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The quality of precession electron diffraction data is higher than necessary for structure solution of unknown crystalline phases

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Four crystal structures are solved from precession electron diffraction data using either the highest-quality experimental data available or voluntarily deteriorated data. The data quality was lowered by changing the intensity of each individual reflection by as much as a factor of 2, even for strong reflections, while taking care to keep strong reflections strong and weak reflections weak. The crystals have been chosen for their different characteristics, ranging from small to large unit cells, low to high symmetry, and containing heavy and light atoms. In each case the structure solution succeeded with the deteriorated data provided that the completeness of the data was high enough. The accuracy of the atom positions obtained for the cations was comparable to that for the best experimental data. Light-atom positions were sometimes less accurate but still satisfactory for a possible subsequent refinement.

1. Introduction

Because of the ever-increasing importance of nano-structured materials for applications as well as for the understanding of fundamental physics, structure-analysis methods need to cope with smaller and smaller crystals. The traditional methods of single-crystal X-ray or neutron diffraction are limited to samples of well over 1 μm^3. If such samples are not available one has to resort to powder diffraction, which has yielded the structures of many unknown phases. However, even this technique reaches its limits when the powder sample contains several phases and/or highly complex structures. The first case can arise either voluntarily, *e.g.* nanometre-sized precipitates in a matrix, or it can be due to the elaboration method, for example when exploring new synthesis parameters under high-pressure and high-temperature conditions. Complex structures can lead to peak overlap in the powder diffraction pattern which then makes the structure determination very difficult or even impossible. In some cases, these patterns do not even allow determination of the cell parameters of the unknown phase(s).

Recently, electron diffraction has been used to overcome the size limitations with regard to single-crystal diffraction of X-rays and neutrons. In a transmission electron microscope the electron beam can be focused on an individual nanometre-sized particle of a powder and a single-crystal diffraction pattern of the particle can be recorded. Rotating the crystal inside the transmission electron microscope allows exploration of a large part of the reciprocal space of a single particle. However, until recently electron diffraction has been regarded as not suitable for structure solution, since electron diffraction is highly dynamical owing to the strong interaction of electrons with matter. This has changed with the availability of the precession electron diffraction (PED) technique (Vincent & Midgley, 1994).

In this technique, the number of simultaneously excited reflections is reduced with respect to the standard in-zone-axis diffraction and therefore the number of paths for multiple diffraction is greatly reduced. An increasing number of structures are being solved by data obtained by this technique (Gjønnes *et al.*, 1998; Gemmi *et al.*, 2003, 2010; Own, Marks & Sinkler, 2006; Own, Sinkler & Marks, 2006; Weirich *et al.*, 2006; Nicolopoulos & Weirich, 2007) where the data have either been considered to be pseudo-kinematical or the two-beam approximation of dynamical theory (Gemmi & Nicolopoulos, 2007) has been used. In spite of the successes of PED for structure solution the data obtained are not kinematical. This can easily be seen for example in the $[1\bar{1}0]$ zone axis of Si where the 222 reflection, which is forbidden in the kinematical theory for the diamond structure, is not only observed, but can be extremely strong.

In the present work we present the structure solutions obtained from PED data sets whose data quality was voluntarily decreased in order to show that data quality is not an issue for structure solution from PED data. For this study we have chosen four structures which range from simple to very complex, they cover different symmetry classes from monoclinic to cubic and they contain very light atoms, heavy atoms or both. All these structures have been solved from PED data.

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2. Experimental

The PED data have been obtained on a Philips CM300ST operated at 300 kV and equipped with the ‘Spinning star’ precession unit (Nanomegas) and a Gatan Multiscan 1k × 1k CCD camera. The precession angle was chosen for each zone axis in order to avoid overlap of the higher-order Laue zone on the zero-order Laue-zone reflections. Data sets with and without the application of the geometric Lorentz-type correction were tested and for each sample the measured intensities were either interpreted in terms of a pseudo-kinematical approximation ($I \sim |F_{hkl}|^2$) or in terms of a dynamical two-beam approximation ($I \sim |F_{hkl}|$). The data set that gave the most reasonable structure solution (or indeed the only structure solution) was retained. Using these optimized parameter sets all the structures were successfully solved from the experimental data.

