High Pressure Collaborative Access Team

A scanning angle energy-dispersive X-ray diffraction (SA-EDXD) technique for studying the structure of materials at high pressure in the diamond anvil cell

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Outlines

Experimental setup

Data collection procedure

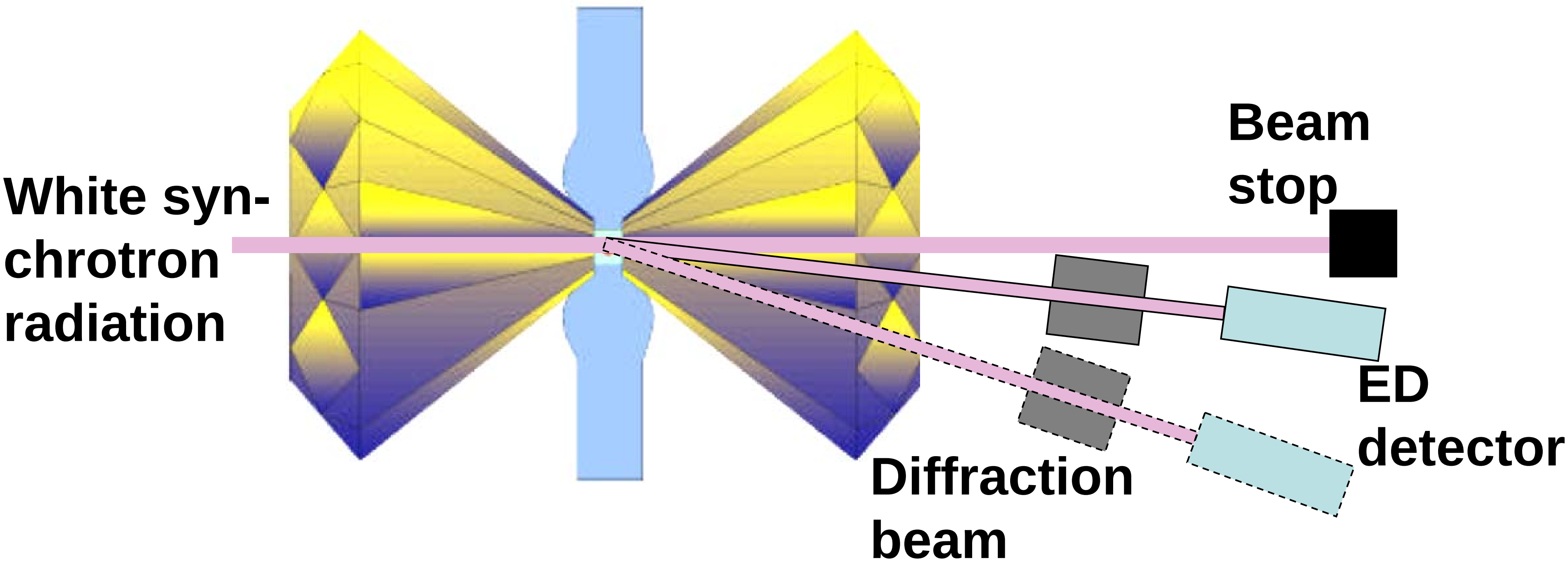
Data Analysis software package

Comparison with routine monochromatic ADX

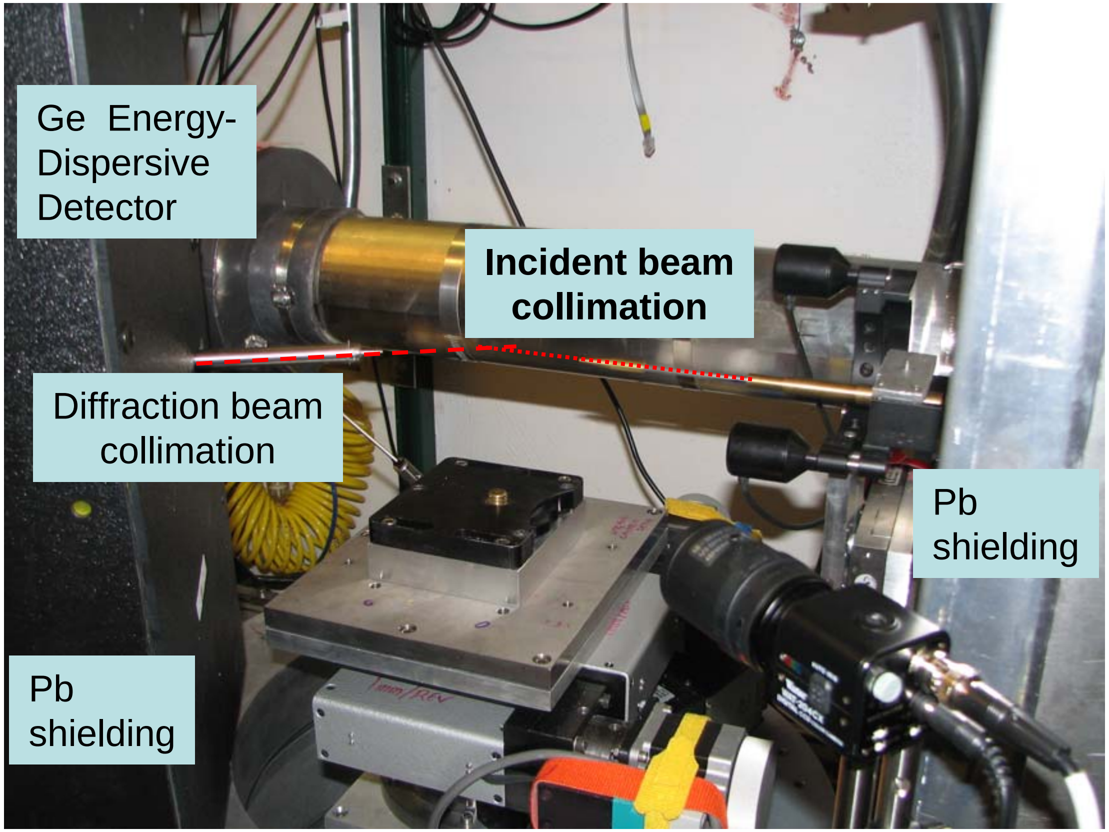
Application with Crystalline and amorphous materials

Summary

Experimental setup



HPCAT 16BMB station

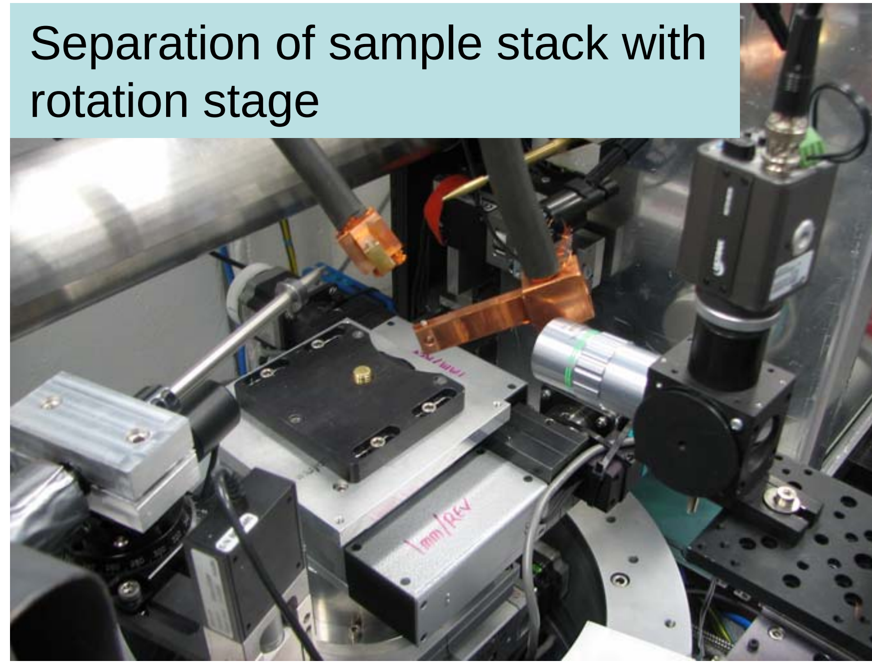


Experimental parameters:

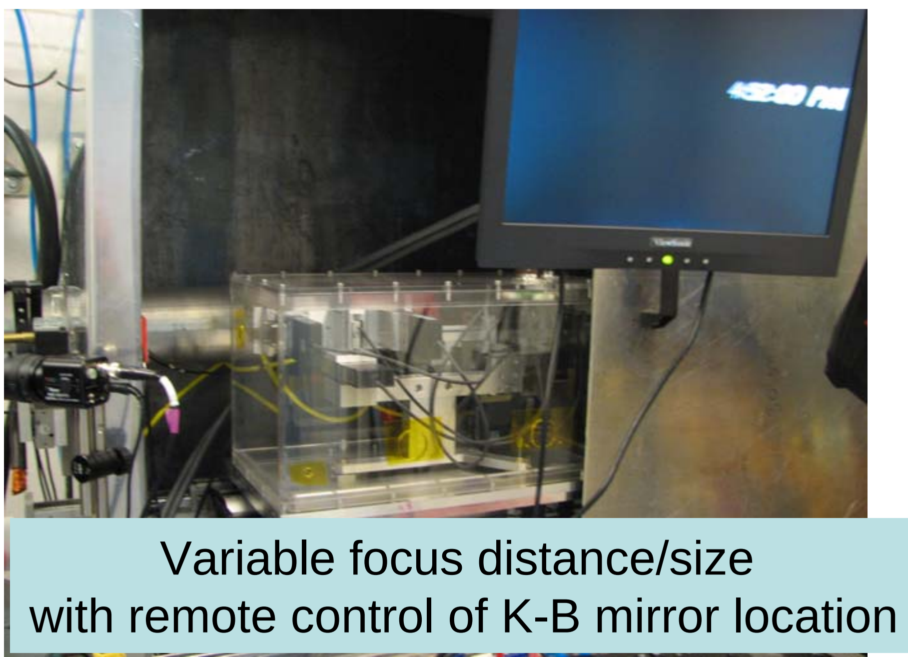
Energy range: 1 – 100 keV
Focus beamsize: 5-50 um
2theta range: 2-40 degrees
eccentricity: ~5 um
Detector to sample distance: ~350 mm
Typical horizontal acceptance: 30-100 um (0.08-0.28 mrad)
Typical vertical acceptance: 150-500 um (0.42-1.4 mrad)
Tip horizontal opening: 30um – 1000 um

Support facilities

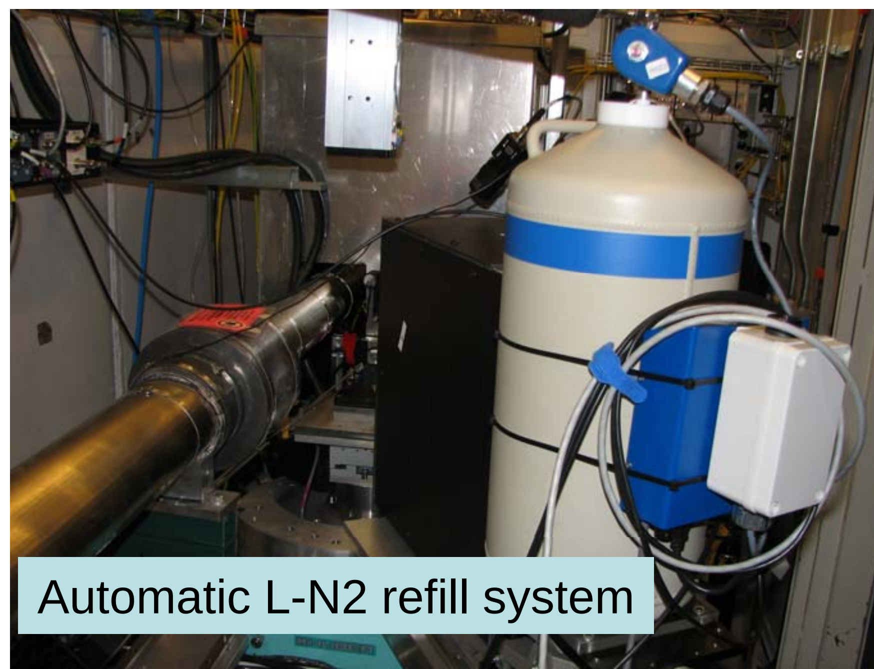
Air-pad and granite for smooth 2 theta change



