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Engineering Multi-scale B2 Precipitation in a Heterogeneous FCC Based Microstructure to Enhance the Mechanical Properties of a $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ High Entropy Alloy

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Abstract

While ordered L_{12} or gamma prime precipitates in face centered cubic (FCC) based microstructures have been extensively used for strengthening nickel or cobalt base superalloys, and more recently in high entropy alloys (HEAs) or complex concentrated alloys (CCAs), the possibility of exploiting ordered B2 precipitates in FCC-based systems has been relatively less investigated. The present study shows the propensity of developing a heterogeneous microstructure, consisting of two different distributions of FCC grain sizes, and two different size scales of B2 precipitates, within an FCC-based $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ HEA/CCA. This alloy composition has been designed using solution thermodynamics-based modeling such that it has a high phase fraction and solvus temperature of the B2 phase. The resulting heterogeneous microstructure exhibited an approximately 400% increase in yield strength with respect to the single-phase FCC solid solution condition of the same alloy while maintaining very good tensile ductility ~20 %.

Keywords: High entropy alloys, Complex concentrated alloys, Heterogenous microstructure, Intermetallic precipitates, Mechanical properties

Introduction

Conventional alloy design has been largely based on one principal element alloyed with small additions of multiple other elements, to obtain desired properties like corrosion resistance, toughness, strength, ductility and thermal properties. However, this strategy limits alloy design to the corners of phase diagrams, leaving unexplored the vast central regions of multi-component phase space. About fifteen years ago, a new strategy emerged, shifting the alloy design paradigm to include equi-atomic or concentrated mixtures of multiple principal alloying elements, leading to the concept of high entropy alloys (HEAs) or multicomponent alloys [1,2]; another widely accepted name is complex concentrated alloys (CCAs) [3]. Four core effects have been attributed to these alloys; sluggish diffusion effect, lattice distortion, high configurational entropy and the cocktail effect [1,4]. Using the HEA/CCA design strategy has expanded the scope of alloy design to include the central unexplored regions of complex multi-component phase diagrams.

The effects of Al addition to CoCrFeNi based alloys has been extensively studied in the past decade [5–9]. With increasing Al content, it was shown that the phase composition of the alloy changes from FCC/L1₂ to FCC/L1₂+BCC/B2 to BCC/B2. In addition to Al, minor Ti additions has also proven beneficial in stabilizing γ' precipitates, increasing both their phase fraction and solvus temperature, as well demonstrated in previous work on Ni-base superalloys [10]. Similarly, in case of HEAs/CCAs, the addition of Ti results in higher γ' phase fractions. This has led to the recent

development of a number of HEAs exhibiting an excellent combination of strength and ductility. He et.al. developed a CoCrFeNi base alloy containing 6 at.% Al+Ti that produced a microstructure containing fine L1₂ coherent nano-precipitates in an FCC matrix [11]. The tensile yield strength and ductility of this microstructure were reported to be over 1 GPa and 17% respectively. Yang et.al. [12] designed a CoFeNi base alloy with Al and Ti constituting 14 at.%. Thermomechanical processing of this alloy led to a high phase fraction of L1₂ nanoparticles in FCC matrix that exhibited a yield strength of 1.03 GPa and ~50% ductility.

In addition to the well-known $\gamma+\gamma'$ microstructures, HEA/CCAs also offer a variety of phase transformation pathways that can lead to novel microstructures containing intermetallic phases such as L1₂, B2, σ , laves and χ phases [13–16]. Al_{0.3}CoCrFeNi, in particular, received much attention owing to its tunability of microstructure and mechanical properties [17]. The traditional thermomechanical processing technique namely, cold working, solution treatment followed by precipitation annealing on Al_{0.3}CoCrFeNi results in a homogenous distribution of L1₂ precipitates in the FCC matrix and grain boundary B2 precipitates. In contrast, when the same alloy is rolled and directly precipitation annealed, concurrent recrystallization of FCC grains and precipitation of B2 and σ phases takes place. When compared to single phase FCC condition, the traditional processing technique results in an improvement in tensile yield stress by 250 MPa and the direct annealing improves the yield stress by ~600 MPa. Using Al_{0.3}CoCrFeNi as a prototype, the importance of B2 precipitation in FCC based HEA/CCAs was studied by Choudhuri et.al. [18] Their study reveals that enhanced local stress levels around B2 precipitates trigger deformation twinning thereby improving strain hardenability of the material. Nonetheless, the investigation and fundamental understanding of FCC+B2 microstructures in these alloy systems has been relatively

low [19–22], especially since they are rarely observed in more conventional alloys based on a single principal element. HEA/CCAs offer the opportunity of exploring such novel phase combinations and microstructures.