In order to test the robustness of the structure solution with respect to poor data quality, we voluntarily decreased the quality of the data available. In each data set optimized as described above, we have ordered the reflections according to their intensities and divided them into three groups: strong, medium and weak reflections. In each group the mean intensity of the reflections in this group has been attributed to each reflection in the group. In this way the intensities of individual reflections have been radically changed, for example the strongest reflection has been attributed an intensity about half the measured value while the weakest reflection of the ‘strong’ group has been attributed an intensity about twice as high as the measured one. In the other reflection groups the changes are comparable. This method of deteriorating the data quality keeps, however, the strong intensities strong, the medium intensities medium and the weak ones weak. The data sets obtained were then used as input for the direct-methods program *SIR2008* of the software package *IL MILIONE* (Burla *et al.*, 2007) in order to solve the structures.

3. Results

3.1. $AgCoO_2$

This hexagonal structure ($a = 2.9$, $c = 12.2$ Å) of space group $P6_3/mmc$ is very simple and contains only three different atom positions (Muguerra *et al.*, 2008). The coordinates of two of these positions are completely determined by the symmetry of the space group and only one positional parameter is free. A total of 39 of the possible 46 independent reflections with $d >$ 0.8 Å obtained from ten zone axes were measured with a precession angle of 1.3° and were used in the structure solution. No Lorentz correction was applied. The internal R values for merging the data from the different zone axes ranged from 7 to 21% with a mean of 15%. The B values obtained from the Wilson plot were negative (−2.83 Å^2 for the as-measured data, −3.41 Å^2 for the deteriorated data) and were automatically set to 0.001 Å^2 by *SIR2008*.

The three reflections with intensity more than 50% of the highest measured intensity were considered ‘strong’, those between 20 and 50% of the highest intensity were considered ‘medium’ (total of 23 reflections), and the rest were the ‘weak’ reflections. Table 1 shows the strongest and weakest reflections of each group with their measured and deteriorated intensities.[1]

Table 1
Reflections of the $AgCoO_2$ phase with their intensities as measured by PED and deteriorated by calculating the mean value in each reflection group.

	h	*k*	*l*	Measured intensities	Deteriorated intensities
Strong	0	0	4	2458.07	1706.70
	0	1	6	1431.05	1706.70
	1	1	0	1230.78	1706.70
Medium	0	0	8	1172.39	757.56
	0	1	2	1138.78	757.56
	0	1	1	1094.65	757.56
	...	...	...	...	...
	0	1	0	556.16	757.56
	1	2	6	508.01	757.56
	0	2	3	500.06	757.56
Weak	0	1	11	458.20	341.26
	0	2	0	457.01	341.26
	0	2	4	443.02	341.26
	...	...	...	...	...
	1	2	3	241.52	341.26
	0	3	3	210.13	341.26
	1	2	0	117.24	341.26

Table 2
Comparison of the atom positions obtained with X-ray data, PED data and deteriorated PED data.

	X-ray diffraction			PED as-measured			PED deteriorated		
Atom	*x*	*y*	*z*	*x*	*y*	*z*	*x*	*y*	*z*
Ag	1/3	2/3	1/4	1/3	2/3	1/4	0	0	0
Co	0	0	0	0	0	0	1/3	2/3	1/4
O	1/3	2/3	0.080	1/3	2/3	0.080	1/3	2/3	0.081

Table 2 summarizes the results of the structure solutions with the different data sets. The atomic positions obtained are almost identical in both cases with the X-ray-refined positions. However, the identification of Ag and Co atoms is inverted in the case of the deteriorated PED data.

3.2. Mn_2O_3

This cubic structure has a rather large unit-cell volume (a = 9.41 Å, V = 833 Å^3) but owing to its high symmetry (space group $Ia3$) only three independent atomic positions are occupied representing four free positional parameters. A total of 126 independent reflections with $d > 0.8$ Å out of a possible 145 were measured from eight zone axes with a precession angle of 4.1°. The mean internal R value for merging the different zone axes was 9%. No Lorentz correction was applied. Ten of these reflections were labelled as strong, 40 as medium and the remaining 76 as weak (Table 3).