Separation of sample stack with rotation stage



Variable focus distance/size with remote control of K-B mirror location



Automatic L-N2 refill system

Data collection procedure

Take EDXD patterns at each 2θ angle, with increment Δ2θ;

In order to achieve the regular Angle-dispersive x-ray diffraction resolution, only a coarse 2 theta scan is needed;

Only scan of 2 theta in horizontal direction is available

…

Data Analysis package

Intensity correction:

$$I^{obs}(Q) = PAG[I^{coh}(Q) + I^{inc}(Q) + I^{mul}(Q) + I^{back}(Q)]$$

P: the polarization factor, *A:* the absorption factor, *G:* the geometric factor, I^{coh}, I^{inc}, and I^{mul}: the coherent, incoherent, multiple scattering intensities, I^{back} is the background scattering from the surrounding materials

Diffraction volume correction:

$$\frac{\sin(2\theta)}{w_i w_d}, \quad when\ L \le D$$

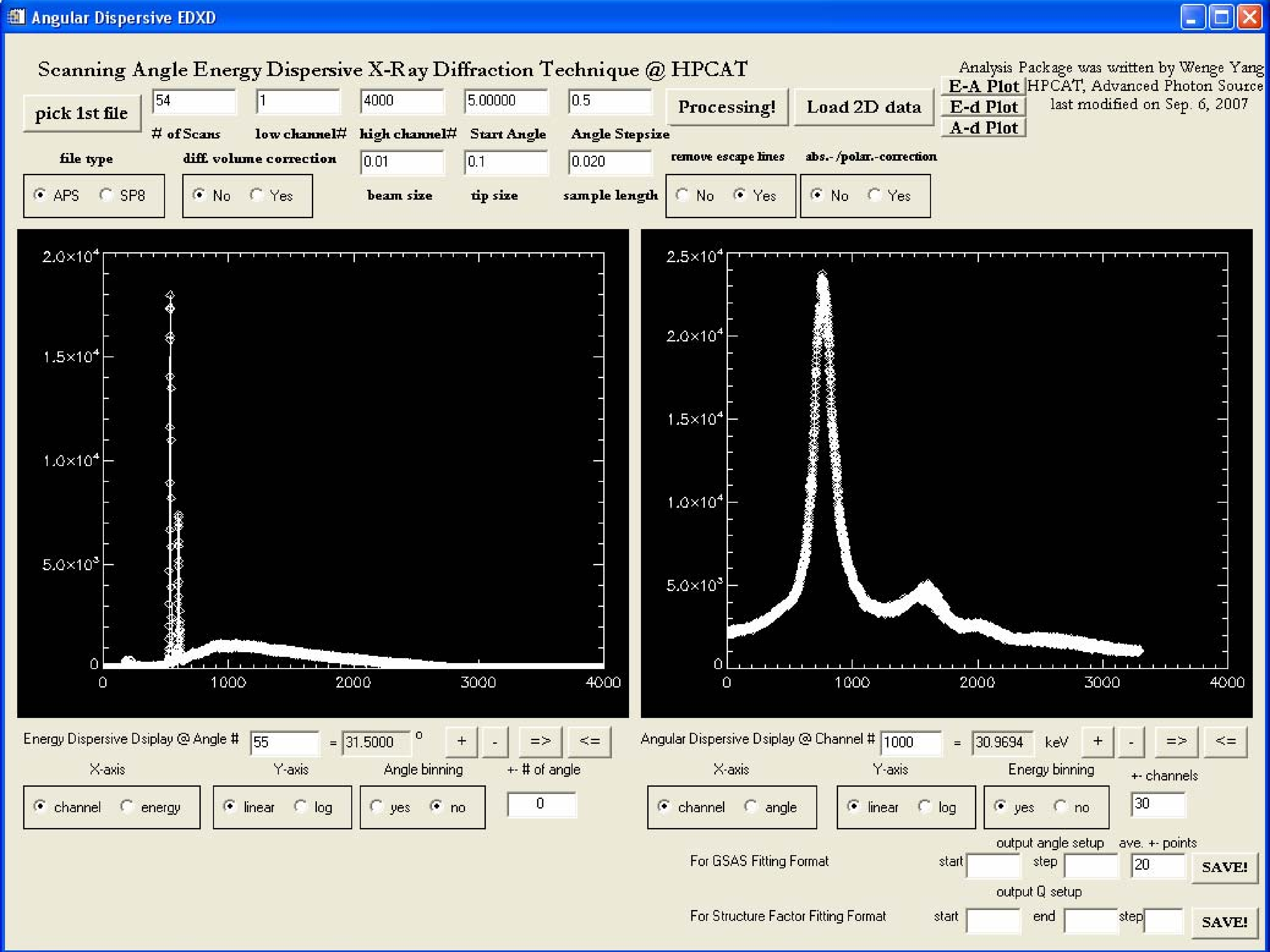
$$L = \frac{w_i}{\tan(2\theta)} + \frac{w_d}{\sin(2\theta)}$$