Interestingly, FCC+B2 microstructure has also been used to design heterogeneous grain structures [23]. Over the past decade, heterogeneous materials have become a fast-emerging area after multiple reports showing strength – ductility synergy as opposed to trade-off [24–26]. This is achieved by having both soft and hard domains in the material. The constriction of plasticity in soft domains by relatively harder regions imparts additional strength and the back-stresses produced at interfaces help in enhancing work-hardening and prevent necking of the material. Previous studies on heterogeneous microstructures have largely focused on bimodal or multimodal distributions of grain size within a single-phase material. Specifically, in the context of precipitation strengthened HEAs/CCAs, these heterogeneous microstructures consist of a complex mixture of fine-grained recrystallized FCC + B2 regions co-existing with coarser non-recrystallized FCC grains with refined B2 precipitates decorating the deformation bands/twins [23].

The extensive previous work on the $\text{Al}_{0.3}\text{CoCrFeNi}$ alloy introduced the competition between homogeneous nucleation of metastable L_{12} precipitates versus defect-mediated heterogeneous nucleation of equilibrium B2 precipitates [17]. However, this alloy suffers from two predominant weaknesses; firstly, the solute content of 7at% Al is insufficient for achieving a substantially high phase fraction and solvus temperature of second phase intermetallic precipitates, and secondly,

this alloy is prone to precipitation of the embrittling σ phase at temperatures as high as 700°C. The principal objectives of this paper can be listed as follows:

- i. Investigate the competition between the $L1_2$ and B2 phases in an appropriate alloy which has the propensity to precipitate a higher phase fraction of these intermetallics, based on solution thermodynamic predictions.
- ii. Investigate the influence of the nature of the defect (grain boundaries versus prior deformation bands/twins) on heterogeneous B2 precipitation.
- iii. Compare and contrast overall mechanical behavior and tensile properties of homogeneous FCC + $L1_2$ microstructures versus heterogeneous FCC + B2 microstructures.

Materials and Methods

Alloy with a nominal chemical composition $Al_{0.5}Co_{1.5}CrFeNi_{1.5}$ (at%: 9.1Al-27.27Co-18.18Cr-18.18Fe-27.27Ni) was prepared using a conventional vacuum arc melter. The alloy was flipped at least three times to ensure homogenization of the melt. Microstructural characterization was done using Scanning Electron Microscopy (FEI Nova-NanoSEM 230TM/ Hitachi SU8010 FE-SEM), fixed with energy dispersive spectroscopy (EDS) and electron backscattered diffraction (EBSD) detectors. TSL OIMTM software was used to analyze the EBSD data and no clean-up procedures were employed for the maps used in this report. Transmission Electron Microscopy (FEI Tecnai G2 TF20TM operating at 200 kV/ JEOL JEM-3000F HRTEM operating at 300 kV) was used to obtain crystallographic information from the phases present in the material. For analysis of compositional partitioning, Atom Probe Tomography (APT) was performed using Cameca LEAP 5000XS 3D Atom Probe Microscope operating at a temperature of 30K with pulse energy of 50nJ, pulse rate of 100 kHz, and a detection rate of 0.5-1 in laser mode. The lift outs for TEM and APT

were prepared using a FEI Nova 200 dual beam focused ion beam (FIB). Three heat treated conditions of the alloy were studied. The cast alloy was homogenized at a temperature of 1150°C for 6 hours in a vacuum furnace before annealing or rolling treatments. The homogenized condition is referred to as single phase FCC condition, and SA750 condition corresponds to the samples that have undergone subsequent annealing treatment at 750°C for 50 hrs after homogenizing. In case of the third condition, CR750, the homogenized samples were cold rolled to 60 % reduction and annealed at 750°C for 50 hrs. Annealing treatments at 750°C were performed in encapsulated quartz tubes backfilled with argon. The tensile samples were machined using Kent wire saw submerged EDM with following dimensions: gauge length of 5 mm, width of 1 mm and thickness of 0.7-1 mm. The tensile samples were tested at room temperature under quasi-static conditions at a strain rate of 10^{-3} s^{-1} using a computer controlled mini tensile testing machine. The tensile tests were performed on at least three samples for each condition. Thermodynamic predictions were performed using ThermoCalc and the TCHEA4 database.

Results

The isopleth obtained by varying Al content in CoCrFeNi, given in Fig. 1 (a) shows that increasing the Al content raises the solvus temperature and volume fraction of B2 phase. However, increasing Al beyond ~10 at% results in a change in primary solidification phases from FCC to FCC + B2. The absence of such single phase (FCC) field deters the alloy in terms of thermomechanical processing and thus in achieving equilibrium phase compositions. Fig. 1 (b), an isopleth obtained by varying Al in $\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ indicates that the single-phase FCC field can be retained at higher Al content by raising the relative contents of Co and Ni. The composition $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ was chosen to utilize the solid solution strengthening from higher Al content and improved solvus

temperature and phase fractions of intermetallic B2 phase. The temperature vs. phase fraction plots in Fig. 1 (c) and (d) compares and contrasts the extensively studied $\text{Al}_{0.3}\text{CoCrFeNi}$ system with $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$. It can be noted that B2 solvus temperature has improved by 70°C , and the volume fraction of B2 has increased by 20 to 80% within the temperature range of $700^\circ\text{C} - 1000^\circ\text{C}$. Chang et al. [27] have previously reported an FCC + β (BCC based) phase field for the $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ alloy based on x-ray diffraction results, without any detailed investigation of the phase stability, transformations, or mechanical properties.

