Fatigue Behavior of a Wrought $\text{Al}_{0.5}\text{CoCrCuFeNi}$ Two-Phase High-Entropy Alloy

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Abstract

Fatigue behavior of a cold-rolled two-phase $\text{Al}_{0.5}\text{CoCrCuFeNi}$ high-entropy alloy (HEA) was studied. Some specimens were fabricated, using commercial-purity raw materials, while others were manufactured with high-purity components. Scatter in the fatigue life of the commercial-purity samples was found in the stress vs. lifetime plot (S-N curve). However, the high-purity samples showed less scatter, and fatigue life is predictable using fatigue statistics. The fatigue property of the alloy is comparable with and may even outperform many commercial alloys. Fatigue cracking is promoted by shrinkage pores with a size of $\sim 5\ \mu\text{m}$, while mechanical nanotwinning were found to be the main deformation mechanism before crack initiation and during crack propagation by transmission electron microscopy (TEM). Two orientations of dense nanotwins were found at the crack initiation site, while less-dense nanotwins were found away from the crack initiation site. The nanotwinning behavior resulted in strengthening of the alloy and, consequently, high fatigue strength ($383 \pm 71\ \text{MPa}$). Moreover, statistical models were applied to predict fatigue life, suggesting that using improved fabrication processes and/or high-

purity raw materials may enhance the fatigue behavior and scatter by reducing the number of fabrication microcracks and pores in the test samples.

Keywords: high-entropy alloys; fatigue behavior; deformation nanotwins; statistical models

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1. Introduction

Metallic structural alloys with excellent mechanical properties at elevated temperatures always remain in high demand for the aerospace and nuclear industries. Modern and advanced technologies not only strongly depend on the existing properties of metals, but urgently call for even better metals with unique properties [1]. Recently, a new strategy for the development of high-strength and high-temperature alloys, called high-entropy alloys (HEAs) and/or multi-principal-element alloys (MPAs), has been proposed and developed [2-12]. Researchers generally assume that such alloys will lead to complex and brittle intermetallics. However, single face-centered-cubic (FCC), body-centered-cubic (BCC), and/or hexagonal-close-packed (HCP) phases have been found in many HEAs systems [3, 5, 6, 8, 10, 11, 13-20]. When the number of elements, N , is equal or larger than 5 and the concentration of each of the alloying elements is between 5 and 35 atomic percent (at. %), the high mixing entropy can significantly reduce the Gibbs free energy and stabilize solid-solution-like phases with relatively simple crystal structures, compared to intermetallic phases, especially at high temperatures [4, 21, 22].

If we seek the applications in the aerospace industry or other fields, besides monotonic loading, the fatigue behavior and lifetime prediction are one of the most influential factors, which are required to be studied and explored, yet rarely reported in HEA systems. The first and only publication so far concerning the fatigue behavior of $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEAs came from our group, Hemphill et al [2]. Their study showed promising fatigue-resistance characteristics due to the prolonged fatigue lives of various samples at relatively-high stresses. Compared with conventional alloys, such as steels, Ti-based alloys, and advanced bulk metallic glasses (BMGs), their results suggested that the fatigue behavior of HEAs compared favorably and could be superior to other alloys.

However, three main issues remain to be explored further. The first issue is that the fracture mechanism in four-point-bending fatigue has not been clearly defined [2]. The precise knowledge of the fracture mechanism is essential for understanding their promising fatigue resistance and developing life-prediction capabilities. Thus, microstructure evolution and crack initiation during fatigue tests was investigated. The second issue is that a large amount of scatter was observed in the stress vs. lifetime plot (also called S-N curve) [2]. These authors believe that the reason of the scatter is due to microstructural defects, such as aluminum-oxide inclusions and microcracks, which were introduced during sample fabrication and thermal heat treatments, including initial casting and cold rolling. Thus, based on what has been found [2], two new sets of HEAs were successfully fabricated: (1) shrinkage pores and macrosegregation in the as-cast ingot were mechanically removed by machining and cold-rolling; (2) besides removing pores and segregation, high-purity elements were used as raw materials in order to reduce the extent of oxide inclusions and microcracks.

The third issue that was investigated is the connection between defects and fatigue-endurance limits of HEAs by means of statistic fatigue-life modeling [2]. The fatigue-endurance limit is usually treated as a constant for a material, and it is a common practice to report the endurance limit of a material to be the stress level below which failure will not occur before an arbitrary large number of cycles, usually 10^7 cycles is reached. Moreover, the S-N data are usually analyzed by a simple linear regression approach, which cannot adequately take into account the significant scatter in the data. Nelson [23] first suggested modeling the endurance limit of a material as a random variable instead of a fixed constant; that is, test specimens have different endurance limits according to some “strength distribution.” Pascual and Meeker [24] later developed a random endurance-limit fatigue-life model to describe (1) the dependence of fatigue life on the stress level and (2) the scatter in the S-N data. They applied their model to analyze the four-point-bending fatigue data of the carbon eight-harness-satin/epoxy laminate, and their results provided evidence suggesting a random nature of the fatigue-endurance limit. They argued that the randomness in the endurance limit is due in part to the location, orientation, size, and number of defects (e.g., cracks) in the material, which are random themselves. Wallin [25] also examined the statistical aspects of the fatigue-endurance limit, and observed that the variation in the endurance limit has a pronounced effect on the scatter in the fatigue life close to the endurance limit. Lognormal, normal, and Weibull distributions have been proposed in the literature to describe the randomness in the endurance limit [25]. In order to partially-understand the scatter in the fatigue life and the connection between defects and fatigue-endurance limits of HEAs, this study adopts the random endurance-limit fatigue-life model to analyze the S-N data of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA obtained from three conditions of samples (two from this study

and one from [2]), using different fabrication processes and/or different purity grades of the raw materials.

