Poster Presentations

[MS16-P02] Structure refinement based on absorption corrected RED data

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Structure determination based on electron diffraction data is of great progress during the last few years. Different electron crystallographic methods were employed in difficult structure determination cases, and different shortage of X-ray crystallography such as size limitation, stacking default and overlapping problem can be overcome by electron crystallography. For instance, rotation electron diffraction (RED)[1] which is a powerful automatic 3D electron crystallography techinique, can collect single crystal X-ray diffraction like data and vigorously used in structure determination for nano-size crystals. More than 100 different structures were solved based on this method until now. For most of the cases, after getting the structure model against on RED data, Retiveld refinement against powder X-ray diffraction or density function theory (DFT) calculation were frequently used for structure refinement to confirm the structure model. The reason for using these methods to do the structure refinement other than to do the refinement based on the electron diffraction data is due to the drawbacks of the electron diffraction data at the moment. These are represented by the high R_{int} value for the original data and poor refinement R_1 value compared to single crystal X-ray diffraction data. Although the reasons caused the drawbacks are not well understood until now, it was discussed that the poor data quality could be contributed by the dynamic effect, the absorption effect, inelastic scattering, beam damage problems, etc. All these effects in electron crystallography are ambiguous problems at the moment, and need to be further studied in the future. For some of these effects such as dynamic effect, a lot of debates were raised regarding to its influence to the intensity accuracy, and microscopists have different views regarding to it. Some of the TEM experts even claimed that due to the dynamic effect, the intensity from electron diffraction is totally mean less. Of course, this is not true since numerous structures can be solved by no matter manually or automatic electron diffraction data based on the recently reported results, although the achieved R value is not so reasonable compared to X-rays. For dynamical effects, Lukáš Palatinus *et al*[2] did the full dynamical refinement based on manually collected PED data, and it shows the improvement on data quality and lower *R* value, the structure and occupancy can be refined accurately. For Lorenz correction, Holger Klein[3] did some work on it; However, The results are quite ambiguous, he claimed that in some of the cases the Lorenz correction is helpful for structure determination but some are not. All of these two corrections were done based on PED data, which was believed to have more accurate intensity compared to SAED data. And also, no one studied the absorption effect which is an important factor influenced the accuracy of the intensity a lot as known in X-ray crystallography. Here, we used rotation electron diffraction data based on SAED mode TEM studied a novel plate-like new vanadium oxide, which shows a size of 220nm ×320nm × 400nm and believed to have anisotropic absorption effects. The structure can be easily solved and refined in SHEX97 program[4] based on the original data, and all the atoms shows elongated temperature factors along the same direction. The original data shows a R_{int} value of 31%. Numerical absorption correction method was employed for absorption correction for the original data, after the absorption correction, the data achieved a R_{int} of 18% and all the anisotropic temperature factors for the atoms in the refined structure improved to be more isotropic. This study indicated the absorption effects in the RED data significant influenced the data quality and refinement results, and the data quality can be

improved by using proper absorption correction.

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Keywords: structure refinement; rotation electron diffraction; absorption correction