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# Influence of hydrogen on the low cycle fatigue behavior of the equiatomic CrMnFeCoNi high entropy alloy

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## ABSTRACT

True plastic strain-controlled low cycle fatigue (LCF) tests were performed on the CrMnFeCoNi high-entropy alloy in two conditions: thermally precharged with hydrogen (H-precharged) and non-charged. Serrated flow was observed during the first cycle in the H-precharged condition, likely due to hydrogen pinning mobile dislocations. This behavior is suppressed with further straining, most likely by the increase in dislocation density. Internal hydrogen increases the cyclic strength of this alloy at all plastic strain amplitudes by enhancing the effective component of the flow stresses. Furthermore, the evolution of back stresses during LCF testing at different strain amplitudes is not influenced by the presence of hydrogen, suggesting that the strengths of the dislocation structures evolve similarly in both conditions. However, transmission electron microscopy reveals that dislocation cell formation is similar in specimens with and without hydrogen at high amplitudes but is limited at low amplitudes in the presence of hydrogen. Primary fatigue crack(s) initiate earlier in the H-precharged than in the non-charged condition, and the presence of hydrogen at the concentration achieved in this study severely reduces the LCF lifetime of this alloy. Fracture surface observations reveal that, although plastic deformation processes are active in the H-precharged condition, the introduction of hydrogen leads to a transition from intragranular to intergranular failure.

## 1. Introduction

High entropy alloys (HEAs) represent an important design concept for metallic materials in which compositions toward the center of multi-component phase diagrams are employed for potentially improved properties [1–3]. One of the most extensively studied HEA compositions is the equimolar CrMnFeCoNi alloy, also known as the Cantor alloy [4]. It has been shown to possess excellent damage tolerance at low temperature [5–8], and early work suggested that this characteristic was preserved under exposure to low hydrogen concentrations, implying excellent resistance to hydrogen embrittlement. [9,10]. The CrMnFeCoNi alloy also displays good tensile strength and ductility at room temperature [7]. In addition, several studies of fatigue crack growth [11,12], high cycle fatigue [13–15] and low cycle fatigue [16–19] in the CrMnFeCoNi alloy at room temperature have suggested superior fatigue performance compared to that of conventional austenitic stainless steels.

This favorable combination of tensile and fatigue properties has motivated studies of this alloy's performance in extreme working environments, such as the transport and handling of gaseous hydrogen in the energy sector.

Numerous studies have examined the monotonic tensile ductility of the CrMnFeCoNi alloy across various hydrogen concentrations and testing strain rates. Luo et al. [20] reported that electrochemical charging to hydrogen concentrations between 8 and 33 wt ppm enhanced strength and ductility in this material by promoting nanotwin formation. They also stated that this improvement in ductility in the presence of hydrogen is unique to the CrMnFeCoNi alloy compared to most other conventional alloys at similar or lower hydrogen concentrations. Zhao et al. [21] conducted gaseous and electrochemical hydrogen charging of CrMnFeCoNi alloys to concentrations of 76.5 wt ppm and 45 wt ppm, respectively. Only a very small reduction in tensile ductility (~5 %) was observed in the gaseous charged condition

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compared to the non-charged condition with no significant change in strength, suggesting that the CrMnFeCoNi alloy may have favorable hydrogen embrittlement resistance compared to austenitic stainless steels. Zhou et al. [22] performed theoretical predictions of the ductile-to-brittle transition in different families of stainless steels and HEAs based on the concept that hydrogen induces changes in dislocation emission and blunting processes at crack tips. They showed that CrMnFeCoNi is less susceptible to hydrogen embrittlement than 304 stainless steel but more so than 316L, indicating that the compositional complexity of HEAs does not necessarily result in superior performance in hydrogen environments compared to austenitic stainless steels.

In contrast, several other studies have reported a severe hydrogen induced decrease in ductility and transition from intragranular to intergranular fracture in the CrMnFeCoNi alloy under monotonic tensile deformation [23–32]. While grain size and strain rate may impact ductility at room temperature, Ichii et al. [23] and Li et al. [33] concluded that hydrogen concentration is the most important factor that controls the improvement or deterioration of ductility, which may explain the apparent conflict in these results. An et al. [27] examined ‘hydrogen’ embrittlement in CrMnFeCoNi electrochemically charged with deuterium at concentrations between 45 and 275 wt ppm. They observed increased tensile strength and decreased ductility at high deuterium concentrations, with apparent brittle zones in the fracture surface. The authors attributed this behavior to a reduction in stacking fault energy, which accelerated dislocation accumulation and twin growth, increasing local stresses. The work of Nygren et al. [25] and Bertsch et al. [24] demonstrated that hydrogen enhances the constraint of localized deformation between neighboring grains and weakens grain boundaries, thereby leading to a reduction in monotonic ductility and promoting intergranular failure. Thus, a consistent picture appears to be emerging regarding the influence of hydrogen on monotonic deformation of CrMnFeCoNi, although the underlying mechanisms are still not fully described.

Hydrogen energy systems are often subjected to repeated pressurization and depressurization cycles; hence, good fatigue resistance is necessary for materials in such applications. The importance of cyclic loading notwithstanding, there is only a single published paper on the influence of hydrogen on fatigue of CrMnFeCoNi [34]. In that work, Koyama et al. evaluated fatigue crack growth resistance by testing compact specimens. They showed a threefold increase in fatigue crack growth rate in specimens thermally precharged with hydrogen (113 wt ppm) compared to the non-charged counterparts and suggested intergranular crack growth to be controlled by dislocation emission from grain boundaries at crack tips. However, there is presently no information in the current literature regarding the influence of hydrogen on cyclic deformation under stress control (high cycle fatigue or HCF) or strain control (low cycle fatigue or LCF) testing of CrMnFeCoNi. Because plastic strain is known to correlate with damage accumulation and failure under cyclic loading [35], strain-controlled LCF tests in particular could help elucidate the influence of hydrogen on these processes and reveal any similarities or differences of cyclic deformation compared to monotonic tensile deformation. Therefore, the goal of the current study is to explore the fatigue behavior and mechanisms of cyclic deformation in the CrMnFeCoNi alloy, with and without internal hydrogen (H-precharged and non-charged, respectively). True plastic strain-controlled tests, although significantly more challenging to conduct than total strain-controlled tests, were performed to better assess the influence of internal hydrogen on low cycle fatigue mechanisms. Of particular importance is the need to determine whether the emerging understanding of the role of hydrogen in monotonic deformation also applies to cyclic deformation of CrMnFeCoNi.

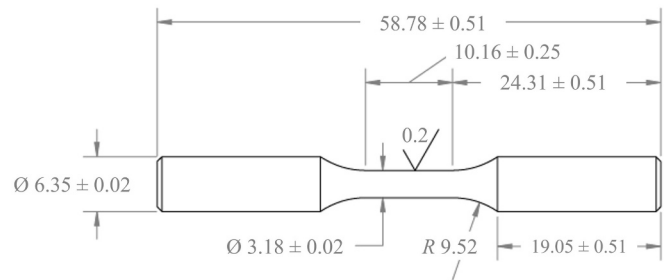


Fig. 1. LCF specimen geometry and dimensions in mm.