Comparing the high temperature phase stability for a high Al containing CoCrFeNi base alloy system, $\text{Al}_{0.5}\text{CoCrFeNi}$ and the current alloy, $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$, SEM backscatter micrographs from 1150°C annealed samples of both alloys are shown in Fig. 2. While the $\text{Al}_{0.5}\text{CoCrFeNi}$ alloy exhibits a dual phase microstructure, FCC grains with substantial phase fractions of the B2 phase (Fig. 2 (a)), the $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ alloy exhibits a single-phase microstructure (subsequently referred to as single phase FCC condition) as shown in Fig. 2 (b). Due to the long-term homogenizing treatment, the FCC grains in this condition have reached a size of $\sim 253.3 \mu\text{m}$. The discrepancy in the predicted single phase FCC field at 1200°C (refer to Fig. 1(d)) and the experimentally verified temperature of 1150°C is possibly due to the inaccuracies in solution thermodynamics models for these complex concentrated alloys. The precipitation anneal temperature of 750°C was selected for a high B2 volume fraction based on the ThermoCalc predicted isopleth and phase fraction diagrams shown in Figs. 1 (b) and (d) respectively.

The micrographs in Fig. 3 (a-b) shows the backscatter images of SA750 condition. A coarse FCC grain structure with grain boundary precipitates is visible in the low magnification image shown

in Fig. 3(a). The backscatter high magnification image, shown in Fig. 3(b), reveals fine scale precipitation within the FCC matrix grains and a bright region around the grain boundary precipitates, presumably due to depletion of Al in matrix surrounding the precipitates. An SEM micrograph from an etched condition of the SA750 sample is shown in Fig. 3(c) revealing both grain boundary precipitates, as well as finer scale homogeneously distributed intragranular precipitates (inset in Fig. 3(c)). The crystal structures of these two types of precipitates, coarse grain boundary and fine scale intra-granular, were revealed by TEM characterization. A bright-field TEM image from the SA750 sample is shown in Fig. 3(d), revealing a collection of coarse grain boundary precipitates and refined intragranular precipitates. The high symmetry selected area diffraction pattern (SADP) in Figure 3(e) is recorded from one such grain boundary precipitate and is unambiguously indexed as [001] BCC zone axis. The superlattice reflection at {001} positions clearly establish the ordering of BCC lattice in the precipitates (B2 phase). An SADP from [001] FCC zone axis, recorded from one of the matrix grains is shown in Fig. 3(f). The {001} superlattice reflections in this diffraction pattern confirm that the intragranular precipitates are ordered FCC phases also known as L_{12} or γ' (gamma prime) precipitates. APT was carried out to determine the composition of L_{12} precipitates in the SA750 sample. The superimposed ion map consisting of all ions, shown in Fig. 4 (a), which is a ~2 nm thick slice of the atom probe 3D reconstruction, clearly shows the enrichment of Ni and Al within the L_{12} precipitates. Iso-concentration surfaces at 13.13 at.% Al along with Al ions, have been plotted in the 3D reconstruction shown in Fig. 4(b). This reconstruction illustrates the 3D distribution of these precipitates within the FCC matrix. The proxigram constructed across the same iso-surface reveals the preferential partitioning of Ni and Al to L_{12} precipitates and Co, Cr and Fe to the FCC matrix. The volume fraction and average size of L_{12} precipitates was found to be and 12.1% and ~10.2

μm respectively. The composition of the matrix and the precipitates was determined by averaging the atoms within a 20 nm radius sphere embedded within each phase. The composition of the L1₂ precipitates was determined to be 21.5Al-13.9Co-6.6Fe-4.4Cr-53.6Ni (at%) whereas the composition of the FCC matrix was found to be 8Al-30.1Co-19.4Cr-18.2Fe-24.3Ni (at%). Based on the experimentally measured precipitate composition and assuming an Ni₃Al (3Ni:1Al) stoichiometry, the site occupancies within this L1₂ or γ' phase can be conjectured to be (Ni,Fe,Co)₃(Al,Cr) [28].