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[1] Supplementary data for this paper are available from the IUCr electronic archives (Reference: SH5125). Services for accessing these data are described at the back of the journal.

Table 3
Reflections of the Mn_2O_3 phase with their intensities as measured by PED and deteriorated by calculating the mean value in each reflection group.

	h	*k*	*l*	Measured intensities	Deteriorated intensities
Strong	0	4	4	4112.26	1992
	2	2	2	3934.47	1992
	2	2	6	2457.08	1992
	...	...	...	...	...
	2	3	3	1076.2	1992
	2	6	6	965.54	1992
	1	3	4	951.74	1992
Medium	1	2	7	938.42	465
	1	4	3	896.67	465
	0	2	8	868.78	465
	...	...	...	...	...
	2	3	5	250.92	465
	2	7	3	248.94	465
	3	9	4	248.77	465
Weak	1	2	5	231.73	115
	0	2	2	228.03	115
	1	2	3	226.47	115
	...	...	...	...	...
	1	7	2	43.63	115
	1	9	2	41.4	115
	1	8	5	29.45	115

SIR2008 evaluated the *B* values as 2.69 $Å^2$ (as-measured) and 2.30 $Å^2$ (deteriorated data). The output of the *SIR2008* program for the data set of experimental intensities and the one obtained from the deteriorated intensities both give the correct structure solution. Table 4 gives a comparison of the atomic coordinates obtained in both cases and the positions refined by X-ray diffraction (Inorganic Crystal Structure Database, ICSD No. 61271).

3.3. $Sr_5BiNi_2O_{10}$

This tetragonal structure (a = 5.36, c = 17.52 Å) of space group *I*4/*mmm* is slightly more complex than the two preceding structures, since it contains eight different atom positions with five free positional parameters. A total of 109 independent reflections with $d > 0.8$ Å out of a possible 181 were obtained from eight zone axes which were measured with a precession angle of 2.4° except for the [111] zone which was measured at 3.4°. The mean internal *R* value for merging the different zone axes was 6%. The Lorentz correction was applied in order to solve the structure. The five reflections with intensity more than 50% of the highest measured intensity were considered ‘strong’, those between 20 and 50% of the highest intensity were considered ‘medium’ (total of 12 reflections), and the rest were the ‘weak’ reflections. Table 5 shows the strongest and weakest reflections of each group with their measured and deteriorated intensities. *SIR2008* evaluated the *B* values as 0.77 $Å^2$ (as-measured) and 2.69 $Å^2$ (deteriorated data).

Table 5
Reflections of the $Sr_5BiNi_2O_{10}$ phase with their intensities as measured by PED and deteriorated by calculating the mean value in each reflection group.

	h	*k*	*l*	Measured intensities	Deteriorated intensities
Strong	2	2	0	396739.78	308000
	0	2	0	390011.41	308000
	1	1	4	324453.41	308000
	0	2	6	227599.25	308000
	0	4	8	201198.34	308000
Medium	1	1	8	174238.56	125197
	1	3	4	170048.08	125197
	2	4	0	148549.33	125197
	...	...	...	...	...
	2	2	12	100025.3	125197
	1	3	10	93962.84	125197
	1	1	2	90216.83	125197
Weak	1	3	8	78207.91	13146
	2	4	6	72396.05	13146
	1	5	4	68486.95	13146
	...	...	...	...	...
	3	6	9	157.18	13146
	1	8	1	140.04	13146
	4	5	17	101.73	13146

Table 6 summarizes the results of the structure solutions with the different data sets. In both cases the PED data yielded a structure solution containing all the cation positions but only three of the four oxygen positions. In both solutions the distances between the positions obtained and the X-ray-refined positions are below 0.1 Å, except for one oxygen position where the difference is close to 0.2 Å.

