



Short communication

# Influence of pre-strain on the gaseous hydrogen embrittlement resistance of a high-entropy alloy



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## ABSTRACT

The effect of pre-strain on the resistance to gaseous hydrogen embrittlement of CoCrFeMnNi high-entropy alloy (HEA) was investigated through mechanical testing and thermal desorption analysis. The results reveal that prior plastic deformation does not affect either the hydrogen contents or the excellent hydrogen embrittlement resistance of the HEA.

## 1. Introduction

High-entropy alloys (HEAs), which by definition comprise of at least five metallic constituent elements in (nearly-)equal amounts (in at%), have been attracting considerable attention in the recent past. This is because of their exceptional mechanical properties such as high specific strength combined with good ductility and excellent mechanical performance over a wide range of temperatures [1–5]. For future structural applications of HEAs, the influence of hydrogen (H) on their mechanical behavior is one of the crucial and essential topics of research since many potential applications of HEAs may lead to H exposure [6–8] and thus there is the possibility of hydrogen embrittlement, which could manifest as severe loss in ductility, unpredictable cracking that leads to premature fracture, and stress corrosion cracking [9–12]. Hence a close examination is warranted, but HEAs' resistance to hydrogen embrittlement was not examined in detail. As an early step of the research, very recently we explored the effect of H on the mechanical behavior of CoCrFeMnNi HEA (which is one of the most widely investigated HEAs to date [13–18]), by subjecting it to either electrochemical or gaseous charging [6,7]. The experimental results show that its resistance to gaseous hydrogen embrittlement is considerably more than face-centered cubic (fcc) austenitic stainless steels. This is in spite of HEA's strong ability to absorb H. We showed that higher hydrogen embrittlement resistance is due to the H content upon charging being below

the threshold value for triggering the mechanism of H-enhanced localized plasticity (HELP) [7,9]. An important issue, which remains un-addressed, is the influence of H on the mechanical performance of *deformed* HEA, which is crucial for two reasons. First, any pre-straining can possibly change the microstructure and, in turn, affect the hydrogen embrittlement resistance. Second, most of structural metals experience some level of straining during the final step of manufacturing before being deployed. Here, it is worth noting that published data shows that such pre-strain accelerates the hydrogen embrittlement in austenitic stainless steels [19–21]. Keeping this in view, we examine the effects of pre-strain on the gaseous hydrogen embrittlement resistance of the CoCrFeMnNi HEA.

## 2. Experimental

The HEA samples were prepared by vacuum induction casting of nominal mixtures of the corresponding metals, each with a purity > 99 wt%. The cast ingot was hot-rolled and then solution annealed at 1100 °C for 1 h to reach an equilibrium state of single phase fcc microstructure. Four different levels of pre-strain (10%, 20%, 30%, and 40% true strain) were introduced by recourse to interrupted tensile tests on dog-bone-shaped plate-type samples with gauge length and width of 25 and 5 mm, respectively (following ASTM E8). Tests were performed under displacement rate control (1.5 mm/min) [4]. The

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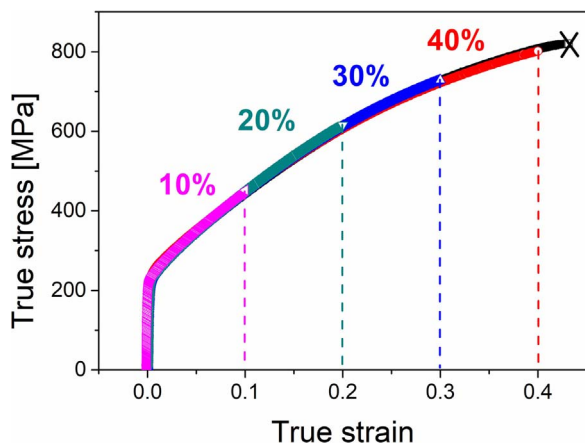


Fig. 1. The representative true stress-true strain curves obtained from the normal and interrupted tensile tests of CoCrFeMnNi HEA sample.

tensile elongation (failure strain) of the uncharged sample was  $\sim 43\%$  (as shown in Fig. 1) and uniform deformation was observed almost until the failure without necking. Note that such tensile tests with large-sized samples were only to prepare pre-strained samples but not directly for studying H effects.

