Oblique Incidence Correction and Instrument Error Adjustment

Yu-Chun Chuang (莊裕鈞), Lai-Chin Wu (吳來錦), Chou-Fu Sheu (許朝富), I-Jui Hsu (許益瑞), and Yu Wang (王瑜)

Department of Chemistry, National Taiwan University, Taipei, Taiwan

Using tunable and high energy synchrotron source to do the electron density study becomes more universal. The powerful x-ray source provides a quicker and more efficient experiment. However, accuracy electron density data should include not only high resolution but also great data precision. High resolution data can be achieved easily by using synchrotron high energy source and large CCD detector or image plate. The large redundancy and high precision reflection data is believed to get more accuracy electron density. In our experience, the data precision R_{int} value is usually lower than 5% even 3% by using in house x-ray diffractrometer system. But the data quality measured in BL12B2 is not good as in house data. We suppose there are two factors could lead the intensity inaccuracy (bad oblique correction and instrument error).

For oblique incidence correction, two methods are used. The first way is to measure the transmittance of phosphor and the other one is to monitor the reflection intensity at different position of the CCD detector. The phosphor in Quamtum-4R CCD is optimized 1 Angstrom. We bought the phosphor form ADSC Company to measure the transmittance in different energy range. The chemical composition of phosphor is Gd_2O_2S doped with heavy atom Tb. The thickness of the phosphor is about 0.015mm-0.025mm. The perpendicular transmission was measured at BL01C in NSRRC using 0.5, 0.6, 0.7, 0.8, 0.9 and 1.0 Å radiation. The perpendicular transmission is 0.818, 0.696, 0.554, 0.488, 0.369 and 0.236, respectively. Based on the transmission results, even 1 Angstrom wavelength there is more than 20% photon will pass through the phosphor layer directly. The correction factor K is shown in Fig. 1. The second method, we measured multi-reflections at different incident angle (α angle) range and monitored the intensity variation of each reflection at different position of the CCD detector to get the calibration curve.

In our past synchrotron reflection data, the R_{int} value is about 6-8%. It is too large to get accuracy electron density. After correcting the oblique incidence problem, the data precision is still not good enough. It probably has some instrument error in our system. We find the spindle axis do not rotate perfectly. For instance, after centering the crystal, the crystal position still moves as the phi axis rotating. It has to be fixed before measuring any reflection data. In order to get better data, the whole instrument should be aligned well. It is believed that if all the calibration and sensitivity files are included to the integration process, we will get better electron density data.

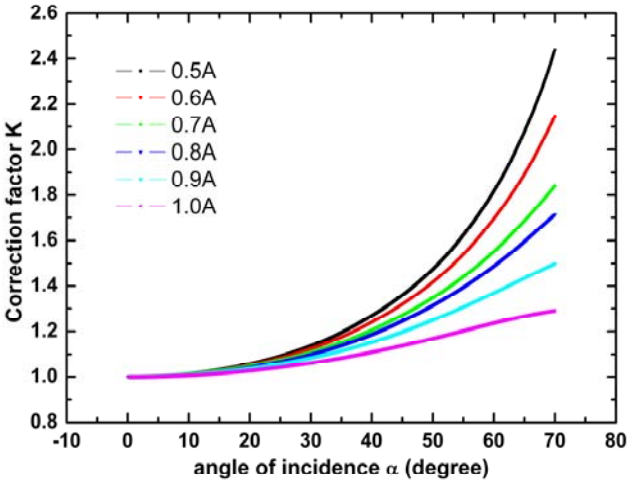

Figure 1. Correction factor K for different wavelengths, such that $I_{corr}=I_{obs}/K$