Tensile ductility of an AlCoCrFeNi multi-phase high-entropy alloy through hot isostatic pressing (HIP) and homogenization

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Abstract

The microstructure and phase composition of an AlCoCrFeNi high-entropy alloy (HEA) were studied in as-cast (AlCoCrFeNi-AC, AC represents as-cast) and homogenized (AlCoCrFeNi-HP, HP signifies hot isostatic pressed and homogenized) conditions. The AlCoCrFeNi-AC alloy has a dendritrictal structure in the consisting primarily of a nano-lamellar mixture of A2 [disorder body-centered-cubic (BCC)] and B2 (ordered BCC) phases, formed by an eutectic reaction. The homogenization heat treatment, consisting of hot isostatic pressed for 1 hour at 1,100 °C, 207 MPa and annealing at 1,150 °C for 50 hours, resulted in an increase in the volume fraction of the A1 phase and formation of a Sigma (σ) phase. Tensile properties in as-cast and homogenized conditions are reported at 700 °C. The ultimate tensile strength was virtually unaffected by heat treatment, and was 396 ± 4 MPa at 700 °C. However,

homogenization produced a noticeable increase in ductility. The AlCoCrFeNi-AC alloy showed a tensile elongation of only 1.0 %, while after the heat-treatment, the elongation of AlCoCrFeNi-HP was 11.7 %. Thermodynamic modeling of non-equilibrium and equilibrium phase diagrams for the AlCoCrFeNi HEA gave good agreement with the experimental observations of the phase contents in the AlCoCrFeNi-AC and AlCoCrFeNi-HP. The reasons for the improvement of ductility after the heat treatment and the crack initiation subjected to tensile loading were discussed.

Keywords: high-entropy alloys; microstructures; tensile properties; heat treatment; thermodynamic modeling; crack initiation

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1. Introduction

Nuclear, turbine, and aerospace industries currently place high demands on high-temperature structural-alloy properties [1], and look for materials that could be superior to conventional nickel-based superalloys. In the last decade, a new class of materials, called high-entropy alloys (HEAs) or multi-principal-element alloys (MPEAs), has been proposed and developed [2-23]. Controlled by solid-solution-strengthening, with a second phase in some cases, the yield strength of HEAs can be very high, and may be comparable to bulk metallic glasses (BMGs) [4, 15, 23]. While BMGs have high strength only at relatively low temperatures (below the glass-transition/crystallization temperatures), crystalline HEAs may retain their high strength at higher temperatures [12, 15, 24]. Figure 1 presents an Ashby map showing ultimate tensile strength (UTS), σ_{UTS} , versus (vs.) density, ρ , for structural metals, along with the data for bulk metallic glasses (BMGs) and HEAs [23, 25-29]. Grey dashed contours (arrow indication) label the specific strength, σ_{UTS} / ρ , from low (right bottom) to high (left top). HEAs are among the materials that have better combination of mechanical properties of strength and density, compared to other materials. Figure 1 shows that the σ_{UTS} vs ρ data for HEAs lay outside the benchmarks established by most conventional alloys and are comparable to Mg-alloys, Ti- alloys, and BMGs.

The present work focuses on one of the earliest quinary HEAs, AlCoCrFeNi, first reported in 2007 [4]. It belongs to the $Al_xCoCrFeNi$ system, which is one of the most well-developed and refined HEA systems [4, 15, 16, 30-41]. The AlCoCrFeNi alloy shows a high compressive yield stress ($\sigma_Y = 1250$ MPa at room temperature and 990 MPa at 500 °C) with a reasonable compressive ductility ($\varepsilon = \sim 33$ % at room temperature and ~ 63 % at 500 °C) [31, 42]. This high strength at elevated temperatures makes it a promising structural material. Meanwhile,

20 atomic percent (at. %) of Al and 20 at. % of Cr might also bring this alloy great corrosion-resistance and oxidation-resistance properties. However, most publications focus on as-cast condition, finding that as-cast AlCrFeCoNi has a BCC + B2 crystal structure [30, 31]. The as-cast alloy decomposes into dendrites and interdendrites, both containing CrFe-rich precipitates embedded in an NiAl-rich matrix [31, 43]. The NiAl-rich matrix shows quite a homogeneous distribution of all alloying elements, while the CrFe-rich precipitates exhibit a small-scale spinodal-like decomposition within the phase [43]. However, there are no publications in the open literature on the homogenized phase stability and high-temperature tensile properties of this important HEA. In this paper, the effect of the homogenization heat treatment on the as-cast microstructure is studied, and the influence of this substantial change in microstructures on tensile properties is investigated. Comparison between the predicted phase equilibria and phases observed after homogenization heat treatment is made. Crack initiation subjected to tensile loading at elevated temperature is also explored for the first time in the HEA family.

2. Thermodynamic modeling

The as-cast HEAs may have different phases than the homogenized HEAs, since rapid cooling can suppress equilibrium phases, which often require a significant elemental redistribution. An effective approach is necessary to determine and/or predict multi-component phase diagrams for HEA systems. Traditionally, binary and ternary phase diagrams have relied upon experimental methods. The first-principles alloy theory was applied to predict the structures of the $\text{Al}_x\text{CoCrFeNi}$ HEA system, and it was concluded that alloys around the equimolar AlCoCrFeNi composition have superior mechanical performance, as compared to the single-phase regions [16]. Meanwhile, Widom et al. applied a hybrid Monte Carlo and molecular-dynamics (MD) method to study the temperature-dependent chemical order for the

refractory MoNbTaW HEA [44]. They found that the Monte Carlo species swaps allow for the equilibration of the structure, which cannot be achieved by conventional MD simulations [44]. In addition, the phenomenological CALculation of PHase Diagrams (CALPHAD) approach [45] is proved to be an effective aid in materials design [46-49], and has recently been successfully used in the AlCoCrFeNi HEA system [50].

The essence of the CALPHAD approach is to obtain self-consistent thermodynamic descriptions (Gibbs energy functions) of lower-order (binary and ternary) systems by fitting to known thermodynamic and phase-equilibrium data. A thermodynamic description represents a set of Gibbs-energy functions with optimized thermodynamic-model parameters for all the phases in a system. The thermodynamic description for higher-order systems can be obtained via an extrapolation method [51]. This description enables estimates of phase diagrams and thermodynamic properties of multi-component systems that are experimentally unavailable.