## 2. Materials and methods

### 2.1. Material

The CrMnFeCoNi alloy investigated in this work was arc melted, drop cast and homogenized in vacuum at 1473 K for 24 h, using procedures similar to those described by Otto et al. [7], including the addition of a small amount of excess Mn to account for its evaporation during arc melting. Unlike that study, where rectangular cross-section castings were rolled to sheet, here cylindrical specimens were extruded to rods from which cylindrical LCF specimens were machined. After casting and homogenization, the 25.4 mm diameter × 127 mm long cylindrical ingots were inserted into tight-fitting holes drilled in stainless steel cans, their back faces welded shut with stainless steel plates, and the combination extruded at 1273 K at an (area) extrusion ratio of 9:1, resulting in an extrusion ratio for the CrMnFeCoNi alloy in the range of 6:1 to 7:1. Three extruded rods were used in this study.

### 2.2. Specimens

Cylindrical LCF specimens with a 3.2 mm gage diameter and a 10.2 mm gage length were machined from the extruded rods using a CNC lathe following the design specified by ASTM E606 [36] (Fig. 1). The gage sections were mechanically ground using SiC paper, then fine polished with diamond compound to 0.25  $\mu\text{m}$ . Each polishing step started along the circumferential direction, and the resulting marks on the surface were eliminated by repeating the same grade of SiC or diamond along the longitudinal direction. The polished specimens were vacuum annealed at 1173 K for 1 h, followed by furnace cooling. The resulting microstructure for one rod is shown in Fig. 2 and consists of recrystallized equiaxed grains with an average grain size of 31  $\mu\text{m}$  and a relatively high density of annealing twins. The other two rods exhibited similar features, but with slightly larger grains (average value of  $\sim 36 \mu\text{m}$ ). Additionally, Cr- and Mn-rich particles (black spots in Fig. 2) in low concentration and approximately 4  $\mu\text{m}$  in diameter were identified using energy dispersive x-ray spectroscopy (EDS). These particles are believed to be oxides (or cavities from oxides removed during polishing) as previously seen in this alloy when processed in the same manner [37].

### 2.3. Hydrogen precharging

Hydrogen-precharging of annealed CrMnFeCoNi specimens was conducted in a chamber containing gaseous hydrogen (research grade, 99.9999 % purity) at a pressure of 138 MPa and a temperature of 573 K for 13 days. The final hydrogen concentration in the precharged (H-precharged) specimens of 87 wt ppm was measured through inert gas fusion-infrared absorbance. Specimens were stored in a freezer at  $-18^\circ\text{C}$  (255 K) after charging for times of one week to a maximum of two months until they were tested. The non-charged and H-precharged conditions are referred to here as HEA and H-HEA, respectively. While other studies that used different precharging conditions suggest that this alloy is able to accommodate a higher concentration of hydrogen than conventional austenitic stainless steels [21,24], that was not the case in

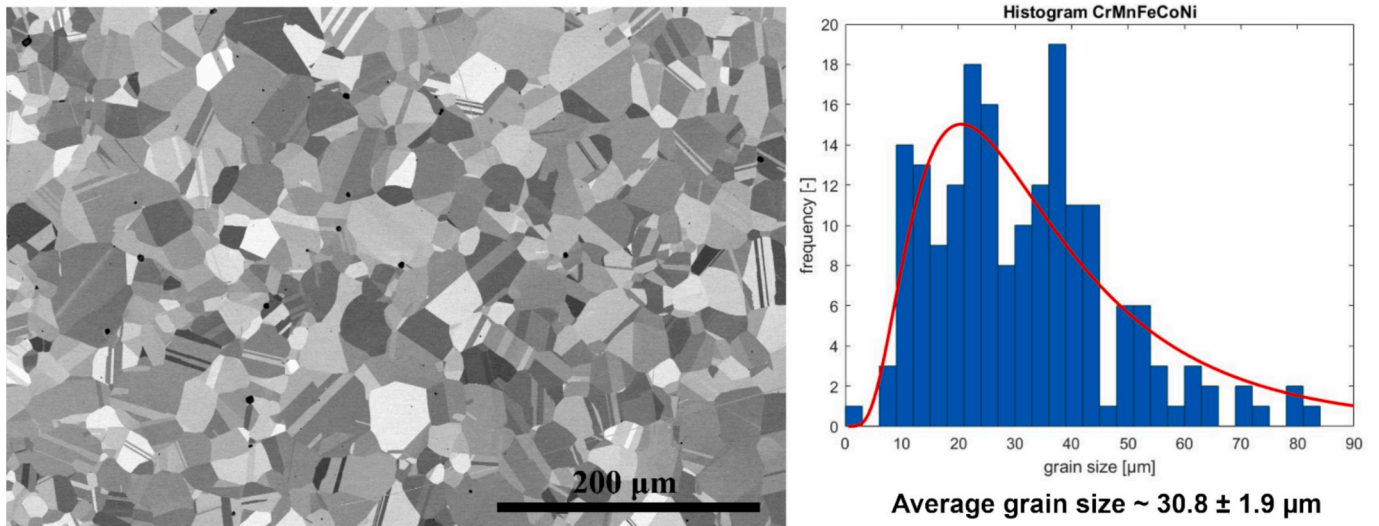


Fig. 2. Backscattered electron micrograph of the initial grain morphology obtained after recrystallization and the resulting grain size distribution.