3.4. $PbMnO_{2.75}$

This monoclinic phase, which can be described as derived from the perovskite structure by crystal shear planes (Bougerol *et al.*, 2002), has a low symmetry (space group $A2/m$) and a large unit cell (a = 32.232, b = 3.81, c = 35.671 Å, β = 130°, V = 3374 $Å^3$). Consequently, the number of independent atoms is high and only the 14 Pb positions and the 15 Mn positions were correctly found by PED. These positions represent a total of 56 free positional parameters. Since the *b* parameter is small and the space group only allows atomic positions with $y = 0$ or $y = \frac{1}{2}$

Table 4
Atom coordinates of the Mn_2O_3 phase as obtained by X-ray diffraction (ICSD No. 61271), from the deteriorated data from PED and from the as-measured PED data.

The distances Δ from the positions from PED data to the X-ray data positions are given in Å.

	X-ray diffraction			PED as-measured				PED deteriorated			
Atom	*x*	*y*	*z*	*x*	*y*	*z*	Δ to X-ray	*x*	*y*	*z*	Δ to X-ray
Mn1	0	0	0	0	0	0	0	0	0	0	0
Mn2	1/4	0.27918	0	1/4	0.276	0	0.073	1/4	0.284	0	0.045
O	0.125	0.15	0.912	0.125	0.151	0.915	0.025	0.123	0.144	0.938	0.252

Table 6
Atom coordinates of the $Sr_5BiNi_2O_{10}$ phase as obtained by X-ray diffraction, from the deteriorated data from PED and from the as-measured PED data. The distances Δ from the positions from PED data to the X-ray data positions are given in Å.

	X-ray diffraction			PED as-measured				PED deteriorated			
Atom	*x*	*y*	*z*	*x*	*y*	*z*	Δ to X-ray	*x*	*y*	*z*	Δ to X-ray
Bi	1/2	1/2	0	1/2	1/2	0	0	1/2	1/2	0	0
Sr1	0	0	0	0	0	0	0	0	0	0	0
Sr2	1/2	0	0.150	1/2	0	0.151	0.02	1/2	0	0.151	0.02
Ni	1/2	1/2	0.252	1/2	1/2	0.250	0.04	1/2	1/2	0.250	0.04
O1	1/4	1/4	0.250	1/4	1/4	0.250	0	1/4	1/4	0.250	0
O2	0	0	0.137	0	0	0.127	0.18	0	0	0.133	0.07
O3	1/2	1/2	0.124	1/2	1/2	0.125	0.02	1/2	1/2	0.136	0.21

without creating too short distances, the structure solution can be treated as a two-dimensional problem (Klein, 2005). The single [010] zone axis obtained at a precession angle of 2.4° was used yielding 1965 reflections of type *h*0*l* with $d > 0.8$ Å, 991 of which were independent. In the *h*0*l* plane the data completeness was 92%. No Lorentz correction was applied.

The deteriorated intensity data set was created by putting the reflections with intensities higher than 50% of the maximum intensity in the 'strong' reflections group, those between 20 and 50% of $I_{\max}$ in the 'medium' reflections group, and the rest in the 'weak' reflections (Table 7). *SIR2008* evaluated the *B* values as 2.51 Å^2 (as-measured) and 7.15 Å^2 (deteriorated data). Table 8 reports the *x* and *z* coordinates of the atoms as obtained from the two PED data sets as well as the X-ray powder diffraction refined coordinates (Bougerol *et al.*, 2002), and the distances between the PED and the X-ray positions. There is no significant difference in the accuracies of the structural models obtained from the as-measured and the deteriorated intensities data sets.

Table 7
Reflections of the $PbMnO_{2.75}$ phase with their intensities as measured by PED and deteriorated by calculating the mean value in each reflection group.

	h	*k*	*l*	Measured intensities	Deteriorated intensities
Strong	11	0	−6	364.63	281.23
	3	0	−12	360.61	281.23
	8	0	6	279.51	281.23
	4	0	4	217.23	281.23
	14	0	−18	184.18	281.23
Medium	7	0	−10	147.28	105.33
	14	0	−16	146.46	105.33
	7	0	−8	141.64	105.33
	...	...	...	...	...
	6	0	−22	81.03	105.33
	11	0	−4	79.00	105.33
	17	0	−30	74.36	105.33
Weak	15	0	−2	72.72	2.77
	18	0	−14	72.68	2.77
	4	0	0	68.54	2.77
	25	0	−22	68.18	2.77
	...	...	...	...	...