A thermodynamic database for the Al-Co-Cr-Fe-Ni system was developed [50]. Different from traditional alloys, which usually focus at one key element corner, the HEAs have multiple key elements. This trend requires the database to be valid in the entire composition region. The current database was developed, using the available experimental information for the 10 constituent binaries and 10 constituent ternaries. In this database, the substitutional solution model describes the disordered solution phases, such as A1 and A2; the ordered intermetallic phases, such as sigma and B2, are described by the compound-energy formalism, and the line compounds are described by the stoichiometric model. Details of these thermodynamic models can be found in References [52, 53], and will not be repeated here. It should also be pointed out that the Gibbs energy of all the phases in the 5-component system were obtained from those of binaries and ternaries [50], and no thermodynamic model parameters were optimized using the

experimental data of the present work for the 5-component system. The PANDAT software performed all the calculations in the present work.

3. Experimental procedures

The as-cast samples of AlCoCrFeNi were prepared by vacuum arc-melting the constituent elements in a water-cooled, copper hearth, using a Large Bell Jar (ABJ-900) arc melter of Materials Research Furnaces, Inc. The elements were all at least 99.9 weight percent (wt. %) pure, and the melting process was conducted in a vacuum of at least 0.01 torr (~ 1.3 Pa) under a Ti-gettered high-purity argon atmosphere. Melting and solidification were repeated several times to improve sample homogeneity. Finally, the re-molten ingot was cast into 6-mm-diameter rods with a high cooling rate [54]. As-cast samples are referred to as AlCoCrFeNi-AC. Several samples were heat treated after casting. These samples are referred to as AlCoCrFeNi-HP. The heat treatment consisted of hot isostatic pressing (HIPing) and homogenization.

To close porosity in as-cast samples, they were HIPed at 1,100 °C for 1 hour under 207 MPa ultra-high-purity argon pressure. The HIP process consisted of several steps. First the samples were placed in a HIP vessel, which was vacuumed to $\sim 10^{-6}$ torr (10^{-4} Pa) and filled with the ultra-high purity argon to the atmospheric pressure. Then the temperature in the chamber was increased to 1,100 °C at a heating rate of 15 °C / min, with a simultaneous linear increase in the argon pressure to 207 MPa, and the samples were soaked at 1,100 °C, 207 MPa for 1 hour. Finally, the furnace power was shut down, the samples were cooled down to 500°C under the argon pressure of 207 MPa, followed by the pressure release to 101 kPa (1 atm) and cooled to room temperature. The HIPed samples were then placed in a horizontal tube furnace, evacuated with a rough pump, and, then, the tube was filled with high-purity argon at a continuous flow

rate of ~ 200 ml/min. The samples were heated to 1,150 °C at 10 °C/min, held at 1,150 °C for 50 hours, and, then, cooled to $T = 50$ °C at 10 °C/min. Alloy densities were measured with a helium pycnometer AccuPyc 1330 V1.03. The sample phases were analyzed using high-energy synchrotron X-ray diffraction in the 6-ID-D beamline, Advanced Photon Source (APS), Argonne National Laboratory. X-ray diffraction samples were in the form of powders from a cross-sectional slice of the sample to make sure that the sample surface was not characterized. The microstructures were investigated by scanning and transmission electron microscopy (STEM and TEM), scanning electron microscopy (SEM), including energy-dispersive spectroscopy (EDS) and electron backscatter diffraction (EBSD). Combined EDS and EBSD techniques were used to analyze the dendritic structure, since automatic indexing procedures used during orientation imaging microscopy cannot differentiate A2 and B2 phases. Cylindrical specimens were prepared for tensile tests according to the American Society for Testing and Materials (ASTM) Standard [55]. Uniaxial tensile tests were conducted in air using an 810 Material Test System (MTS) machine equipped with a box furnace. The tensile tests were conducted at 700 °C with a strain rate of $1 \times 10^{-4} \text{ s}^{-1}$. The nanoindentation experiments were performed in a Hysitron TriboScope (Minneapolis, MN) mounted on a Quesant atomic force microscope (AFM) (Agoura Hills, CA). The diamond tip was used with a cube-corner indenter. Constant loading and unloading rates were used in the indentation tests. The penetration depth and applied load were employed in the characterization of the near-surface mechanical behavior of the AlCoCrFeNi alloy.

4. Results

4.1. Tensile property

Figure 2 shows the engineering stress-strain curves of the AlCoCrFeNi alloy in both the as-cast (AlCoCrFeNi-AC) and homogenized (AlCoCrFeNi-HP) conditions for tensile testing at 700 °C. The values of the elongation to fracture (ϵ), yield strength (σ_y), ultimate tensile strength (σ_{UTS}), and specific strength (σ_{UTS}/ρ) of the samples are given Table 1. The values of σ_y for the as-cast and heat-treated alloy are 395 MPa and 295 MPa, respectively. A noticeable increase in tensile elongation occurs after the heat treatment: the elongation increases from 1.0 % for as-cast samples to 11.7 % for heat-treated samples. The values of σ_{UTS} for as-cast (400 MPa) and homogenized (393 MPa) conditions are comparable and may remain within the experimental error. For comparison, the tensile properties of the reported HEAs and conventional structural materials at 700 °C are also listed in Table 1 and Figure 3. Compared to others, the present alloy exhibits the relatively high σ_y , σ_{UTS} , and plastic deformation.

4.2. Microstructures of the alloy in the as-cast condition

Figure 4 presents the image quality, inverse pole-figure and phase-identification maps of grains and dendrites, as well as the elemental maps of the AlCoCrFeNi-AC alloy. The microstructure consists of equiaxed grains. Flowery, branched dendrites are seen inside the grains. The dendrites span is $\sim 40 \mu\text{m}$, and the arm thickness is $\sim 20 \mu\text{m}$. The data were collected simultaneously with the EBSD and EDS techniques. Randomly oriented equiaxed matrix grains are clearly recognized on the image quality and inverse pole figure maps (Figure 4a-b). The phase map (Figure 4c) and EDS element mapping (Figure 4d-h) reveal two distinct compositions. The dendrites with the volume fraction of 45 % are enriched with Al and Ni (called NiAl-rich dendrites), and the interdendritic regions with the volume fraction of 55 % are enriched with Cr

and Fe (called NiAl-poor interdendrites), while Co is uniformly distributed inside these phases. To further investigate phase structures of NiAl-rich dendrites and NiAl-poor interdendrites, bright-field and dark-field TEM images of the interior of the matrix grains are exhibited in Figure 5. The dark-field TEM images were produced using a fundamental spot of the A2 and B2 phases [Figure 5(b)] and a superlattice spot of the B2 phase [Figure 5(c)]. They reveal a lamellar structure of the matrix grains consisting of fine, nanometer-sized lamellae of A2 and B2 phases. These results correspond well to the literatures [31, 43] concerning this as-cast alloy. The authors believe that an eutectic reaction rather than the spinodal decomposition is responsible for the formation of the nano-scaled mixture of the A2 and B2 phases, which is discussed in Section 5.3.