2. Experimental procedures

The material investigated in the present study was a cold-rolled $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA. Totally three conditions of samples were prepared (Table 1). Three ingots of $\text{Al}_{0.5}\text{CoCrCuFeNi}$ (mole percent) were prepared by arc-melting the constituent elements on a water-cooled copper hearth. The individual ingredients came from two sources: one was fabricated using commercial-purity raw materials for the first and second ingots (Condition-1 and Condition-2), while the other was manufactured with high-purity raw materials for the third ingot (Condition-3). The melting was accomplished at a vacuum level of 0.01 torr. The melting and solidification processes were repeated at least five times to improve the homogeneity of the sample, and then cast into a copper mold. The three cast ingots were annealed at 1,000 °C for six hours and water quenched. Shrinkage pores and macrosegregation on surfaces of second and third ingots were mechanically removed by machining. The three ingots were cold-rolled at room temperature, and the rolling reduction was 84 % with a final thickness of 3 mm. These three cold-rolled plates were, subsequently, machined into three conditions of rectangular specimens with dimensions of 25 x 3 x 3 mm. The longitudinal direction of machined specimens was maintained parallel to the rolling direction. To remove any surface with imperfections produced during the fabrication process, the samples were ground, polished, and finished by 1,200 grit SiC paper, prior to fatigue tests.

High-cycle four-point-bending fatigue experiments were conducted on the HEAs at different applied loads. Tests continued until the failure of the specimen, or to 10^7 cycles were attained, using a computer-controlled material test system based on a servohydraulic-testing

machine. The results of each test were plotted on a typical stress versus the number of cycles to failure curve (also called S-N curve). The stress, σ , on the tensile surface within the span was calculated, using the following beam-theory relationship,

$$\sigma = \frac{3P(S_o - S_i)}{2BW^2} \quad (2)$$

where P is the applied load, S_o is the outer span length of 20 mm, S_i is the inner span length of 10 mm, B is the thickness, and W is the height, both 3 mm, respectively. Samples were subjected to four-point-bending fatigue tests with a stress ratio (R) of 0.1 (R = minimum stress/maximum stress, and note that the maximum stress is up to 1,250 MPa in the present study) under a load-controlled mode, using a sinusoidal waveform at a frequency of 10 Hz. Accordingly, the maximum stress amplitude [(maximum stress – minimum stress)/2] in the present study is 563 MPa.

All cross-sectional examinations [Figure 1a (it only shows samples in the Condition-3 here, and those in the Conditions 1 and 2 show the same phenomenon)] were conducted in the as-received condition (after cold rolling, before fatigue testing) using optical microscopy. More than 10 cross-sections of each condition were examined for pores and microcracks. To obtain the lattice structures, synchrotron X-ray diffraction (XRD) was performed using the 5-ID beam line at the Advanced Photon Source (APS), the Argonne National Laboratory. The specimens were ~ 1 mm thick using a beam energy of 60 keV with a beam size of 1 x 1 mm. The scattered, transmitted X-rays were collected, using a Mar345 image plate. To examine the nature of the cyclic-strain localization and damage accumulation mechanisms under cyclic loading, the focus-ion-beam (FIB) technique was performed on the samples. Platinum (Pt) was deposited on the fracture surface area for protection before applying FIB. The microstructures before and after the

fatigue tests and fracture surfaces after the fatigue tests were analyzed using scanning electron microscopy (SEM) with the energy-dispersive spectroscopy (EDS), and transmission electron microscopy (TEM).

3. Results

3.1. Microstructural characterization before fatigue testing

Microstructural characterization before fatigue tests was performed, using SEM with the EDS elemental mapping, TEM, and synchrotron XRD. Figure 2 shows Condition-3 $\text{Al}_{0.5}\text{CoCrCuFeNi}$ microstructures on three sides [the cold-rolled surface, electric-discharge-machining (EDM) cut surface, and cross section] with SEM-EDS elemental mapping of six principal elements, including Al (red), Co (green), Cr (blue), Cu (yellow), Fe (cyan), and Ni (magenta). Cu segregation is clearly seen from the elemental mapping. Suffering the same fabrication process (cast + annealed + cold-rolled), the microstructures of three conditions are very similar, consisting of two phases: (1) a continuous matrix, formed from the primary dendrite phase (dark color), and (2) a Cu-rich second phase (light color), developed from the Cu-rich interdendritic phase, which is consistent with previous studies [2, 3, 13, 15, 26]. For further investigation of the Cu-rich interdendritic phase, TEM was performed, as shown in Figure 3. The EDS results in our previous publication (Table 1 in Hemphill et al. [2]) are shown for the compositions of the matrix phase and Cu-rich phase. In addition, image and diffraction analyses reveal that the Cu-rich phase seen on the SEM image in fact has a high density of nanoprecipitates, which have an FCC structure, and a particle size of 5 - 10 nm. The nanoprecipitates are very probably Ni-, Al-rich clusters, since Ni and Al has strong bonding with each other and tend to segregate to form the final NiAl-rich B2 (ordered BCC) structure. Figure 4 shows synchrotron X-ray line profiles of three conditions of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA: black

for Condition-1, blue for Condition-2, and red for Condition-3. Synchrotron X-ray profiles of three conditions are similar, only showing a single FCC structure with a lattice parameter of 0.362 ± 0.002 nm. As two phases can be distinguished on SEM images (Figure 2), it demonstrates that the two phases are FCC and enjoy a close match in the lattice parameters (0 – 1 %). The matrix and minor are hereafter denoted by FCC-1 and FCC-2. An ordered structure indicated by the (100) super-lattice peak is not present in the synchrotron XRD patterns, which reveals that no ordered FCC (called $L1_2$) phase is found in this alloy. The inserted synchrotron two-dimensional (2-D) diffraction pattern in Figure 4 (it only shows the Condition-3 here, and the Conditions 1 and 2 present the same phenomenon) shows a texture, which is due to cold rolling.

