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Decoupling the roles of constituent phases in the strengthening of hydrogenated nanocrystalline dual-phase high-entropy alloys

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ABSTRACT

Nanocrystalline (NC) dual-phase Al_{0.7}CoCrFeNi HEAs containing face-centered cubic (FCC) and bodycentered cubic (BCC) microstructural phases were fabricated by high-pressure torsion (HPT). The influences of hydrogen on the thermal desorption and nanoindentation responses of NC HEA were compared with the coarse-grained alloy. The plastic zone size and indentation size effects were carefully considered to identify the distinct contributions of the constituent phases to the hardness and its variation with hydrogen charging. Results show that the FCC phase is susceptible to a larger degree of hydrogen-induced hardening than the BCC phase. Such difference is negated in the NC samples. These results are discussed in terms of the distinct responses of FCC and BCC HEA phases to hydrogen and the governing deformation mechanisms in coarse grained and NC samples.

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the mechanical behavior in a hydrogen environment is a challenge.

Keeping this in view, the contributions of each constituent phase

The "Cantor" family high-entropy alloys (HEAs), in particular Al_x CoCrFeNi, were extensively investigated with a view to tailor their strength-ductility combinations [1–4]. It was suggested that an intermediate Al level (e.g., x = 0.7) yields an optimum property combination [2,5]. The dual phase microstructure of $Al_{0.7}$ CoCrFeNi HEA is lamellar in nature and is a result of the (near-)eutectic reaction. The crystal structures of the two constituent phases are face-centered cubic (FCC) and body-centered cubic (BCC) [6–9]. Further strengthening of HEAs can be achieved through significant grain refinement to nanocrystalline (NC) level (average grain size, d < 100 nm) by high-pressure torsion (HPT), one of severe plastic deformation (SPD) processing methods [10–12].

While hydrogen effects on mechanical behavior of FCC and BCC HEAs is extensively investigated [13–15], the response of dualphase HEAs is not probed in detail yet. It could be rather complex in view of the disparate diffusivities and solubilities of hydrogen in different phases and thus the distinct mechanical responses [16]. Decoupling the distinct roles of the two (or more) phases in

t in a dual-phase Al_{0.7}CoCrFeNi HEA to the overall strength changes due to nano-graining (induced via HPT process) and then hydrogenation are investigated in this work. A robust protocol to estimate the hardness of the microstructural phase, which takes both the plastic zone volume underneath indenter and indentation size effect (ISE) into account, was utilized for this purpose. An arc-melted Al_{0.7}CoCrFeNi HEA ingot was used for this study. It was homogenized, cold rolled, and annealed shortly, and referred

It was homogenized, cold rolled, and annealed shortly, and referred to as "as-received (AR)." Disk samples (10 mm diameter, ~0.83 mm thickness) were cut from the AR samples, and then subjected to HPT with an applied pressure of 5 GPa for 1, 2, and 5 turns at a rotation speed of 1 rpm. The distribution of the Vickers hardness across the diameter of each disk was recorded using HMV-2 equipment (Shimadzu, Japan) with a peak load $P_{\rm max}$ of 980 mN.

Microstructural characterization was conducted using X-ray diffraction (XRD; Bruker D8, Germany) and scanning electron microscopy (SEM; Verios G4 XHR, Thermo Fisher Scientific, USA) equipped with energy dispersive X-ray spectroscopy (EDS) and electron backscattered diffraction (EBSD; Hikari, EDAX, USA). The Young's modulus, *E*, and Poisson's ratio, ν , of all the samples were evaluated by ultrasonic pulse-echo testing following the standard ASTM E-494. Nanoindentation experiments were conducted on AR







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Fig. 1. Microstructural evolution in Al_{0.7}CoCrFeNi HEAs (a) AR, (b) after 1, (c) 2, and (d) 5 turns. In addition to SEM images, each set includes BSE images and EDS map of Aluminum element.

and edges of the HPT specimens using Nanoindenter-XP (KLA, USA) equipped with a Berkovich tip. Peak loads, P_{max} , of 5 and 10 mN and constant indentation strain rate of 0.025 s^{-1} were employed. Continuous stiffness measurement (CSM) mode was utilized to obtain hardness values at various indentation depths. Before nanoindentation tests, all sample surfaces were polished to a mirror finish using the 0.05 μ m colloidal silica. The polished specimens were electrochemically hydrogen charged [17] at room temperature (RT, ~25 °C) with a potentiostat/galvanostat equipment (HA-151A, Hokuto Denko, Japan) with a current density of 100 mA/cm² for 24 h in an electrolyte containing 0.1 mol/L NaOH and 0.5 wt.% NH₄SCN. Thermal desorption spectroscopy (TDS) was performed using a gas chromatograph (JTF-20A, J-Science Lab, Japan) at a constant heating rate of 100 °C/h.

