Examining Site Occupancy in $Co_{1-x}Ni_xO$ Single Crystals using Dynamical *Simulations of EBSD Patterns*

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Electron backscatter diffraction (EBSD) is a widely used technique for characterizing the microstructure of different crystalline materials. The diffracted incident electron beam from the sample surface creates an EBSD (Kikuchi) pattern on the detector, which is then used to index the orientation of the crystal. The standard indexing algorithms predominantly focus on the interplanar angles to extract crystallographic information. There has been work in the literature examining the extraction of lattice parameters as well [1-3]. The simulation and analysis of full, dynamical diffraction patterns may also allow for more detailed analyses that include sub-lattice occupancy.

This paper examines the effects of site occupancy on a well characterized, model material: Co1−xNixO. A series of these single crystals with $0 \leq x \leq 1$ was previously produced and characterized using x-ray diffraction and electron microscopy [4,5]. The whole compositional series possesses the 225 space group with a linear shift in lattice parameter as the ratio of Co:Ni increases on the cation sub-lattice. Kinematic diffraction predicts a straightforward change in bandwidths based upon lattice parameter shifts, but how should the band intensities change for dynamical diffraction? For this set of structures (Co1−xNixO), we generated full dynamical EBSD pattern simulations using the open-source EMsoft software platform [6]. This approach generates a master EBSD pattern, covering the entire Kikuchi sphere using the detailed crystal structure information, including space group, lattice parameters, and atomic positions/occupancy. Incorporating the geometrical parameters of the experimental setup, individual EBSD patterns can be dynamically simulated from this master pattern for any crystal orientation. EBSD patterns were dynamically simulated for the CoO (a=0.4263 nm), NiO (a=0.4181 nm), and Co33Ni67O (a=0.4235) phases at the same orientation (Figs.1a and 1b). Subtracting Figure 1a from 1b illustrates the EBSD pattern variances between NiO and CoO (Figure 1c). The center of the $< 0\bar{1}1 >$ zone axis shows a large difference in internal band intensity due to the change in the Co:Ni ratio on the cation sublattice. Similar features are also visible along the (200) and (222) bands. The band intensity and width of the simulated patterns were investigated by extracting line profiles through three, low-index bands: (200), (022), and (222) around the $< 0\bar{1}1 >$ zone axis. The line profiles were measured at the same positions in all patterns, and Figures 1d-f shows noticeable differences between these patterns due to the variance of the lattice parameters and the site occupancy. The center of the (222) band suggests that the ratio of Co:Ni might be determined from careful analysis of the pattern intensity in this portion of reciprocal space. In this presentation, we will compare the dynamically simulated EBSD patterns to experimentally measured patterns from actual Co1−xNixO single crystals. We will also discuss the potential for assessing the lattice parameter and site occupancy shifts from simulated and experimental EBSD patterns.
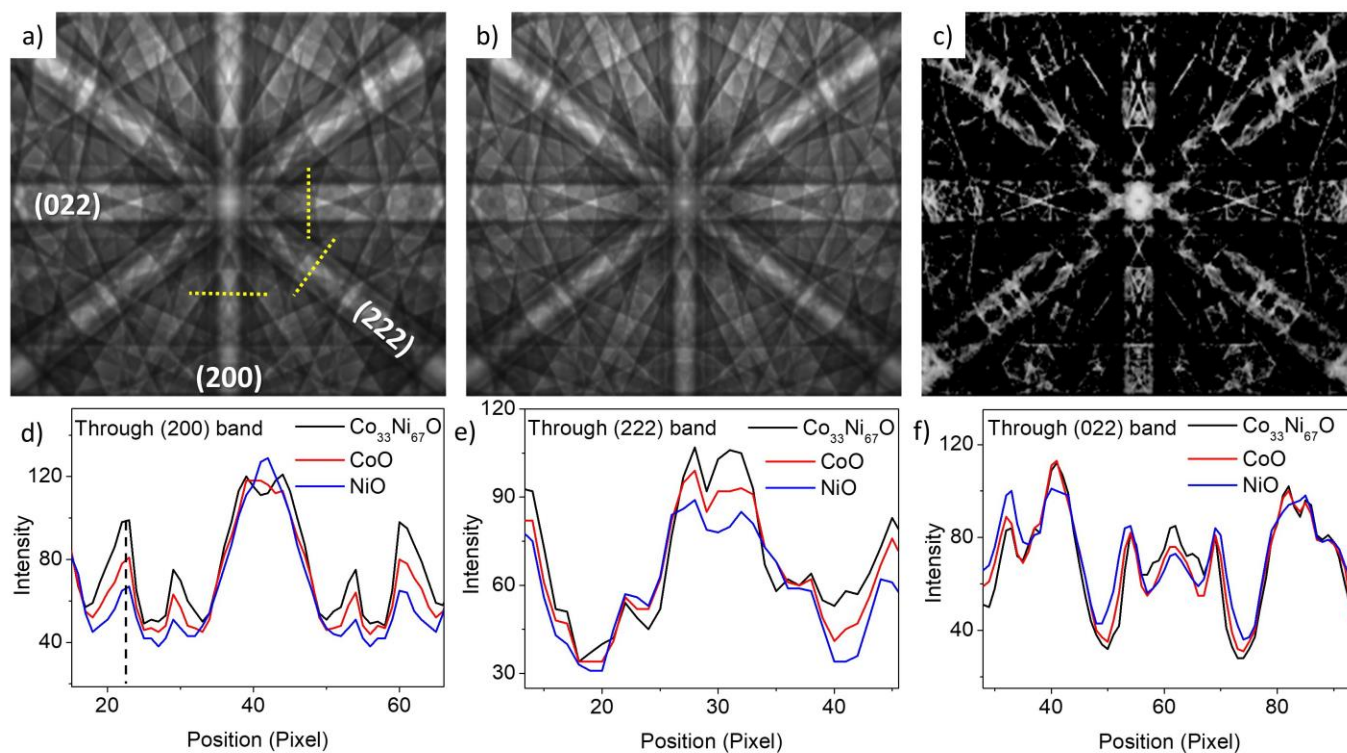
Figure 1. Figure 1. The dynamically simulated pattern by EMsoft for (a) NiO and (b) CoO and (c) shows the pattern difference between them. The band profiles were measured along the yellow dashed lines through the (200) (d), (222) (e), and (022) (f) bands, respectively.

References

References:
[1] L. Li. and M. Han, J. Appl. Cryst. **48** (2015), p. 107.
[2] J.R. Michael and J.A. Eades, Ultramicroscopy **81** (2000), p. 67.
[3] G. Nolze et al., J. Appl. Cryst. **53** (2020), p. 435.
[4] L. N. Brewer et al., J Mater. Res. **17** (2002), p. 760.
[5] L. N. Brewer et al., J Mater. Res. **17** (2002), p. 768.
[6] P.G. Callahan and M. De Graef, Microscopy and MicroAnalysis, **19** (2013), p. 1255.