Microstructure characterizations of the gauge parts of the interrupted samples were conducted by using X-ray diffraction (XRD; D/MAX-2500, Rigaku-Denki, Japan), electron backscattered diffraction (EBSD; CrystAlign system with the e-FlashHR detector, Bruker, Germany) mounted on a field emission scanning electron microscope (FE-SEM; S-4300SE, Hitachi, Japan), and transmission electron microscopy (TEM; Talos F200X, FEI Co., Hillsboro, OR, USA).

From the gauge parts of the interrupted specimens, sub-sized flat tensile specimens (with gauge length and width of 10 and 1 mm, respectively [7]) as well as small rectangular pieces were extracted, and were ground with colloidal silica ( $0.05 \mu\text{m}$ ) to a mirror finish, resulting in a uniform thickness of  $\sim 300 \mu\text{m}$ . Gaseous H charging was performed on both sets of specimens in a custom-made Sieverts apparatus at  $300^\circ\text{C}$  under a constant pressure of 15 MPa, which is the maximum capacity of the apparatus, for 72 h. This charging condition is known to be effective in evaluating the hydrogen-induced change in mechanical behavior of austenitic stainless steels [22,23] (that have the same fcc structure and similar main elements as CoCrFeMnNi HEA [3,7]). To minimize the influence of outgassing, all the charged samples were immediately immersion stored in liquid nitrogen until subsequent experiments, which were completed, in any case, within  $\sim 24$  h after charging. Sub-sized tensile tests were carried out on both uncharged and charged samples using a micro-tensile tester (MTest 300, Gatan Inc., Pleasanton, CA, USA) at a cross-head speed of  $0.1 \text{ mm/mm}$  (which corresponds to an initial strain rate of  $\sim 1.7 \times 10^{-4} \text{ s}^{-1}$ ). Nanoindentation experiments were conducted on the rectangular samples using Nanoindenter-XP (formerly MTS; now Keysight Technologies, Santa Rosa, CA, USA) equipped with a Berkovich indenter. All the indentation tests were performed to a peak load,  $P_{\text{max}}$ , of 100 mN with constant indentation strain rate,  $\dot{\epsilon} = (dh/dt)/h = 0.025 \text{ s}^{-1}$  where  $h$  is indentation depth and  $t$  is time. For the quantitative analysis of the H content in the charged rectangular samples, thermal desorption spectroscopy (TDS) was performed with a quadrupole mass spectroscope (EX0014, R-DEC Company, Tsukuba, Japan) at a constant heating rate of  $5^\circ\text{C/min}$  to the maximum temperature of  $\sim 800^\circ\text{C}$ .

### 3. Results and discussion

The true stress vs. true strain response of the HEA, superposed with those obtained from the interrupted tensile tests, is displayed in Fig. 1. After yielding at a stress,  $\sigma_y$ , of  $\sim 220$  MPa, the HEA exhibits

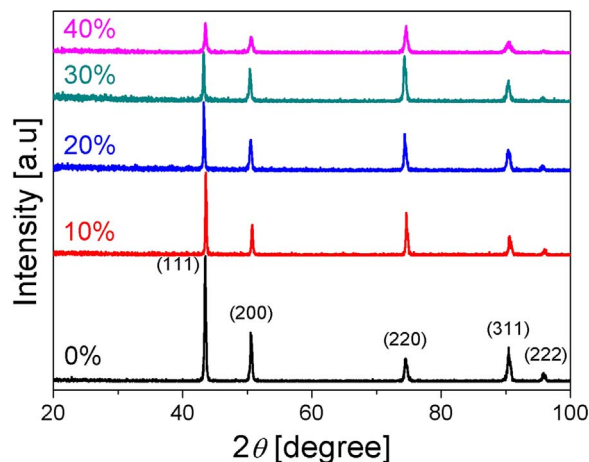


Fig. 2. The representative results from XRD.

considerable work hardening before failing at a stress of  $\sim 820$  MPa and 43% strain.