3.2. S-N curves of four-point-bending fatigue

High-cycle four-point-bending fatigue test results are plotted as the stress amplitude [the stress amplitude calculated from Equation (2) by replacing P with $P_{\max} - P_{\min}$, where $P_{\max}$ and $P_{\min}$ are the applied maximum and minimum loads, respectively] vs. the number of cycles to failure or run-out (10^7 cycles) to give the S-N curve, as shown in Figure 5. In general, the shape of the S-N curve of the $Al_{0.5}CoCrCuFeNi$ HEA is similar to those for other crystalline metals and amorphous metals [27-33]. The S-N curve shows a distinct endurance limit of 383 ± 71 MPa, which is below the yield strength of this alloy (1,284 MPa [2]), and the fatigue ratio of the fatigue limit to tensile strength (1,344 MPa [2]) is about 0.29. These results indicate that HEAs have favorable and/or greater endurance limits, and fatigue ratios, compared to steels and other conventional alloys, as will be discussed later. Above the endurance limit, the stress amplitude generally exhibits a relationship with the number of fatigue cycles, i.e., decreasing the stress amplitude tends to increase the fatigue life.

As seen from Figure 5, there is a noticeable extent of scatter at each stress amplitude for regular-purity samples (black dots for Condition-1 and blue triangles for Condition-2, respectively). However, the Condition-3 (red squares) samples showed less scatter. For example, at a stress amplitude of 495 MPa, two fatigue failures of the Condition-2 samples are within an order of magnitude between 2.7×10^4 and 1.3×10^5 cycles while two failures of Condition-3 samples only between 6.0×10^4 and 9.8×10^4 cycles. At a stress amplitude of 540 MPa, most failures are within an order of magnitude of each other, between 3.0×10^4 and 1.0×10^5 cycles for Conditions-2, and 3. As the stress decreases, especially near the range of the endurance limit, the spread in failures becomes more pronounced than at a higher stress amplitude, which is characteristic of fatigue behavior [34]. Estimations of the endurance limits based on the stress amplitude are within a lower bound of 270 MPa and an upper bound of 473 MPa. These values were chosen because the specimens reached 10^7 cycles without failure. The results reveal the general trend of the S-N data: the fatigue life increased with decreasing stress amplitude, as is normally observed for crystalline materials.

3.3. Fractography

The fracture surfaces were examined using SEM. Figure 6 presents the fracture surface of a Condition-3 specimen tested at a relatively high stress amplitude of 495 MPa, with an R ratio of 0.1, in air and at room temperature. The topography of the fracture surface possesses very crystallographic features: (i) a crack-initiation region, (ii) a slow and stable crack-propagation region with a crystallographic appearance and striations, and (iii) a fast-fracture region with dimpled-overload [Figure 6(a)]. Under four-point-bending cyclic loading, the crack-initiation site could usually be identified at a corner of the tensile stress surface, by tracing the radial marks on the fracture surface, which pointed toward the origin of the fracture. Voids and/or inclusions

were found in the higher-magnification micrograph of the fatigue-crack-initiation region [Figure 6(b)]. It was confirmed that the cracks initiated from pores but the direction of striations were random, because the microcracks may form from several pores. Following crack initiation, the fatigue crack propagates randomly through and around pores, which indicates that the direction of fatigue striations has a variable path, which has been changed owing to existing pores. The characteristic feature of the crack-propagation region exhibits a stair-step appearance with fatigue striations that formed due to the four-point-bending cyclic loading, as revealed by the SEM examination in Figure 6(c). Some areas in Figure 6(c) also exhibit a cleavage-like fracture surface, which means that the crack propagates in both ductile and brittle modes. The random directions of the fatigue striation in Figure 6(c) are consistent with those in the crack-initiation region. These trends indicate that the crack propagation is crystallographic. The fast fracture region [Figure 6(d)] contains typical ductile fracture features, such as dimples, which indicates that some ductility in the alloy is present. The local plastic deformation can be seen as the development of slip lines and slip bands after a significant number of loading cycles, which eventually reach a size such that they can act as the initiator of the fatigue crack, followed by crack propagation, as shown in Figure 7. Some oxides were found at high magnification in the crack-initiation region. As shown in Figure 8(b-c), the area adjacent to the crack-initiation site experiences significant plastic deformation due to stress concentration, followed by crack propagation with slip bands. Found in Figure 8(b), peaks (extrusions) and troughs (intrusions) of the surface of the sample were formed due to the movement of material along slip planes, which serve as stress risers to create microcracks and facilitate crack propagation. Microcracks nucleated along planes of the high shear stress can be found, as shown in Figure 8-Figure 10.

3.4. Deformation substructures

To examine the nature of cyclic strain localization and damage accumulation mechanisms under cyclic loading, TEM-FIB specimen was removed from a failed specimen with a lifetime of 2,069,447 cycles at a stress amplitude of 540 MPa in Condition-1. Three different locations, including crack initiation site, adjacent to the crack-initiation site, and away from the crack initiation site, were the major focus, and the TEM analyses are shown in Figure 11. Figure 11(c) indicates that the low nanotwin density associated with tangled dislocations already exists in the cold-rolled specimens before fatigue testing, since the location away from the crack-initiation site and free from plastic deformation shows such a structure. From Figure 11(a) and (b) taken from the locations with severe cyclic deformation (at the crack-initiation site) and moderate deformation (adjacent to the crack-initiation site), respectively, it can be found that more nanotwins were induced during cyclic loading before crack initiation. In particular, two sets of nanotwins were formed at the crack-initiation site as compared to one set of twinning seen in Figure 11(b) and (c). Figure 12 shows the further investigation on FIB specimens at the crack initiation site. Two different-orientation sets of dense nanotwins and a high density of tangled dislocation [Figure 12(a)] were clearly identified. Diffraction patterns [Figure 12(b)] with the zone axis [011] and three chosen spots [Figure 12(c)] for dark images indicate that the orientations or twinning direction of two-set twins are $[2\bar{1}1]$ (green color) [Figure 12(e)] and $[21\bar{1}]$ (blue color) [Figure 12(f)], respectively.

