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Scanning Nano Beam Electron Diffraction and Applications to Characterization of High Entropy Alloys

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Acquiring the texture information of polycrystals is essential to further understand the properties of many technologically important materials. The conventional technique to characterize crystallite orientation is bright and dark field transmission electron microscopy (TEM), and electron backscatter diffraction (EBSD) performed in a scanning electron microscope (SEM). While EBSD allows a quantitative determination of crystal orientations, it has the disadvantage of relatively low spatial resolution and sensitive to crystal surfaces. TEM has the advantage of high resolution, but diffraction contrast recorded in bright or dark field TEM is difficult to interpret. Therefore, it is required to develop new imaging techniques in TEM in order to detect the crystal orientation and structure information for nanocrystalline materials. Nano beam diffraction (NBD) is a TEM-based diffraction technique that can obtain diffraction patterns using electron probes of few nanometers in diameter [1]. Here, we report a novel technique called scanning NBD to study nanostructures in TEM and its applications to high entropy alloys (HEAs) of $Al_{0.5}CoCrCuFeNi$. This technique is based on automated recording of the electron diffraction patterns on a CCD camera while scanning the area of interest with a nanometer-sized electron beam. The implementation in TEM allows a combined study together with bright or dark-field TEM, which is an advantage over Scanning transmission electron microscopy (STEM) based nanodiffraction [2]. HEAs generally have five or more major metallic elements and possess a very high configurational entropy of mixing. Since solid-solution phases can be more stable than intermetallic compounds or other complex-ordered phases during solidification, HEAs usually possess excellent mechanical properties, thermal stability, and corrosion resistance together with low fabricated costs [3].

In our study, the scanning NBD was performed at a JEOL 2100 Cryo TEM, the electron probe was formed using a condenser aperture of 10 micron and an additional condenser mini-lens[4]. The probe had a diameter of about 3 nm in FWHM. The scanning NBD was performed using a step size of 20 nm, and we recorded 20 x 20 data points in an local area of the size 400nm x 400nm inside the specimen. Fig. 1 shows the bright field image of the HEAs sample, the rows and columns of blue dots mark some of the positions of the electron beam at which electron diffraction patterns were automatically recorded using a CCD camera. Fig. 2 shows one selected area diffraction pattern in a set of scanning electron diffraction data, the annular area marked with white lines was respectively selected for Bright field (BF), Dark field (DF), Annular Bright field (ABF), Annular dark field (ADF) imaging. Fig. 3 shows the ADF image calculated from Fig. 2 and corresponded to Fig. 1. The electron diffraction patterns were respectively selected from each slice in the scanning electron diffraction data.

The BF image obtained from scanning NBD patterns agrees with the direct TEM BF image (Fig. 1), while it also provides diffraction information, ADF, ABF and DF images. Thus, scanning NBD enables a complete structure characterization from which we can further analyze the crystal and structure.

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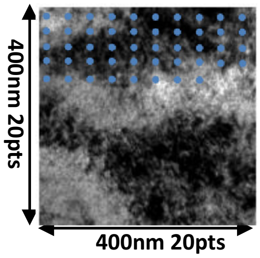

Figure 1. Bright field image of HEAs, the rows and columns of blue dots are a few of the positions of the nano electron beam at which electron diffraction patterns were automatically recorded by the CCD camera.

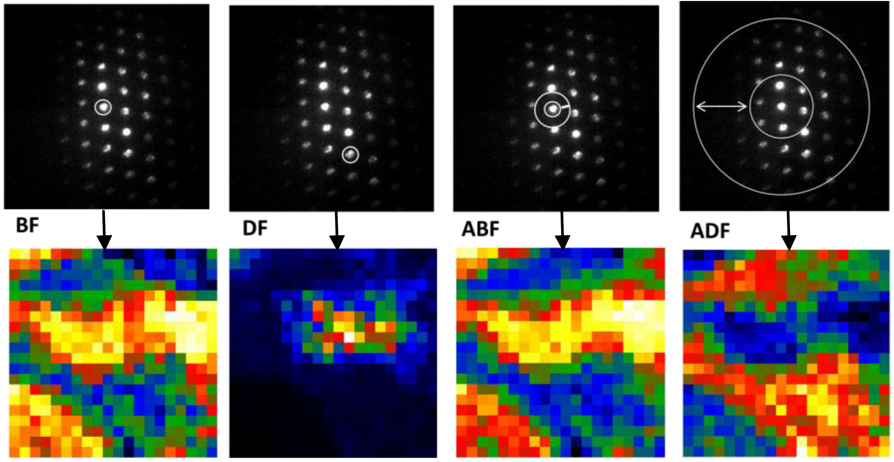

Figure 2. One selected area diffraction pattern in a set of scanning electron diffraction data, the annular area marked with white lines was respectively selected for Bright field (BF), Dark field (DF), Annular Bright field (ABF), Annular dark field (ADF) imaging.

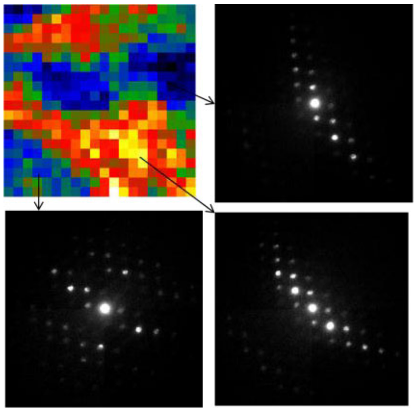

Figure 3. ADF image calculated from Figure 2 and corresponded to Figure 1, The electron diffraction patterns were respectively selected from each slice in the scanning electron diffraction data, from which we can further analyze the crystal orientation and structure.