Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Electronic Supporting Information

Used procedure for Rietveld refinement with X´Pert HighScore Plus is given in table S1. The fitted parameter in every refinement cycle are marked with an x.

Table S1: Used procedure for Rietveld refinement with X´Pert high score plus of as prepared samples.

	1	2	3	4	5	6	7	8	9	10	11	12
Zero shift	x	x	x	x	x	x	x	x	x	x	x	x
Flat background	x	x	x	x	x	x	x	x	x	x	x	x
Coefficient 1	x	x	x	x	x	x	x	x	x	x	x	x
Coefficient 3	x	x	x	x	x	x	x	x	x	x	x	x
Coefficient 3	x	x	x	x	x	x	x	x	x	x	x	x
Scale factor	x	x	x	x	x	x	x	x	x	x	x	x
Lattice parameter a		x	x	x	x	x	x	x	x	x	x	x
Lattice parameter c		x	x	x	x	x	x	x	x	x	x	x
Preferred orientation (001)			x	x	x	x	x	x	x	x	x	x
Anisotropic Broadening (001)			x	x	x	x	x	x	x	x	x	x
W				x	x						x	x
Atomic coordinates z(O)					x	x	x	x	x	x	x	x
V						x	x	x	x			x
B isotropic O							x		x	x	x	x
B isotropic Co								x	x	x	x	x
U										x		

DiFFAX simulation shows shifting and broadening of the (012), (015) and (107) reflection for C19-type (Figure S1 a) and 2H-type (Figure S1 b) faults in the resulting XRPD pattern.

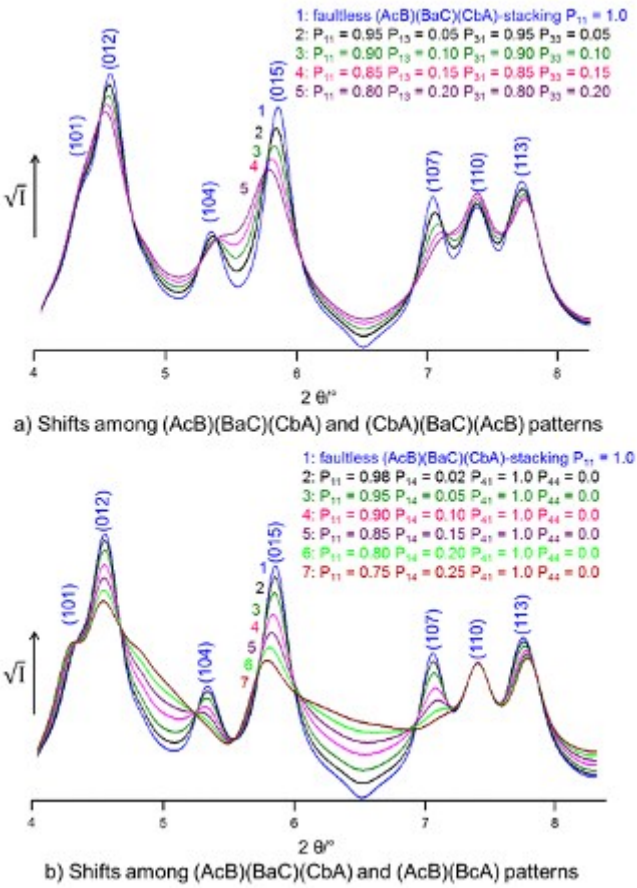

Figure S1: Simulated XRPD-patterns of CoOOH containing variable probabilities (table 6) for transitions between (AcB)(BaC)(CbA)- and a) (CbA)(BaC)(AcB)- and b) (AcB)(BcA)-stacking pattern.

Figure S 2 and S 3 display the best Rietveld fit for a 4 c and 6 c supercell. Applying a comparatively small supercell (4 *c*, 12 layers) leads to a good fit of the powder pattern and to acceptable agreement factors (table 4). A enlargement in *c*-direction of (6 *c*, 18 layers) yields improvements in the fit of the powder pattern and in the final agreement factors.

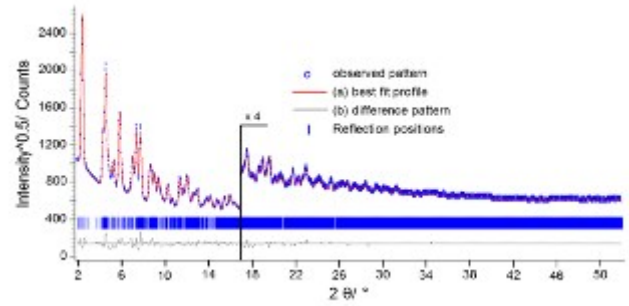

Figure S2: Scattered X-ray intensities CoO(OH), sample CoOOH-O_2 at ambient conditions as a function of diffraction angle 2θ. The observed pattern (circles) measured in Debye-Scherrer geometry, the best Rietveld fit profiles (line) using the method for microstructure refinement[3] with a 4 c supercell and the difference curve between the observed and the calculated profiles (below) are shown. The high angle part starting at 17.0 2θ is enlarged for clarity.

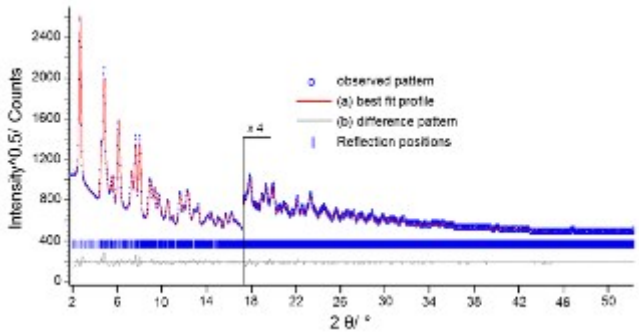

Figure S3: Scattered X-ray intensities CoO(OH), sample CoOOH-O_2 at ambient conditions as a function of diffraction angle 2θ. The observed pattern (circles) measured in Debye-Scherrer geometry, the best Rietveld fit profiles (line) using the method for microstructure refinement[3] with a 6 c supercell and the difference curve between the observed and the calculated profiles (below) are shown. The high angle part starting at 17.0 2θ is enlarged for clarity.