4.3. Microstructures of the alloy after the homogenization treatment

The homogenization treatment led to dramatic changes in the alloy microstructure (Figure 6). The homogenized alloy is called AlCoCrFeNi-HP. Instead of dark dendrites (NiAl-rich) in a bright matrix (NiAl-poor), light-color precipitates are now recognized inside the grey-color matrix and along grain boundaries. The different grey levels of the constituents in the backscattered-electron (BSE) images result from different compositions, e.g., brighter contrasts correspond to higher concentrations of heavier elements. The combined EBSD/EDS analysis reveals the presence of four phases in the homogenized alloy (Figure 7). The matrix grains have a B2 crystal structure. Two other phases, one with an A2 crystal structure and another with a tetragonal crystal structure (σ phase), precipitate inside the large B2 grains. The fourth phase, with the A1 crystal structure, mainly precipitates along grain boundaries, although some particles are also present inside the grains. The elemental analysis of these phases reveals that the B2 phase is rich in Ni and Al, the A2 and σ phases are rich in Cr and Fe, and the A1 phase is slightly enriched with Al, Co, and Cr. The chemical compositions of four phases (A2, B2, A1, and σ)

determined by SEM-EDS are shown in Table 2, and the volume fractions of these four phases determined through the EBSD analysis, and thermodynamic calculations are shown in Table 3. Bright-field TEM images of the AlCoCrFeNi-HP alloy are shown in Figure 8. Nanoprecipitates with an A2 structure (indicated by red arrows) inside the B2 matrix are easily recognized. As shown in Figure 8(c-g), STEM-EDS X-ray maps of the microstructures of the AlCoCrFeNi-HP alloy (in a homogenized condition) confirm that the A2 nanoprecipitates is enriched with Co, Cr, and Fe, while the B2 matrix is enriched with Ni and Al. The chemical compositions of these phases are consistent with those in the homogenized condition (Table 2), showing that A2 has 19 at. % Co, 43 at. % Cr, and 30 at. % Fe. In all, based on the electron microscopic investigation, the AlCoCrFeNi-HP is confirmed to be composed of four major phases: A2, B2, A1, and σ .

4.4. Characterizations using synchrotron X-ray diffraction

High-energy synchrotron X-ray diffraction shows that the as-cast alloy contains two phases: the A2 phase with a lattice parameter of $a = 0.288$ nm, and the B2 phase having a very similar lattice constant and crystal structure with respect to the A2 (Figure 9). A (100) superlattice peak confirms the presence of the B2 phase. The A2+B2 structures were consistent with previous publications [4, 31]. The trend above is also very common in other HEA systems, where Tsai et al. found that the lattice constants among the three phases in $\text{Al}_{0.3}\text{CoCrCu}_{0.5}\text{FeNi}$, measured using the TEM images, were very similar [56].

Four phases were identified in the AlCoCrFeNi-HP: A2 and B2 having the same lattice parameter $a = 0.287$ nm, A1 with $a = 0.359$ nm, and σ with $a = 0.880$ nm and $c = 0.454$ nm. The heat treatment slightly alters the lattice parameter of the A2 and B2 phases from 0.288 nm to 0.287 nm. This trend may be caused by the diffusion of solute atoms and then creation of

vacancies among A2+B2 phases in order to form another two new phases (A1 and σ). Such diffusion will lead to chemical-composition changes, possibly contributing to the slight change of lattice parameters. The σ phase is an ordered intermetallic compound with $P4_2/mnm$ (#136) [57]. The three strongest peaks belonging to this phase, (410), (411), and (331), as well as (002), are found in the synchrotron pattern. Three other σ peaks, (330), (202), and (212), are shielded by peaks from other phases. In sum, phase identification for both the as-cast (A2+B2) and homogenized (A2+B2+A1+ σ) conditions using synchrotron X-ray diffraction well agrees with the electron microscopic results in Sections 4.1 and 4.2.

4.5. Thermodynamic properties and phase equilibrium

Figure 10a presents the calculated solidification path for the AlCoCrFeNi alloy by the Scheil model [58], which indicates that the primary solidified phase is B2. Then the A2+B2 phase will form with decreasing temperature. According to the simulation shown in Figure 10a, only 5 % volume fraction of the liquid is consumed to form the primary B2, and the next 45 % volume fraction of the liquid forms A2+B2 mixture phases. Then, the rest of the liquid solidifies into A2+B2+A1 structures. An equilibrium line calculation is also performed for the AlCoCrFeNi alloy, as shown in Figure 10b. All four phases, A2+B2+A1+ σ , are all presented in the equilibrium phase diagram. As exhibited in the calculation, the A1 phase is in equilibrium with the A2 phase after the heat treatment, which is consistent with the experimental data in the present work and those in the literature [32, 34]. The calculated volume fractions of A2 and B2 are similar to the experimental results, while those of A1 and σ are different from the experimental results (Figure 10b and Table 3). Note that the σ phase does form in the temperature range of 500 - 810 °C (Figure 10b), and it is also observed in the AlCoCrFeNi-HP

microstructure (Figure 7). However, AlCoCrFeNi-HP was annealed beyond the thermodynamically predicted temperature range (500 - 810 °C). Several possible reasons are proposed in Section 5.3. Overall, the thermodynamics prediction, presented in Figure 10, agree qualitatively with the experimental observation in this investigation. The validity of thermodynamic modeling and differences between modeling and experimental results are discussed in Section 5.3.

5. Discussion

5.1. The reason for the improvement of ductility

The present work shows a significant effect of homogenization heat treatment on the microstructure and tensile properties of the AlCoCrFeNi HEA. Most importantly, the number and amount of phases increase and the tensile elongation at 700 °C increases from 1.0 % to 11.7 % following the homogenization heat treatment. How the high-temperature properties of HEAs are related to those of other structural materials and how they depend on heat-treatment conditions and compositions are shown in Table 1 and Figure 3. For example, the tensile behavior of AlCoCrCuFeNi HEA in as-cast and forged conditions is very different: from high strength with limited elongation to low strength with decent elongation. The tensile behavior of Al_{0.5}CoCrCuFeNi HEA in rolled and annealed conditions is also quite different. This trend indicates that proper heat treatments can be applied to HEA system by changing microstructures and tailoring properties. This is also exactly the same situation in the present study. Compared to the AlCoCrFeNi-AC alloy, the AlCoCrFeNi-HP alloy exhibits higher combined properties.

