



Regular article

Comparative study of hydrogen embrittlement in stable and metastable high-entropy alloys

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ABSTRACT

We investigated the effects of hydrogen on the mechanical response and embrittlement behavior of Fe₂₀Mn₂₀Ni₂₀Cr₂₀Co and Fe₃₀Mn₁₀Cr₁₀Co (at.%) alloys precharged with 100 MPa hydrogen gas. These alloys can be classified as stable and metastable austenite-based high entropy alloys (HEAs), respectively. We carried out tensile tests at initial strain rates of 10⁻⁴ and 10⁻² s⁻¹ at ambient temperature. Both HEAs revealed hydrogen embrittlement associated with localized plasticity-assisted intergranular crack initiation. It is important to note that hydrogen-assisted cracking of the metastable HEA occurred via localized plasticity for both the austenite and ε-martensite phases – an unusual observation for the latter.

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High-entropy alloys (HEA) have been drawing interest recently because of reported property improvements, e.g. in strength-ductility combination [1] and cryogenic fracture resistance [2]. Similar to steels, reducing the phase stability of the austenite in HEAs was shown to improve tensile properties via the transformation-induced plasticity (TRIP) effect [3]. It should be noted here that the TRIP effect results from the ε-martensitic transformation from a face centered cubic (FCC) structure to a hexagonal close-packed structure (HCP), and not from α'-martensitic transformation from a FCC to a body centered cubic (BCC) structure [3,4].

In recent studies, a Fe₂₀Mn₂₀Ni₂₀Cr₂₀Co HEA with stable austenite was reported to show high hydrogen embrittlement resistance [5], and hydrogen uptake can even be expected to have a positive effect with respect to elongation and strength [6]. These results are consistent with the conventional results for austenitic steels [7–9]. Particularly, most of the stable austenitic steels show a low hydrogen embrittlement susceptibility because of a low diffusivity of hydrogen. A positive effect of hydrogen on tensile ductility has been reported for a high-Mn austenitic steel, and is owing to a reduction in the stacking fault energy [8,9]. Furthermore, even if the austenite is unstable, an influence of the ε-martensitic transformation is not critical for susceptibility to hydrogen embrittlement when compared with the α'-martensitic transformation [10,11]. The hydrogen embrittlement resistance in the ε-phase of the metastable austenitic steels is attributed to its lower diffusivity compared to that in the BCC or FCC phases [12].

These previous studies indicate that the HEA concept, including an effect of the austenite stability in FCC alloys, can be used for synthesizing new hydrogen resistant structure materials. In this regard, we attempted to evaluate the hydrogen embrittlement susceptibility by introducing hydrogen in the form of a 100 MPa-high-pressure gas. This is one of the most severe conditions to which hydrogen-energy-related infrastructures are exposed. Subsequently, we comparatively characterized the hydrogen embrittlement behavior of the HEAs.

In this work, we prepared two types of HEAs: Fe₂₀Mn₂₀Ni₂₀Cr₂₀Co and Fe₃₀Mn₁₀Cr₁₀Co (at.%). The details of the chemical compositions of the HEAs are listed in Table 1. The first alloy is a HEA with stable austenite, and is referred to as the stable HEA. The second one has a dual-phase microstructure consisting of metastable austenite and ε-martensite, and is referred to as the metastable HEA. The metastable HEA shows the TRIP effect arising from the ε-martensitic transformation as mentioned above [3,4]. Ingots of mass 50 kg of the two alloys were prepared by vacuum induction melting and hot-rolled to 52% thickness at 1273 K followed by homogenization at 1473 K for 2 h in an Ar atmosphere and furnace cooling. The homogenized bars were further hot-rolled to obtain a thickness reduction to 33% (from 60 to 20 mm). The rolled bar was solution-treated at 1073 K in an air atmosphere for 1 h, followed by water-quenching. To introduce hydrogen, the specimens were exposed to a 100 MPa hydrogen gas atmosphere at 543 K for 200 h. Tensile tests were carried out at ambient temperature (293 K) at initial strain rates of 10⁻⁴ and 10⁻² s⁻¹. Note that Nygren et al. [13] recently reported significant hydrogen embrittlement in the stable HEA pre-charged with 120 MPa hydrogen gas, but the initial strain rate in their study (10⁻⁶ s⁻¹) is two-order and four-order lower than

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Table 1

Chemical compositions of the high entropy alloys (HEA) (mass %).

Steels	C	Mn	P	S	N	O	Al	Cr	Co	Ni	Fe
Stable HEA	0.002	19.77	0.002	0.006	0.0065	0.007	0.018	18.23	20.85	20.21	20.90
Metastable HEA	0.009	29.80	0.004	0.007	0.0087	0.015	0.028	9.29	10.46	0.01	50.37

the present experimental conditions. After the tensile tests, microstructures and fracture surfaces were characterized by electron backscatter diffraction (EBSD) and secondary electron imaging, respectively. The EBSD measurements were conducted at 20 kV and a beam step size of 50 nm after mechanically polishing the specimen surface. The fracture surface observations were carried out at 15 kV.