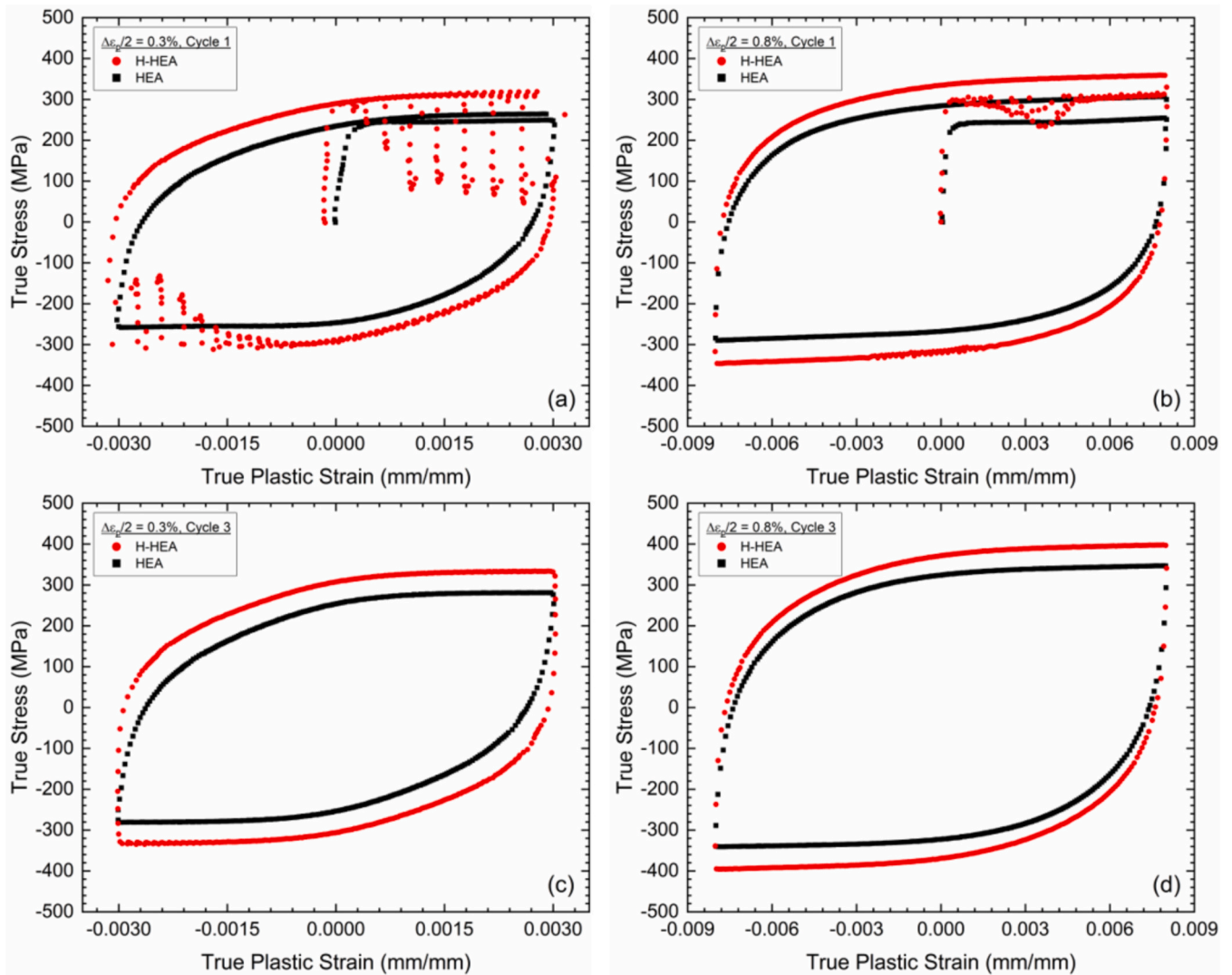
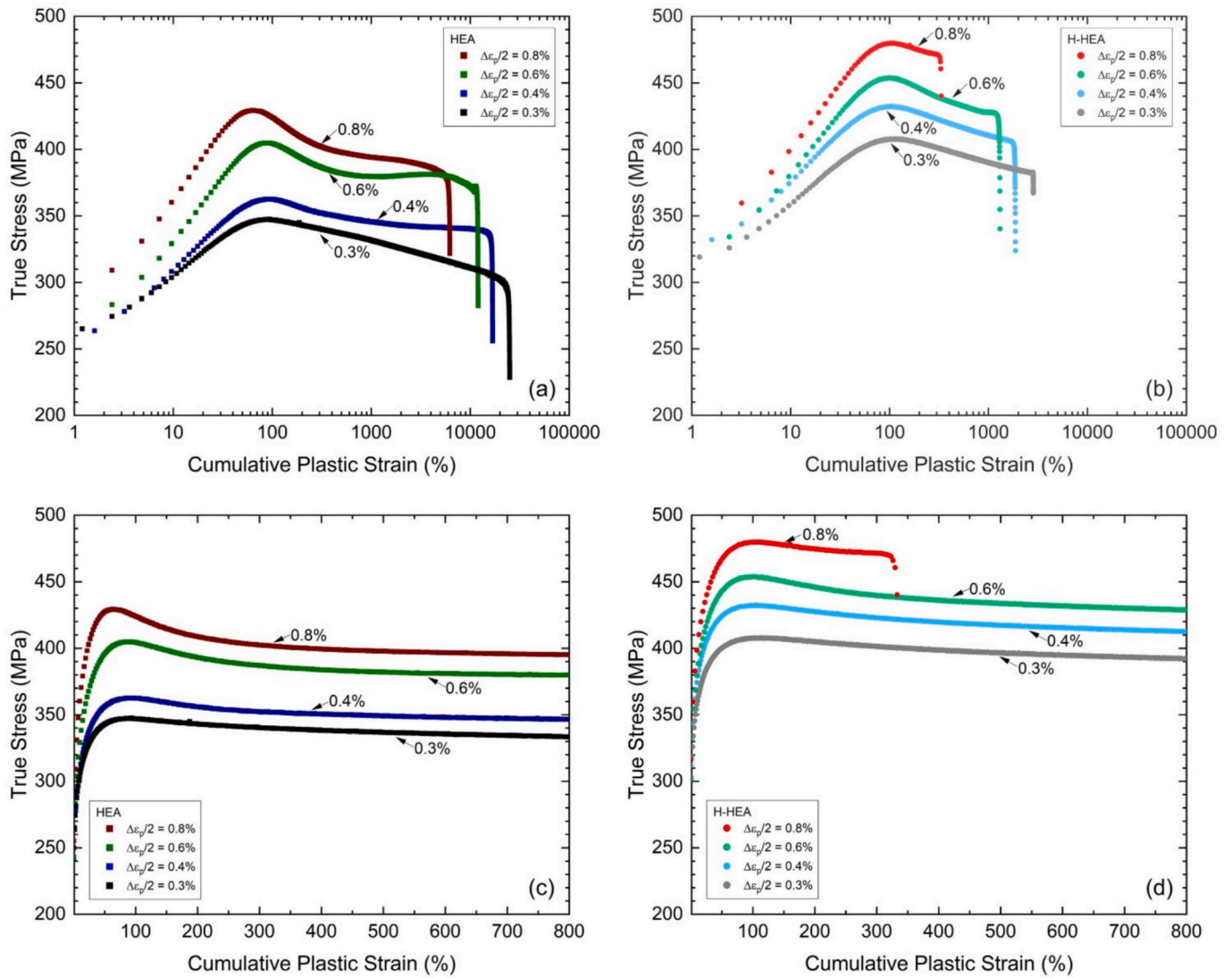


Fig. 3. True stress-true plastic strain hysteresis loops for the HEA and H-HEA material conditions at the (a, b) first and (c, d) third cycles obtained during LCF tests at (a, c) 0.3 % and (b, d) 0.8 % plastic strain amplitudes.



**Fig. 4.** Cyclic tension stress response curves for the HEA (a, c) and H-HEA (b, d) conditions on (a, b) linear-log axes and (c, d) truncated data on linear-linear axes. Cumulative plastic strain equals  $4 \times \Delta\epsilon_p/2 \times N$ . Different colors represent different plastic strain amplitudes. Only one test for each condition is shown for clarity.

this study. The hydrogen concentration in strain-hardened 316L stainless steel after gaseous precharging in the same thermal environment as described above was 155 wt ppm [35], almost twice the value measured in the annealed CrMnFeCoNi alloy. In addition to these initial hydrogen concentrations, it is important to consider whether the concentration changes before and during the test. Previous work on Type 316L stainless steel has shown that there is no significant loss of hydrogen under similar low cycle fatigue testing conditions [35,38]. Since the diffusivities of hydrogen in CrMnFeCoNi and Type 316L stainless steel are similar [39], we expect a similar result for the HEA. Furthermore, studies of hydrogen desorption from CrMnFeCoNi [23,25,28] have demonstrated that it occurs at very low rates at the temperature at which the specimens were stored and that even at room temperature there would not be significant hydrogen loss in 8–10 days [25]. Since LCF tests of the CrMnFeCoNi were generally short (approximately 1 h of setup and 2 h of testing), hydrogen loss in this work should be insignificant.