$$\frac{1}{\dfrac{w_i w_d}{\sin(2\theta)} - \dfrac{(L-D)^2 \tan(2\theta)}{4}}, \quad when\ L > D$$

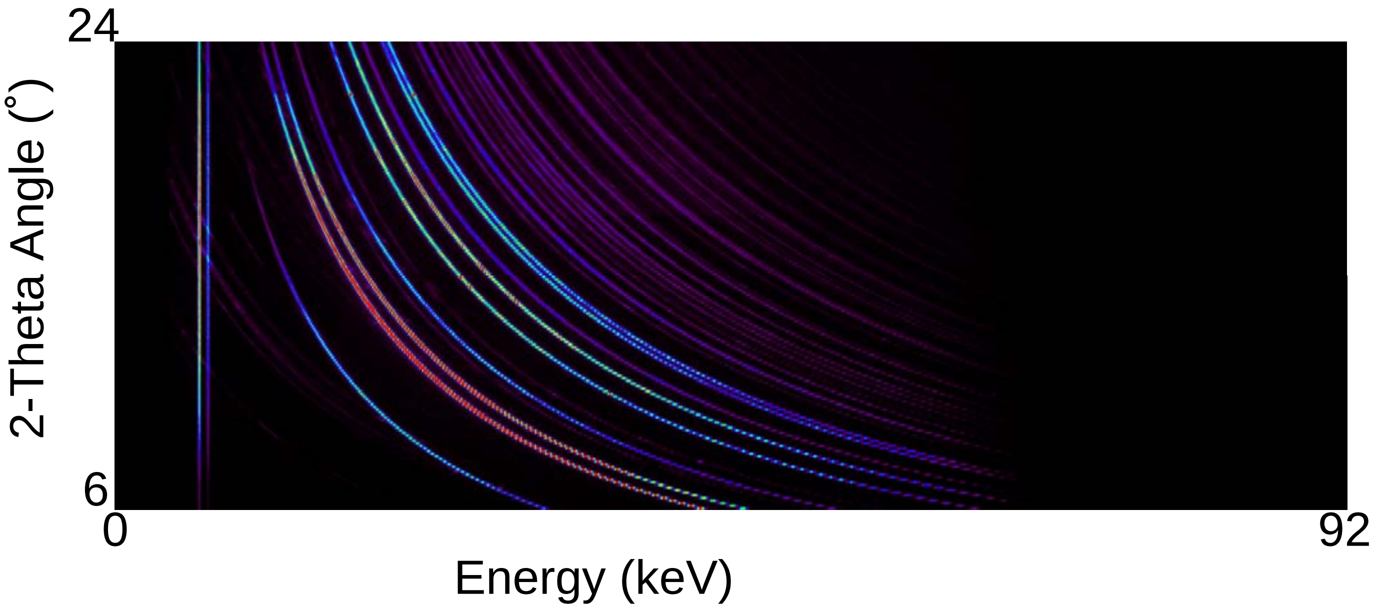
w_i, w_d are widths of incident and diffraction beam