Previous work by Gwalani et.al. [17] on the Al_{0.3}CoCrFeNi HEA/CCA established that in the absence of a large number density of heterogenous nucleation sites within the FCC matrix, B2 precipitates cannot easily nucleate and consequently reach a near equilibrium phase fraction. Instead, the competing mechanism of homogeneous precipitation of the metastable L1₂ phase might occur within the FCC matrix [17]. However, cold-rolling followed by directly annealing the sample at the target second phase precipitation temperature, resulted in fine recrystallized FCC grains with the recrystallization process concurrently taking place with B2 precipitation at the new grain boundaries, consequently leading to a substantially higher B2 phase fraction within the FCC matrix. The same strategy was adopted in case of the present Al_{0.5}Co_{1.5}CrFeNi_{1.5} alloy. Therefore, the homogenized alloy was cold-rolled to 60 % reduction in thickness and then directly annealed at 750°C for 50hrs, with this condition being subsequently referred to as the CR750 condition.

The partially recrystallized microstructure of this CR750 condition can be observed in the low magnification SEM micrographs shown in Figs. 5(a-b). The recrystallized regions have been outlined for convenience in these images. A montage of SEM micrographs, covering a 1×1 mm²

area, was used to determine the average area fraction of recrystallized regions and was found to be ~17%. A high magnification backscatter SEM image from the recrystallized region shown in Fig. 5(c) clearly reveals fine FCC grains with grain boundary precipitates. A high magnification backscatter SEM image from the non-recrystallized region (Fig. 5(d)) shows clearly reveals a high number density of fine scale precipitates within the coarser non-recrystallized FCC grains. These fine scale precipitates are geometrically aligned along bands (Fig. 5(d)). Both the recrystallized and non-recrystallized regions have been investigated in further detail employing SEM-EBSD, TEM and APT based techniques, for determining the identity of the second phase precipitates, their orientation relationship with the FCC matrix, and other details of the overall structure and composition of phases present.

Results from the orientation image microscopy (OIM) from CR750 consisting of an inverse pole figure (IPF) and the corresponding phase map from a recrystallized region are shown in Figs. 6(a-b). The inset in Fig. 6(a) shows the image quality (IQ) map of the region. These maps reveal fine recrystallized FCC grains with intragranular and intergranular precipitates which were indexed as a BCC based phase. While the FCC grain size in recrystallized regions is ~3.5 μm , the size of B2 precipitates is ~0.76 μm . Further characterization was performed using TEM. A [011] FCC zone axis electron diffraction pattern from a recrystallized FCC grain in this region, shown in Fig. 6(c), clearly indicates that there is no $L1_2$ precipitation inside these FCC grains. The electron diffraction pattern from [011] BCC zone axis, recorded from one of the second phase precipitates (Fig. 6(d)) clearly reveals superlattice reflections at $\{001\}$ locations, confirming that these BCC based precipitates are ordered B2 precipitates. A bright-field image and a $\{001\}$ superlattice B2 dark-field TEM image, recorded from a precipitate, are shown in Figs. 6(e) and (f) respectively.

The IPF and phase maps shown in Figs. 7(a-b) were recorded from the non-recrystallized region of the CR750 sample. These EBSD images show two coarse non-recrystallized FCC grains with a grain boundary and a homogeneous distribution of well-aligned fine scale needle-like precipitates within these grains. The fine-scale second phase precipitates, both at the grain boundary as well as within the grains, are indexed as BCC-based precipitates (refer to Fig. 7(b)). These precipitates have a multi-modal distribution with size ranging from 80 nm to 1.2 μm , as determined by ImageJ software. The IQ and kernel average misorientation (KAM) maps are shown in the insets of Fig. 7(a) and (b) respectively. The KAM map, which has strain-free recrystallized grains at the prior grain boundary, shows that some areas in the non-recrystallized region are relatively strain-free. This is an indication that recovery has occurred in some non-recrystallized regions. Similar to the observations from the recrystallized regions, there was no $L1_2$ precipitation within these FCC grains, and the fine-scale aligned precipitates have a B2 crystal structure, as revealed by the respective electron diffraction patterns, recorded from an FCC grain (Fig. 7(c)) and the microdiffraction pattern recorded from a fine-scale precipitate (Fig. 7(d)). Corresponding bright-field and $\{001\}$ B2 superlattice dark-field TEM images, recorded from the needle-like precipitate are shown in Figs. 7(e) and (f) respectively.

Detailed analysis of the SEM-EBSD results from this recrystallized region in the SA750 sample, indicated the presence of multiple orientation relationships between the B2 precipitates and the adjacent FCC grains. Two distinct types of B2 precipitates are observed, those nucleating at grain boundaries or triple junctions (referred to as inter-granular) and those located within a single FCC grain (referred to as intra-granular). Fig. 8 shows pole figure analyses of two cases of intra-granular

B2 precipitates within FCC grains. Based on pole figure analysis, as shown in Fig. 8, the orientation relationship (OR) between both the intra-granular B2 precipitates and the surrounding FCC matrix grain was determined to be:

$$\{111\}_{\text{FCC}} // \{011\}_{\text{B2}} \text{ and } \langle 011 \rangle_{\text{FCC}} // \langle 111 \rangle_{\text{B2}}$$

This is referred to as the Kurdjumov-Sachs (K-S) OR [29].