#### 2.4. Low cycle fatigue testing

Fully-reversed true plastic strain-controlled LCF tests (ratio of minimum to maximum plastic strain  $R = -1$ ) were conducted on an MTS 810 servo-hydraulic testing machine running MTS TestStar and

Testware-SX software codes adapted from Walley et al. [40]. The control variable, true plastic strain ( $\epsilon_p$ ), was calculated in real time using the measured load ( $L$ ), total strain ( $\epsilon_t$ ) determined from an extensometer attached to the specimen, initial cross-sectional area ( $A_0$ ) and elastic modulus ( $E$ ):

$$\epsilon_p = \ln(1 + \epsilon_t) - (1 + \epsilon_t) \times \frac{L}{A_0 \times E} \quad (1)$$

LCF tests were performed at room temperature and a constant plastic strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$ , probing plastic strain amplitudes (defined as  $\Delta\epsilon_p/2$ ) of 0.3, 0.4, 0.6 and 0.8 %. The effective elastic moduli used for plastic strain calculations were initially derived from tensile tests conducted at a total strain rate of  $2 \times 10^{-4} \text{ s}^{-1}$ , then refined to ensure that the loading and unloading segments of the stress-plastic strain hysteresis loops were nearly vertical. To increase statistical significance, two tests were conducted at each plastic strain amplitude. Failure was defined here as the number of cycles necessary to fracture the specimens or to induce a 20 % drop in peak tension stress compared to peak compression stress, whichever was reached first. The collected stress-plastic strain data were used to calculate Bauschinger stresses considering the reverse yield strength at a true plastic strain offset of 0.1 %. In addition, a decrease of the tension elastic unloading slope by 1 % was used to

estimate the fatigue crack initiation lifetime. Details of the test configuration and data analysis procedures (including the Bauschinger stress calculations) are reported elsewhere [35]. In this work, the extensometer knife edges were machined to include a radius equal to that of the specimen and were then glued to the specimen using cyanoacrylate adhesive to ensure that they remained in contact and did not serve as crack nucleation sites in this initially soft material.

### 2.5. Microscopic characterization

To evaluate the effect of hydrogen on the crack path and fracture mode, fracture surfaces were characterized using a Thermo Fisher Quattro S scanning electron microscope (SEM) operating at an accelerating voltage of 10–15 kV in the secondary electron imaging mode. Additionally, dislocation arrangements were investigated after fatigue failure by transmission electron microscopy (TEM). Several thin discs were sliced from the gauge sections at locations 2–3 mm from the fracture surfaces of the failed samples normal to the loading direction after fatigue testing at plastic strain amplitudes of 0.3 % and 0.8 % using a low-speed saw. The discs were then thinned by mechanical polishing to  $\sim 100 \mu\text{m}$  followed by electropolishing in a 10 vol% sulfuric acid in methanol electrolyte at  $-30^\circ\text{C}$  using a Fischione Model 120 twin jet electropolisher operating at a DC current of 80–100 mA. TEM imaging was performed on a JEM-2100F-AC TEM at 200 kV under two-beam conditions near the [110] and [112] zone axes. Diffraction contrast imaging-scanning transmission electron microscopy (DCI-STEM) was performed on slightly thicker TEM samples using an FEI Tecnai F20 UT.

## 3. Results and discussion

### 3.1. Stress response

Fig. 3 shows representative hysteresis loops obtained for the CrMnFeCoNi alloy with and without hydrogen (H-HEA and HEA, respectively) at plastic strain amplitudes of (a) 0.3 % and (b) 0.8 % during the first cycle. These loops illustrate typical changes in stress as the plastic strain increases within a cycle. For the non-charged HEA, loops are well formed and symmetric along the plastic strain axis at all plastic strain amplitudes. In contrast, the stress-plastic strain response of H-HEA is characterized by serrated flow during the initial cycle. The serrations observed in these results are significantly larger than those observed in conventional displacement-controlled tensile tests. For example, Fu et al. [41] detected serrated flow in CrMnFeCoNi at elevated temperatures, which showed stress fluctuations of only a few percent of the applied stress. In true plastic strain-control in a servo-hydraulic testing machine that has been tuned to be highly responsive in order to conduct the LCF tests described in this work, the stress fluctuations are especially dramatic as strain bursts and the corresponding load drops affect the real time calculation of true plastic strain (represented in Eq. (1)). As the control system attempts to maintain the constant true plastic strain rate in the presence of these rapid changes, the system can border on instability. Since the stress fluctuations depend on the machine dynamics, it is not possible to directly ascribe any physical significance to their magnitude in this case, but their presence reflects the influence of hydrogen on dislocation motion in the first cycle.

Serrated flow has been reported to take place during monotonic [41–46] and cyclic [47] deformation of medium and high entropy alloys at high temperatures. This phenomenon is often observed in ranges of temperature and strain rate in which mobile dislocations are pinned by solute atoms due to dynamic strain aging [48–50]. When the dislocations break away, the solute mobilities are high enough to allow them to catch up with and re-pin the dislocations in solute strengthened materials. The observation of serrations in the hydrogen-precharged condition tested at room temperature in the present study, but not in the non-charged condition, suggests that this behavior likely arises from periodic dislocation pinning by hydrogen atoms in the H-HEA condition. Serrated

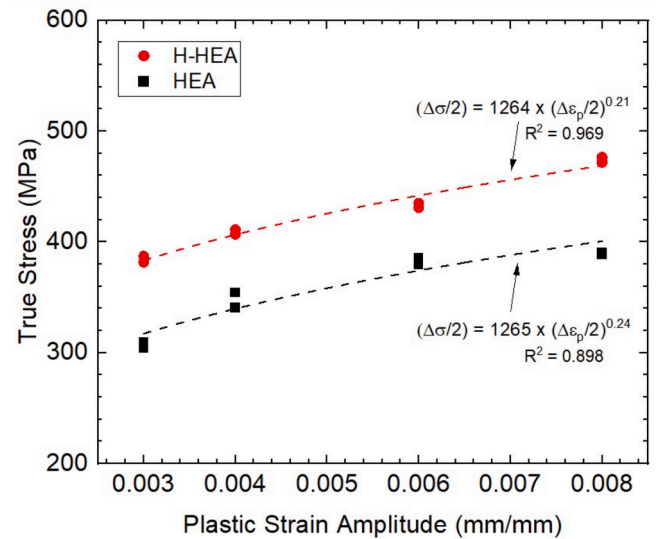
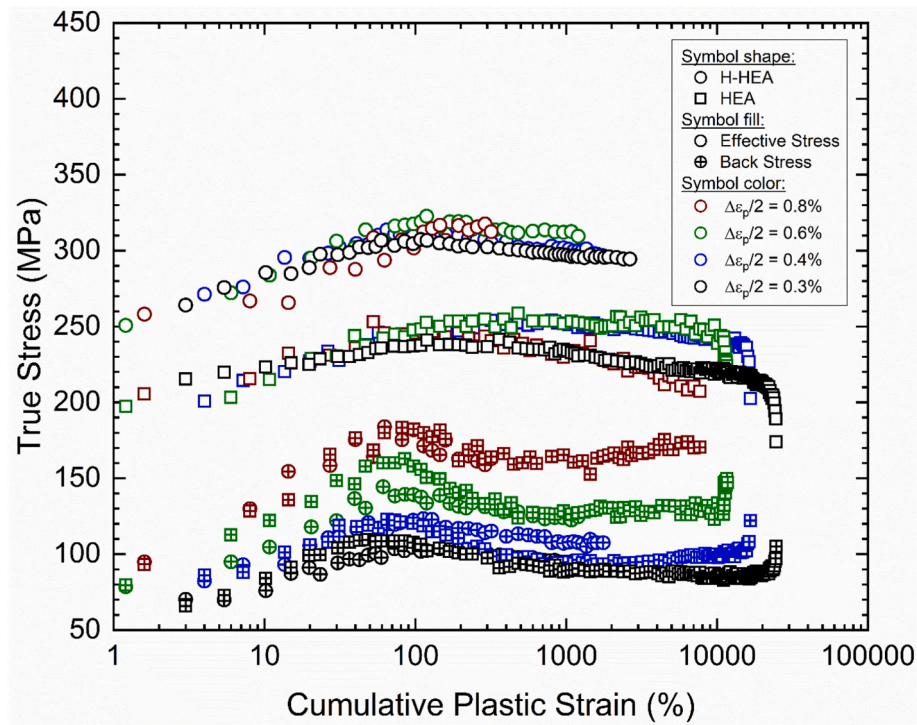


Fig. 5. Cyclic stress–strain curve for the CrMnFeCoNi alloy in the non-charged (HEA) and H-precharged (H-HEA) conditions.

flow was present at all plastic strain amplitudes in the forward and reverse directions, although less prominent at 0.8 % (Fig. 3(b)) due to the large driving forces for dislocation motion at this amplitude. Furthermore, the serrations were present only during the first cycle, as shown by their absence in the hysteresis loops for the third cycle in Fig. 3 (c–d); this change is most likely a consequence of increased total dislocation density as the material hardens during the early stages of cyclic deformation, as demonstrated by Shams et al. in the absence of hydrogen [17]. Thus, it appears that in the H-HEA condition the available hydrogen solutes segregate to all dislocations – mobile and immobile – and the concentration is no longer sufficient to pin all mobile dislocations after the first cycle.

