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Deformation of CoCrFeNi high entropy alloy at large strain

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ABSTRACT

The deformation behavior of an equi-atomic face-centered-cubic CoCrFeNi high entropy alloy was investigated by in-situ neutron diffraction under tensile loading up to 40% applied strain. A three-stage deformation behavior was fully captured by lattice strain and texture evolution. In spite of the chemical complexity, the deformation in CoCrFeNi is dominated by dislocation activities. Analysis of diffraction and microscopy data shows that the deformation progresses from dislocation slip to severe entanglement, where a sharp increase in dislocation density was observed. The neutron diffraction data, corroborated by transmission electron microscopy analysis, provided microscopic insights of the previously reported three-stage hardening behavior.

High entropy alloys (HEAs), defined as alloys containing multiprincipal elements with elemental compositions ranging from 5 at.% to 35 at.% [1], are gaining intensive attention as they represent a novel approach to engineer a new material. Conventional physical metallurgy suggests HEAs should have more complex compositions than traditional alloys, with multiple phases and intermetallic compounds [2]. However, some HEAs show a simple single phase, face-centered-cubic (FCC) structure, with excellent ductility at room temperature [3] and cryogenic temperatures [4]. A number of studies on deformation behavior have been conducted on FCC-based HEAs [2–9], but most of them were performed at ex-situ conditions, or over a very limited region in specimens. Neutron diffraction is an ideal method for characterizing structural changes within a bulk sample owing to its high penetration depth. In-situ neutron diffraction measurements under loading have been conducted to reveal deformation mechanisms in various materials such as stainless steels [10, 11], nano- to ultrafine-grained Ni [12], and HEAs [13, 14]. Wu et al. [13] investigated the structural changes in FCC CoCrFeNiMn HEA during tensile loading at room temperature. The HEA shows strong crystal elastic and plastic anisotropy, and its plasticity comes from the operation of a mixture of edge and screw dislocations. The lattice strain and texture evolutions were found to be similar to those of conventional FCC alloys up to ~12% applied strain. Additionally, Woo et al. [14] studied deformation mechanisms of CoCrFeNiMn alloy at elevated temperatures. The evolution of lattice strain, peak width, and peak intensity indicate that the dominant deformation mode is dislocation slip at 800 K and diffusion-controlled dislocation creep at 1000 K. Although in-situ neutron diffraction has been applied to HEA, the measurements mostly stopped at between 10%~20% applied strain. Evidence indicating a change of deformation mechanisms in HEA has been reported by Liu et al. [3] at large applied strains (up to 40%). A three-stage work hardening behavior was observed in CoCrFeNi HEA at room temperature. The work hardening rate decreases in stage A, followed by an increase in stage B and a decline again in stage C [3]. This phenomenon had also been reported in austenite Cu-Al alloy [15] and stainless steels [16]. The observed work hardening behaviors were attributed to deformation-induced twins [15] and/or martensitic transitions in austenite stainless steels [16],

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mostly based on post-mortem microscopy analysis. In this paper, we report in-situ neutron diffraction measurements on CoCrFeNi HEA to elucidate the deformation mechanisms at large tensile strains.

The as-cast CoCrFeNi alloy was produced by arc-melting method with pure metals (purity ≥ 99.5%wt.) under high purity argon atmosphere and casting into copper mold with an ingot dimension of 60 mm × 15 mm × 15 mm. Tensile specimens were cut from the ingot by electron discharge machine with a gauge dimension of 22 × 4 × 1 mm, and polished down to 2000-grit SiC paper. Transmission electron microscopy (TEM) characterization was performed using FEI Tecnai G^2 Spirit operating at 120 kV. The TEM specimens, cut near the center of the gauge length, were ground down to ~25 μm and punched to ϕ3 mm foils. Subsequent thinning of TEM foils was performed by a twin-jet method using an etching solution of 20%vol. HNO_3, and 80% vol. CH_4O at a voltage of 25 V under a temperature of ~233 K.

In-situ neutron diffraction measurements under tensile load were carried out at TAKUMI beamline [17] in Materials and Life Science Experimental Facilities at J-PARC, Japan. The tensile loading direction is 45° relative to incident neutron beam direction, with two detectors located at ±90° collecting diffraction patterns with the scattering vector in the tensile and transversal directions respectively. CoCrFeNi specimens were first step-loaded in the elastic regime and then continuously loaded in the plastic regime up to 40.9% applied strain with a nominal strain rate of ~2.27×10^{-5}/s. Several unloading steps were undertaken at specified strain values to allow ample time for collecting high-quality data for peak width analysis. The neutron diffraction data were analyzed by Z-Rietveld [18, 19] and Convolutional Multiple Whole Profile (CMWP) [20, 21] software. Neutron diffraction data for CMWP analysis under loading state were binned with a time interval of 30 min, which leads to a strain uncertainty of ~4%.

A representative neutron diffraction pattern of CoCrFeNi HEA is presented in Fig. 1, showing a set of FCC peaks, indicating that the present HEA is of single-phase FCC structure. Lattice strain evolution as a function of true stress is shown in Fig. 2. In the elastic regime, lattice strain increases linearly with applied stress. Values for elastic modulus E_{hkl}, determined from the slope of the linear relationships, are summarized in Table 1. They are in general consistent with that of the quinary CoCrFeNiMn alloy [13]. It can be seen that {200} is the most compliant orientation in the present HEA.