Overall, TEM observations suggest that the main crack-initiation mechanism: the severe cyclic stress action on the maximum-tensile-stress sites of specimens will induce more nanotwins and more sets of nanotwins before crack initiation. The increased density of nanotwins brings larger work hardening at the sites and gives higher resistance to crack initiation due to smaller stress concentration by providing more twin boundaries. As a result, the fatigue strength level is

expected to be higher. This trend basically explains the reasons why the present alloys have favorable and/or greater endurance limits, and fatigue ratios, as compared to steels and other conventional alloys. Further discussion on this trend will be presented later.

4. Statistical Modeling

4.1. Fatigue-life modeling

This section outlines the fatigue-life model with a random endurance limit. A commonly-used analytical representation of the S-N curves, considering the fatigue-endurance limit, is given by [35]

$$n = \begin{cases} c(s - \gamma)^{-d}, & s > \gamma, \\ \infty, & s \leq \gamma, \end{cases} \quad (3)$$

where n , s , and γ , are, respectively, the cycles to failure, the applied stress, and the fatigue-endurance limit, and c and d are positive material parameters. Taking the natural logarithm on Equation (3) results in

$$\log(n) = \begin{cases} \beta_0 + \beta_1 \log(s - \gamma), & s > \gamma, \\ \infty, & s \leq \gamma, \end{cases} \quad (4)$$

where $\beta_0 = \log(c)$ and $\beta_1 = -d$. To account for the statistical scatter in the observed fatigue-life data, a random error term, ε , is introduced to Equation (4) such that the fatigue life, n , is modeled by

$$\log(n) = \beta_0 + \beta_1 \log(s - \gamma) + \varepsilon, \quad s > \gamma. \quad (5)$$

It is assumed that the fatigue-endurance limit, γ , is a random variable following a certain probability distribution. Let $V = \log(\gamma)$, and suppose that V has a probability density function (pdf) given by

$$f_V(v; \mu_\gamma, \sigma_\gamma) = \frac{1}{\sigma_\gamma} \phi_V\left(\frac{v - \mu_\gamma}{\sigma_\gamma}\right), \quad (6)$$

where μ_γ and σ_γ are the location and scale parameters, respectively. If $\phi_V(\cdot)$ is the standardized normal pdf, i.e., $\phi_V(z) = \exp(-z^2/2)/\sqrt{2\pi}$, the endurance limit, γ , follows the lognormal distribution. If $\phi_V(\cdot)$ is the standardized smallest extreme value pdf, the endurance limit is, then, a Weibull random variable.

Let $x = \log(s)$, and $W = \log(n)$. Then conditioned on a fixed value of $V < x$, the conditional pdf of W is assumed to be given by

$$f_{W|V}(w; \beta_0, \beta_1, \sigma, x, v) = \frac{1}{\sigma} \phi_{W|V}\left(-\frac{w - [\beta_0 + \beta_1 \log(\exp(x) - \exp(v))]}{\sigma}\right), \quad (7)$$

with a location parameter, $\beta_0 + \beta_1 \log(\exp(x) - \exp(v))$ and scale parameter, σ . Again $\phi_{W|V}(\cdot)$ may be assumed to be the standardized normal or smallest extreme value of pdf. The marginal pdf of W is, then, given by

$$f_W(w; x, \theta) = \int_{-\infty}^x \frac{1}{\sigma \sigma_\gamma} \phi_{W|V}\left(\frac{w - [\beta_0 + \beta_1 \log(\exp(x) - \exp(v))]}{\sigma}\right) \cdot \phi_V\left(\frac{v - \mu_\gamma}{\sigma_\gamma}\right) dv, \quad (8)$$

where $\theta = (\beta_0, \beta_1, \sigma, \mu_\gamma, \sigma_\gamma)$. The marginal cumulative distribution function (cdf) of W is given by

$$F_W(w; x, \boldsymbol{\theta}) = \int_{-\infty}^x \frac{1}{\sigma_\gamma} \Phi_{W|V} \left(\frac{w - [\beta_0 + \beta_1 \log(\exp(x) - \exp(v))]}{\sigma} \right) \cdot \phi_V \left(\frac{v - \mu_\gamma}{\sigma_\gamma} \right) dv, \quad (9)$$

where $\Phi_{W|V}(\cdot)$ denote the conditional cdf of W , given V . The distribution functions determined by Equations (8) and (9) do not have closed forms. Numerical methods will be used to evaluate them.

Pascual and Meeker [24] showed that this random endurance-limit model possesses the characteristics that are usually observed in the fatigue-life data. Their results demonstrated that this model can adequately describe the curvature in the S-N relationship and the increase in scatter in the fatigue life as the stress decreases.

