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Imaging Lattice Distortions in High Entropy Alloys at Multiple Length Scales Using Electron Nanodiffraction and 4D-STEM

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High entropy alloys (HEAs) have attracted tremendous interest in the materials research community due to the vastly expanded phase space created by multi-component alloys and the demonstration of superior mechanical properties. Unlike conventional alloys which use a base element as the matrix, HEAs are composed of multiple principle elements in an equal, or nearly-equal molar fraction. Because of the mixture of atoms of different sizes, severe lattice distortion has been suggested to as one of four major characteristics of HEAs, as well as leading to unconventional dislocation dynamics during deformation [1]. To elucidate the fundamental strengthening mechanisms in HEAs, it is crucial to understand the crystallographic details of lattice distortions. However, the characterization on lattice distortion is very challenging. Previous studies based on pair distribution function analysis by X-ray and neutron diffraction but such analysis only provides the average lattice distortion [2]. The critical information regarding the local structure, the size, and the magnitude of fluctuations in lattice distortions is still missing.

Here, we utilize electron diffraction and 4D-STEM approaches to examine lattice distortions at Å to 10^2 nm scale. The diffraction experiments were carried out at 300 kV in micro-probe mode in a FEI Titan Themis Z scanning transmission electron microscope. Scanning electron nanodiffraction (SEND) is used to detect and visualize lattice distortions. Using the built-in (S)TEM deflection coils in a TEM, a nm-sized electron beam is rastered over the region of interest and a series of diffraction patterns are acquired from the scanned area. Diffraction patterns were energy-filtered by a post-column energy filter (Gatan Quantum ER 965 GIF) and recorded with the CCD camera. The positions of diffraction spots in the recorded patterns are robustly identified using circular Hough transformation (CHT) algorithm [3]. By measuring the distance between diffraction spots, local elastic strain can be calculated and mapped out from the SEND dataset. Figure 1 illustrates the method. The obtained strain maps demonstrate distortion domains of tens of nanometers in size.

We use 4D-STEM to image local crystal rotation. An electron probe of a larger convergence angle is used with a reduced depth of focus. The diffraction spots became disks as shown in Figure 2. 4D-STEM patterns were recorded by a FEI Ceta 16M digital camera. We detect the center of mass (CoM) within a central disk. The results show that CoM varies across the scanned region, indicating that out-of-plane crystal tilts of 1 mrad or less exist in the sample. Fig. 2c displays an example map of crystal tilts, where the colors and the length of arrows represent the tilt orientations and the tilt magnitude, respectively.

Overall, our results demonstrate the different aspects of lattice distortions as seen by different electron probes in diffraction mode. The strain caused by lattice distortions can accumulate over a distance. The accumulated strain is accommodated by inducing local crystal tilts and the formation of small domains, leading to a hierarchical domain structure in HEAs. Thus, scanning electron nanodiffraction and 4D-STEM with different probe convergence angles provide critical insights about complex alloy structure.