The lamellar structure explains the poor ductility. The AlCoCrFeNi-AC alloy shows 50 - 100 nm lamella of A₂+B₂ phases. Dislocation motion through these finely-spaced interfaces is

very difficult. Similarly, TiAl-based alloys also have very limited tensile ductility at room temperature because of their fully-lamellar structures ($\alpha_2+\gamma$) generally observed in cast conditions [59]. However, the brittleness even up to 700 °C in the AlCoCrFeNi-AC material is probably due to the difficulty of forcing dislocation motion through the alternating thin lamella of different crystal structures. Our TEM results indicate that the boundary planes between the A2 and B2 lamellas are often of {100} plane with $\langle 010 \rangle$ in the long directions of the lamella. The slip systems active in these highly alloyed phases are unknown, but based upon extrapolations from the knowledge of B2-NiAl and A2-Fe, the dominant slip planes are likely {110}-type. Burger's vectors are similarly unknown, but $a\langle 110 \rangle$ in B2 and $a/2\langle 111 \rangle$ in A2 are likely and assumed here. Atom probe tomography (Figure 11a) indicates that the lamellas contain high densities of fine precipitates of the opposite phase. A sketch of AlCoCrFeNi-AC specimen shows an A2+B2 lamellar structure at the atomic level (Figure 11b). Both the lamella boundaries and the fine precipitates likely act as strengthening obstacles to reduce the ductility of the material. For example, if a lamella is 50 nm wide and oriented with $\langle 100 \rangle$ in its long direction, then {110}-plane dislocation will have only ~ 50 or ~ 70 nm of the distance from one edge of the lamella to the other. This distance will be further interrupted by the fine particles, which will require dislocation climb, cross slip, cutting, or bowing, to bypass. The assumed $a\langle 110 \rangle$ B2 dislocations will not easily transfer into the assumed $a/2\langle 111 \rangle$ burger's vector in A2, and vice-versa. Thus, dislocations will probably have to climb, cross-slip, or bow around the nanoparticles, rather than cut the particles. Those dislocations that do pass through the particle forest will then probably pileup at the lamella interface, again due to the difficulty of the shear transfer from the $a\langle 110 \rangle_{B2}$ to $a/2\langle 111 \rangle_{A2}$ burger's vectors. All of these factors together will result in the increased shear stress required to drive dislocations through the structure, and, therefore, reduced ductility.

However, the size scales of the microstructures are very different in two conditions (as-cast and homogenized). In the homogenized condition instead, due to the breakage of the A2+B2 lamella into larger domains (Figure 8), giving the dislocations longer glide-lengths before hitting an obstacle may be the main reason for the improvement of ductility but lower yield strength (Figure 2). Furthermore, the other important reason may be the occurrence of the relatively ductile A1 phase free of precipitates. As shown in Figure 12, the nanoindentation results revealed that the A1 phase has a hardness of 4.3 ± 0.2 GPa, which is less than half of the B2 phase with a hardness of 8.9 ± 0.4 GPa at room temperature. Liu et al. also confirmed that the A1 phase has a lower yield strength than the B2 matrix in the $\text{Al}_{0.8}\text{CoCrCuFeNi}$ HEA at room temperature [60]. Generally, a phase with a lower hardness and a lower yield strength will possess a higher ductility, even more at elevated temperatures. This trend may also happen in our case, which means that the appearance of a large amount of a relatively ductile A1 phase along grain boundaries in the homogenized condition contributes to the improvement of the ductility, compared with the as-cast condition with a very few amount of A1 nanoprecipitates. In addition, the thermal treatment (HIP+homogenization) after casting may reduce defects produced by casting and relieve residual stresses during fast cooling drop casting.

5.2. Crack initiation subjected to tensile loading

The relationship between microstructure and crack initiation of tensile testing is a fundamental principle of materials science, especially shedding some light on new alloy design. For example, TG6 high-temperature titanium alloy, the fine α_L phase increased the resistance to dislocation slip, which delayed the crack nucleation and decreased the crack-propagation velocity [61, 62]. For brittle samples, cracks usually initiated at grain boundaries and triple-junctions, and lead to intergranular fracture [63]. This trend happened in the AlCoCrFeNi-AC

alloy (**Figure 13**). The formation of intergranular cracks under tensile loading in the as-cast condition has two possibilities: (a) cracks formed at the intersections of several grain boundaries, and (b) the formation of voids at grain boundaries aligned perpendicular to the principal tensile stress [63]. It is particularly intriguing that cracks did not form around the existing casting pores in the dendrite as shown in **Figure 13b**, while some bead-like and wedged-like cracks only formed along grain boundaries and triple-junctions, as shown in **Figure 13b-c**. This trend indicates that a small volume of casting pores did not influence the tensile performance at elevated temperatures, because the cracks initiated along grain boundaries instead of the existing casting pores. The intergranular cracks formed during the tensile loading at 700 °C also found in other cast dendritical HEAs subject to room-temperature tensile loading (cast Al₈Co₁₇Cr₁₇Cu₈Fe₁₇Ni₃₃ [64]), and high-temperature compression loading (cast AlCoCrCuFeNi [65], cast Cr-Nb-Ti-V-Zr system [12]), which indicate that the weak parts in cast dendritical HEAs usually locate at the intersections of several grain boundaries.

As for AlCoCrFeNi-HP alloy, no obvious casting pores with gathered precipitates were found in the cross-sections parallel to the loading direction (**Figure 14**). The cracks initially launch on the interface between the B2 and A1 phases, as shown in **Figure 14b-c**. The interfacial crack is first initiated for the AlCoCrFeNi-HP alloy. For the interface between B2 and A1, the following discontinuities in properties need to be considered: (1) crystallography: structures and lattice-parameter variation; (2) moduli: difference in the elastic modulus; (3) coefficients of thermal expansion (CTE). In the AlCoCrFeNi-HP alloy, the lattice parameter of B2 is $a = 0.287$ nm, and the lattice parameter of A1 is $a = 0.359$ nm, which show a large difference with distinct structures (one is B2 and another is A1). In order to investigate the

difference in elastic moduli, the nanoindentation experiment of the two phases (B2 and A1) was performed. As shown in **Figure 12**, the hardness of the B2 phase is more than twice of that of the A1 phase, and the elastic modulus of the B2 phase is $\sim 8\%$ higher than that of the A1 phase. As we know, cracks nucleate at the interfaces as a result of the stresses produced by the elastic incompatibility of the adjacent phases [66]. Because of the difference in compositions and orientations, the elastic moduli of B2 and A1 phases are different, which result in different elastic strains. Although no open literatures to the authors investigated crack initiation of multi-phase HEAs subjected to high-temperature tensile loading, the crack-initiation sites in our case would be expected to be associated with, if not due to, the interface between B2 and A1.