Another case of intra-granular B2 precipitates is shown in Fig. S1 where two conjoint B2 precipitates are located within an FCC grain. The B2 precipitate on the bottom clearly exhibits the Nishiyama-Wasserman (N-W) OR [29] with the surrounding FCC matrix:

$$\{111\}_{\text{FCC}} // \{011\}_{\text{B2}} \text{ and } \langle 011 \rangle_{\text{FCC}} // \langle 001 \rangle_{\text{B2}}$$

The B2 precipitate on the top of the same conjoint pair has only a $\{111\}_{\text{FCC}} // \{011\}_{\text{B2}}$ but no $\langle 011 \rangle_{\text{FCC}} // \langle 111 \rangle_{\text{B2}}$.

A set of conjoint B2 precipitates, located at a triple junction, in the recrystallized region, are shown in Fig. 9. While the B2 precipitate on top exhibits a K-S OR with the FCC grain on top, the B2 precipitate on the bottom exhibits a K-S OR with the bottom FCC grain. Finally, the SEM-EBSD analysis of a B2 precipitate located within a FCC grain of the non-recrystallized region is shown in Fig. 10. This B2 precipitate also exhibited a K-S OR with the surrounding FCC grain.

To compare the composition of B2 precipitates from the recrystallized and non-recrystallized regions in the CR750 sample, site-specific APT experiments were performed on both these regions and these results have been summarized in Fig. 11. Fig. 11(a) is an Al ion map showing the 3D atom probe reconstruction of the section of a coarser B2 precipitate and its interface with the adjacent FCC matrix from a recrystallized region. An 7.8 at.% Al iso-concentration surface together with Al ions, are shown in the same figure to clearly delineate the Al-rich B2 regions

from the FCC matrix region. The proxigram in Fig. 11(b), plotted across the Al iso-surface, reveals the partitioning of elements between the matrix and B2 phases. The respective compositions of the B2 precipitate and FCC matrix, averaged over a sphere of 25 nm radius, are 22.7Al-13.3Co-11Cr-10Fe-43Ni (at%) and 4.6Al-30.5Co-21.5Cr-19.8Fe-23.6Ni (at%) respectively. Similarly, Fig. 11(c) shows the APT reconstruction from the non-crystallized region capturing a section of a fine scale B2 precipitate. A 9.8 at.% Al iso-concentration surface together with the Al ions have been plotted in the same reconstruction (Fig. 11(c)). The corresponding proxigram from this iso-surface is shown in Fig. 11(d). The compositions of the B2 precipitate and the FCC matrix, determined by a similar sphere analysis as described earlier, are 23.1Al-14Co-10.2Cr-9.1Fe-43.6Ni (at%) and 5.3Al-29.5Co-21.6Cr-19.1Fe-24.5Ni (at%) respectively. Based on the experimentally determined B2 precipitate composition, and assuming an NiAl (1Ni:1Al) stoichiometry, the site occupancies within the B2 structure can be conjectured to be (Ni,Co)(Al,Co,Cr,Fe) [28,30].

The tensile engineering stress-strain curves for the single-phase FCC, SA750, and CR750 conditions are shown in Fig. 12(a). The single-phase FCC condition exhibited a yield stress (YS) of 180 MPa, ultimate tensile stress (UTS) of 453 MPa with ~62% ductility. The SA750 condition which has an FCC+L1₂ microstructure, exhibited an yield stress of 360 MPa, UTS of 720 MPa, and 48% tensile ductility. The partially recrystallized FCC+B2 microstructure in case of the CR750 sample, exhibited the best balance of properties with the highest yield stress of 790 MPa, UTS of 1020 MPa, and 20% tensile ductility. The corresponding work-hardening curves for all three conditions are shown in Fig. 12(b). It can be observed that the hardening rate of CR750 starts at a much higher value compared to the other two conditions. However, the work hardening rate after stage I is increasing in the case of single phase FCC and CRSA750 presumably due to

dynamic Hall-Petch effect [31] and decreasing in the case of CR750. Additionally, the steady state hardening or stage II which is an indication of deformation twinning appears to be prominent in single phase FCC and CRSA750 conditions. The deformation mechanisms in the three conditions associated with tensile loading is a subject of future study.

