Supplementary material

Deformation mechanisms and work-hardening behavior of transformationinduced plasticity high entropy alloys by in-situ neutron diffraction

S. Fu^{a,b}, H. Bei^{c*}, Y. Chen^a, T.K. Liu^{a,c,d}, D. Yu^{a,d}, and K. An^{a*}

^aNeutron Scattering Division, Oak Ridge National Laboratory, Oak Ridge, USA; ^bSchool of Chemical Engineering and Technology, Tianjin University, Tianjin, China; ^cMaterials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, USA; ^dDepartment of Materials Science and Engineering, The University of Tennessee, Knoxville, USA

* Corresponding author: Hongbin Bei, hbei1@utk.edu; Ke An, kean@ornl.gov

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S-1 Methods of tensile test

The sample for tensile test was a bone-shaped bar with 20 mm in gauge length and 3 mm in gauge diameter. Its loading axis lied along the rolling direction. The tensile test was conducted on the neutron diffractometer VULCAN at low loading rate for sufficient acquisition of diffraction patterns. Note that a short hold (~20 min) was introduced at 730 MPa (15%) for the change of control mode because the extensometer had to be reset manually. The tension was under force control at 0.05 MP/s before 730 MPa (15%), and then switched to slower displacement control for better statistics till its fracture. Correspondingly, the strain rate changed from ~5.0 × 10-5 /s to ~1.1 × 10-5 /s. Diffraction in both loading and transverse directions reflected the lattice spacing and intensity in ~3×3×8 mm³ gauge volume of the sample, from which the bulk average information of transformation, lattice strain and grain reorientation were obtained. A series of diffraction patterns in the loading direction at varied stresses are given in Figure S1. Note that each diffraction pattern reflects the average information of 10-minutes neutron data.



Figure S1. Diffraction patterns in LD under varied stresses.

S-2 Methods of intensity correction

The intensity change caused by such gauge volume reduction is corrected by multiplying the ratio of original cross-section area to the current area. Based on the assumption of volume incompressibility, the ratio of original cross-section area to the current area is equal to (1+e) where *e* is the engineering strain.

S-3 Methods of EBSD

EBSD measurements were performed using a JEOL 6500F field-emission gun scanning electron microscope (FEG-SEM) equipped with a TSL OIM EBSD system for the initial TRIP-HEA sample and the deformed sample with ~6% strain. The spatial resolution of the measurement is 0.3 μ m.

S-4 Methods of pole figure measurements

Pole figures (PFs) were measured by neutron diffraction at the VULCAN Diffractometer for the initial and fractured samples by uniaxial tension as shown in Figure S2. To accomplish the PF measurement, the sample was rotated around the normal axis of diffraction plane from 0° to 45° denoted by Ψ , and the loading axis of the sample from 0° to 360° denoted by Φ . The diffraction data was collected every 5° in Ψ and every 30° in Φ by two detectors in transverse and loading directions. The neutron diffraction data were fitted by single-peak-fitting method using the VRDIVE program (K. An, VDRIVE-Data Reduction and Interactive Visualization Software for

Event Mode Neutron Diffraction, ORNL Report ORNL-TM-2012-621, Oak Ridge National Laboratory, 2012). The integral intensity of {111}, {200} and {311} diffraction peaks for the FCC phase and {10.0}, {10.1}, {10.2} and {10.3} peaks for the HCP phase were used to calculate the full orientation distribution function (ODF) by the Matlab toolbox MTEX (R. Hielscher, H. Schaeben, J. Appl. Crystallogr. 2008, 41:1024-1037). Then the PF with any crystallographic direction could be obtained based on the ODF by MTEX.



Figure S2. Initial texture of the TRIP-HEA and the deformed texture after tensile fracture measured by VULCAN. The rolling direction is at the center of the pole figures.