4.2. Computations and Results

This section presents the computational results of applying the random endurance limit model to analyze the S-N data of three conditions of $\text{Al}_{0.5}\text{CoCrCuFeNi}$ samples. The fatigue-life data of different conditions of $\text{Al}_{0.5}\text{CoCrCuFeNi}$ samples were analyzed separately, using the random endurance-limit model, as shown in Section 4.1. For each condition, we denote the fatigue-life data by (n_i, s_i, δ_i) , $i = 1, 2, \dots, m$, where m is the total number of observations in that condition, s_i is the applied stress amplitude for the i th sample, and the binary indicator, δ_i , specifies whether n_i is a failure or a censored (runout) observation. Herein $\delta_i = 1$, when n_i is the observed cycles to fatigue failure, and $\delta_i = 0$ when n_i is a censored observation, i.e., the fatigue test is terminated after n_i cycles but the sample has not failed. The unknown model parameters, $\boldsymbol{\theta}$, are estimated by the maximum likelihood method [36], which maximizes the likelihood function of the data given by

$$L(\boldsymbol{\theta}) = \prod_{i=1}^m [f_W(w_i; x_i, \boldsymbol{\theta})]^{\delta_i} [1 - F_W(w_i; x_i, \boldsymbol{\theta})]^{1-\delta_i}, \quad (10)$$

where $w_i = \log(n_i)$ and $x_i = \log(s_i)$, for $i = 1, 2, \dots, m$, and $f_W(w_i; x_i, \boldsymbol{\theta})$ and $F_W(w_i; x_i, \boldsymbol{\theta})$ are defined by Equations (8) and (9), respectively. Note that in the computation, n_i and s_i are scaled to have units of million cycles and 10^3 MPa, respectively, to improve numerical stability.

We assumed that $\varphi_V(\cdot)$ and $\varphi_{WW}(\cdot)$ are both standardized normal pdf. Table 2 lists the maximum likelihood estimates of the model parameters for the three conditions of samples. Using the maximum likelihood estimates of the model parameters, we can predict the S-N behavior by estimating the p -quantile fatigue life as a function of the stress. The p -quantile fatigue life at the stress level, s , denoted by $n_p(s)$, is defined as

$$\Pr(n \leq n_p(s); s, \boldsymbol{\theta}) = p = F_W(\log(n_p(s)); \log(s), \boldsymbol{\theta}). \quad (11)$$

when $p = 0.5$, $n_{0.5}(s)$ is the median fatigue life at the stress level, s , which can be used to describe the average stress-life behavior. Figure 5 shows the predicted $n_{0.5}(s)$ for the three conditions of HEA samples. As shown in Figure 5, the random endurance limit model can reasonably capture the stress-life relationship for all three conditions.

The endurance limit is of particular interest in this study. The endurance limit, γ , is assumed to follow the lognormal distribution with the location parameter, μ_γ , and scale parameter, σ_γ . Once the two parameters are estimated, we can compute some characteristics of the random endurance limit. The median endurance limit computed by $\exp(\mu_\gamma)$ and the standard deviation of the endurance limit computed by $\sqrt{\exp(\mu_r) \exp(\sigma_\gamma^2) (\exp(\sigma_\gamma^2) - 1)}$ can be used to measure the average endurance limit and the variability in the endurance limit, respectively [37].

Table 3 shows the median endurance limits and the standard deviations of the endurance limits for the three conditions of the samples. Because V , the logarithm of the endurance limit, is assumed to follow the pdf given by Equation (6), we can derive the pdf of the endurance limit itself, which is given by

$$f(\gamma; \mu_\gamma, \sigma_\gamma) = \frac{1}{\gamma \sigma_\gamma} \varphi_V \left(\frac{\log(\gamma) - \mu_\gamma}{\sigma_\gamma} \right). \quad (11)$$

Figure 13 depicts the $f(\gamma; \mu_\gamma, \sigma_\gamma)$ functions for the three conditions of HEA samples.

5. Discussion

5.1. Fatigue-strengthening mechanism in relationship to twinning behavior

A low density of existing nanotwins with tangled dislocations in the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ alloy was believed to be introduced during the cold-rolling process, as mentioned in Section 3.4. Therefore, elastic and/or plastic strains were accommodated in structures with existing nanotwins and tangled dislocations prior to fatigue-crack initiation. This process also occurs in the case of the supersaturated carbon in martensites, which increases the twinning to accommodate the strains in the Fe lattice due to carbon [38]. Then, fatigue cracks were initiated at heterogeneous nucleation sites, either preexistent defects (inclusions, gas pores, or local soft spots) [34], or twin boundaries, or both. Since more nanotwins were found at and adjacent to the crack initiation site where severe and moderate deformation occurred, respectively, as shown in Figure 11(a-b), nanotwinning is expected to be the preferred mode for deformation before crack initiation and during propagation, and, thus, be the principal mechanism for cyclic plastic deformation in the present alloy.

Nanotwins in the alloy were formed during the cold-rolling and cyclic loading. In particular, those nanotwins during cyclic loading brought strengthening for the alloy and, consequently, high fatigue strength (383 ± 71 MPa in terms of the stress amplitude by averaging seven data points at 10^7 cycles in Figure 5). The reasons for this trend are further considered. First, the formation of nanotwins results in continuous grain fragmentation because of the formation of new interfaces. The new twin boundaries act like high-angle grain boundaries, effectively reducing the dislocation mean free path, causing twinning-induced strengthening [15, 39]. The larger work-hardening due to the dynamic microstructure refinement is also referred to as the dynamic Hall–Petch effect [40].

Conventional deformation twins are usually in the same order of grain size and also found to be microtwins and nanotwins in some special alloys with the low stacking fault energy, such as Hadfield manganese steels and 316L steels [41]. Therefore, nanotwins in this HEA is unique. Nanotwins were also seen at cryogenic temperatures in another HEA, CoCrFeMnNi [39]. The formation of nanotwins is believed mainly due to two principal reasons: low stacking fault energy (SFE) and high strength, which has been discussed for the present alloys [15]. The low SFE could reduce the critical stress needed to nucleate twins, and, thus, preferably promotes the nucleation of nanotwins. The low SFE is attributable to the fact that the FCC matrix in HEAs contains an extremely large amount of solutes, which can effectively lowering the overall energy of stacking fault by Suzuki interactions [42, 43].

