of 5 or more elements (with concentrations of 5–35 atomic percent) to create a single solid solution [7–12]. However, it has eventually been realized that while at elevated temperatures the higher configurational entropy of mixing dominates often resulting in a single solid solution, at lower temperatures, the enthalpy of mixing starts manifesting via ordering and/or phase separating tendencies, eventually resulting in decomposition into multiple phases [2–5]. Furthermore, it has been observed that in many cases multiple generations of phases occur in these alloys giving rise to a hierarchical microstructure [2–5]. It is also noteworthy that most of the phases formed at room temperature still contain all the constituent elements, hence the configurational

* Corresponding author. Advanced Materials and Manufacturing Processes Institute, University of North Texas, Denton, TX, 76207, USA.

E-mail address: rajarshi.banerjee@unt.edu (R. Banerjee).

entropy does appear to play an important role in their stabilization. Instead of forming a single phase solid solution, most HEA systems tend to form a dual or triple High Entropy Phases (HEPs).

Single solid solution (SSS) *fcc* phase in high entropy alloys have been shown to be stable in $\text{Al}_{0.1}\text{CoCrFeNi}$ and CoCrFeMnNi at room temperature. Recent reports by Otto et al. [13] and Stepanov et al. [14] independently showed that CoCrFeMnNi disintegrated into Cr rich sigma phase, NiMn rich L_{10} phase, FeCo B2 phase and a Cr-rich body-centered cubic, *bcc*, phase after longer annealing times at 500 °C or above or under strain ageing conditions. Addition of a fifth element to CoCrFeNi (quaternary alloy) in equi-proportion so far has not been shown to stabilize a SSS. Especially Al, Ti and Cu beyond a certain small solubility limit have resulted in more number of phases and ordered structures [15,16].

The present study focuses on microstructural evolution and wear resistance of the $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ HEA. CoCrFeNi forms the base alloy composition for many *fcc*-based HEAs. CoCrFeNi offers a single solid solution *fcc* phase with very good work hardenability and the possibility of precipitation strengthening on addition of minimum amounts of Al, Ti and Mo. $\text{Al}_{0.3}\text{CoCrFeNi}$ [16–21], $\text{Ti}_x\text{CoCrFeNi}$ [22,23], and $\text{Mo}_x\text{CoCrFeNi}$ [24,25] have been extensively studied as precipitation hardenable HEA systems where the matrix is a single solid solution *fcc* phase and fine scale ordered *fcc* or *bcc* based precipitates are formed, thus strengthening the alloy. $\text{Nb}_x\text{CoCrFeNi}$ [26–28] systems have been shown to form eutectic high entropy alloys. AlCoCrFeNi and CoCrFeNiTi alloys have also been studied showing *bcc*+B2 [29–31] and *fcc*+laves phase structures [32] respectively. Choudhuri et al. [33] reported stabilization of the Ni_2AlTi (L_{21}) phase by adding Ti in a high entropy alloy. Al and Ti can be considered to be the elements disrupting the entropic stabilization of single solid solution phase and promote ordered phases. This is due to the highly negative enthalpy of mixing of these elements with some of the other constituent elements such as Ni, Fe and Co, resulting in the formation of intermetallic phases. Most cases report the intermetallic precipitate phase to be a minor phase while a disordered *bcc* or *fcc* solid solution phase forms the majority of the microstructure. Contrastingly, in a recent report, Tsai et al. [34] reported the formation of an intermetallic sigma phase matrix in an $\text{Al}_{0.3}\text{CrFe}_{1.5}\text{MnNi}_{0.5}$ HEA. The present study discusses the results from as cast $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ HEA where the resultant microstructure was found to be a Cr-Fe-Co based large lattice parameter *bcc*-based intermetallic phase (*chi* phase) and the Ni_2AlTi based DO_3 (L_{21} ordered) phase as the major constituent phases and an *fcc* phase as a minor phase.