4. Discussion

The usual procedure of determining an unknown crystal structure goes through two phases. The first step is to solve the structure, *i.e.* to find the positions of most if not all of the atoms in the asymmetric unit. The second step is the refinement of this first model in terms of accuracy of the atomic positions, the occupancy of each position and the thermal displacement parameters. While it is evident that the accuracy of the diffraction data is paramount for the second step, we focus here on the importance of data accuracy for the determination of the first structural model of an unknown phase.

Direct methods proceed to a normalization of the experimental intensities, taking into account the thermal displacement of the atoms. This thermal displacement causes a decrease in intensity of the reflections at high $\sin\theta/\lambda$. After determining a mean thermal displacement parameter by statistical means, the direct-methods programs correct for this attenuation. This correction being well taken into account in the case of X-ray or neutron scattering, its importance in the case of structure solution (and not refinement) from electron diffraction is much diminished. This can be easily understood when considering the remaining multiple scattering in PED. The mean effect of the multiple scattering in this geometry is to transfer intensity from low-angle reflections to high-angle reflections, which is qualitatively similar to the correction for thermal displacements. In some cases this multiple scattering effect can be strong enough so that the geometric Lorentz correction, which also increases the intensities at high angles, not only is not helpful but can actually inhibit the structure solution.

As a consequence, not too much attention should be paid to the results of the Wilson plot in direct methods when considering electron diffraction data. In some cases the thermal displacement parameters can even be determined to be negative (as is the case for the structure of $AgCoO_2$) while the structure solution still works well.

From the results of four different structures that have been solved from PED data one can see that the success of a structure solution does not depend on the data set used. The deteriorated intensities are always sufficient to solve the structures. This observation is true for very different crystals having small unit cells ($AgCoO_2$, V = 89 Å^3) or large unit cells ($PbMnO_{2.75}$, V = 3374 Å^3), high symmetry ($AgCoO_2$:

Table 8
Atom positions obtained for $PbMnO_{2.75}$ from PED data (deteriorated or as-measured) compared to the positions refined against X-ray powder diffraction data (Bougerol *et al.*, 2002).

The atom names in the first column are those given by Bougerol *et al.* (2002). The columns Δ show the distances between the PED positions and the X-ray-refined positions in Å.

X-ray diffraction			PED as-measured				PED deteriorated		
	x/*a*	*z*/*c*	Atom	*x*/*a*	*z*/*c*	Δ	*x*/*a*	*z*/*c*	Δ
Pb10	1.000	0.000	Pb1	1	0	0.00	1	0	0.00
Pb7	0.273	0.482	Pb2	0.2641	0.4829	0.31	0.26370	0.4822	0.31
Pb4	0.466	0.528	Pb3	0.4717	0.5349	0.19	0.47086	0.5353	0.20
Pb2	0.788	0.445	Pb4	0.7915	0.4471	0.09	0.79201	0.4461	0.11
Pb9	0.937	0.061	Pb5	0.9427	0.0688	0.21	0.94145	0.0691	0.23
Pb6	0.325	0.411	Pb6	0.3216	0.4142	0.20	0.32083	0.4121	0.16
Pb3	0.413	0.103	Pb7	0.4135	0.1024	0.03	0.41519	0.1054	0.07
Pb1	0.151	0.120	Pb8	0.1502	0.1200	0.03	0.15044	0.1225	0.10
Pb8	0.114	0.365	Pb9	0.1145	0.3630	0.08	0.11092	0.3599	0.14
Pb5	0.373	0.342	Pb10	0.3798	0.3470	0.17	0.37521	0.3418	0.08
Pb11	0.641	0.324	Pb11	0.6405	0.3270	0.12	0.63870	0.3251	0.10
Pb14	0.916	0.314	Pb12	0.9067	0.3122	0.26	0.90119	0.3067	0.37
Pb12	0.826	0.703	Pb13	0.8302	0.7054	0.10	0.83248	0.7065	0.16
Pb13	0.422	0.269	Pb14	0.4320	0.2766	0.26	0.42672	0.2758	0.19
Mn11	0.690	0.260	Mn1	0.6957	0.2600	0.18	0.68905	0.2586	0.04
Mn15	0.952	0.243	Mn2	0.9567	0.2434	0.14	0.95021	0.2420	0.04
Mn10	0.578	0.057	Mn3	0.5777	0.0662	0.33	0.57880	0.0654	0.28
Mn7	0.640	0.496	Mn4	0.6327	0.4910	0.18	0.63107	0.4896	0.22
Mn4	0.226	0.228	Mn5	0.2105	0.2312	0.58	0.21333	0.2263	0.37
Mn6	0.323	0.586	Mn6	0.3132	0.5842	0.28	0.31724	0.5851	0.17
Mn9	0.521	0.135	Mn7	0.5226	0.1392	0.12	0.52645	0.1398	0.15
Mn14	0.839	0.549	Mn8	0.8404	0.5471	0.10	0.84095	0.5470	0.12
Mn3	0.898	0.483	Mn9	0.8955	0.4721	0.34	0.89330	0.4708	0.36
Mn5	0.252	0.657	Mn10	0.2607	0.6593	0.24	0.26607	0.6593	0.41
Mn13	0.788	0.622	Mn11	0.7872	0.6212	0.02	0.78838	0.6196	0.09
Mn1	−0.008	0.172	Mn12	−0.003	0.1780	0.16	0.00402	0.1780	0.30
Mn2	0.948	0.397	Mn13	0.9489	0.3968	0.03	0.95076	0.4008	0.10
Mn8	0.525	0.295	Mn14	0.5300	0.2849	0.48	0.52515	0.2914	0.13
Mn12	0.725	0.194	Mn15	0.7334	0.1963	0.23	0.73680	0.1914	0.44