Fig. 4(a, b) shows the cyclic tension stress response curves for specimens in the HEA and H-HEA conditions tested at plastic strain amplitudes between 0.3 % and 0.8 %. For both material conditions, there is an initial cyclic hardening during the first several cycles, commonly attributed to dislocation multiplication in the annealed microstructure [51]. Hydrogen increases the strength of this alloy at all plastic strain amplitudes; peak stresses are 10–20 % larger in H-HEA compared to HEA. This strengthening effect was also observed in previous work on hydrogen-precharged 316L stainless steel, in which hydrogen increased the yield strength by approximately 13 % at a concentration of 155 wt ppm [35]. After reaching a peak value of cyclic stress, cyclic softening occurs in the HEA condition and the extent of softening decreases as amplitude decreases. This cyclic softening, which is more evident in Fig. 4(c, d) where the cumulative strain data are truncated and plotted on a linear scale, is followed by a region of stress saturation. The H-HEA condition exhibited similar behavior, although the rate and extent of cyclic softening are less than those in the HEA condition at high amplitudes. The softening and saturation stages encompass most of the lifetimes for both conditions.

Lu et al. [16] and Shams et al. [17] reported similar cyclic deformation behavior in non-charged, annealed CrMnFeCoNi tested under total strain control. According to these authors, the initial cyclic hardening was due to dislocation multiplication and solute hardening, with planar slip as the dominant mode as evidenced by slip bands and stacking faults. The softening stage was characterized by a transition to wavy slip, during which dislocations rearranged into more stable substructures (such as ill-defined veins and walls observed via TEM), and screw dislocations were mutually annihilated within the depleted channels. The subsequent stress saturation regime was interpreted by these authors as a dynamic equilibrium between dislocation

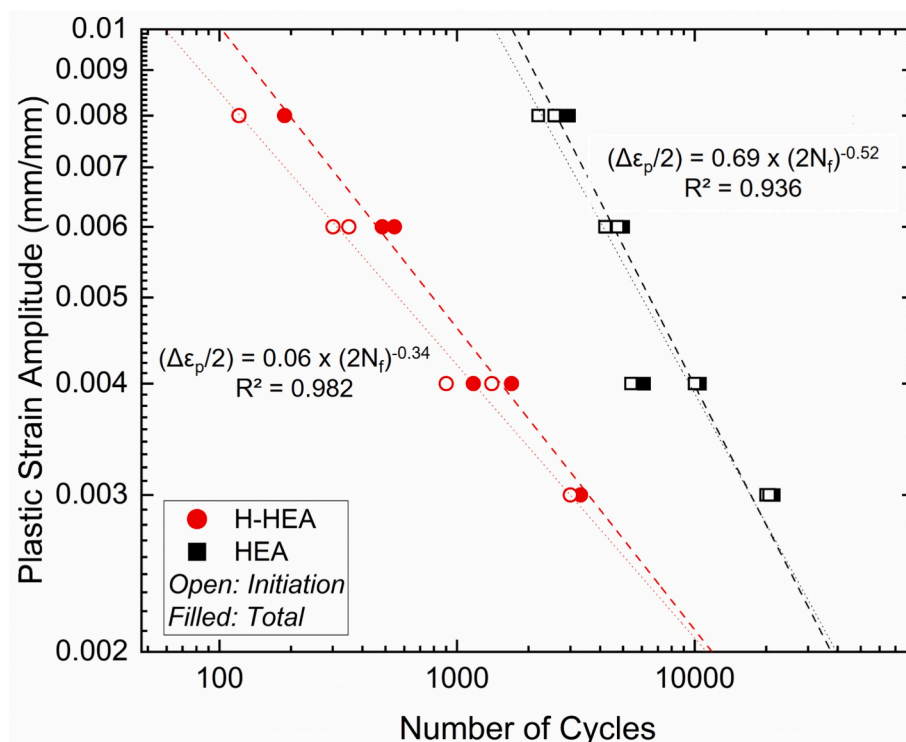


**Fig. 6.** Bauschinger stresses as a function of cumulative plastic strain for the HEA (squares) and H-HEA (circles) conditions. Different colors represent different plastic strain amplitudes as in Fig. 4. Only one test for each condition is shown for clarity.

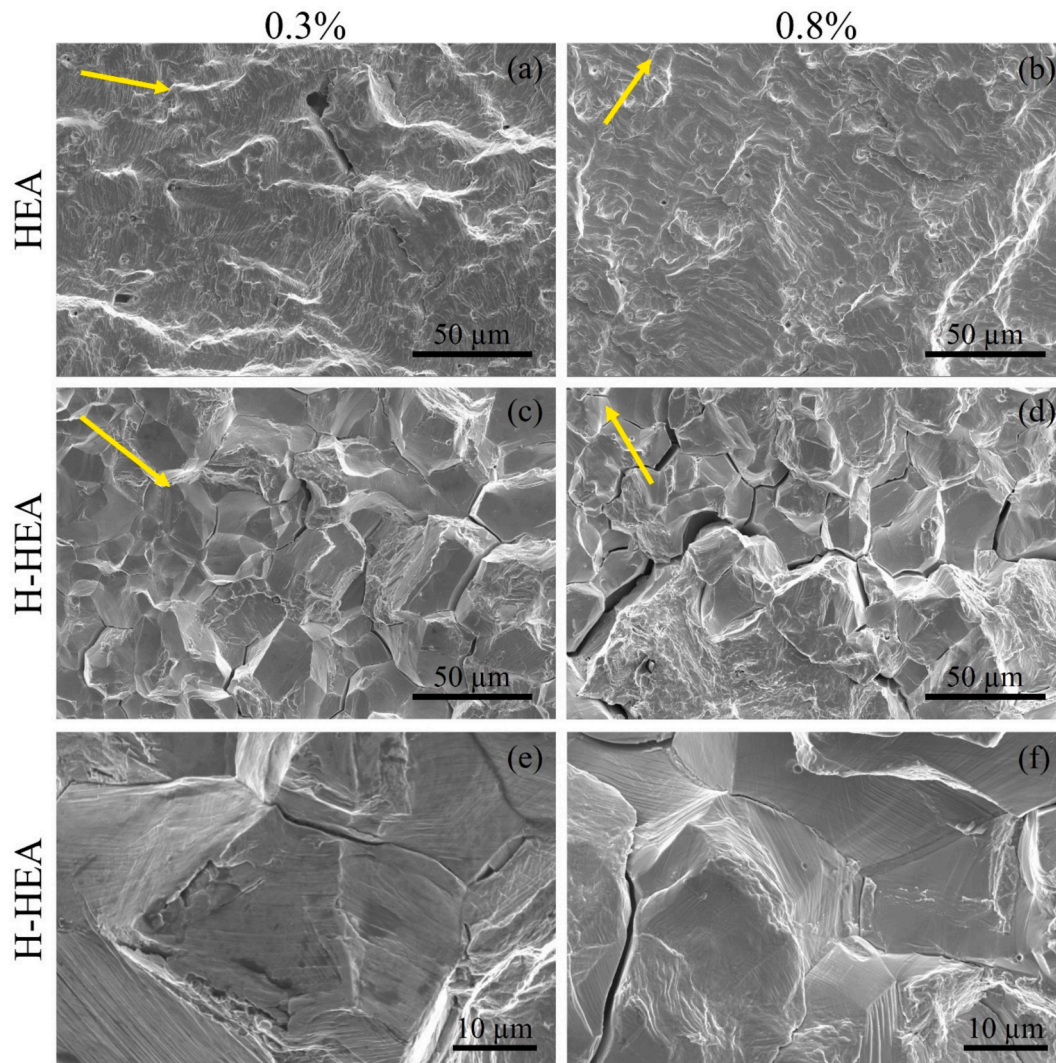
multiplication and annihilation, supported by TEM observations of well-developed dislocation structures and deformation twins.