Representative SEM images of the AR and HPT-processed samples are displayed in Fig. 1, where backscattered electron (BSE) micrographs and corresponding EDS element maps for Al are also provided. The AR sample (Fig. 1a) exhibits a lamellar structure composed of two alternating phases of FCC and BCC/B2 ("A2/B2" strictly speaking), as widely reported for Al_{0.7}CoCrFeNi [1,3]. Since ordering is not supposed to significantly affect the hydrogenrelated properties and not the focus of our present work, BCC and B2 phases will not be specifically differentiated hereafter, as often done in prior studies [9]. While each phase in AR sample can easily be identified with the aid of the EBSD maps (as shown in Fig. S1 of the Supplementary Information, SI), it is difficult to apply typical EBSD analysis to very fine phases and thus EDS mapping was adopted for the phase identification of HPT-processed samples. As the BCC/B2 phase is enriched with Al and Ni (which are thus depleted in FCC phase) [7,18,19], comparison of BSE image with EDS map confirms that the darker lamellar microstructure is indeed the BCC/B2 phase. Upon HPT process (Figs. 1b-d), the regions of BCC/B2 phase become disconnected and the individual domain gets smaller with increasing N. Values of d for FCC and BCC/B2 phases in the AR sample, obtained from EBSD, were 14.2 and 5.6 μm respectively (Table 1).

XRD scan results, displayed in the inset of Fig. 2, also confirm the FCC+BCC/B2 dual-phase structure in all the samples. The *d* values of both phases in the HPT-processed samples were estimated

Table 1

Summary of average grain size, d, of Al_{0.7}CoCrFeNi, where the data for AR sample are obtained from EBSD and those for HPT samples are the crystallite sizes from XRD results.

Grain size [nm]	AR	N = 1	N = 2	<i>N</i> = 5
FCC	14,200	28	7	3
BCC/B2	5,600	37	30	5



Fig. 2. The variations in dislocation density of FCC and BCC/B2 phases (with inset showing X-ray diffraction scan results).

by XRD and are listed in Table 1. Despite the possibility of an underestimation, it is obvious that the *d* of both phases are reduced to NC level with increasing *N*. The variation in dislocation density, ρ , upon HPT process can also be estimated from the XRD results from the full width at half maximum (FWHM) values of the diffraction peaks using the modified Williamson–Hall method (with the aid of the modified Warren–Averbach method), details of which are provided elsewhere [20–22]. ρ in each FCC or BCC/B2 phase was thus independently estimated using the reflection, and



 $\ensuremath{\textit{Fig. 3.}}$ TDS results obtained on the hydrogen-charged AR and HPT processed samples.

the results are plotted as a function of *N* in Fig. 2. Two features are evident form it. In all the cases, ρ in FCC phase is slightly higher than that in BCC/B2 phase, which agrees with previous results on similar dual-phase alloys [23,24]. HPT process enhances ρ in both the phases, as expected, by ~4–5 times compared with that before HPT process, which is consistent with that reported in literature on HEAs [25,26]. ρ in FCC and BCC/B2 phases saturate at ~1.4 × 10¹⁵ and ~1.1 × 10¹⁵ m⁻², respectively, at *N* = 1.

Representative TDS results are displayed in Fig. 3. The hydrogen desorption peak at a lower (or higher) temperature represents that the corresponding hydrogen-trapping sites bind hydrogen in a weaker (or stronger) manner [27,28]. The major peak occurs around 120 °C in all the samples. It reflects the hydrogen trapped in an interstitial site of the lattice. A secondary peak of much smaller intensity (and partly overlapping with the major peak) appears around 260 °C, but only for HPT-processed samples. They are conceivably due to the largely increased number of dislocations and/or grain boundaries that trap hydrogen [27,29]. With N, the hydrogen content markedly increases from ~9.13 weight ppm (wppm) in AR to 62.47 wppm after N = 5. Variation of microstructural factors such as the phase fraction (*X*), ρ , and *d*, with *N* could be responsible for this. The X of FCC and BCC/B2 phases, evaluated using SEM micrographs (Fig. 1), indicate no obvious change in the relative X values; the fraction of FCC phase always remains \sim 70%. Therefore, the possibility for a phase transformation (since FCC generally shows higher hydrogen solubility than BCC does [16]) can be ruled out. Since ρ values in both phases saturate beyond N = 1while the hydrogen content continues to increase, ρ might not be the reason as well. Moreover, it was reported earlier that dislocations do not contribute to enhancing hydrogen solubility [30]. Thus, increased number of grain boundaries, which act as effective hydrogen trapping sites, is the most likely reason behind the enhanced solubility of hydrogen with N. This agrees with our prior work, which highlighted the significant role played by the grain boundaries in trapping hydrogen [15].

