A simple recipe to create three-dimensional reciprocal space maps

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Combinations of advanced X-ray sources and zero-noise detector of enormous dynamic range have significantly increased the opportunity of mapping the reciprocal space of crystal lattices. It is particularly important in the design of new devices based on epitaxial thin films. In this work, we present a simple approach to the three-dimensional geometry involved in plotting reciprocal space maps, along with an example of computer codes to allow X-ray users to write their own codes. Experimental data used to illustrate an application of the codes are from antimony telluride epitaxial film on barium difluoride substrate.

Keywords: Synchrotron X-ray diffraction, nanostructured devices, area detectors, epitaxial films, topological insulators

I. INTRODUCTION

Acquisition of X-ray diffraction data with area detectors has become extremely popular nowadays. However, data analysis in the three-dimensional (3D) reciprocal space can be quite challenging for new users of X-ray facilities doted with this capability. It is particularly relevant to study nanostructured devices made of different materials and crystalline domains. In this very short work, we put together the most straightforward information that a beginner X-ray user needs to know for processing their diffraction data into 3D plots of the reciprocal space, that is, to create a 3D reciprocal space map (3D-RSM).

II. RSM BASIC 3D GEOMETRY

As schematized in Fig. [1] the spatial position of pixel \( n_0 m_0 \) in the lab reference frame is \( \vec{R}_d \), and of any other pixel given as \( \vec{R}_{nm} = \vec{R}_d + \vec{r}_{nm} \). In terms of the sample-detector vector \( \vec{D} \),

\[
\vec{R}_{nm} = D \hat{s}_d - (n - n_0) p \hat{y}_d + (m - m_0) p \hat{x}_d
\]

(1)

where the unit vectors are written as a function of the instrumental angles \( 2\theta_d \) and \( \varphi_d \), as also depicted in Fig. [1].

\[
\hat{s}_d = \cos(2\theta_d)[\cos(\varphi_d) \hat{x} + \sin(\varphi_d) \hat{y}] + \sin(2\theta_d) \hat{z},
\]

\[
\hat{x}_d = \sin(\varphi_d) \hat{x} - \cos(\varphi_d) \hat{y}, \quad \text{and}
\]

\[
\hat{y}_d = -\sin(2\theta_d)[\cos(\varphi_d) \hat{x} + \sin(\varphi_d) \hat{y}] + \cos(2\theta_d) \hat{z}.
\]

(2)

The directions from the origin of the lab reference frame where the X-rays hit the sample, \([X, Y, Z] = [0, 0, 0]\), to the matrix of pixels define the set of scattering vectors

\[
\vec{k}_{nm} = \frac{2\pi}{\lambda} \frac{\vec{R}_{nm}}{|\vec{R}_{nm}|}.
\]

(3)

as well as the accessible \( Q \)-space

\[
\vec{Q}_{nm} = \vec{k}_{nm} - \vec{k} = \frac{2\pi}{\lambda} \left( \frac{\vec{R}_{nm}}{|\vec{R}_{nm}|} - \hat{x} \right)
\]

(4)

for the detector at a \((2\theta_d, \varphi_d)\) position. In the lab frame, this accessible \( Q \)-space is just a section of the Ewald sphere, and it remains constant as long as the direct beam direction, X-ray energy, and detector position are kept fixed during RSM data acquisition. On the other hand, the accessible \( Q \)-space in the crystal’s frame of reference has a volume that depends on how the crystal’s rotation is performed for data acquisition.

In a simple rocking curve of \( hkl \) reflection, the 3D-RSM technique aims to map the intensity distribution around a selected \( hkl \) node. Its diffraction vector

\[
\vec{Q}_{hkl} = Q_{hkl} [-\sin(\theta) \hat{x} + \cos(\theta) \hat{z}]
\]

(5)

is coplanar with the \( xy \) plane of incidence, and it rotates around the \( y \) axis with angle \( \alpha \) as defined in Fig. [2]. The accessible \( Q \)-space in Eq. (4) is projected in the crystal’s frame \( \hat{x}', \hat{y}', \hat{z}' \), as follow

\[
\Delta Q_x(n, m) = \vec{Q}_{nm} \cdot \hat{x}'
\]

\[
\Delta Q_y(n, m) = \vec{Q}_{nm} \cdot \hat{y}'
\]

\[
\Delta Q_z(n, m) = \vec{Q}_{nm} \cdot \hat{z}'.
\]

(6)