5.2. Reasons for extensive scatter

The variability in fatigue behavior is well known. This trend is true for cycles to failures in our case, but if the materials are stable, S-N should not have extensive scatter, as shown in Condition-3 of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA (Figure 5). The reason for the extensive scatter is that

in addition to three basic factors (stress variation, peak values, and cycles), there are other variables, such as stress concentration, corrosion, temperature, overload, metallurgical structures, and residual stresses, which can affect the propensity for fatigue.

Bending fatigue produces the stress gradient, which is tensile on the bottom surface, decreases with increasing the distance into the sample, and then become compressive. Fatigue cracks generally initiate on a tensile surface, and a certain size of shrinkage pores acting as stress risers decrease the time to nucleation and, thus, degrade fatigue life. Dislocations usually play a major role in the fatigue crack initiation phase [34]. In the present study, since there might have stress risers such as shrinkage pores and preexisting cracks, due to the fabrication process on or near the surface. As shown in Figure 1, stress concentration at these defects will cause the local microscopic stress to exceed the yield strength (1,284 MPa), while the maximum stresses of the remaining area were still below the yield strength of the material. In this case, upon repeated fatigue loading, plastic deformation occurred locally in the alloy [44, 45]. Then, extensive scatter occurred depending if the specimens had shrinkage pores and preexisting cracks. After carefully examining cross sections of as-received specimens, the content of microcracks is much less in Condition-3, while porosity is barely seen in all three conditions (Figure 1). This is the principal reason for the reduced scatter in Condition-3.

Besides, some oxides were found, as shown in Figure 8c. They were formed during melting and heat treatment. If many oxides were located near the bottom surface in the specimen, the tensile stresses were more easily triggering cracks than the specimens without or having less oxides. The cycles to failure of the former would be much lesser than the latter, resulting in extensive scatter [2].

5.3. Fatigue-life modeling

As shown in Table 2, the maximum likelihood estimates of the model parameters differ substantially among the three conditions of samples, indicating that the fabrication process and the purity level of the raw materials may have significant and complex effects on the fatigue behavior of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEAs. All the β_1 estimates are negative, implying that as the stress increases, the fatigue life, on average, decreases. No obvious trend was discovered concerning how improving the fabrication process and/or the raw materials affects the parameters, β_0 , β_1 , or σ . However, as shown in Table 2 and Table 3 and Figure 13, improving the fabrication process and/or the raw materials clearly has positive effects on the fatigue-endurance limits. Conditions-2 and 3 have comparable median endurance limits, and they are higher than the median endurance limit of Condition-1. Moreover, the standard deviations show a clear decreasing trend from Condition-1 to Condition-3, indicating a reduction of variability in the fatigue-endurance limit from Condition-1 to Condition-3. Figure 13 also conveys the same information. In Figure 13, the endurance limits corresponding to the peaks of the $f(\gamma; \mu_\gamma, \sigma_\gamma)$ functions for Conditions 2 and 3 are almost identical and higher than that of Condition-1. The $f(\gamma; \mu_\gamma, \sigma_\gamma)$ function of Condition-3 has the least variability; while the $f(\gamma; \mu_\gamma, \sigma_\gamma)$ function of Condition-1 has the most variability.

The defects in the HEA samples, therefore, may have a significant effect on the fatigue-endurance limits (Figure 1 and **Figure 7**). A reduction of the defects may result in improved fatigue-endurance limits of HEAs. Because the median endurance limit measures the average endurance limit of the sample, it may be correlated with the average number of defects in the test samples. On the other hand, the standard deviation measures the dispersion in the endurance limits among different samples within the same condition, and it may be correlated to the

homogeneity of defects in the cast material. Using an improved fabrication process and high-purity raw materials may reduce the average number of defects as well as enhance the uniformity of defect distribution in the material. This trend results in less scattering of fatigue data in Condition-3 than Condition-1 and 2 (Figure 5 and Figure 13, and Table 3). Because the variation in endurance limit has a pronounced effect on the scatter in fatigue life, especially at low stress amplitudes, the effect of defects on the fatigue-endurance limits may partially explain the scatter in the fatigue-life data.

5.4. Comparisons with conventional fatigue-resistant materials

Figure 14a presents three conditions of the HEAs with a lower-bound endurance limit comparable with the 15-5PH stainless steel and Ti-6Al-4V, and an upper bound only surpassed by some bulk-metallic glasses (BMGs). Figure 15a shows the same trend that the HEAs have relative high fatigue-endurance limits, compared with conventional structural counterparts. Such surprisingly high values of the fatigue-endurance limit are attributed to nanotwinning and the extremely-high value of the ultimate tensile strength (UTS, 1,344 MPa [2]) of the two-phase FCC $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA. Note that in Figure 15a, increasing UTS generally increases fatigue-endurance limit. To better compare the fatigue performance of the HEAs with other structural counterparts with respect to their UTS, the fatigue ratios (defined as the ratio of the fatigue endurance limit, based on the stress amplitude, to the UTS) are used and shown in Figure 14b and Figure 15b. The lower bound of the fatigue ratios of the HEAs compares favorably with those of steels, Ti-alloys, and Ni-alloys. The upper bound of the fatigue ratio is significantly higher than that of other conventional structural counterparts and even BMGs (Figure 14b and Figure 15b), showing that the optimization of the fabrication process decreased crack initiation sites (e.g., microcracks) and, therefore, enhanced the fatigue performance. Three conditions of

HEAs lie outside the benchmarks established by conventional alloys and BMGs (Figure 14b and Figure 15b). The fatigue and yield strengths of some BMGs may be greater than HEAs because BMGs do not have crystal defects, such as grain boundaries and dislocations. As expected, HEAs usually have much greater ductility than BMGs [8]. Overall, HEAs have better combination of mechanical properties of both fatigue and yield strengths, compared to conventional structural counterparts.