Discussion

As compared to the extensively investigated $\text{Al}_{0.3}\text{CoCrFeNi}$, the $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ HEA/CCA offers a substantially improved solvus temperature and phase fractions of strengthening intermetallic phases. The present study on the $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ alloy, demonstrates the ability to engineer widely different microstructures; a single-phase FCC solid solution, a typical precipitation-strengthened FCC+ L_{12} microstructure (SA750), and a partially recrystallized FCC+B2 microstructure (CR750), within the same HEA/CCA with widely varying mechanical properties. While the single-phase FCC condition exhibited a relatively low YS of 180 MPa, it had excellent strain hardenability leading to a UTS of 453 MPa and ~62% tensile ductility.

Regarding second phase precipitation in this alloy, although the ThermoCalc based solution thermodynamic modeling suggests that the equilibrium phases are FCC and B2 at 750°C, the SA750 condition exhibited a homogenous distribution of L_{12} precipitates within an FCC matrix, and a small fraction of grain boundary B2 precipitates. This unexpected precipitation of a metastable L_{12} phase at 750°C, can be rationalized via more detailed thermodynamic analysis. Starting from a homogeneous single-phase FCC solid solution, using ThermoCalc, the onset driving forces have been computed for all the competing intermetallic phases, B2, L_{12} and σ , for this specific alloy composition. These driving forces, as function of temperature, have been plotted

in Fig. 13. While B2 is the equilibrium phase at 750°C, there is a positive driving force for the precipitation of the L1₂ phase at the same temperature. Despite the highest driving force for B2 precipitation at 750°C and an expected equilibrium phase fraction ~35%, there is only a very small fraction of grain boundary B2 precipitates observed in the SA750 condition. This is due to the high interfacial energy barrier associated with B2 nucleation within FCC matrix, as discussed previously [17]. In comparison, there is a finite onset driving force for L1₂ precipitation at 750°C (referring to Fig. 13), while there is likely to be a rather low nucleation barrier associated with L1₂ nucleation within the FCC matrix. The underlying reasoning for this difference in nucleation barriers is the substantially lower FCC/L1₂ interfacial energy as compared with the FCC/B2 interfacial energy. While these interfacial energies have not been determined and are very difficult to compute in case of these five component alloys, it is possible to qualitatively compare them based on the degree of structural similarity across the interface. Thus, since L1₂ ordering is based on the parent FCC crystal lattice, the FCC/L1₂ interfacial energy is typically very low for a coherent interface, ~10 – 40 mJ/m² for Ni-base and Co-base superalloys [10]. In contrast, since the B2 phase is based on a parent BCC crystal structure, the FCC/B2 interfacial energy is expected to be much higher. While there are no reports in the literature of FCC/B2 interfacial energies in any alloy system, the closest approximation can be considered to be FCC/BCC interfaces in case of austenite/ferrite microstructures which typically have interfacial energies in the 180 – 660 mJ/m² range [17]. Considering the precipitation tendencies for the two competing phases at 750°C, while the driving force for the precipitation of the B2 phase is more than 2 times higher than for L1₂, and approximately estimating the FCC/B2 interfacial energy to be ~10 times higher than that of the FCC/L1₂ interface, results in a nucleation barrier ~200 times lower for the L1₂ phase as compared to the B2 phase. Therefore, while L1₂ precipitates can homogeneously nucleate within

FCC, it is nearly impossible for B2 precipitates to homogeneously nucleate within an FCC matrix. Only heterogeneous nucleation of B2 precipitates is possible and in case of the SA750 condition, grain boundaries are the only available heterogeneous nucleation sites. However, the small fraction of grain boundary surface area per unit volume available in the coarse-grained SA750 condition limits the B2 nucleation density and results in a small phase fraction of this phase, much lower than the expected equilibrium phase fraction of ~35%. Instead, the strong chemical ordering tendency introduced via the introduction of Al in the $\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ base alloy results in the homogenous precipitation of a metastable L_{12} phase. The yield stress in SA750 is thus nearly doubled (enhanced by over 180 MPa) when compared to the single FCC phase condition, due to the homogenous L_{12} intermetallic precipitation in the former case.

Recently, there has been a lot of interest on heterogeneous microstructures in HEAs, with multiple studies reporting very good combinations of strength and ductility resulting from such microstructures [23,26]. Heterogeneous microstructures in HEAs can have different connotations, such as a mixture of different grain size distributions in a single-phase microstructure, or a mixture of different phases in the microstructure, or a combination of both of these. CR750 exhibits such a heterogeneous microstructure with a mixture of ~17% recrystallized regions and ~83% non-recrystallized regions. From the SEM micrographs (Fig. 3(a-b)), as expected, the recrystallization primarily occurs at prior grain FCC boundaries and at sites of high dislocation activity. While the recrystallized regions consisted of fine grains of FCC with B2 precipitates, on the same size scale as the grains, the non-recrystallized region contained a homogenous distribution of refined B2 precipitates, which were crystallographically aligned along deformation bands and twins. Thus, the heterogeneous microstructure in case of the CR750 condition of the $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ alloy