To perform the dot products, these vectors have to be written in a common frame, that is

\[
\hat{x}' = \cos(\theta) \hat{x} + \sin(\theta) \hat{z}
\]

\[
\hat{y}' = \hat{y}
\]

\[
\hat{z}' = -\sin(\theta) \hat{x} + \cos(\theta) \hat{z}
\]

(7)

resulting in

\[
\Delta Q_x(n, m) = (\vec{Q}_{nm} \cdot \hat{x}) \cos(\theta) + (\vec{Q}_{nm} \cdot \hat{z}) \sin(\theta)
\]

\[
\Delta Q_y(n, m) = \vec{Q}_{nm} \cdot \hat{y}
\]

\[
\Delta Q_z(n, m) = (\vec{Q}_{nm} \cdot \hat{z}) \cos(\theta) - (\vec{Q}_{nm} \cdot \hat{x}) \sin(\theta)
\]

(8)
FIG. 1. Lab reference frame $\hat{x}$, $\hat{y}$, and $\hat{z}$. Incident wavevector $\vec{k} = (2\pi/\lambda)\hat{x}$ (along axis $x$) for X-ray of wavelength $\lambda$. Spatial position of pixel $nm$ at the $N_y \times N_x$ pixel array of the detector area given as $\vec{r}_{nm} = (n_0 - n)p\hat{y}_d + (m - m_0)p\hat{x}_d$ where $p$ is the pixel size, and $n_0m_0$ stands for the pixel hit by the direct beam when $2\theta_d = 0$ and $\varphi_d = 0$.

FIG. 2. Crystal reference frame $\hat{x}'$, $\hat{y}'$, and $\hat{z}'$ that undergoes a $\theta$ rotation around $y$ axis, $\hat{y}' = \hat{y}$. Diffraction vector $\vec{Q}_{hkl} = (2\pi/d_{hkl})\hat{z}'$ where $d_{hkl}$ is the atomic interplane distance of reflection $hkl$.

as three matrices of reciprocal space coordinates of the pixels for each $\theta$ position of the rocking curve to which there is one matrix of intensity $I_{nm}$ from the detector image. To generate the 3D-RSM, it is necessary to compute the $\vec{Q}_{nm} \cdot \hat{\alpha}$ dot products ($\alpha = x, y, z$). According to Eq. (1), these products are constant values as a function of the $\theta$ rotation:

\[
\vec{Q}_{nm} \cdot \hat{x} = \frac{2\pi}{\lambda} \left( \frac{\vec{R}_{nm} \cdot \hat{x}}{|\vec{R}_{nm}|} - 1 \right),
\]

\[
\vec{Q}_{nm} \cdot \hat{y} = \frac{2\pi}{\lambda} \left( \frac{\vec{R}_{nm} \cdot \hat{y}}{|\vec{R}_{nm}|} \right), \quad \text{and (9)}
\]

\[
\vec{Q}_{nm} \cdot \hat{z} = \frac{2\pi}{\lambda} \left( \frac{\vec{R}_{nm} \cdot \hat{z}}{|\vec{R}_{nm}|} \right).
\]

III. COMPUTER CODES RECIPE

Basic input data for coplanar ($\varphi_d = 0$) 3D-RSM plots:

1. Wavelength $\lambda$.
2. Elevation of the detector arm: $2\theta_d$.
3. Sample-detector distance $D$.
4. Pixel size $p$.
5. Number of pixels $N_y$ (index $n = 1, 2, \ldots, N_y$, running from top to bottom) and $N_x$ (index $m = 1, 2, \ldots, N_x$, running from left to right) along $\hat{y}_d$ and $\hat{x}_d$ directions, respectively, as defined in Eq. (1).
6. Central pixel indexes $n_0m_0$ determined by setting both detector angles to zero.
FIG. 3. (a) 3D-RSM of 006 reflection of Sb$_2$Te$_3$ thin film on BaF$_2$ (111). (b) Projection of the RSM in the $xz$ plane of incidence. See §IV for details. DESY Photon Science, PetraIII beamline P08 (proposal I-20211588).

7. 1D array $\mathbf{th}(1:N)$ containing the $N$ experimental $\theta_j$ angles of the rocking curve where $j = 1, 2, \ldots, N$.

8. 2D arrays of detector images $\mathbf{I}_j(1:Ny,1:Nx)$ with the intensity data linked to each $j$ step.

For the $\theta$ rotation axis as defined in Fig. 2, the ideal detector position is $2\theta_d = 2\arcsin(\lambda/2d_{hkl})$ and $\phi_d = 0$. Matrices to be created, common to every $j$ step, are:

1. 1D arrays $\mathbf{sd}(1:3)$, $\mathbf{xd}(1:3)$, and $\mathbf{yd}(1:3)$ as the unit vectors in Eq. (2). Positions 1, 2, and 3 as the $x$, $y$, and $z$ componentes.