It is well known that bending fatigue usually leads to an increased fatigue-endurance limit, as compared to tension fatigue (uniaxial cyclic loading), but a reduced endurance limit one in comparison to compression fatigue [46, 47]. The bending produces stresses, which are tensile at the surface, decrease with increasing the distance into the sample, and at the neutral axis become compressive. Therefore, in the bending-fatigue there is a stress gradient with respect to the sample thickness (or diameter), while in the tension-fatigue the stress is uniform throughout the sample thickness (or diameter). Thus, in tension-fatigue, all defects in the gauge diameter can initiate fatigue cracks and thus cause crack growth, but in the bending-fatigue test, initially one half of the sample is in compression. Discontinuities and stress raisers also tend to become more influential, and cracks tend to initiate more readily under tension loading, as compared to compression loading [47]. These trends generally result in fatigue-endurance limit in this order: tension-fatigue < bending-fatigue < compression-fatigue. However, this may not be always the case. In some instances, fatigue performance in cyclic bending is exacerbated compared to those under tension cyclic loading, especially as when the test volume in the four-point-bending-fatigue specimen is greater than that in the notched and tapered tension-fatigue mode [48]. A larger test volume potentially contains more defects and stress raisers to promote crack initiation, which decreases the fatigue-endurance limit [46, 48]. This is also similar to the fatigue in three-

point cyclic bending wherein fatigue performance is improved, as compared to that under four-point cyclic bending, because of the reduced volume test in the three-point bending cyclic loading [47]. Furthermore, the stress ratio (R), cyclic frequency, surface condition, and sample size can also influence the fatigue-endurance limit [32, 46, 47, 49].

Therefore, the performance of the HEAs (in bending fatigue), shown in Figure 14-16, may or may not present higher levels than their performance under tension fatigue, depending on certain fatigue parameters, such as test volume, stress ratio (R), surface condition, etc. This trend needs to be recognized in comparison with other materials, since some data on other alloys are derived from tension fatigue with different testing parameters. However, it must be noted that the current HEA materials exhibit a tensile ductility of the order of 7.6 % (see Table 4), which may not effectively accelerate crack initiation and propagation because they may be less sensitive to defects and stress raisers, in comparison to more brittle materials [46]. The stress amplitude is a standard parameter in the material fatigue considerations and is also used in the current study in order to provide a comparison between the HEAs and conventional structural counterparts with different specimen geometries and testing procedures. Therefore, considering the lack of literature on the tension fatigue behavior of the HEAs, the bending fatigue of the HEAs can still provide a compromise linking to the behavior of other materials under cyclic tension loading. Thus, a focus of continuing research will be placed on tension fatigue in order to provide a better comparison between the HEAs and their conventional structural counterparts.

6. Conclusions

The two-phase FCC $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA shows a very promising mechanical property with a high yield strength (1,284 MPa), decent fatigue endurance limit (383 ± 71 MPa), and

significant ductility (7.6 %) (Table 4). Based on the observation in this study, the following conclusions can be drawn.

- (1) The fatigue life decreases, when the applied stress amplitude increases, as is normally observed. The high-cycle fatigue tests of the Condition-3 samples (fabricated by high-purity elements) generally gives higher fatigue strength than Condition-1, and less scatter than Conditions 1 and 2 (fabricated by commercial-purity elements). The attributed reasons are due to preexisting microcracks, pores, and oxides.
- (2) The TEM observations suggest that nanotwinning is the main deformation mechanism before crack initiation and often during fatigue crack propagation. The main crack initiation mechanism is: the severe cyclic stress action on the maximum-tensile-stress sites of specimens will induce more nanotwins and more sets of nanotwins before crack initiation. The increased density of nanotwins brings larger work hardening at the sites and gives higher resistance to crack initiation due to smaller stress concentration by providing more twin boundaries.
- (3) Nanotwins formation during the fatigue process results in strengthening for this alloy, which gives greater endurance limits, and fatigue ratios, as compared to steels and other conventional alloys.
- (4) By employing a random endurance-limit fatigue-life model to explore the effects of defects on the fatigue-endurance limits of HEAs, the results suggest that using improved fabrication processes and/or high-purity raw materials enhance the fatigue behavior by reducing the preexisting microcracks in the test samples.

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Table 1. Summary of three conditions of the cold-rolled Al_{0.5}CoCrCuFeNi HEA.

Condition	Raw materials	Processing	Machining
1*	Commercial purity: > 99 %	Homogenized at 1,000 °C for 6 h, water quenched and then cold rolled.	Shrinkage pores and macrosegregation were remained in some portions.
2	Commercial purity: > 99 %	Homogenized at 1,000 °C for 6 h, water quenched and then cold rolled.	Shrinkage pores and macrosegregation were removed before cold rolling.
3	High purity: > 99.9 %	Homogenized at 1,000 °C for 6 h, water quenched and then cold rolled.	Shrinkage pores and macrosegregation were removed before cold rolling.

* used in the literature [2]

Table 2. Maximum likelihood estimates of the model parameters.

Parameter	Maximum likelihood estimate		
	Condition-1	Condition-2	Condition-3
β_0	-3.884	-5.981	-3.726
β_1	-1.111	-1.590	-0.643
σ	1.034	0.006	0.216
μ_γ	-1.176	-1.009	-1.033
σ_γ	0.373	0.170	0.053

Table 3. Characteristics of the endurance limit distribution (Unit: MPa).