L diffraction length along incident beam

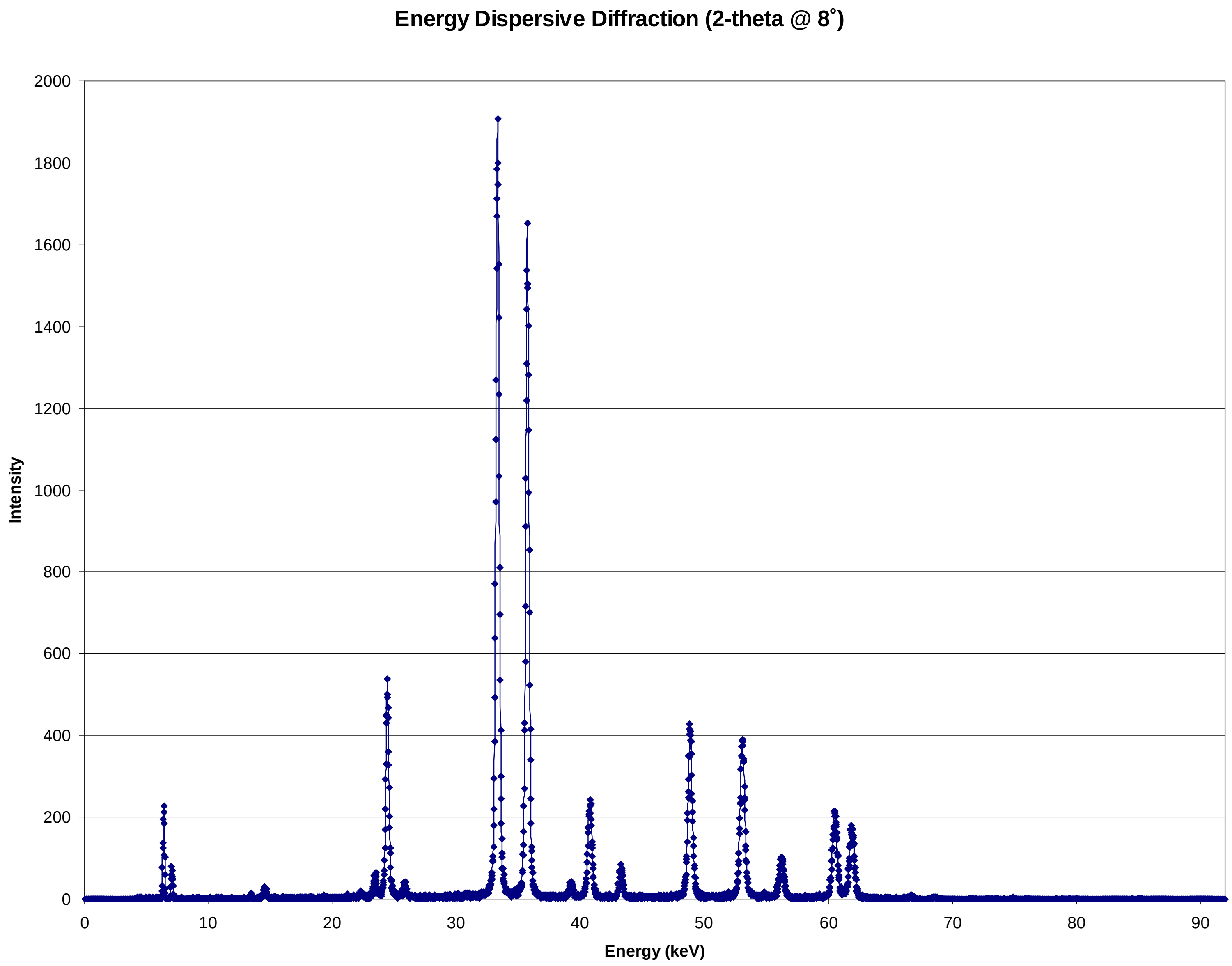
D sample diameter


Angular Dispersive EDXD
Scanning Angle Energy Dispersive X-Ray Diffraction Technique @ HPCAT
Analysis Package was written by Wenge Yang
HPCAT, Advanced Photon Source
last modified on Sep. 6, 2007
E-A Plot
E-d Plot
A-d Plot
pick 1st file
54
# of Scans
1
low channel#
4000
high channel#
5.00000
Start Angle
0.5
Angle Stepsize
Processing!
Load 2D data
file type
APS
SP8
diff. volume correction
No
Yes
0.01
beam size
0.1
tip size
0.020
sample length
remove escape lines
No
Yes
abs.- /polar.-correction
No
Yes
Energy Dispersive Dsiplay @ Angle #
55
= 31.5000
Angular Dispersive Dsiplay @ Channel #
1000
= 30.9694
keV
X-axis
channel
energy
Y-axis
linear
log
Angle binning
yes
no
+- # of angle
0
X-axis
channel
angle
Y-axis
linear
log
Energy binning
yes
no
+- channels
30
output angle setup
ave. +- points
For GSAS Fitting Format
start
step
20
SAVE!
output Q setup
For Structure Factor Fitting Format
start
end
step
SAVE!