2. 2D arrays $\mathbf{R}_x(1:Ny,1:Nx)$, $\mathbf{R}_y(1:Ny,1:Nx)$, and $\mathbf{R}_z(1:Ny,1:Nx)$ as the matrices of $xyz$ components of the pixel positions $\tilde{R}_{nm}$ in real space as in Eq. (1), and a 2D array $\mathbf{R}(1:Ny,1:Nx)$ as the matrix of their modulus, $|\tilde{R}_{nm}|$.

3. 2D arrays $\mathbf{Q}_x(1:Ny,1:Nx)$, $\mathbf{Q}_y(1:Ny,1:Nx)$, and $\mathbf{Q}_z(1:Ny,1:Nx)$ as the matrices of $xyz$ components of the pixel position $\tilde{Q}_{nm}$ in reciprocal space as defined in Eq. (9).

Matrices to be created subsequently as a function of the rocking curve angle $\theta_j$ are:

1. 3D arrays $\mathbf{DQ}_x(1:Ny,1:Nx,1:N)$, $\mathbf{DQ}_y(1:Ny,1:Nx,1:N)$, and $\mathbf{DQ}_z(1:Ny,1:Nx,1:N)$, exactly as shown in Eq. (8), for instance $\mathbf{DQ}_x(:, :, j) = \mathbf{Q}_x(:, :) \cos(\theta_j) + \mathbf{Q}_z(:, :) \sin(\theta_j)$.

2. 3D array $\mathbf{A}(1:Ny,1:Nx,1:N)$ with all concatenated images from the detector, that is $\mathbf{A}( :, :, j) = \mathbf{I}_j( :, :)$.

When these four 3D arrays: $\mathbf{DQ}_x$, $\mathbf{DQ}_y$, $\mathbf{DQ}_z$, and $\mathbf{A}$ are stored in the computer’s memory, available MatLab or Python packages can be used to plot the RSM. Next section shows an example using MatLab. General scripts to create 3D-RSMs by using either MatLab or Python are available for download at [GitHub: Reciprocal space maps generator](https://github.com/ReciprocalSpaceMaps).

### IV. MATLAB CODES AND EXAMPLE

```matlab
function ReciprocalSpaceMaps

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Basic input info %
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
wl = 1.2401446; % wavelength (Angstrom)
twoth_d = 14.037; % elevation of detector arm (deg)
phi_d = 0; % azimuth of detector arm (deg)
D = 1005.2; % sample-detector distance (mm)
p = 0.172; % pixel size (mm)
Ny = 195; % number of pixels along $yd$ (vertical in Fig. 1)
```

When these four 3D arrays: $\mathbf{DQ}_x$, $\mathbf{DQ}_y$, $\mathbf{DQ}_z$, and $\mathbf{A}$ are stored in the computer’s memory, available MatLab or Python packages can be used to plot the RSM. Next section shows an example using MatLab. General scripts to create 3D-RSMs by using either MatLab or Python are available for download at [GitHub: Reciprocal space maps generator](https://github.com/ReciprocalSpaceMaps).
\(\text{Nx} = 487; \) % number of pixels along \(\text{xd}\) (horizontal in Fig. 1)
\(n_0 = 95; m_0 = 230; \) % central pixel (hit by the direct beam)
\(nb = 87; mb = 36; \) % dead pixel
\text{folder}=\text{'BiTe22040_00118\p100k';} % location of image files
\text{fnamepng} = \text{'BiTe22040L06';} % output file name
\text{sclfactor} = [.6 .45 .3]; % isoimage curve levels
\text{Az} = -60; \text{El} = 20; % viewing angle of 3D plot
\text{tifFiles} = dir([\text{folder} '\*.tif']); % number of image files
\text{dth} = 0.01; % increment in the rocking curve angle \(\theta\) (deg)
\text{th0} = 4.01852; % initial \(\theta\) value (deg)
\text{th} = \text{th0}+(0:N-1)*\text{dth}; % array of \(\theta\) values