The microstructural characterization of the pre-strained specimens was performed with XRD, EBSD, and TEM. In all the XRD scans (Fig. 2), identical peak positions, all of which correspond to a single fcc phase, confirm the absence of stress induced phase transformation [8,15,24]. The lattice parameters estimated ( $\sim 3.591\text{--}3.594 \text{ \AA}$ ) are well within the reported range ( $3.59\text{--}3.61 \text{ \AA}$  [25–27]) for this HEA. Microstructural analysis performed with EBSD and TEM, Fig. 3a and b, shows only a few annealing twins and low dislocation density in the unstrained sample. Upon straining to 20%, grain distortion and a significant increase in dislocation density within the grains [28] were noted. Further straining to 40% leads to elongation of the grains along the tensile direction; the grains are almost filled with highly tangled dislocations, which is in agreement with the prior reports on HEAs [29,30].

Results of the micro-tensile tests performed on the 0–40% pre-strained HEA samples before and after gaseous H charging are displayed in Fig. 4, from which the following two observations can be made. (a) H charging does not affect the strain hardening behavior and the ductility of the HEA in any significant manner. (b) In 0% and 20% strained samples, H charging does not seem to affect  $\sigma_y$  whereas it slightly increases in samples strained to 30% and above. Laplanche et al. [30] estimated the threshold stress for the initiation of mechanical twinning in the same HEA as  $\sim 720 \pm 30$  MPa. Fig. 4 indicates that  $\sigma_y$  in samples strained to 30% and beyond is either similar or higher to this value. Since H is known to reduce the stacking fault energy, which, in turn, promotes twinning [31,32], the H-induced slight strengthening observed in highly pre-strained HEA samples is possibly due to pronounced development of mechanical twinning [8].

Variations in the hardness, estimated by using the Oliver-Pharr method [33] on the nanoindentation data, with pre-strain are plotted in Fig. 5 for both before- and after-charging conditions. From it, we noted that H charging does not alter the hardness in any substantial manner at all pre-strain levels. The absence of a significant hardness increase in 30% and 40% pre-strained samples (unlike the results of tensile tests, especially for 40% pre-strained samples) may be explained by the differences in stress condition and deformation size between nanoindentation and micro-tensile tests; i.e., the stress condition underneath an indenter is much more complex than that in a uniaxial tensile sample, and the deforming volume during the nanoindentation is much smaller than that in the tensile tests of micro-sized (but still bulky) samples. Combined with the possibility that twinning may prefer some grains (having specific orientations) and deformation mode, these differences may suppress the increase in local hardness.

In an earlier study [7], we observed that gaseous charging of H does not embrittle CoCrFeMnNi HEA and thus rationalized it on the basis that the local H content being below a certain threshold level required