In the HPT process, the applied equivalent strain ε_{eq} within the disk is given by $\varepsilon_{eq} = \frac{1}{\sqrt{3}} \frac{2\pi Nr}{t}$ where r and t are the radius and thickness of the disk, respectively [31]. Vickers hardness (HV) was measured across each HPT disk and plotted in Fig. 4a function of r and N. Inset shows that HV increases markedly with ε_{eq} before saturating at ε_{eq} of ~20 (corresponding approximately to the edge part of N = 1 sample). HV of AR sample is also included at $\varepsilon_{eq} = 0$.

The results from the nanoindentation tests conducted with $P_{\text{max}} = 10$ mN are displayed in Fig. 4. Here, indentation plastic-



Fig. 4. Variations in nanoindentation hardness, and the ratio of the final displacement (h_f) to maximum displacement (h_{max}) of uncharged and hydrogen-charged states. Inset shows variations in Vickers hardness of the AR sample and HPT-processed discs with different equivalent strain, and indentation locations.

ity is captured with $h_{\rm f}/h_{\rm max}$, where $h_{\rm f}$ is the final indentation displacement after unloading and h_{\max} is the maximum displacement at a given P_{max} . See [14] for details. Thus, hardness and h_f/h_{max} reflect strength and ductility, respectively. They are described for two independent sample conditions, i.e., HPT (nano-graining) and hydrogenation. Upon HPT, the hardness significantly increases after merely one turn (N = 1), and remains almost unchanged with further increase in N. As expected from the strength-ductility tradeoff, an opposite trend was observed for $h_{\rm f}/h_{\rm max}$ with N. The HPT process induces a large decrease in h_f/h_{max} at the early stage, but further straining does not bring a significant reduction. Note that $h_{\rm f}/h_{\rm max}$ in HPT samples are still reasonably high (~0.7), which may imply ductility loss is insignificant upon HPT. With hydrogen charging, an increase in hardness and decrease in h_f/h_{max} are noticed in the AR sample. Such divergence is relatively smaller in HPT samples, suggesting that the mechanical properties of HPT samples are less sensitive to hydrogen charging.

To decouple the contribution of each phase to the overall strength and then to explore the hydrogen effects on it, following protocols were adopted in consideration of the indentation-induced plastic zone volume and ISE. Fig. 5a schematically illustrates it.

Nanoindentation hardness of the FCC phase considering plastic zone size: All the indentation impressions produced at $P_{\text{max}} = 10$ mN include both FCC and BCC/B2 phases. Therefore, additional tests at $P_{\text{max}} = 5$ mN were performed on only the FCC phase, as shown in the inset of Fig. 5a and Fig. S2. Note that the BCC/B2 phase (especially in HPT-processed samples) is too small to be directly indented. The radius of the plastic deformation zone during indentation (r_p), which is actually much larger than the visible triangular impression, should be considered while estimating the nanoindentation hardness of the FCC phase, H_N , to ensure the exclusion of any adjacent BCC/B2 phase. Following Johnson's expanding-cavity-based model [32], r_p can be estimated as:

$$r_{p} = a_{c} \left\{ \frac{1}{6(1-\nu)} \left[\frac{E}{\sigma_{y}} \tan(90^{\circ} - \theta) + 4(1-2\nu) \right] \right\}^{1/3}$$
(1)



Fig. 5. (a) Schematic diagram of ISE calculation process, the solid line is theoretical value of nanoindentation data, and the dashed line is the curve after ISE correction, (b) changes in estimated macroscopic hardness of uncharged and hydrogen-charged samples, and (c) hardening ratio of H_0 by hydrogen charging and nano-graining via HPT (inset).

where a_c is the contact radius, θ is the half cone angle to the surface (and equivalent θ for a three-sided pyramidal Berkovich indenter is 70.3°) and σ_y is the yield strength. Values of r_p are estimated with a_c for each indentation determined with the Oliver-Pharr method [33], v = 0.34, E = 190 GPa (both obtained from ul-

trasonic pulse-echo tests), and $\sigma_y = 701$ MPa [5]. By drawing the circle with estimated r_p surrounding each indentation, the indentations excluding the influence from BCC/B2 phase were selected and the H_N value of only the FCC phase was determined.