We measured the diffusible hydrogen content by thermal desorption spectroscopy at a heating rate of 400 K h^{-1} . The diffusible hydrogen contents of the stable and metastable HEAs, which are defined to be the cumulative hydrogen content from 313 to 873 K, were 113 and 178 mass ppm, respectively. The hydrogen desorption profile is shown in

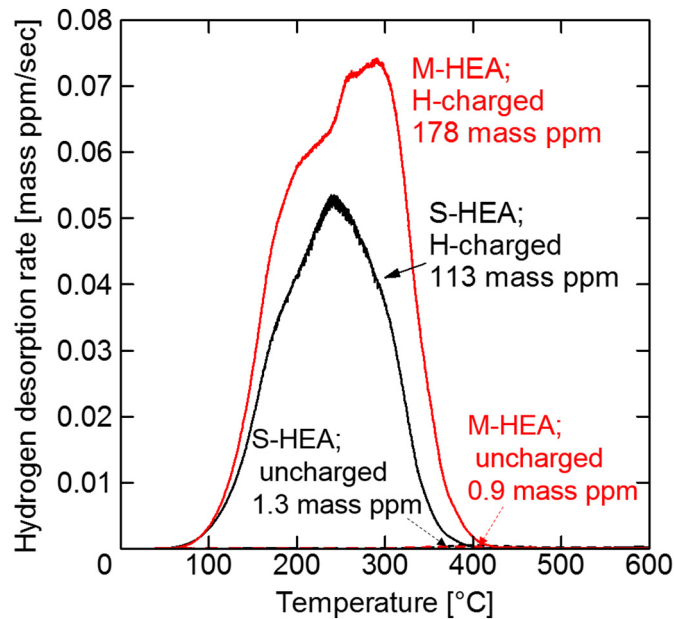


Fig. 1. Hydrogen desorption profiles of the stable and metastable HEAs with and without hydrogen charging. S-HEA: stable high entropy alloy; M-HEA: metastable high entropy alloy.

Fig. 1. The higher diffusible hydrogen content of the metastable HEA is perhaps due to the presence of thermally induced ϵ -martensite. Specifically, the γ/ϵ interface acts as a trap site of diffusible hydrogen, which can be a cause of severe hydrogen embrittlement [14].

Fig. 2(a) shows the engineering stress-strain curves of the stable HEA with and without hydrogen charging. In contrast to the previous work [5] that used a 10 MPa-hydrogen gas-pressure at an identical strain rate, the severe hydrogen charging conditions using a 100 MPa-hydrogen-gas pressure significantly deteriorated the tensile ductility of the stable HEA. It is also noted that hydrogen uptake increased the yield strength, perhaps owing to solution hardening of the hydrogen. Similar to the results observed for the stable HEA, the metastable HEA showed a degradation in tensile ductility and an increase in yield strength, as shown in **Fig. 2(b)**. In both HEAs, the tensile ductility recovered slightly with increasing strain rate. We noted the following two facts. (1) Hydrogen embrittlement occurs even in the stable HEA when hydrogen is introduced using a 100 MPa-gas pressure. (2) The hydrogen-charged metastable HEA showed a lower elongation but similar tensile strength even with the higher diffusible hydrogen content when compared to the hydrogen-charged stable HEA. We will further discuss the underlying mechanisms of hydrogen embrittlement in the stable HEA, and the reasons for the lower elongation and comparable strength in the metastable HEA.

Fig. 3 shows sets of EBSD images around the subcracks of the fractured specimens at an initial strain rate of 10^{-4} with hydrogen charging. In the uncharged condition, subcracks were not observed on the specimen surface after failure for both alloys. **Fig. 3(a–c)** shows that the stable HEA specimen exhibits intergranular crack initiation with orientation rotation without any phase transformation. The grain reference orientation deviation (GROD) shown in **Fig. 3(d)**, which qualitatively indicates plastic strain distribution [15,16], indicates more clearly that cracks were initiated with the formation of the orientation gradient. This fact suggests that hydrogen-assisted intergranular crack initiation is associated with localized plasticity. The occurrence of localized plasticity-assisted intergranular crack initiation is discussed in comparison with the previous studies [5,13]. The experimental conditions and associated fracture mode are summarized in **Table 2**.

The test mentioned in the work of Zhao et al. [5] was carried out at an initial strain rate of 10^{-4} s^{-1} for specimens precharged with a

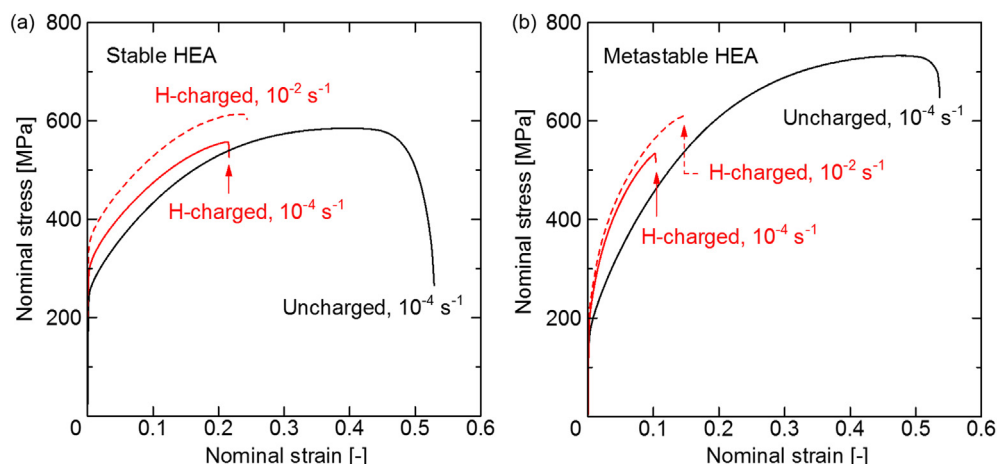


Fig. 2. Engineering stress-strain curves of the (a) stable and (b) metastable HEAs with and without hydrogen gas charging.

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