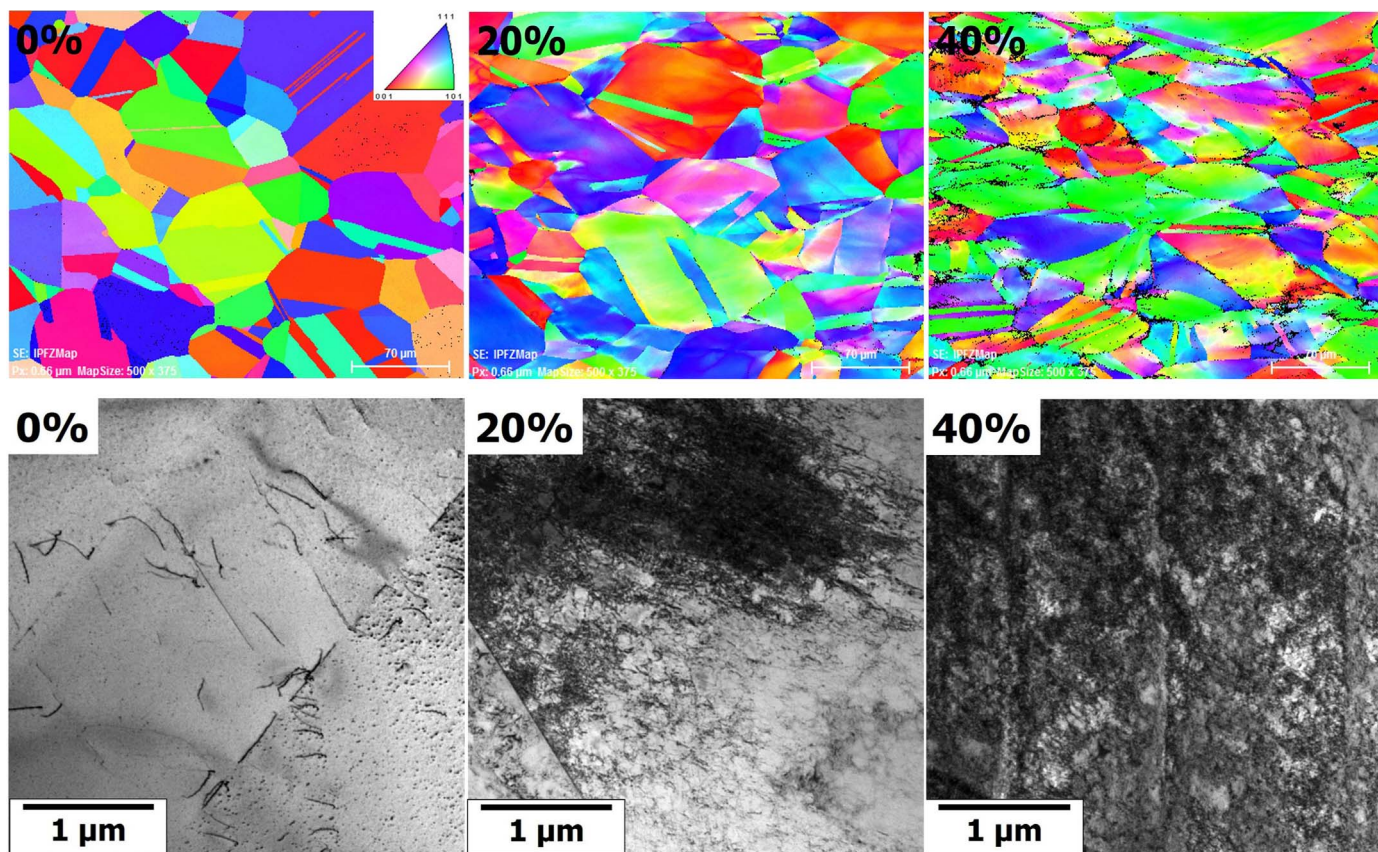


Fig. 3. Microstructural evolution in the pre-strained samples; the representative results from (a) EBSD, and (c) TEM.

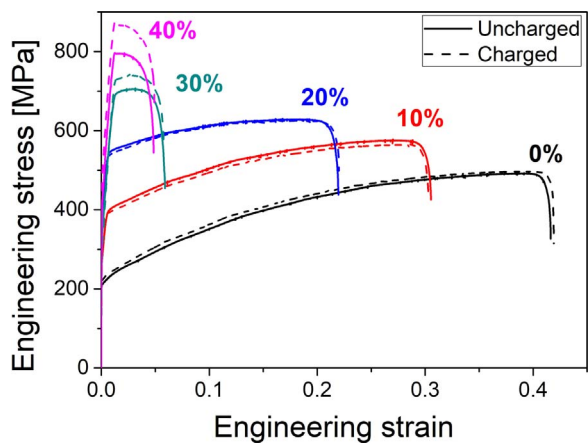


Fig. 4. Typical engineering stress-engineering strain curves from micro-tensile tests.

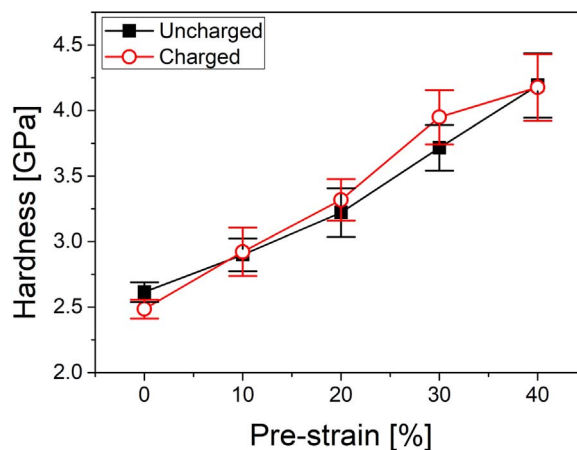


Fig. 5. Variation in nanoindentation hardness with pre-strain.

for triggering HELP. The results of the present study, in particular the invariance of ductility in pre-strained and H charged samples, further establish the excellent resistance of the HEA under investigation to gaseous H embrittlement. In addition, they imply that pre-straining does not significantly alter the threshold H levels for triggering the HELP mechanism.

