A.8: Data reduction procedure for correction of geometrical factors in the analysis of specular x-ray reflectivity of small samples

For small samples, the modification of the XRR profile by the geometrical factors manifesting due to profile and size of the beam and the size of the sample is significant. Geometrical factors extend till spill over angle which is often greater than critical angle for small samples. Since geometrical factor is a smoothly varying function and extends beyond critical angle, it is impossible to determine the spill over angle from XRR profile of small samples. We have shown by comparing the normal XRR profile of a small sample with the XRR profile taken with a surface contact knife edge on the same sample, that the spill over angle can be determined. Figure A.8.1 shows the configuration of knife edge placement. Unlike hitherto used methods which have drawbacks, this is a self consistent method for data reduction.

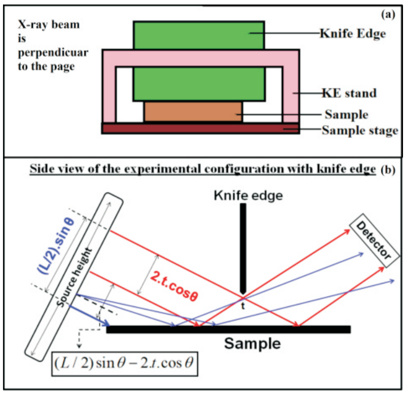

Fig. A.8.1: (a) Schematic drawing of the knife edge assembly and (b) side view to the knife edge configuration with ray diagram.

The XRR curves with and without knife edge simultaneously are used for data reduction, which can be represented as:

$$E(\theta) = g(\theta)\,R(\theta)\,C_1 \quad (1)$$

$$E_{KE}(\theta) = g_{KE}(\theta)R(\theta)C_2 \quad (2)$$

$E(\theta) = g(\theta)$ and $R(\theta)$ are the experimental curve, geometrical factor and Fresnel reflectivity respectively. The constants C_1 and C_2 preserve the information of the incident intensity in the mathematical manoeuvring of the problem.

$$\text{Hence,}\quad \frac{E(\theta)}{(C_1\ /C_2)} = E_{KE}(\theta);\quad \text{for } \theta > \theta_{so} \quad (3)$$

Following equation 3, $E(\theta)$ is divided by a constant so that the resultant function $E'(\theta)$ matches with $E_{KE}(\theta)$ at higher angles. The constant is identified to be C_1/C_2. The angle at which $E'(\theta)$ and $E_{KE}(\theta)$ start matching is identified as the spill over angle (Figure A.8.2(a) to (c)).

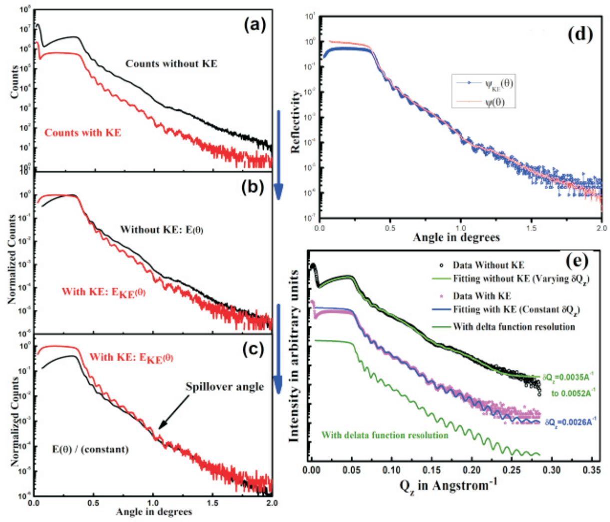

Fig. A.8.2: (a)-(c) Sequence of steps to calculate spill over angle (d) normalized curves after data reduction (e) fitting of both the curves.

Knowing spill over angle and sample size we generate a Gaussian beam profile that obeys the relation: $\sin\theta_{so}=1.274\ (T/L)$, where T and L are the width of the beam and sample size, respectively. This is used to calculate the geometrical factor $g(\theta)$. The normalized data are calculated as follows for without knife edge:

$$\Psi(\theta) = E(\theta)/[\ g(\theta)\,R(\theta1)C_1]; \quad (=R(\theta)/\ R(\theta1)); \quad (4)$$

Similarly for XRR with knife edge we find:

$$\Psi_{KE}(\theta) = E_{KE}(\theta)/[g_{KE}(\theta)R(\theta1)C_2] = E_{KE}(\theta)/[R(\theta1)C_2] \quad [\because g_{KE}(\theta)\approx 1] \quad (=R(\theta)/\ R(\theta1)) \quad (5)$$

Plots of $\Psi(\theta)$ and $\Psi_{KE}(\theta)$ and the fitting of the curves are shown in Figure A.8.2(d) and 2(e), respectively. This is a heuristic solution that is supported by intuitive modelling of the geometrical factor. It is applied on Er_2O_3 thin film samples prepared by PLD where a change of lattice parameter was noticed below 5×10^{-3} mbar of O_2 partial pressure. This change must be associated with a change of density that was successfully detected by performing the data reduction proposed here. This small change in density (~6.2%) occurring precisely when there is a change of lattice parameter would have gone unnoticed using the conventional methods. More details can be found in *J. Appl. Cryst., 51, 1295-1303 (2018)*.

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