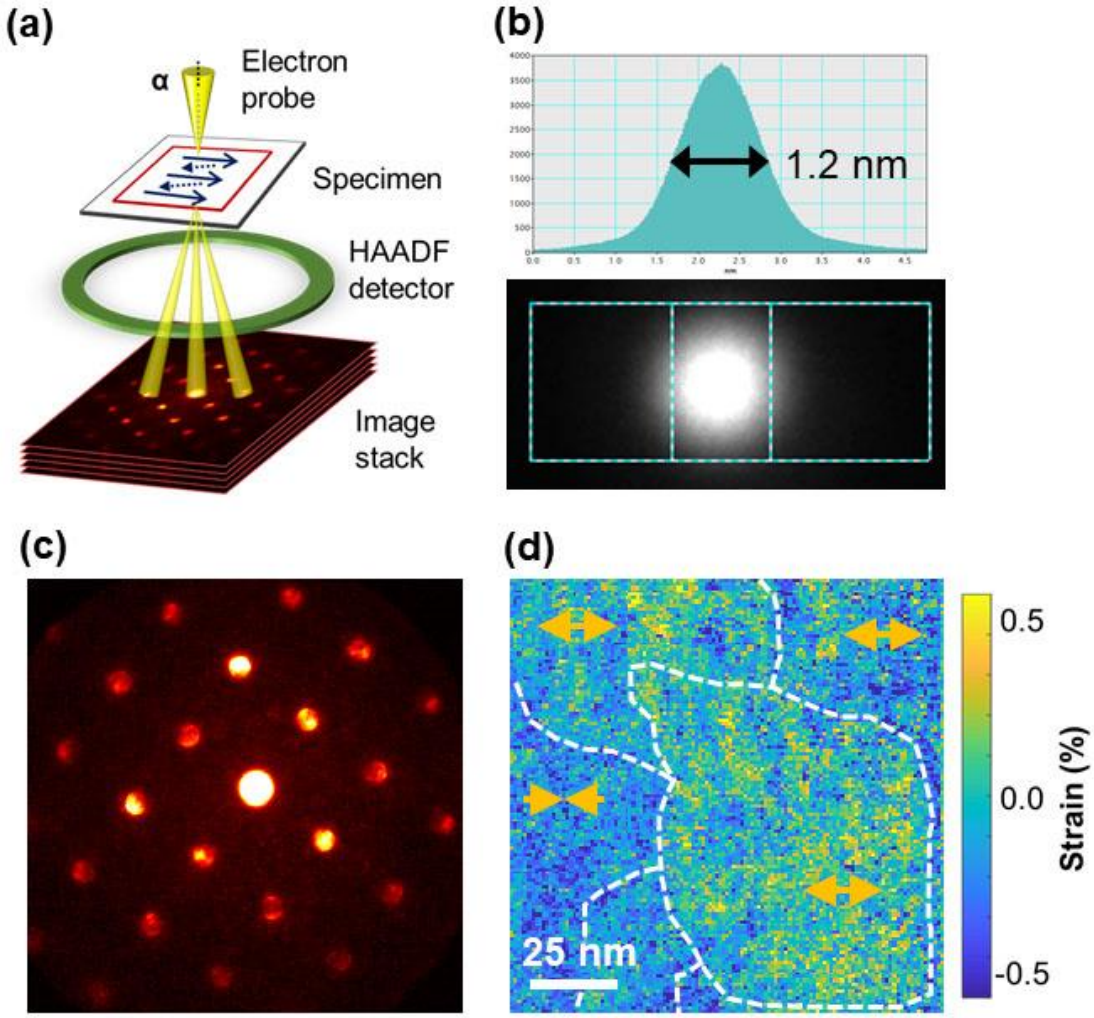

Figure 1. (a) A schematic diagram of SEND and 4D-STEM (b) An electron probe image with a convergence angle of 0.8 mrad as well as the corresponding intensity profile. (c) An example diffraction pattern from a SEND dataset (d) An example map of strain along (100) direction.

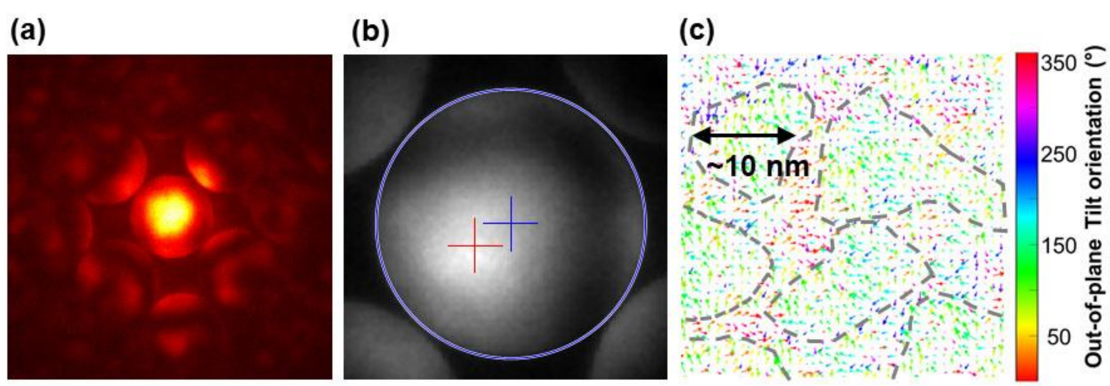

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Figure 2. (a) An example CBED pattern from a 4D-STEM dataset (b) An example of center of mass identification, where the blue cross is the center of a central disk and the red cross is the identified location of center of mass (c) An example map of crystal tilts within the scanned area.

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