$P6_3/mmc$; Mn_2O_3: $Ia3$) or low symmetry ($PbMnO_{2.75}$: $A2/m$) with or without heavy elements. Even the most complex structure we have solved up to now by PED data representing 56 free positional parameters for the cations was successfully solved with the deteriorated data.

The accuracy of the cation positions found by the deteriorated PED data compared to the X-ray-refined positions is as good as for the as-measured PED data. This remains true in the very complex structure $PbMnO_{2.75}$, where only the cation positions were found. The accuracy of the oxygen positions is sometimes less good when using the deteriorated PED data. However, it is never worse than 0.25 Å off the X-ray-refined position. This is sufficient for a subsequent X-ray refinement provided good X-ray powder diffraction data are available.

The situation changes when the available data set contains fewer reflections than the sets used in this work. Using, for example, 126 independent reflections with less redundancy obtained from five zone axes of Mn_2O_3 is not sufficient for solving the structure with the deteriorated data set, although the as-measured intensities do yield the correct structural model (Klein, 2011).

The procedure we used to deteriorate the intensities of the PED data sets changed the measured intensities of individual reflections by up to a factor of 2 in some cases, but did not mix strong reflections with medium or weak ones. For structure solution with direct methods this is important since the invariant triplets are based on strong reflections. So as long as the strong reflections remain strong and can be identified as strong due to the presence of the weak reflections in the data set, the correct phases can be attributed and the structure can be solved.

This is precisely the achievement of the precession technique for electron diffraction (Marks & Sinkler, 2003; Own, Marks & Sinkler, 2006; Eggeman *et al.*, 2010; Sinkler & Marks, 2010). By suppressing many of the paths for multiple diffraction the strong reflections remain strong, even though they are still affected by dynamical diffraction effects. The experimental data are still not kinematical and it is often not clear if a pseudo-kinematical approximation is better than a two-beam dynamical approximation for the determination of the structure-factor amplitude from the measured intensities. The intensity rank of the reflections, however, is sufficiently preserved.

In contrast, in classical on-zone-axis diffraction, multiple diffraction weakens strong reflections and increases the intensities of weak reflections up to a point where it becomes difficult to distinguish between them. Accordingly, data sets from this classical electron diffraction do not solve the structures presented above.

5. Conclusion

We have shown that voluntarily deteriorated PED intensities mimicking low-quality experimental data are sufficient for solving unknown crystal structures with satisfactory accuracy provided that the data set contains a large number of reflections. Experimental effort should therefore be put into the acquisition of as many reflections as possible as opposed to fewer reflections with higher accuracy.

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