is a complex mixture of two different grain size distributions of the parent matrix FCC phase, together with multiple scales of distribution of intermetallic B2 precipitates. Within the recrystallized regions, concurrent recrystallization and second phase B2 precipitation take place due to the competition between, recovery followed by strain-free grain nucleation dictated by the prior strain arising from cold-rolling, and the large thermodynamic driving force for B2 precipitation at the same temperature. This phenomenon restricts the size of recrystallized FCC grains due to the grain boundary pinning effect arising from the B2 precipitates. On the other hand, in the non-recrystallized regions, the alignment of B2 precipitates is presumably along specific crystallographic directions, dictated by the deformation bands and twins produced by prior cold rolling. Due to the availability of large number of heterogenous nucleation sites in CR750, the volume fraction of B2 is substantially higher than that observed in case of the SA750 condition. Interestingly, nearly identical compositions of B2 precipitates are observed in both recrystallized and non-recrystallized regions, as determined using APT. Any minor differences are within the local composition variations. The grain boundary diffusion in case of the recrystallized regions, and the pipe diffusion at deformation bands and twins helped attain the equilibrium composition of B2 precipitates in both recrystallized regions as well as non-recrystallized regions. This heterogeneous microstructure exhibited the highest yield stress of ~790 MPa, nearly double that of the SA750 (FCC + L1₂) condition, primarily arising from the high phase fraction of B2 precipitates coupled with the mixture of fine and coarse FCC grains. The stress-strain curves shown in Fig. 12(a), and the corresponding work hardening curves shown in Fig. 12(b), also indicate that the work hardening is significantly higher in case of the CR750 condition, as compared to the other two conditions, single phase FCC and SA750. This could be related to the effect of the complex multi-phase heterogeneous microstructure consisting of two distinct regions;

recrystallized refined FCC grains with B2 precipitates at grain boundaries and triple junctions, and the partially recovered coarser prior FCC grains with intra-granular B2 precipitates. The refined recrystallized FCC grains presumably exhibits a high strength due to the combined effect of Hall-Petch hardening together with the B2 precipitation at grain boundaries and triple junctions. The prior FCC grains in the non-recrystallized region have at least partially recovered and softened due to the 750°C annealing treatment, however, the high density of deformation bands and twins, decorated with B2 precipitates, act as barriers to dislocation movement, consequently increasing the strength of these coarse FCC grains. While it is difficult to assess which of these two regions is stronger, the overall yield strength of this composite heterogeneous microstructure is substantially higher. Subsequent to yielding, the deformation produced in the relatively softer regions of this microstructure is restricted by the surrounding plastically hard material, thereby producing back stresses at the interface. This phenomenon often termed back stress hardening strengthens the softer regions, raising the work-hardening rate of the material [32]. Further detailed experimental investigations coupled with computational modeling are required for a better assessment of the relative strengths of the two different regions in this heterogeneous microstructure, which will be the subject of future research.

Conclusions

1. $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ is a candidate alloy to study FCC+B2 microstructures in HEA/CCAs because of the high B2 solvus temperature ($\sim 1200^\circ\text{C}$) and phase fractions ($\sim 35\%$ at 750°C), as predicted by solution thermodynamic modeling.
2. A lack of heterogeneous nucleation sites for the equilibrium B2 phase, results in this alloy precipitating metastable L_{12} precipitates within the FCC matrix grains during annealing at

750°C. The relatively lower thermodynamic nucleation barrier for L1₂ precipitation as compared to B2 precipitation drives this phase transformation.

3. Annealing the alloy directly after cold-rolling produces a large number of heterogenous B2 nucleation sites, and results in a complex multi-phase heterogeneous microstructure consisting of two distinct regions; recrystallized refined FCC grains with B2 precipitates at grain boundaries and triple junctions, and partially recovered coarser prior FCC grains with intragranular B2 precipitates decorating the prior deformation bands and twins resulting from cold-rolling. This condition exhibited a ductility of ~20% and a yield strength of ~780 MPa, which is approximately a 400% increase from the single phase FCC condition of the same alloy.

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Competing Interests

None

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Figure Captions

Fig. 1: (a) Pseudo-binary isopleth for $\text{Al}_x\text{CoCrFeNi}$ system (b) Pseudo-binary isopleth for $\text{Al}_x\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ system. (c) Phase fraction vs temperature diagram for $\text{Al}_{0.3}\text{CoCrFeNi}$ and (d) Phase fraction vs temperature diagram for $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$.

Fig. 2: (a) Backscatter SEM micrograph of $\text{Al}_{0.5}\text{CoCrFeNi}$ showing a two-phase microstructure. High magnification picture is provided in the inset (b) Backscatter SEM micrograph of $\text{Al}_{0.5}\text{Co}_{1.5}\text{CrFeNi}_{1.5}$ showing a single phase microstructure. The inset shows high magnification picture of a grain boundary.