5.3. Validity of thermodynamic modeling

The simulation shows that the structure is a mixture of the A2+B2 owing to this special solidification (note that NiAl-rich dendrites solidified first, and then NiAl-poor interdendritic regions formed), which is consistent with the experimental observation for the microstructures of AlCoCrFeNi-AC alloy in the present work. Note that the volume fraction of the A1 phase is $\sim 50\%$ (Figure 10b), and we may not be able to obtain this amount, since the AlCoCrFeNi-HP alloy may not yet reach equilibrium due to the sluggish diffusion of elements in HEAs and the short annealing period (only 50 hours in the present study).

There is one main difference between thermodynamic-modeling predictions and microstructural characterizations. It is the presence of σ phases in the homogenized condition. As mentioned previously, the σ phase is observed in the microstructure of the AlCoCrFeNi-HP alloy, which was annealed at $1,150^\circ\text{C}$ for 50 hours, beyond the thermodynamically predicted existing temperature range ($500 - 810^\circ\text{C}$). There are two possible reasons for this discrepancy.

One reason may be due to unreasonable thermodynamic parameters for the σ phase in the present thermodynamic model. The other reason may result from the localization of certain alloying elements within this alloy forming the σ phase. For example, a certain amount of Co can improve the stability of the σ phase at higher temperatures, up to 1,283 °C (such as the binary Co-Cr σ phase [67, 68]), which is higher than the annealing temperature of 1,150 °C. Indeed, some amounts (24 at. %) of Co are observed in the σ phase, as presented in Figure 7(d) and shown in Table 2. Of course, it is also likely that the σ phase precipitated during slow furnace cooling (10 °C/min). Chou et al. [34] did not observe the σ phase at 500 °C and 550 °C, which is not surprising, since the precipitation is a diffusion-controlled process, and a certain amount of phases are necessary for the phase identification by the regular lab X-ray diffraction.

Another issue is regarding either spinodal or eutectic decomposition. In several publications on HEAs, the netlike structure, shown in Figure 5, was suggested to form by a spinodal decomposition of a high-temperature solid phase [3, 43]. However, our results show that an eutectic reaction rather than the spinodal decomposition is responsible for the formation of the nano-scale mixture of the A2 and B2 phases. This conclusion is based on the results of the microstructure analysis, which show (a) the presence of the primary B2 grains inside the fine A2+B2 eutectic in the as-cast alloy (Figure 5); (b) the well-developed eutectic structure (Figure 4 and Figure 7); and (c) the precipitation of the secondary A2 phase particles inside the B2 grains in the homogenized alloy (Figure 8). All these observations are in good agreement with the CALPHAD results shown in Figure 10a. The good agreement of our experimental data with thermodynamic calculation results suggests that our interpretation of the microstructure evolution is valid. We however do not exclude the possibility of the spinodal decomposition in the alloy at temperatures below 1,000 °C, but low-temperature phase reactions require an

additional study, which is beyond the scope of the present work.

Above all, the microstructures and mechanical behaviors of one alloy, AlCoCrFeNi, have been carefully studied in two conditions (as-cast and homogenized), as summarized in Figure 15. As confirmed by both experiments (SEM/EDS, EBSD, TEM/STEM, APT, and synchrotron X-ray) and simulations (CALPHAD), the homogenization treatment results in phase evolution: from two major phases (A2+B2) in the as-cast condition to four major phases (A2+B2+A1+ σ) in the homogenized condition. The very different microstructures in the two conditions exhibit similar σ_{UTS} but a noticeable increase in the tensile ductility after the heat treatment (HIP and homogenized).

6. Conclusions

In summary, we discuss how as-cast and homogenized phases can be identified, what phases are usually found in the as-cast and homogenized conditions, and what the thermodynamics and kinetics of phase transformations are in the AlCoCrFeNi HEA, as shown in a summary cartoon of Figure 15.

(1) A noticeable increase in the tensile ductility occurs after the HIP and homogenized treatment. During tensile testing at 700 °C, the elongation of the homogenized alloy is 11.7 %, while the as-cast alloy show the elongation of only 1.0 %. The ultimate tensile strength at 700 °C is almost unaffected by the heat treatment, 400 MPa and 393 MPa, respectively, for the as-cast (AlCoCrFeNi-AC) and homogenized (AlCoCrFeNi-HP) conditions.

(2) AlCoCrFeNi-AC consists primarily of a nano-lamellar mixture of A2 [disorder body-centered-cubic (BCC)] and B2 (ordered BCC) phases. An eutectic reaction rather than

a spinodal decomposition is responsible for the formation of the nano-scale mixture of the A2 and B2 phases. The CALPHAD thermodynamic modeling is well consistent with the experimental data.

(3) Four phases, A2, B2, A1, and σ phases, are found in the AlCoCrFeNi-HP. A2 nanoprecipitates embedded in the B2 matrix are also found in the homogenized condition, while blocky wall-like A1 phases along boundaries are identified and free of precipitates.

(4) The reason for the limited elongation of the AlCoCrFeNi-AC alloy may be that dislocation motion through these finely-spaced interfaces (50 - 100 nm) is very difficult, resulting in higher yield strength. The improvement of ductility for the AlCoCrFeNi-HP alloy may be due to the breakage of the A2+B2 lamella into larger domains, the occurrence of relatively ductile FCC phase, reduced casting defects, and relieved residual stress.

(5) For the AlCoCrFeNi-AC alloy, intergranular beadlike cracks were found to initiate along grain boundaries rather than the existing casting pores. For the AlCoCrFeNi-HP alloy, initial cracks formed on the interface between B2 and A1 phases, because the crystallography, moduli, and CTE were discontinuous and different at the interface.