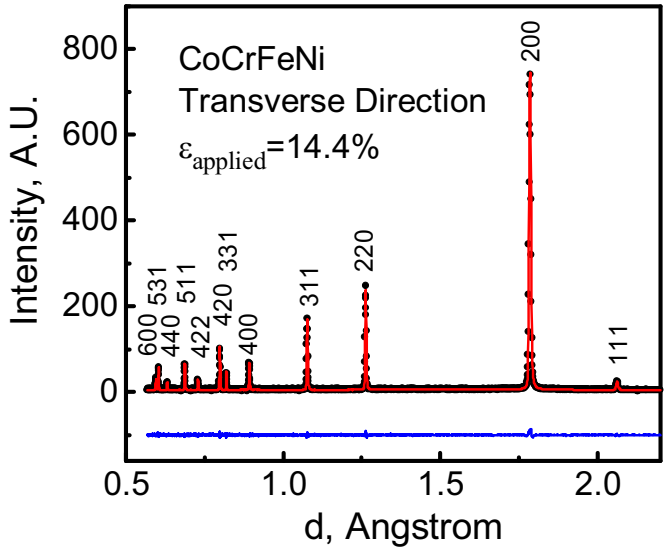

Fig. 1. Neutron diffraction data (solid black circle) of a CoCrFeNi alloy along with CMWP fitted pattern (solid red line) at 14.4% applied strain. The difference (solid blue line) between measured data and fitted pattern is shown at the bottom. Note that the difference plot is shifted by −100 downwards for clarity. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

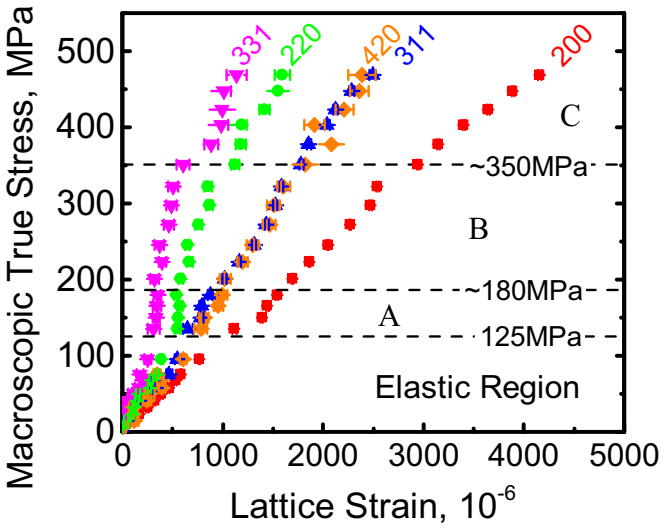

Fig. 2. Lattice strain evolution in the tensile direction as a function of true stress during tensile loading.

The linear relationship between lattice strain and true stress breaks down as macroscopic yield begins, similar to that in stainless steel [22]. In a single-phase material, grains with dislocation slip planes preferentially oriented to the loading axis would deform plastically first, while other grains still deform elastically [23]. The deviation from linear response results from load partitioning dependent on crystallographic orientation, and is determined by both elastic and plastic anisotropy [24]. In the present HEA, {220} and {331} orientations show smaller increase in lattice strain, while {200} orientation shows larger increase, after the macroscopic yielding point. This indicates that {220} and {331} grains yielded first, transferring the load to {200} grains. The value of lattice strain in {200} grains is the largest through the whole deformation regime. {200} grains bear more load and the value of E_{200} is the lowest one among the as-studied orientations. Lattice strains of {311} and {420} show minimal deviation from the linear response. At early deformation stages (Stage A and B in Fig. 2), the lattice strains show continuous increase with applied stress. As stress increases to ~350 MPa (Stage C), lattice strains of all orientations shifted towards a higher value, and milder slopes relative to the previous region are observed, indicating the start of a secondary yield, where all grains start to carry more load at large deformation strains.

Fig. 3(a) reveals multi-stage features of the work hardening behavior in the present HEA. In particular, three deformation regimes were identified in work hardening evolution with the macroscopic true strain, similar to what had been reported in a previous study by Liu et al. [3]. Stage A presents a continuous decrease in work hardening rate, until it reaches a minimum at ~4% true strain. The work hardening rate shows an increase at Stage B, and gradually goes into a plateau until 23.3% true strain. Following Stage B is a moderate work hardening rate decrease till the end of deformation, which is denoted as Stage C in Fig. 3(a).

The evolution of lattice strain and peak intensity corresponds well with the work hardening rate, as shown in Fig. 3(b). The intensity values were normalized with those recorded before loading except for that of {111}, which is very weak in the as-cast condition. The intensity of {111} was instead divided by 15 for a better comparison in the plot. The normalized intensities of all {hkl} orientations are close to 1 before

Table 1
Elastic modulus of different {hkl} of present HEA.

E_{200}/GPa	E_{220}/GPa	E_{311}/GPa	E_{331}/GPa	E_{420}/GPa
120 ± 2	217 ± 11	156 ± 10	269 ± 37	152 ± 14