	Median (MPa)	Standard deviation (MPa)
Condition-1	308.6	230.0
Condition-2	364.5	104.9
Condition-3	355.8	31.4

Table 4. Microstructures and mechanical properties of the wrought $\text{Al}_{0.5}\text{CoCrCuFeNi}$ two-phase HEA. The data for yield strength, ultimate tensile strength, and tensile strain come from Condition-1 [2]. The fatigue strength is in terms of the stress amplitude by averaging seven data points at 10^7 cycles in Figure 5, while the fatigue ratio is calculated by the fatigue strength over the ultimate tensile strength.

Material	Microstructure	Yield strength	Ultimate tensile strength	Tensile strain	Fatigue strength	Fatigue ratio
$\text{Al}_{0.5}\text{CoCrCuFeNi}$	FCC-1 and FCC-2	1,284 MPa	1,344 MPa	7.6 %	383 ± 71 MPa	0.29

Figure 1. (a) A macrograph showing the cross-sectional examination conducted in the as-received condition (before fatigue testing), and preexisting cracks in (b) Condition-2 and (c) Condition-3, detected by optical microscopy.

Figure 2. Macrograph (a) of a Condition-3 cold-rolled $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA and micrographs of three sides [(b) cold-rolled surface, (c) electric-discharge-machining (EDM) cut surface, and (d) cross section] with SEM-EDS elemental mapping of six principal elements, including Al (red), Co (green), Cr (blue), Cu (yellow), Fe (cyan), and Ni (magenta)

Figure 3. Bright-field TEM image (a) and dark-field TEM image (b) show that the Cu-rich phase (also called as FCC-2) has a nanostructure with a nano-grain size of 5 - 10 nm. The diffraction pattern (c) confirms that the phase is FCC.

Figure 4. Synchrotron X-ray line profiles of three $\text{Al}_{0.5}\text{CoCrCuFeNi}$ conditions showing the FCC structures with a lattice parameter of 0.362 ± 0.002 nm. The inserted synchrotron two-dimensional (2-D) diffraction pattern indicates a texture in the sample. The results of the Condition-1 from literature [2].

Figure 5. The S-N curves of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA under high-cycle four-point-bending fatigue with an R ratio of 0.1 in air at room temperature. Note that the arrow indicates a run-out without failure. Dashed lines show the modeling-predicted median fatigue life vs. stress amplitude relationship for the three conditions of HEAs.

Figure 6. Fracture morphologies of a Condition-3 $\text{Al}_{0.5}\text{CoCrCuFeNi}$ specimen failed at a stress amplitude of 495 MPa, R = 0.1, room temperature, in air, fatigue life of 97,715 cycles, and 10 Hz: (a) an overview, (b) a crack-initiation region with a mixed morphology of fatigue striations and

pores, (c) a crack-propagation region with fatigue striations and cleavages, and (d) a fast-fracture region with dimples

Figure 7. The tensile-stress surface of a Condition-3 $\text{Al}_{0.5}\text{CoCrCuFeNi}$ specimen failed at a stress amplitude of 495 MPa, $R = 0.1$, room temperature, in air, fatigue life of 97,715 cycles, and 10 Hz: (a) an overview of tensile-stress surface; (b) a high-magnification micrograph of (a) showing slip bands; (c) a high-magnification micrograph of (a) showing shrinkage pores; (d) a high magnification micrograph of (b); (e) a high-magnification micrograph of (c).

Figure 8. Crack initiation of a Condition-3 $\text{Al}_{0.5}\text{CoCrCuFeNi}$ specimen failed at a stress amplitude of 495 MPa, $R = 0.1$, room temperature, in air, fatigue life of 97,715 cycles, and 10 Hz: (a) an overview of crack initiation region; (b) a high-magnification micrograph of (a) showing slip bands; (c) a high-magnification micrograph of (a) showing extensive plastic deformation

Figure 9. High-magnification micrographs of a microcrack (a) in Figure 7a on the tensile-stress surface of $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA sample after four-point-bending fatigue: (b) a high-magnification micrograph of (a) showing a pore; and (c) a high-magnification micrograph of (a) showing microcrack.

Figure 10. SEM images with EDS elemental mapping of six principal elements, including Al (red), Co (green), Cr (blue), Cu (yellow), Fe (cyan), and Ni (magenta)

Figure 11. TEM-FIB images removed from a fatigue-failed specimen with a lifetime of 2,069,447 cycles at a stress amplitude of 540 MPa in Condition-1 at three different locations, including (a) at the crack-initiation site, (b) adjacent to the crack-initiation site, and (c) away from the crack-initiation site. Tangled dislocations and nanotwins coexist in the microstructure.

Figure 12. Two different-orientation sets of dense nanotwins with a high density of tangled dislocation (a) are clearly identified. Three sets of diffraction patterns with a zone axis of [011] (b), the matrix and two orientations of twins, are indicated. Three chosen spots (c) for dark field images are also indicated. Three dark-field images are the matrix in combination with the first set of twins (red color) (d), the first set of twins (green color) (e), and the second set of twins (blue color) (f).

Figure 13. Probability distributions of the endurance limits for the three conditions of HEAs

Figure 14. S–N curves comparing (a) the fatigue endurance limits at 10^7 cycles and (b) the fatigue ratios of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA, other conventional alloys, and BMGs. Colored dashed lines denote the endurance limit of each alloy [2].

Figure 15. Plots comparing (a) the endurance limits in terms of stress amplitude and (b) the fatigue ratios of the $\text{Al}_{0.5}\text{CoCrCuFeNi}$ HEA as a function of the ultimate tensile strength (UTS) of other structural materials and BMGs [2].

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

Figure 6.

Figure 7.

Figure 8.

Figure 9.

Figure 10.

Figure 11.

Figure 12.

Figure 13.

Figure 14.

Figure 15.