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References

- [1] K. Lu. *Science* 328 (2010) 319-320.
- [2] B. Cantor, I.T.H. Chang, P. Knight, A.J.B. Vincent. *Mater. Sci. Eng. A* 375 (2004) 213-218.
- [3] J.W. Yeh, S.K. Chen, S.J. Lin, J.Y. Gan, T.S. Chin, T.T. Shun, C.H. Tsau, S.Y. Chang. *Adv. Eng. Mater.* 6 (2004) 299-303.
- [4] Y.J. Zhou, Y. Zhang, Y.L. Wang, G.L. Chen. *Appl. Phys. Lett.* 90 (2007) 181904.
- [5] O.N. Senkov, G.B. Wilks, D.B. Miracle, C.P. Chuang, P.K. Liaw. *Intermetallics* 18 (2010) 1758-1765.
- [6] Y. Zhang, X. Yang, P.K. Liaw. *Jom-Us* 64 (2012) 830-838.
- [7] M.A. Hemphill, T. Yuan, G.Y. Wang, J.W. Yeh, C.W. Tsai, A. Chuang, P.K. Liaw. *Acta Mater.* 60 (2012) 5723-5734.
- [8] F. Otto, Y. Yang, H. Bei, E.P. George. *Acta Mater.* 61 (2013) 2628-2638.
- [9] O.N. Senkov, S.V. Senkova, C. Woodward, D.B. Miracle. *Acta Mater.* 61 (2013) 1545-1557.
- [10] C. Zhu, Z.P. Lu, T.G. Nieh. *Acta Mater.* 61 (2013) 2993-3001.
- [11] Y. Zhang, T. Zuo, Y. Cheng, P.K. Liaw. *Sci. Rep.* 3 (2013) 1455.
- [12] O.N. Senkov, S.V. Senkova, D.B. Miracle, C. Woodward. *Mater. Sci. Eng. A* 565 (2013) 51-62.
- [13] W. Guo, W. Dmowski, J.-Y. Noh, P. Rack, P. Liaw, T. Egami. *Metall. Mater. Trans. A* 44 (2013) 1994-1997.
- [14] Z. Tang, M.C. Gao, H.Y. Diao, T.F. Yang, J.P. Liu, T.T. Zuo, Y. Zhang, Z.P. Lu, Y.Q. Cheng, Y.W. Zhang, K.A. Dahmen, P.K. Liaw, T. Egami. *Jom-Us* 65 (2013) 1848-1858.
- [15] Y. Zhang, T.T. Zuo, Z. Tang, M.C. Gao, K.A. Dahmen, P.K. Liaw, Z.P. Lu. *Prog. Mater. Sci.* 61 (2014) 1-93.
- [16] F. Tian, L. Delczeg, N. Chen, L.K. Varga, J. Shen, L. Vitos. *Phys. Rev. B* 88 (2013) 085128.
- [17] D.B. Miracle, J.D. Miller, O.N. Senkov, C. Woodward, M.D. Uchic, J. Tiley. *Entropy* 16 (2014) 494-525.
- [18] Z. Tang, L. Huang, W. He, P. Liaw. *Entropy* 16 (2014) 895-911.
- [19] Y. Zhang, Z.P. Lu, S.G. Ma, P.K. Liaw, Z. Tang, Y.Q. Cheng, M.C. Gao. *MRS Commun.* 4 (2014) 57-62.
- [20] M. Komarasamy, N. Kumar, Z. Tang, R.S. Mishra, P.K. Liaw. *Mater. Res. Lett.* 3 (2014) 30-34.
- [21] L.J. Santodonato, Y. Zhang, M. Feygenson, C.M. Parish, M.C. Gao, R.J. Weber, J.C. Neufeld, Z. Tang, P.K. Liaw. *Nature communications* 6 (2015) 5964.
- [22] J. Antonaglia, X. Xie, Z. Tang, C.W. Tsai, J.W. Qiao, Y. Zhang, M.O. Laktionova, E.D. Tabachnikova, J.W. Yeh, O.N. Senkov, M.C. Gao, J.T. Uhl, P.K. Liaw, K.A. Dahmen. *Jom-Us* 66 (2014) 2002-2008.
- [23] Z. Tang, T. Yuan, C.W. Tsai, J.W. Yeh, C.D. Lundin, P.K. Liaw. *Acta Mater.* (2015) <http://dx.doi.org/10.1016/j.actamat.2015.1007.1004>.
- [24] O.N. Senkov, G.B. Wilks, J.M. Scott, D.B. Miracle. *Intermetallics* 19 (2011) 698-706.
- [25] Y. Lu, Y. Dong, S. Guo, L. Jiang, H. Kang, T. Wang, B. Wen, Z. Wang, J. Jie, Z. Cao, H. Ruan, T. Li. *Sci. Rep.* 4 (2014) 6200.
- [26] C.W. Tsai, M.H. Tsai, J.W. Yeh, C.C. Yang. *J. Alloys Compd.* 490 (2010) 160-165.