The cyclic stress strain curves for the HEA material in the hydrogen-precharged and non-charged conditions were created by determining the stresses at half-life at each of the imposed plastic strain amplitudes, as shown in Fig. 5. The dashed lines represent the fit to the standard

cyclic form of the empirical Hollomon relationship [52]. The fitting parameters are shown near the respective data, and the values for the HEA condition fall within those obtained by Lu et al. [16] for coarser and finer grain sizes. The addition of hydrogen increases the cyclic strength through an offset over the range of amplitudes studied as illustrated in Fig. 4. That is, the extent of hardening that occurs as plastic strain



**Fig. 7.** Strain-life plots containing initiation (open symbols) and total (filled symbols) lifetime data for HEA and H-HEA material conditions.



**Fig. 8.** SEM micrographs of the fracture surfaces in (a, b) HEA and (c-f) H-HEA after LCF testing at (a, c, e) 0.3 % and (b, d, f) 0.8 % plastic strain amplitudes. Arrows indicate the direction of crack propagation.

amplitude increases over the range that was tested is nearly unaffected by the presence of hydrogen in this alloy.

To better understand the different contributions to hardening, the effective ( $\sigma_e$ ) and back ( $\sigma_b$ ) stress components of the peak tension stresses were determined as a function of cumulative plastic strain for both material conditions. These two components were identified using the standard Bauschinger analysis expressed in Eq. (2) [53,54]:

$$\sigma_e = \frac{\sigma_{\max} - \sigma_o}{2}; \sigma_b = \frac{\sigma_o - \sigma_{\min}}{2} \quad (2)$$

where  $\sigma_o$  is the reverse yield stress determined at a true plastic strain offset of 0.1 % [55,56], and  $\sigma_{\max}$  and  $\sigma_{\min}$  represent the maximum and minimum peak stresses in each hysteresis loop, respectively. Fig. 6 demonstrates that effective stresses are greater than back stresses in both material conditions and at all strain amplitudes. This result differs from those reported by Lu et al. [56] at similar plastic strain amplitudes, who observed that the back stresses were greater than the effective stresses in a similar non-charged alloy. This discrepancy is most likely due to differences in testing mode (total strain control in their study versus plastic strain control in the present study), which influences the evolution of these quantities as well as the magnitudes of reverse yield strength. The high values of peak stresses observed in both material conditions at high strain amplitudes are attributed primarily to the increase in back stresses with plastic strain amplitude, consistent with findings of Lu et al. [56].

In other words, changes in plastic strain amplitude primarily affect interactions between mobile dislocations and long-range obstacles, such as dislocation cell walls. However, there are no differences in back stresses between the HEA and H-HEA conditions at a given plastic strain amplitude, revealing that hydrogen does not affect these long-range dislocation interactions.

In addition, this analysis demonstrates that the hydrogen-induced strengthening shown in Figs. 4 and 5 is represented by the increasing magnitude of the effective component of the flow stress for the H-HEA condition. In other words, hydrogen influences short-range interactions between mobile dislocations and solutes, providing additional glide resistance (decreased mobility) reflected in the effective stresses. The serrated flow due to hydrogen pinning observed in H-HEA specimens (Fig. 3) is an example of hydrogen increasing the effective stresses in this alloy. The difference in effective stresses between HEA and H-HEA conditions at different plastic strain amplitudes remains approximately constant at about 50 MPa as illustrated in Fig. 6, indicating that the effect of hydrogen on the evolution of the effective stresses is independent of the plastic strain amplitude and its contribution to strengthening is the same for all plastic strain amplitudes.

### 3.2. Total and crack initiation lifetimes

In addition to strengthening the CrMnFeCoNi alloy, hydrogen

severely degrades its LCF lifetime at all plastic strain amplitudes, as observed from the strain-life plots for the representative HEA and H-HEA specimens shown in Fig. 7. The presence of hydrogen decreases the number of cycles to failure by factors of approximately 5 and 15 at plastic strain amplitudes of 0.3 % and 0.8 %, respectively. The strain-life data were fitted to a power law in the form of the Coffin-Manson equation [57,58] shown in Eq. (3) and represented by the dashed lines in Fig. 7.

$$\frac{\Delta \epsilon_p}{2} = \epsilon'_f (2N_f)^c \quad (3)$$

The values of the fatigue ductility coefficient ( $\epsilon'_f$ ) and fatigue ductility exponent ( $c$ ) for each material condition are also given in Fig. 7 near the respective data. The fitting parameters obtained in this study for the CrMnFeCoNi alloy are consistent with those reported by Shams et al. [17]. Since  $\epsilon'_f$  is often approximately equal to the monotonic true fracture ductility [51], these data imply that hydrogen greatly reduces the monotonic tensile ductility of CrMnFeCoNi. This observation is consistent with the results of the tensile behavior of H-precharged CrMnFeCoNi alloy showing a reduction in monotonic strain to failure of 35–60 % when similar or larger hydrogen concentrations are introduced into the alloy [23–26]. In contrast, lower concentrations of hydrogen have led to a very small decrease (~5 %) or even a slight increase (~1% to 6 %) in ductility when compared to the non-charged condition [20,21]. The present findings help confirm that the influence of hydrogen on the mechanical response of CrMnFeCoNi is strongly dependent on its concentration. We also note that the different slopes of the fatigue-life plots (Fig. 7) suggest that the low cycle data may converge at very low plastic strain amplitude.

Estimated fatigue crack initiation clearly occurs much earlier in the life of the H-HEA condition than the HEA condition, as shown in Fig. 7. Across the range of tested plastic strain amplitudes, crack initiation represents approximately 93 % and 73 % of total lifetime in HEA and H-HEA, respectively. The observed earlier crack initiation in the presence of internal hydrogen is consistent with the loss of ability to accommodate plastic deformation as indicated by the loss of ductility in tensile testing. The crack growth component of fatigue life is the difference between the total cycles to failure and the cycles to initiate a crack. Although the fraction of lifetime corresponding to crack growth is larger in the H-HEA condition, the extent of hydrogen-assisted fatigue crack growth is as much as 10 times greater than without hydrogen. For example, the crack growth component of fatigue life at strain amplitude of 0.8 % is less than 100 cycles for the H-HEA condition compared to nearly 1,000 cycles for the HEA condition at the same test amplitude (Fig. 7).