Macroscopic hardness of the FCC phase considering ISE: Although we now have the H_N for FCC+BCC/B2 and FCC phases, they cannot be directly compared due to ISE [34]. H_N has to be "corrected" to obtain the size-independent, macroscopic hardness, H_0 . The Nix–Gao relation [34] between H_N and h, given below can be utilized for this purpose:

$$H_N^2 = H_0^2 \left(1 + \frac{h^*}{h} \right) \tag{2}$$

where *h* and *h*^{*} are indentation depth and its material-dependent "characteristic" length scale, respectively. By utilizing the CSM mode, *H*_N values at various depths from one single nanoindentation test can be readily obtained. The *H*²_N versus 1/*h* plots are utilized to estimate *H*₀ of FCC+BCC/B2 and FCC phases, referred to as *H*_{0,all} and *H*_{0,F}, respectively. They are illustrated in Fig. 5a.

Macroscopic hardness of BCC/B2 phase considering phase fraction in contact: The SEM images taken before and after nanoindentation were compared to determine the phase fraction, X, within the indentation-induced plastic zone using the r_p values estimated by Eq. (1). Then, the macroscopic hardness of the BCC/B2 phase is estimated by using the simple rule-of-mixtures:

$$H_{0,B} = \frac{H_{0,all} - H_{0,F}X_F}{1 - X_F}$$
(3)

where the subscripts "all," "F," and "B" stand for overall, FCC, and BCC/B2 phases, respectively. Since we already obtained the $H_{0,all}$ and $H_{0,F}$, the remaining value of $H_{0,B}$ can thus be obtained.

The variation in H_0 of each phase as a function of *N* is displayed in Fig. 5b. The HPT-induced increase in H_0 is significant in both FCC and BCC/B2 phases and H_0 is further enhanced upon hydrogen charging. For clarifying the hydrogen-induced hardening, the increment is displayed as the hardening ratio (*HR*), which is the hardness change divided by the original hardness. Fig. 5c summarizes *HR* induced by hydrogenation as well as by HPT (inset). Two important features are evident. First, a significant reduction in *HR* occurs upon HPT process initially, which plateaus as the samples deform more severely (i.e., with increasing *N* or ε_{eq}). In all cases, AR sample has much higher HR value for each phase than HPT processed samples. Second, and more importantly, *HR* of the FCC phase is always higher than that observed in the BCC/B2 phase.

In terms of the different responses to hydrogen of each phase. FCC shows much stronger hydrogen-induced hardening than BCC/B2. FCC phase absorbs much more interstitial hydrogen than BCC/B2 [16]. On the other hand, it also is relatively softer than the BCC/B2 phase, which makes it more-readily hardenable by hydrogen-induced solid solution strengthening mechanism [35,36]. In terms of the grain size effect, the HPT-processed NC samples show much smaller difference between HR values in the FCC and BCC/B2 phases. This is because the deformation mechanism in the NC HEAs is dominated by GB-mediated dislocation activity (dislocation nucleation and/or dislocation depinning at GBs [37]), irrespective of hydrogen [15]. Such a scenario can be indicated by the marginal increase in the overall hardness induced by hydrogen (see Fig. 5) and supported by the unchanged activation volume level in NC HEAs upon hydrogen charging (as reported in [15]). Therefore, when the grain boundaries dominate (viz. in NC regime), the hydrogen-induced hardening effect plays a rather minor role in affecting the strength and thus HR difference between FCC and BCC/B2 phases gets reduced as well.

In summary, nanocrystalline dual-phase Al_{0.7}CoCrFeNi HEA was fabricated by HPT and the influence of hydrogen was investigated through TDS and nanoindentation experiments in the present

work. By considering the plastic zone size and ISE, the sizeindependent hardness values of each microstructural phase in AR and NC samples and its variation upon hydrogen charging were estimated. The FCC phase shows a larger amount of hydrogeninduced hardening than BCC/B2, which is due to its soft nature combined with the ability to absorb higher quantity of hydrogen. Such a difference is much smaller in NC samples where grain boundaries predominate the deformation process.

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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.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2021. 114472.

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