- [27] A.V. Kuznetsov, D.G. Shaysultanov, N.D. Stepanov, G.A. Salishchev, O.N. Senkov. *Mater. Sci. Eng. A* 533 (2012) 107-118.
- [28] J.Y. He, W.H. Liu, H. Wang, Y. Wu, X.J. Liu, T.G. Nieh, Z.P. Lu. *Acta Mater.* 62 (2014) 105-113.
- [29] C. Ng, S. Guo, J. Luan, Q. Wang, J. Lu, S. Shi, C.T. Liu. *J. Alloys Compd.* 584 (2014) 530-537.
- [30] W.-R. Wang, W.-L. Wang, S.-C. Wang, Y.-C. Tsai, C.-H. Lai, J.-W. Yeh. *Intermetallics* 26 (2012) 44-51.
- [31] Y.P. Wang, B.S. Li, M.X. Ren, C. Yang, H.Z. Fu. *Mater. Sci. Eng. A* 491 (2008) 154-158.
- [32] Y.-F. Kao, T.-J. Chen, S.-K. Chen, J.-W. Yeh. *J. Alloys Compd.* 488 (2009) 57-64.
- [33] H.B. Cui, Y. Wang, J.Y. Wang, X.F. Guo, H.Z. Fu. *China Foundry* 8 (2011) 259-263.
- [34] H.P. Chou, Y.S. Chang, S.K. Chen, J.W. Yeh. *Mater. Sci. Eng., B* 163 (2009) 184-189.
- [35] J.M. Zhu, H.M. Fu, H.F. Zhang, A.M. Wang, H. Li, Z.Q. Hu. *J. Alloys Compd.* 509 (2011) 3476-3480.
- [36] S.G. Ma, Y. Zhang. *Mater. Sci. Eng. A* 532 (2012) 480-486.
- [37] F.J. Wang, Y. Zhang, G.L. Chen, H.A. Davies. *J. Eng. Mater. Technol.* 131 (2009).
- [38] J.M. Zhu, H.M. Fu, H.F. Zhang, A.M. Wang, H. Li, Z.Q. Hu. *Mater. Sci. Eng., A* 527 (2010) 6975-6979.
- [39] C. Li, M. Zhao, J.C. Li, Q. Jiang. *J. Appl. Phys.* 104 (2008) 113504.
- [40] C. Li, J.C. Li, M. Zhao, Q. Jiang. *J. Alloys Compd.* 504, Supplement 1 (2010) S515-S518.
- [41] W.-R. Wang, W.-L. Wang, J.-W. Yeh. *J. Alloys Compd.* 589 (2014) 143-152.
- [42] Y. Zhang. *Mater. Sci. Forum* 654-656 (2010) 1058-1061.
- [43] A. Manzoni, H. Daoud, R. Volkl, U. Glatzel, N. Wanderka. *Ultramicroscopy* 132 (2013) 212-215.
- [44] M. Widom, W.P. Huhn, S. Maiti, W. Steurer. *Metall. Mat. Trans. A* 45 (2014) 196-200.
- [45] L. Kaufman, H. Bernstein. *Computer calculation of phase diagrams with special reference to refractory metals*, Academic Press, New York, 1970.
- [46] J.M. Joubert. *Prog. Mater. Sci.* 53 (2008) 528-583.
- [47] H.B. Cao, Y. Pan, L. Ding, C. Zhang, J. Zhu, K.C. Hsieh, Y.A. Chang. *Acta Mater.* 56 (2008) 2032-2036.
- [48] H. Bei, Y. Yang, G.B. Viswanathan, C.J. Rawn, E.P. George, J. Tile, Y.A. Chang. *Acta Mater.* 58 (2010) 6027-6034.
- [49] Y. Yang, C. Zhang, S. Chen, D. Morgan, Y.A. Chang. *Intermetallics* 18 (2010) 574-581.
- [50] C. Zhang, F. Zhang, S. Chen, W. Cao. *Jom-Us* 64 (2012) 839-845.
- [51] K.-C. Chou, Y. Austin Chang. *Berichte der Bunsengesellschaft für physikalische Chemie* 93 (1989) 735-741.
- [52] C. Zhang, F. Zhang, S.-L. Chen, W.-S. Cao, Y. Chang. *Acta Mater.* 59 (2011) 6246-6256.
- [53] C. Zhang, M. Gao, Y. Yang, F. Zhang. *Calphad* 45 (2014) 178-187.
- [54] W. Zhao, Z. Sun, Z. Tang, P.K. Liaw, J. Li, R.P. Liu, G. Li. *Metall. Mater. Trans. A* 45 (2013) 2371-2375.
- [55] ASTM E8. *Standard Test Methods for Tension Testing of Metallic Materials*. West Conshohocken, PA: ASTM International, 2012.
- [56] M.-H. Tsai, H. Yuan, G. Cheng, W. Xu, K.-Y. Tsai, C.-W. Tsai, W.W. Jian, C.-C. Juan, W.-J. Shen, M.-H. Chuang, J.-W. Yeh, Y.T. Zhu. *Intermetallics* 32 (2013) 329-336.
- [57] G. Bergman, D.P. Shoemaker. *Acta Crystallogr.* 7 (1954) 857-865.
- [58] E. Scheil. *Z. Metallkd* 34 (1942) 70-72.

- [59] C.T. Liu, J.H. Schneibel, P.J. Maziasz, J.L. Wright, D.S. Easton. *Intermetallics* 4 (1996) 429-440.
- [60] Z. Liu, S. Guo, X. Liu, J. Ye, Y. Yang, X.-L. Wang, L. Yang, K. An, C.T. Liu. *Scripta Mater* 64 (2011) 868-871.
- [61] T. Wang, H. Guo, L. Tan, Z. Yao, Y. Zhao, P. Liu. *Mater. Sci. Eng. A* 528 (2011) 6375-6380.
- [62] T. Wang, H. Guo, Y. Wang, X. Peng, Y. Zhao, Z. Yao. *Mater. Sci. Eng. A* 528 (2011) 2370-2379.
- [63] C.R. Brooks, A. Choudhury. *Failure analysis of engineering materials*, McGraw-Hill, New York, 2002.
- [64] H.M. Daoud, A. Manzoni, R. Völkl, N. Wanderka, U. Glatzel. *Jom-Us* 65 (2013) 1805-1814.
- [65] N. Nayan, G. Singh, S.V.S.N. Murty, A.K. Jha, B. Pant, K.M. George, U. Ramamurty. *Intermetallics* 55 (2014) 145-153.
- [66] L.E. Murr. *Interfacial phenomena in metals and alloys*, Addison-Wesley Pub. Co., Advanced Book Program, Reading, Mass., 1975.
- [67] K. Ishida, T. Nishizawa. *J. Phase Equilib.* 11 (1990) 357-370.
- [68] ASM International. *Alloy Phase Diagram Committee.*, ASM International. *Handbook Committee.* ASM handbook. Materials Park, Ohio: ASM International, 1992.
- [69] J. Chen, B. Young. *Eng. Struc.* 28 (2006) 229-239.
- [70] V. Venkatesh, H.J. Rack. *Mech. Mater.* 30 (1998) 69-81.
- [71] R. Schaeublin, T. Leguey, P. Spatig, N. Baluc, M. Victoria. *J. Nucl. Mater.* 307 (2002) 778-782.
- [72] R.S. Sundar, S.C. Deevi. *Mater. Sci. Eng. A* 357 (2003) 124-133.
- [73] J.C. Li, T.Y. Wu, Y. Riquier. *Mater. Sci. Eng. A* 174 (1994) 149-156.
- [74] C.T. Liu. *Mater. Chem. Phys.* 42 (1995) 77-86.
- [75] V.K. Sikka, S.C. Deevi, S. Viswanathan, R.W. Swindeman, M.L. Santella. *Intermetallics* 8 (2000) 1329-1337.
- [76] S. Noh, R. Kasada, A. Kimura. *Acta Mater.* 59 (2011) 3196-3204.
- [77] A. Hirata, T. Fujita, Y.R. Wen, J.H. Schneibel, C.T. Liu, M.W. Chen. *Nat. Mater.* 10 (2011) 922-926.