### 3.3. Fracture surfaces

SEM characterization of the fracture surfaces was conducted to probe the changes in fracture mode and crack path induced by the presence of internal hydrogen; these surfaces are depicted in Fig. 8. All images were taken near the center of the specimens away from the initiation sites and from the overload regions. In the HEA condition, fracture was mainly intragranular and fatigue striations are evident in the crack propagation area at both (a) 0.3 % and (b) 0.8 % plastic strain amplitudes. These features are characteristic of fatigue crack growth in ductile materials [51,59]. In contrast, the H-HEA condition displays intergranular fracture at plastic strain amplitude of both (c) 0.3 % and (d) 0.8 %, as commonly observed in brittle solids [51,59]. In addition, secondary cracking along grain boundaries is more prevalent in H-HEA consistent with the intergranular fracture mode. These observations indicate that hydrogen significantly embrittles the CrMnFeCoNi alloy when subjected to these testing conditions and this hydrogen concentration. This embrittlement is also reflected in the significantly shorter fatigue lives in the presence of hydrogen. The effect of hydrogen on fracture mode in

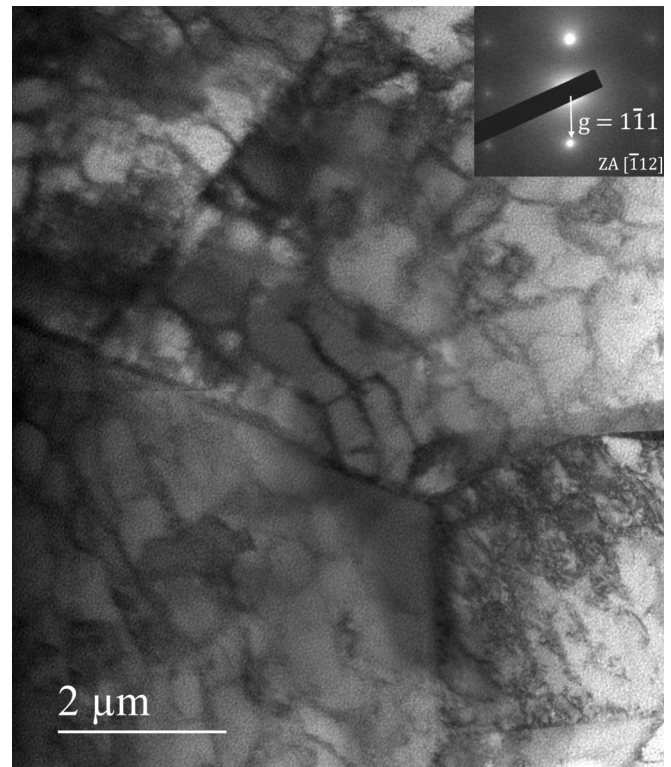


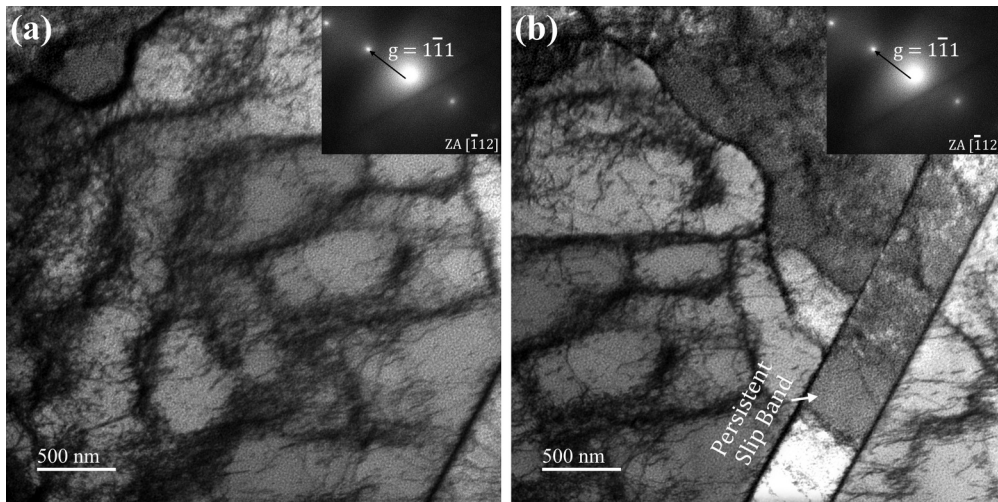
Fig. 9. TEM micrograph showing dislocation cells in the non-charged HEA specimen after LCF testing at 0.3 % plastic strain amplitude ( $N_f = 20,885$ ). The average cell size is  $670 \pm 240$  nm.

this alloy has been reported by other authors [24,25,27–32,34]. One explanation of this effect was proposed by Bertsch et al. [24] who suggested that hydrogen limits the ability of the CrMnFeCoNi alloy to accommodate plastic deformation, leading to strain incompatibilities across the grain boundaries and, ultimately, intergranular failure.

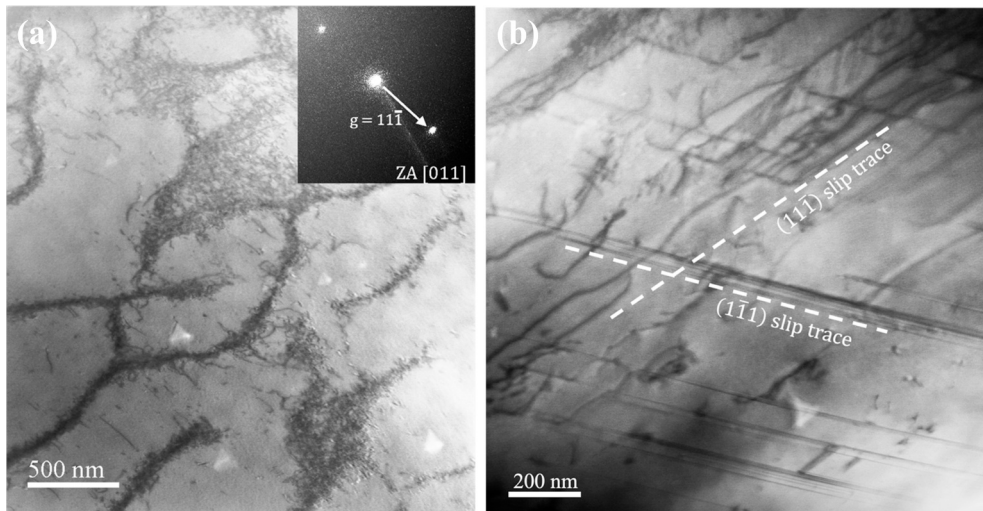
Despite the brittle fracture features, Fig. 8 shows fine slip traces on the grain boundary surfaces of H-HEA after LCF testing at (e) 0.3 and (f) 0.8 % plastic strain amplitudes, demonstrating that cyclic plastic deformation is active in the presence of hydrogen, presumably by dislocation glide on multiple slip systems. Martin et al. [60] observed similar intergranular fracture morphology in hydrogen-precharged nickel after monotonic testing. They proposed that the slip steps on the intergranular facets indicate hydrogen-enhanced plasticity processes, which increase local stresses, facilitate hydrogen transport to grain boundaries and ultimately lead to intergranular fracture by decohesion. Nygren et al. [25] and Fu et al. [61] also attributed slip traces on intergranular fracture surfaces of hydrogen-precharged CrMnFeCoNi to hydrogen-enhanced plasticity mechanisms.

