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# Fracture properties of a refractory high-entropy alloy: In situ micro-cantilever and atom probe tomography studies

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ABSTRACT

Most refractory high-entropy alloys (HEAs) are brittle and suffer from limited formability at ambient temperature. Previous studies imply that grain boundaries affect their fracture behavior, but quantitative studies on the fracture properties of body-centered-cubic HEAs are scarce. Here, using in situ micro-cantilever tests, we show that the fracture toughness of a bi-crystal HEA, $Nb_{25}Mo_{25}Ta_{25}W_{25}$, is one order of magnitude lower than that of single crystalline ones. Atom probe tomography of the bi-crystal HEA reveals element segregation and formation of oxides and nitrides at grain boundaries, suggesting that minimizing grain boundary segregation is critical to improving fracture properties in refractory HEAs.
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Refractory high-entropy alloys (HEAs) are emerging metallic systems that consist of four or more equiatomic refractory elements (e.g., Nb, Mo, Ta, W, and V) and tend to form single solid-solution-like body-centered cubic (bcc) phases with a strong tendency to solid solution strengthening [1–7]. For the last five years, such alloys have attracted significant attention, because of their superior and useful properties at elevated temperatures (above ~1100 °C): high strength and hardness [2,8], enhanced thermal and microstructural stability [9, 10], good oxidation resistance [7,11], etc. A vast majority of them are, however, extremely brittle at room temperature and suffer from poor ductility, rendering them difficult to further process and use [2,6,12, 13]. For example, two typical refractory HEAs (i.e., $Nb_{25}Mo_{25}Ta_{25}W_{25}$ and $V_{20}Nb_{20}Mo_{20}Ta_{20}W_{20}$) have been found to exhibit good ductility above 600 °C, but at room temperature, they fail easily by cracking at low compressive strains (~2.1% and ~1.7%, respectively) [12]. In a recent study, we compressed $Nb_{25}Mo_{25}Ta_{25}W_{25}$ HEA micro-pillar samples and observed that the micro-pillars with a grain boundary (GB) fractured along the GB at a much lower strain than single crystalline (SC) HEA micro-pillars, which can bear large-strain bending (>75°) without any fracture [6]. Our previous observation implies that the refractory HEAs might not be intrinsically brittle, but it is their GBs that significantly reduce their ductility. Thus, two questions arise: how much does a GB influence the fracture properties (i.e., fracture toughness and fracture strength) in a refractory HEA? And what characteristics of the GBs induce the brittleness of the HEAs?

Although face-centered cubic (fcc) HEAs, such as CrMnFeCoNi, have been reported to exhibit promising fracture resistance [14], the inadequate fracture-resistance property of bcc HEAs is, in fact, a bottleneck that limits their usage. So far, to the authors' knowledge, study of the fracture properties of bcc HEAs is still lacking. The main obstacle to measuring their fracture toughness is that the materials are generally too brittle to be fabricated as standard macroscopic fracture toughness specimens (as illustrated in supplementary information Fig. S1). In this work, we have applied the methodology of in situ micro-cantilever fracture tests [15] to study the fracture behavior of SC $Nb_{25}Mo_{25}Ta_{25}W_{25}$ HEAs and bi-crystal (BC) HEAs (i.e., those containing a GB) and evaluate the effect of a GB on the fracture properties of refractory HEAs. Using this technique, combined with finite element method (FEM) simulations, we are able to calculate the fracture toughness and strengths of the HEA micro-cantilevers and also compare them with other reported micrometer-sized materials, such as ceramics [16,17], intermetallics [18,19], and metals [20,21].

A bulk $Nb_{25}Mo_{25}Ta_{25}W_{25}$ HEA was produced using the arc melting technique in an argon atmosphere and then homogenized at 1800 °C for seven days (as described in [6]). The crystal orientations of the as-prepared HEA specimen were characterized using the electron back-

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scatter diffraction (EBSD) technique (Fig. 1a). The specimen shows an equiaxed grain microstructure with grain sizes of a few hundred microns. SC- and BC-cantilevers were fabricated from two adjacent ⟨110⟩-oriented grains (misorientation angle <5°) using the focused ion beam (FIB) technique (Hitachi, FB-2200), as shown in Fig. 1b and c, respectively. Both SC- and BC-cantilever beams have a length ($L_0$) of ~6–8 μm, a width ($W$) and thickness ($B$) of ~1.5–2 μm, as schematically illustrated in Fig. 1d. A notch with a depth of ~0.3–0.5 μm and a tip radius of ~10 nm was fabricated in each cantilever using a fine milling current (5 kV, 5 pA) and a milling time of ~5–10 s. In SC-cantilevers, the notches were close to the cantilever beam support and parallel to {100} planes (primary cleavage planes for bcc metals). In BC-cantilevers, the notches were cut along the GBs. The micro-cantilever specimens were mounted in an indenter holder (Nanofactory Instruments AB, SA2000N), which was fitted to a transmission electron microscope (TEM, JEOL JEM-2100). A sharp diamond tip was used to load at the beam (close to the free end) in a displacement control mode (2 nm/s) by feedback mechanism (the details of the loading apparatus were described in [22]). Four specimens were measured for both SC- and BC-cantilevers. A force-displacement curve during the fracture process was recorded.