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Common matrices to all \(\theta\) values
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
\text{sd} = \text{[cosd(twoth_d)*[cosd(phi_d) sind(phi_d)] sind(twoth_d)];}
\text{xd} = \text{[sind(phi_d) -cosd(phi_d) 0];}
\text{yd} = \text{[-sind(twoth_d)*[cosd(phi_d) sind(phi_d)] cosd(twoth_d)];}
\text{ry} = \text{-(1:Ny) - n0)*p; % vertical pixels, 1st at the upper left corner}
\text{rx} = \text{([1:Nx] - m0)*p; % horizontal pixels}
\text{Rx} = \text{D*sd(1) + repmat(ry*yd(1),1,Nx) + repmat(rx*xd(1),Ny,1);} \text{Ry} = \text{D*sd(2) + repmat(ry*yd(2),1,Nx) + repmat(rx*xd(2),Ny,1);} \text{Rz} = \text{D*sd(3) + repmat(ry*yd(3),1,Nx) + repmat(rx*xd(3),Ny,1);}
\text{R = sqrt(Rx.*Rx + Ry.*Ry + Rz.*Rz);} 
\text{aux} = \text{((2*pi/wl);}
\text{Qx = aux*(Rx./R - 1);}
\text{Qy = aux*Ry./R;}
\text{Qz = aux*Rz./R;}

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% 3D matrices
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
\text{DQx = zeros(Ny,Nx,N); DQy = DQx; DQz = DQx; A = DQx;}
\text{for j=1:N}
\text{DQx(:,:,j) = Qx.*repmat(cosd(th(j)),Ny,Nx) + Qz.*repmat(sind(th(j)),Ny,Nx);} \text{DQy(:,:,j) = Qy;}
\text{DQz(:,:,j) = Qx.*repmat(-sind(th(j)),Ny,Nx) + Qz.*repmat(cosd(th(j)),Ny,Nx);} \text{Ij = double(imread([folder '\tifFiles(j).name])); Ij(nb,mb) = 1;}
\text{A(:,:,j) = Ij;}
\text{end}
\text{A(A<1)=1;}
\text{Amax=max(max(max(A)));}
\text{whalf = log10(0.5*Amax);}
\text{Z = log10(A);}
\text{hf1 = figure(1);}
\text{clf}
\text{set(hf1,'InvertHardcopy','off','Color','w')}
\text{patch(isosurface(DQx,DQy,DQz,Z,sclfactor(1)*whalf),'FaceColor','red',...}
\text{'EdgeColor','none','FaceAlpha',1);} \text{hold on}
\text{patch(isosurface(DQx,DQy,DQz,Z,sclfactor(2)*whalf),'FaceColor','green',...}
\text{'EdgeColor','none','FaceAlpha',.22);} \text{patch(isosurface(DQx,DQy,DQz,Z,sclfactor(3)*whalf),'FaceColor','blue',...}
\text{'EdgeColor','none','FaceAlpha',.04);} \text{hold off}
\text{view(3)} \text{daspect([1 1 1])}
\text{axis tight}
\text{camup([0 0 1])}
\text{camlight}
\text{lighting phong}
\text{set(gca,'Color',[0.97 0.97 0.97],'Box','on',...}
\texttt{camlight}
\texttt{axis tight}
\texttt{view(Az,El); grid;}
\texttt{set(gca,'XLim',[-.05 .05],'YLim',[-.05 .05])}
\texttt{print('-dpng','-r300',[fnamepng '.png'])}

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% 2D projection
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Qz x Qx
hf2 = figure(2); clf
set(hf2,'InvertHardcopy','off','Color','w')
XX=zeros(Ny,N);
ZZ=XX;
II=XX;
for \texttt{n=1:N}
\texttt{S = zeros(Ny,1);} 
\texttt{for \texttt{m=1:Nx}}
\texttt{S = S + A(:,m,n);} 
\texttt{end}
\texttt{II(:,n) = S;} 
\texttt{end}
\texttt{IImax = 0.5*max(max(II));}
\texttt{II(II>IImax) = IImax;} 
\texttt{for \texttt{n=1:N}, XX(:,n) = DQx(:,m0,n); end}
\texttt{for \texttt{n=1:N}, ZZ(:,n) = DQz(:,m0,n); end}
\texttt{contourf(XX,ZZ,log10(II),12)}
\texttt{set(gca,'FontName','Arial','FontSize',10,'LineWidth',1);} 
\texttt{axis image}
\texttt{axis tight}
\texttt{c = colorbar;}
\texttt{c.Label.String = 'log(counts)';}
\texttt{xlabel('\Delta Q_x (\text{\textmu m}^{-1})')}
\texttt{ylabel('\Delta Q_z (\text{\textmu m}^{-1})')} 
\texttt{print('-dpng','-r300',[fnamepng 'QxQz.png'])}

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