### 3.4. Dislocation structures

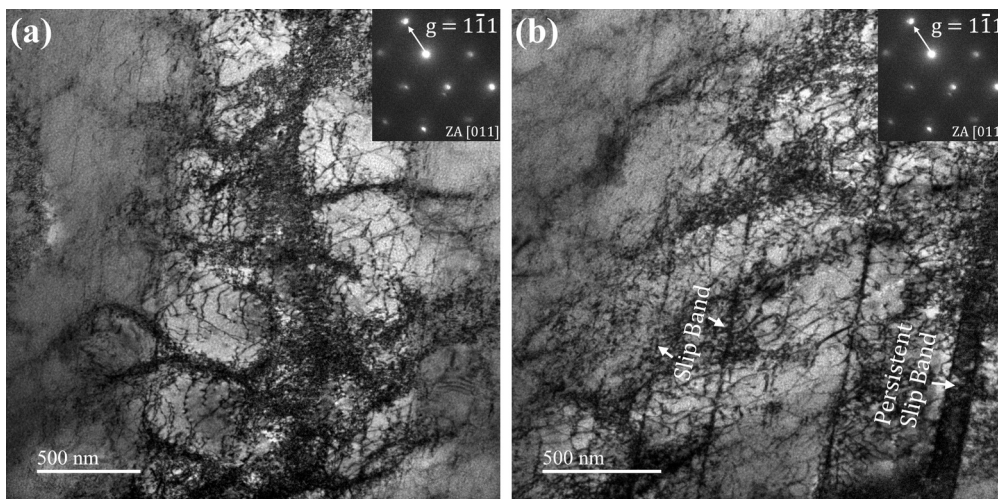
Well-developed dislocation cell structures were observed in non-charged CrMnFeCoNi (HEA) for both 0.3 % (Fig. 9) and 0.8 % (Fig. 10) plastic strain amplitudes in specimens tested to failure. These fatigue microstructures are similar to those shown by Lu et al. [16] and Shams et al. [17] in the CrMnFeCoNi alloy after total strain-controlled fatigue tests. They differ from those observed in tensile deformation of this alloy, which are characterized by more diffuse cell walls and more dislocation tangles within cell interiors [8]. The high strain amplitude (0.8 %) resulted in a slightly smaller dislocation cell size of  $560 \pm 230$  nm compared to  $670 \pm 240$  nm for the 0.3 % amplitude tests. In addition, a few persistent slip bands (PSBs) were observed in the non-charged condition after testing at 0.8 % plastic strain amplitude.



**Fig. 10.** TEM micrographs showing slip band along with dislocation cells in two different locations in the non-charged HEA specimen after LCF testing at 0.8 % plastic strain amplitude ( $N_f = 2,593$ ). The average cell size is  $560 \pm 230$  nm.



**Fig. 11.** DCI-STEM micrograph showing (a) incipient formation of cell structure, and (b) multiple slip on the  $\{111\}$  family in the H-HEA specimen after LCF testing at 0.3 % plastic strain amplitude ( $N_f = 2,366$ ).



**Fig. 12.** TEM micrograph showing slip traces and bands along with dislocation cells in the H-HEA specimen after LCF testing at 0.8 % plastic strain amplitude ( $N_f = 105$ ). The average cell size is  $390 \pm 85$  nm.

The fatigue microstructures in H-HEA samples after LCF testing at 0.3 % (Fig. 11) and 0.8 % (Fig. 12) plastic strain amplitudes contrast with the non-charged ones by an increased tendency for planar slip and a decreased tendency of dislocation cell formation compared to the non-charged condition, especially at low amplitude. At 0.3 %, Fig. 11 shows walls, ill-defined veins and planar slip of dislocations on the  $\{111\}$  planes, but no clearly defined cells. At 0.8 % strain amplitude (Fig. 12), well-defined cell formation was observed along with marked slip traces along  $\{111\}$  planes. Such planar slip traces can penetrate through multiple dislocation cells. These microstructural features are similar to those observed in tensile deformation of the charged HEA [25,27], although the cell walls are generally narrower and the cell interiors generally have fewer dislocations after cyclic deformation.

The TEM results indicate that hydrogen promotes planar slip and suppresses cross-slip of screw dislocations in CrMnFeCoNi, which can in turn promote localized deformation that degrades the fatigue lifetime of the material. Similar behavior was previously reported by Oliveira et al. [38] in 316 L stainless steel, where hydrogen decreased the propensity for dislocation cell formation during cyclic deformation. Kim et al. [29] measured the width of Shockley partials in hydrogen-precharged CrMnFeCoNi after tensile testing and reported a 29 % decrease in stacking fault energy (SFE) compared to the non-charged counterpart at a slightly higher hydrogen concentration than in the present study. Xie et al. [62] used first-principles calculations to investigate hydrogen embrittlement in the CrMnFeCoNi alloy and reported a hydrogen-induced reduction in stacking fault energy. Based on these findings, the reduced cross slip activity in H-HEA compared to HEA samples in the present study is consistent with hydrogen decreasing the SFE. Consequently, the evolution of microstructure in the precharged and non-charged conditions differs. However, despite the different evolution of fatigue microstructures in H-HEA and HEA samples, with hydrogen suppressing the formation of well-defined cells at an amplitude of 0.3 %, there are still hard substructural features at this amplitude (e.g. dislocation walls) that give rise to a similar evolution of back stresses in both material conditions as shown in Fig. 6.

#### 4. Summary

This study reports on the first results of the influence of hydrogen introduced by thermal precharging on cyclic deformation mechanisms in the CrMnFeCoNi high entropy alloy. The fatigue behavior of this alloy in the hydrogen-precharged (87 wppm H) and non-charged conditions was investigated through true plastic strain-controlled LCF tests at plastic strain amplitudes between 0.3 % and 0.8 %. Serrated flow is observed in the presence of internal hydrogen in the first cycle, likely due to hydrogen pinning the mobile dislocations. Internal hydrogen also increases the cyclic strength of the CrMnFeCoNi alloy at all plastic strain amplitudes. This strengthening is associated with greater effective stresses in the hydrogen-precharged condition compared to the non-charged condition, reflecting the influence of hydrogen on dislocation glide in this alloy. In addition, back stresses are similar in both material conditions for each tested amplitude, implying that hard dislocation substructures are present in both material conditions in the CrMnFeCoNi alloy. TEM observations revealed well-defined dislocation cells in the HEA samples at both strain amplitudes. In contrast, H-HEA samples exhibited well-defined dislocation walls but ill-defined cells with extensive planar slip at low amplitude, suggesting that hydrogen suppresses cross slip in CrMnFeCoNi and promotes localized deformation. In addition, hydrogen accelerates the formation of a primary crack(s) and severely decreases the LCF lifetime of this alloy under the current testing conditions and hydrogen concentration. The consequence is a dramatic change from ductile fatigue crack growth in the absence of hydrogen to more brittle, plasticity-mediated intergranular fracture with internal hydrogen.

#### CRedit authorship contribution statement

**Dayane M. Oliveira:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Christopher W. San Marchi:** Writing – review & editing, Resources, Methodology, Funding acquisition, Conceptualization. **Easo P. George:** Writing – review & editing, Resources, Methodology, Funding acquisition. **Ayeman Nahin:** Writing – original draft, Visualization, Software, Investigation, Formal analysis, Data curation. **Mingwei Zhang:** Writing – review & editing, Validation, Supervision, Software, Methodology, Investigation, Funding acquisition. **Jeffery C. Gibeling:** Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Data availability

Data will be made available on request.

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