Fig. 2a–c and e–g present snapshots from movies of typical SC- and BC-cantilevers upon loading, respectively. Fig. 2d and h show their corresponding load–displacement curves. The SC-cantilever exhibits a linear elastic behavior at the initial loading stage (between a and b in Fig. 2d), a slight yielding before reaching the maximum load, and a subsequent gradual force drop. In contrary to the SC-cantilevers, all the BC-cantilevers experienced a catastrophic event at the maximum load. They did not show any plastic yielding before fracture, and the crack tips suddenly opened and advanced along the GBs (as shown in Fig. 2g), indicating that the BC-specimens are more brittle than the SC ones. After in situ cantilever tests, we characterized the fracture surfaces using a scanning electron microscope (SEM). The two types of cantilevers reveal distinct surface morphologies. The SC-specimen shows a quasi-cleavage feature with river markings (Fig. 2i), as being cleavage-like but not along a single sharp plane, suggesting that SC-HEAs fracture in a mode between brittle fracture and ductile fracture. During the bending test, the secondary fracture planes {110} might be also activated due to a few degrees of the misalignment between the notch and the fracture plane. The BC-specimen exhibits an extremely flat and smooth surface along the grain boundary (Fig. 2j), showing a typical feature of brittle intergranular fracture.

Fig. 2 suggests that both the SC- and BC-specimens show a limited amount of crack tip plasticity before fracture because a linear elastic behavior is present and no force-displacement plateau has been observed. To calculate the plane strain fracture toughness, $K_{Ic}$, the following equation, according to linear elastic fracture mechanics (LEFM), can be applied [23]:

$$K_{Ic} = \frac{F_{max} L_f}{B W^{\frac{3}{2}}} f \frac{a}{W} \tag{1}$$

where $F_{max}$ is the maximum load before fracture (i.e., fracture force) and $f(a/W)$ is a geometry factor, which can be calculated using FEM simulations. In this study, two-dimensional extended FEM modeling was used to calculate the values of $K_{Ic}$ for both SC- and BC-cantilevers using the J-integral method. The details of the method are explained in the supplementary data (Fig. S3) and ref. [24]. Additionally, because the

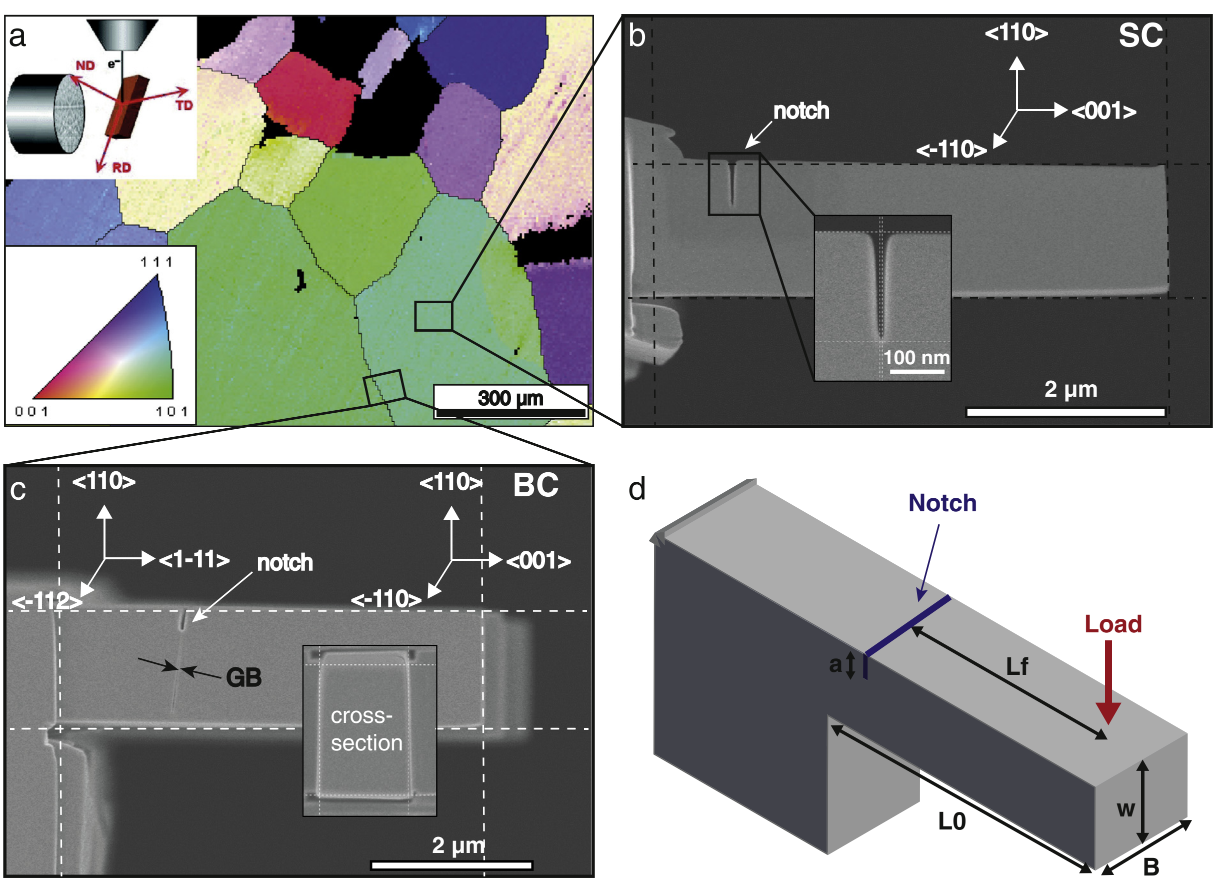


**Fig. 1.** a. An EBSD inverse pole figure map of the cross-section of the HEA bulk specimen. Two adjacent ⟨110⟩-oriented grains were selected to fabricate micro-cantilevers, as indicated by boxes. b. and c. Typical single crystalline (SC) and bi-crystal (BC) cantilevers fabricated by FIB, respectively. The notch, crystal orientation, and grain orientation are indicated in each figure. d. A schematic of the shape and dimension of an FIB-notched cantilever with a beam length, $L_0$, width, $W$, thickness, $B$, loading length, $L_f$, and notch depth, $a$.

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