The often unusual phase combinations present in HEAs, involving hard intermetallic phases, can potentially lead to enhanced surface properties of these alloys and this has been of significant research interest. The ability of HEAs to form complex passivation and tribo-layers has been the subject of ongoing research. Some systems have been reported to have excellent room temperature and high temperature hardness that make them promising materials for high performance structural applications. The current alloy system is unique in terms of its very hard intermetallic matrix, with embedded softer L_{21} phase which may be useful in potential damping applications. Quantifying the wear resistance for such applications is critical. Therefore, in the present study the surface degradation by sliding reciprocating wear under different loading conditions of the $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ HEA was investigated and wear mechanisms involved have been rationalized.

2. Experimental procedure

An alloy of composition $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ was prepared by a conventional arc-melting and casting route. Microhardness was

performed using a Shimadzu Dynamic Ultra-micro Hardness Tester (ModelDUH-211) with a load of 1.96N and a holding time of 10 s. Microstructural characterization was performed using scanning electron microscopy (SEM) (FEI Nova-NanoSEM 230) and Transmission electron microscopy (TEM) (FEI Tecnai G2 TF20 operating at 200 kV). Site-specific samples were prepared using dual beam focused ion beam (FIB) milling on a FEI Nova 200 for both TEM and Atom Probe Tomography (APT) analysis. APT experiments were conducted on a CAMECA local electrode atom probe 3000X HR instrument. All experiments were performed in the temperature range of 20 K–40 K with target evaporation of 0.5% and pulse fraction of 20% of a steady-state applied DC voltage. APT data reconstruction and analysis were carried out using CAMECA IVAS® 3.6.10 software.

Reciprocating sliding wear experiments were carried out for the as-cast alloy. Samples were mounted and polished with 0.1 μm silica to produce a mirror finish. The dimensions and mass of the samples were kept constant between various iterations. Wear tests were performed using an Rtec Universal Tribometer, with a 3 mm WC spherical counterface in ambient condition (~40% RH, 27 °C). WC was selected to eliminate counterface tribochemical phases, e.g., WO_3 , forming in the wear track that could otherwise bias the results. Tests were carried out at three different load conditions, namely, 10N, 15N and 20N corresponding to initial maximum Hertzian contact pressures of ~3200 MPa, 3700 MPa and 4200 MPa, respectively. These high normal loads/pressures were chosen to induce wear in the $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ HEA, since lower loads/pressures resulted in minimal wear making it difficult to quantify wear rates. The sliding stroke length was fixed at 1 mm. The sliding frequency was kept constant at 5 Hz and sliding time was 20 min for all tests, which corresponds to a total sliding distance of 15 m. Counterface surfaces were analyzed after each test iteration. White-light interferometry was used to generate 3D wear images and Gwyddion data processing software was used to calculate the surface volume loss. Wear surfaces were carefully preserved and were analyzed for wear mechanism and wear particle composition to reveal operating mechanisms and surface oxidation respectively. SEM and EDS mapping was used to image the wear tracks and elemental mapping for identifying tribolayer formation.

3. Results and discussion

3.1. Scanning Electron Microscopy

Back-scattered SEM images from the as-cast $\text{Al}_{0.25}\text{Ti}_{0.75}\text{CoCrFeNi}$ HEA are shown in Fig. 1(a–b). The microstructure shows three distinct phases. A lighter contrast (whitish) matrix with darker contrast (blackish) regions exhibiting an amoeba-like appearance (cubic dendrites) and a third greyish phase are visible, also labeled in Fig. 1(b). Energy Dispersive Spectroscopy (EDS) scans revealing the elemental distribution in different phases of the microstructure are shown in Fig. 1(c–h). The lighter contrast and darker contrast regions show strong partitioning of elements. The darker regions are enriched in Ni, Al, Ti and Co whereas the lighter regions are enriched in Cr, Fe and Co.

Electron backscattered Diffraction (EBSD) results from this alloy are shown in Fig. 2. Fig. 2(a) shows a BSED SEM image depicting the region of interest scanned for orientation and phase mapping. Fig. 2(b) shows the pseudo-colored reference stereographic quadrants corresponding to various orientations (inset) and the inverse pole figure (IPF) map corresponding to this region. Fig. 2(c–e) show the individual phase maps. Based on these maps it is evident that the lighter contrast phase is being indexed as the χ (χ) phase, the darker contrast phase as the L_{21} (*fcc* based ordered) phase and the greyish phase as disordered *fcc*. The total area fraction of each phase

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