S-5 Discussion on dislocation slip of the FCC

While TRIP persisted till fracture, dislocation slip was identified as the main accompanying deformation mechanism in the FCC when being compared to the deformation behavior of the FCC single-phase HEA. A classic FCC single-phase HEA, FeMnCoCrNi (Yeh J-W, Chen S-K, Lin S-J, et al. Adv Eng Mater. 2004;6(5):299-303), was *in-situ* tensioned for comparison on VULCAN. The single-phase HEA deforms mainly by dislocation slip at room temperature (Yao MJ, Pradeep

KG, Tasan CC, et al. Scripta Mater. 2014;72-73:5-8. Tasan CC, Deng Y, Pradeep KG, et al. Jom. 2014;66(10):1993-2001), thus its intensity evolution reflects a typical texture by slip-induced grain rotation. The diffraction intensity of the FCC in TRIP-HEA is shown in Figure S3 (a). After the triggering of TRIP, both the {111} grains with high tendency in stacking faults and the {200} grains preferring dislocation slip (Kwon EP, Fujieda S, Shinoda K, et al. Mater Sci Eng A. 2010;527(24-25):6524-6532. Gutierrez-Urrutia I, Zaefferer S, Raabe D. Mater Sci Eng A. 2010;527(15):3552-3560) showed similar evolution of intensity that nearly overlapped that of the normalized FCC fraction. It indicated homogeneous transformation in the bulk material. Thus, by normalizing the intensity to the phase fraction, the intensity variation of the FCC in TRIP-HEA can be traced with the transformation effect excluded, as shown in Figure S3 (b). The normalized intensity evolutions of the FCC grains are comparable to those of the single-phase HEA, indicating similar development of deformation texture in the two materials. This is further proved by the final texture of the TRIP-HEA, as shown in supplemental Figure S2, consisting of <111> and <100> fibers that are known as a result of dislocation slip in FCC materials. A simple deformation mechanism of dislocation slip is thus implied for the FCC in TRIP-HEA besides TRIP. It should be noted that the fiber texture of the TRIP-HEA FCC should be weaker than that of the singlephase HEA, as implied by the "delayed-like" intensity change of TRIP-HEA FCC grains in both LD and TD [Figure S3 (b)]. This could be attributed to the work-hardening effect of TRIP on the parent phase.



Figure S3. (a) The intensity evolution of FCC grains in the TRIP-HEA with macro stress in LD and TD. (b) The normalized intensity evolution of FCC grains in the TRIP-HEA in comparison with that in the single-phase HEA. The intensities of the TRIP-HEA were normalized by the FCC fraction: $I_n = \left(\frac{I}{I_0}\right) / \left(\frac{f}{f_0}\right)$, where I_n is the normalized intensity; I and I_0 are the measured intensity under and before deformation, respectively; f and f_0 are the FCC volume fraction under and before deformation, respectively.

S-6 Calculation of phase stress

Based on the rule of mixture, the applied stress is distributed among the FCC and HCP phases with respect to their volume fractions. Furthermore, the average stress response of individual phase can be estimated from the strain of the Rietveld refined cell parameter following the Hooke's law. Since the elastic modulus of phases would vary with texture development during tension, the phase stress is obtained by solving the following equation set with unknown elastic modulus that kept nearly constant in small deformation range.

$$\sigma_i = E_i \varepsilon_i, i = FCC \text{ or } HCP$$

 f_i , σ_i , E_i , ε_i are the volume fraction, stress, elastic modulus and strain of phase *i* respectively. σ is the macro stress. The calculated elastic modulus of both phases is found in consistency with the texture evolution. As shown in Figure S1, the sample developed from the random texture in both phases to the deformed texture consisting of <111> and <100> fibers in the FCC and <10.1> fiber with c-axis tilted away from the loading axis in the HCP. The calculated elastic modulus of the FCC increases with deformation and reflects the high stiffness of <111> fibers especially at large deformation. The calculated elastic modulus of the HCP is relatively low and barely change with deformation, which reflects the lean c-axis distribution along the loading axis during tension.