Fig. 3: (a-c) SEM of CRSA750 condition, (a) Backscatter micrograph of CRSA750 condition (b) High magnification picture of a region near grain boundary showing two kinds of precipitates (c) Secondary electron image from an etched sample. The inset shows fine scale precipitation within the matrix. (d-f) TEM of CRSA750 condition, (d) Bright field TEM micrograph showing coarse B2 precipitates and fine scale L_{12} precipitation within the matrix. (e) Selected area diffraction pattern (SADP) obtained from [001] BCC zone axis exhibiting B2 superlattice reflections (f) SADP obtained from [001] FCC matrix exhibiting L_{12} superlattice reflections.

Fig. 4: Atom Probe Tomography (APT) of CRSA750 condition. (a) A 2 nm slice of superimposed Al, Co, Cr, Fe, Ni ion maps from the 3D reconstruction, showing the enrichment of Ni and Al in

globular L_{12} precipitates. (b) Al 13.13 at.% iso-concentration surface along with Al ions showing 3D distribution of L_{12} precipitates and in the reconstruction. (c) Proxigram across Al 13.13 at.% iso-concentration surface showing the elemental partitioning between matrix and precipitates.

Fig. 5: Backscatter SEM of CR750 condition (a-b) Low magnification pictures showing the amount of recrystallization in the prior rolled microstructure. Recrystallized regions are outlined with blue color. High magnification pictures from (c) fully recrystallized region and (d) non-recrystallized region.

Fig. 6: (a) IPF map obtained from a fully recrystallized region in CR750 condition using EBSD (color coding for BCC and FCC phases is at the bottom) (b) The corresponding phase map indicating that the precipitates are BCC based (Percentage of phases present is given at the bottom) (c-f) TEM of recrystallized region in CR750 condition, (c) SADP obtained from [001] FCC zone axis showing no superlattice reflections (d) SADP obtained from [011] B2 zone axis (e) Bright field micrograph showing a B2 precipitate surrounded by FCC grains. (f) Dark field micrograph obtained from {001} superlattice reflection in (d).

Fig. 7: (a) IPF map obtained from a non-recrystallized region in CR750 condition using EBSD (color coding for BCC and FCC phases is at the bottom) (b) The corresponding phase map indicating that the precipitates are BCC based (Percentage of phases present is given at the bottom) (c-f) TEM of non-recrystallized region in CR750 condition, (c) SADP obtained from [011] FCC zone axis showing no superlattice reflections (d) Microdiffraction pattern obtained from [001] B2

zone axis (e) Bright field micrograph showing a layer of B2 precipitates in FCC matrix. (f) Dark field micrograph obtained from {001} superlattice reflection in (d).

Fig. 8: Orientation relationship between intragranular B2 precipitates and FCC matrix in recrystallized region of CR750 (a-b) Discrete plots for B2 and FCC respectively for precipitate 1 (IPF map is shown in the inset of (a)) and (c-d) Discrete plots for B2 and FCC respectively for precipitate 2 (IPF map is shown in the inset of (c)).

Fig. 9: Orientation relationship between B2 precipitates at a triple junction and FCC matrix in recrystallized region of CR750 (a-b) Discrete plots for B2 top and FCC top respectively (c) IPF map of the region (d-e) Discrete plots for B2 bottom and FCC bottom respectively.

Fig. 10: Orientation relationship between intragranular B2 precipitate and FCC matrix in non-recrystallized region of CR750 (a-b) Discrete plots for B2 and FCC respectively. IPF map is shown in the inset of (a): green region corresponds to FCC and pink corresponds to B2.

Fig. 11: Atom Probe Tomography (APT) of CR750 condition. (a) Al 7.8 at.% iso-concentration surface along with Al ions showing part of an FCC recrystallized grain and part of a B2 precipitate. (b) Proxigram across Al 7.8 at.% iso-concentration surface showing the elemental partitioning between matrix and precipitate. (c) Al 9.8 at.% iso-concentration surface along with Al ions showing part of an FCC non-recrystallized grain and part of a B2 precipitate. (d) Proxigram across Al 9.8 at.% iso-concentration surface showing the elemental partitioning between matrix and precipitate.

Fig. 12: (a) Tensile engineering stress-strain curves and (b) Work-hardening plots for all the three conditions studied.

Fig. 13: Normalized onset driving force vs temperature plots for the three predicted intermetallic phases (B2, L1₂ and σ) in Al_{0.5}Co_{1.5}CrFeNi_{1.5} alloy system.

Fig. S1: Orientation relationship between intragranular conjoint B2 precipitates and FCC matrix in recrystallized region of CR750 (a-c) Discrete plots for FCC, B2 bottom and B2 top respectively (d) IPF map of the region.