The H content in the pre-strained samples after charging were measured by analyzing TDS curves, and the results are displayed in Fig. 6. The H content of the annealed sample is ~63.2 wppm that is at a similar level to that reported in previous study, ~76.5 wppm [7], confirming the reason for the hydrogen embrittlement resistance being insufficient H content as discussed above. Two features of Fig. 6 are noteworthy. Pre-straining does not appear to alter the temperature of the peak desorption. This observation implies that the main H trapping

sites remain unaltered by the pre-straining. More importantly, the H content, which is given by the area under the desorption curve, is not significantly affected by pre-straining. This indicates that the largely increased dislocation density in the pre-strained samples did not enhance the capacity of the HEA to absorb H, which is what one would expect as dislocations are known to be effective H trapping sites [34–36]; an increase in dislocation density with plastic strain should have lead to larger amounts of trapped H. On the contrary, the H content in deformed samples is lower (by ~8.5–10.5 wppm) than that in the undeformed sample; possible reasons for this are discussed in the next paragraph. We can conclude that the lower H in the deformed HEA is the reason for the sustained good hydrogen embrittlement in them.

Results presented in Fig. 6 suggest that dislocations in the HEAs do not favor H atom trapping, unlike in conventional alloys. A plausible



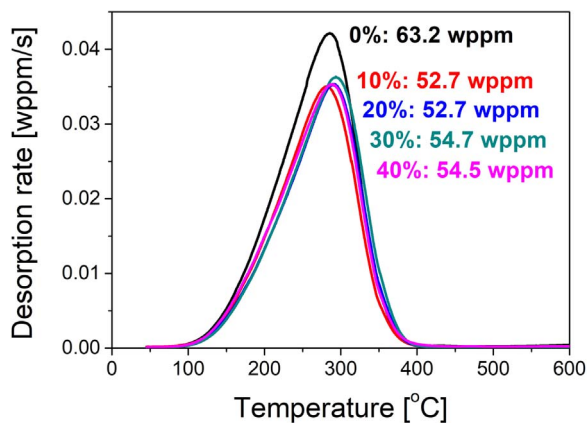


Fig. 6. H desorption curves obtained from TDS measurements of H-charged specimens.

reason for this may be the significant lattice strain energy [1,37] of HEAs, which is also one of the reasons for the high H solubility in CoCrFeMnNi HEA lattice [7]. Since the TDS peak temperatures, and thus the main H trapping sites, are not significantly affected by pre-straining (see Fig. 6), we can conclude it is the lattice rather than dislocations that continues to trap H. In this sense, "cleaner" microstructure of the annealed HEA sample than the pre-strained samples may explain a slightly higher H content in the former than in the latter; that is, probably less H atoms are expected to be trapped in dislocations than in HEA lattice. Recently, Zhao and Nieh [38] suggested that the CoCrFeMnNi HEA has a smaller dislocation core than pure Ni, which, in turn, may not favor preferential H segregation to dislocation cores [39]. Another possible contributing factor is the nanoscale heterogeneity such as co-clusters and/or short-range orders (SROs) that may exist in the lattice of CoCrFeMnNi HEA [40–42]. This is partially supported by the planar dislocation slip characteristics in CoCrFeMnNi HEA [29,30] since SRO typically suppresses cross-slip. Such nanoscale heterogeneities may favor H trapping and thus, at least partially, explain the high H solubility of annealed HEA. Plastic deformation leads to shearing of those co-clusters and SROs, and a reduction in their population [42] could lead to a reduced H solubility.

#### 4. Conclusion

To summarize, the effect of prior-plastic deformation on the gaseous hydrogen embrittlement of CoCrFeMnNi HEA was investigated, results of which show that the excellent resistance of the HEA to hydrogen embrittlement is unaffected by pre-strains. This is because pre-straining does not enhance the H solubility in the HEA. On the contrary, it decreases the H solubility slightly due to the smaller dislocation core and the existence of nanoscale heterogeneity in the HEA that make the lattice more suitable for H atoms' occupation than dislocations.

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#### Prime novelty statement

- Effect of pre-strain on hydrogen embrittlement resistance of high-entropy alloy was studied.
- Prior plastic deformation does not affect the excellent hydrogen

embrittlement resistance of CoCrFeMnNi alloy.

- High-entropy alloy lattice may be more suitable hydrogen-trapping sites than dislocations.

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