Table 1. Tensile properties at 700 °C for AlCoCrFeNi-AC, AlCoCrFeNi-HP, and some other high-temperature structural materials (all of these data were tested in tension at 700 °C): elongation to fracture (ϵ), yield strength (σ_y), ultimate tensile strength (σ_{UTS}), and specific strength (σ_{UTS}/ρ). Densities of all materials are also listed. Note that density (ρ) values of the present work (AlCoCrFeNi-AC and AlCoCrFeNi-HP) were measured with a helium pycnometer, while density values of other HEAs are calculated by the theoretical density of a disordered solid solution [5].

Materials	ϵ (%)	σ_y (MPa)	σ_{UTS} (MPa)	σ_{UTS}/ρ [MPa/(g/cm³)]	ρ (g/cm³)
AlCoCrFeNi-AC (This Work)	1.0	395	400	57.1	7.0
AlCoCrFeNi-HP (This Work)	11.7	295	393	56.1	7.0
AlCoCrCuFeNi As-Cast [27]	4.7	350	360	50.7	7.1
AlCoCrCuFeNi Forged [27]	63	63	91	12.8	7.1
Al _{0.5} CoCrCuFeNi As-Rolled [26]	5.0	180	185	24.3	7.6
Al _{0.5} CoCrCuFeNi As-Annealed [26]	12	160	180	23.7	7.6
Duplex Stainless Steel [69]	2.4	329	366	46.9	7.8
304 Stainless Steel [69]	9.1	179	248	31.8	7.8
Ni-Based Inconel 690 [70]	26	150	478	53.7	8.9
ODS Steel 0.3 % Yttria [71]	11	210	272	34.9	7.8
Intermetallics FeAl 787 [72]	18	345	350	64.8	5.4

Table 2. Chemical compositions (at. %) of each phase in AlCoCrFeNi-HP (A2+B2+A1+ σ) by SEM-EDS (more than 3 different locations and less than 2 at. % standard deviation).

AlCoCrFeNi-HP					
Phase \ Element	Al	Co	Cr	Fe	Ni
A2	3	19	43	30	6
B2	30	19	9	14	27
A1	8	23	27	27	15
σ	5	24	28	31	11

Table 3. Volume fraction of each phase in homogenized (A2+B2+A1+ σ) conditions by the EBSD analysis and thermodynamic modeling, respectively.

AlCoCrFeNi-HP		
Phase	Experiment	Modeling
A2	24 %	43 %
B2	46 %	40 %
A1	16 %	17 %
σ	14 %	-
Total	100 %	100 %

Figure 1. An Ashby map showing ultimate tensile strength (UTS), σ_{UTS} , versus density, ρ , for structural materials, along with the data for bulk metallic glasses (BMGs) and HEAs [23, 25-29]. Grey dashed contours label the specific strength, σ_{UTS} / ρ , from low (right bottom) to high (left top). HEAs are among the materials that have better combination of mechanical properties of strength and density, compared to other materials.

Figure 2. The tensile engineering stress-strain curves of the AlCoCrFeNi alloy, in both as-cast (AlCoCrFeNi-AC) and homogenized (AlCoCrFeNi-HP) conditions, at the temperature of 700 °C. The inserted figure illustrates the cylindrical sub-size specimens for tensile tests.

Figure 3. A scatter plot shows the specific strengths and elongations to failure of the AlCoCrFeNi-AC and AlCoCrFeNi-HP, and other high-temperature structural materials at 700 °C [26, 27, 69-77]. Compared to conventional structural alloys, the AlCoCrFeNi-HP has a comparable combination of mechanical properties at elevated temperatures.

Figure 4. The EBSD (a) image-quality map, (b) inverse pole-figure map, and (c) phase map. Black lines in (b-c) are phase or grain boundaries. Simultaneously-acquired EDS (d-h) X-ray maps of the microstructures of the AlCoCrFeNi-AC alloy.

Figure 5. Bright-field and dark-field TEM images of the interior of the matrix grains: (a) is bright-field TEM, (b) fundamental A2 dark-field TEM, and (c) superlattice B2 dark-field TEM images. Insets to (b-c) are convergent beam electron diffraction patterns from A2 and B2, respectively. Arrows denote the superlattice position of B2.

Figure 6. Back-scattered-electron (BSE) images of the homogenized alloy at two different magnifications.

Figure 7. The phase-map microstructures of the AlCoCrFeNi-HP alloy. EBSD + EDS-derived phase map (a) low magnification and (b) high magnification. The EDS maps are presented in (c-g): B2 [(grey color in (a))], A2 [(red color in (a))], σ phase [(blue color in (a))], and A1 [(green color in (a))].

Figure 8. Bright-field (a) and dark-field (b) TEM images of the AlCoCrFeNi-HP alloy. Simultaneously-acquired STEM-EDS (c-g) X-ray maps of the microstructures of the AlCoCrFeNi-HP alloy confirming that the nanoprecipitates are enriched with Co, Cr, and Fe, while the matrix are enriched with Ni and Al.

Figure 9. The high-energy synchrotron X-ray diffraction patterns of as-cast and homogenized conditions. The identified phases are shown in the legend. For the Sigma (σ) phase, the following lattice parameters were used: $a = 8.800 \text{ \AA}$, $c = 4.544 \text{ \AA}$. Two insets show the diffraction rings for as-cast and homogenized conditions, corresponding to the synchrotron X-ray diffraction pattern.

Figure 10. Thermodynamics modeling of (a) non-equilibrium solidification (fast cooling) by Scheil model [58] and (b) an equilibrium phase diagram.

Figure 11. (a) Atom probe tomography (APT) of the AlCoCrFeNi-AC alloy, and (b) corresponding sketch of different atom clusters, such as A2 and B2.

Figure 12. Hardness and reduced modulus of B2 and A1 phases by nanoindentation.

Figure 13. SEM images of a longitudinal cross-section of the AlCoCrFeNi-AC alloy after tensile deformation at 700 °C. Microstructures below the fracture surface display tensile center cracks, and casting pores. Cracks preferentially initiate along grain boundaries.

Figure 14. Optical light microscopic images (a-b) of a longitudinal cross-section below the fracture surface in the AlCoCrFeNi-HP alloy after tensile deformation at 700 °C; SEM image (c) of cracks with a high magnification of the AlCoCrFeNi-HP alloy. Cracks are found at the interface between B2 and A1.

Figure 15. A summary cartoon of all major results in the present study.

Figure 1.

Figure 2.

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