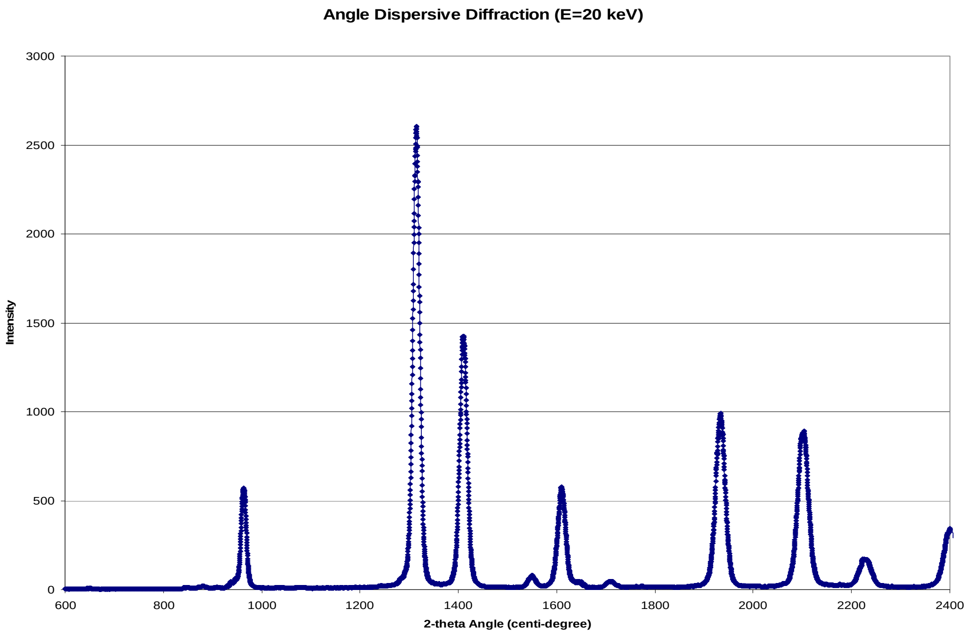
SA-EDXD scan on Fe_2O_3 at 25.4 GPa in DAC

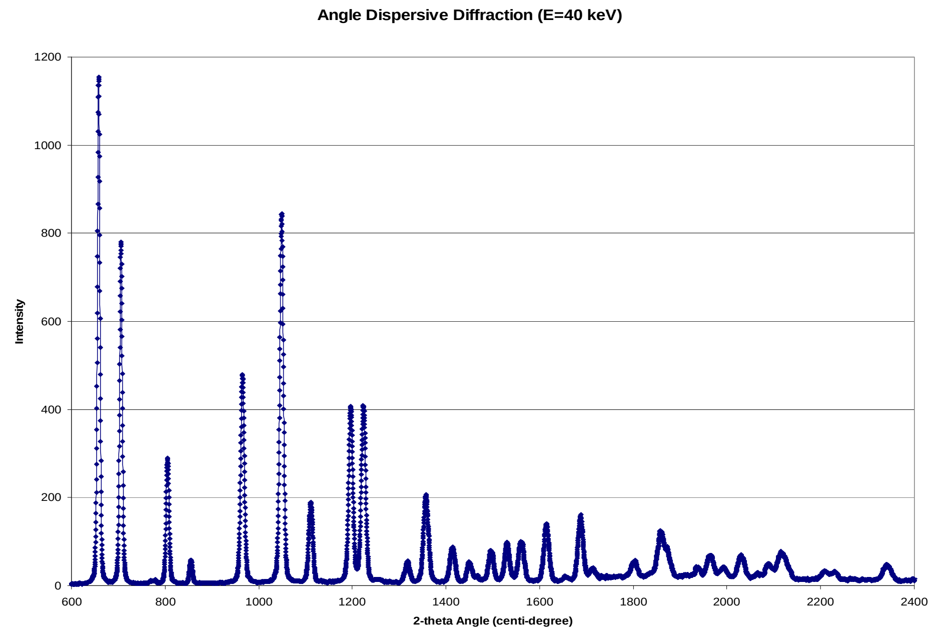


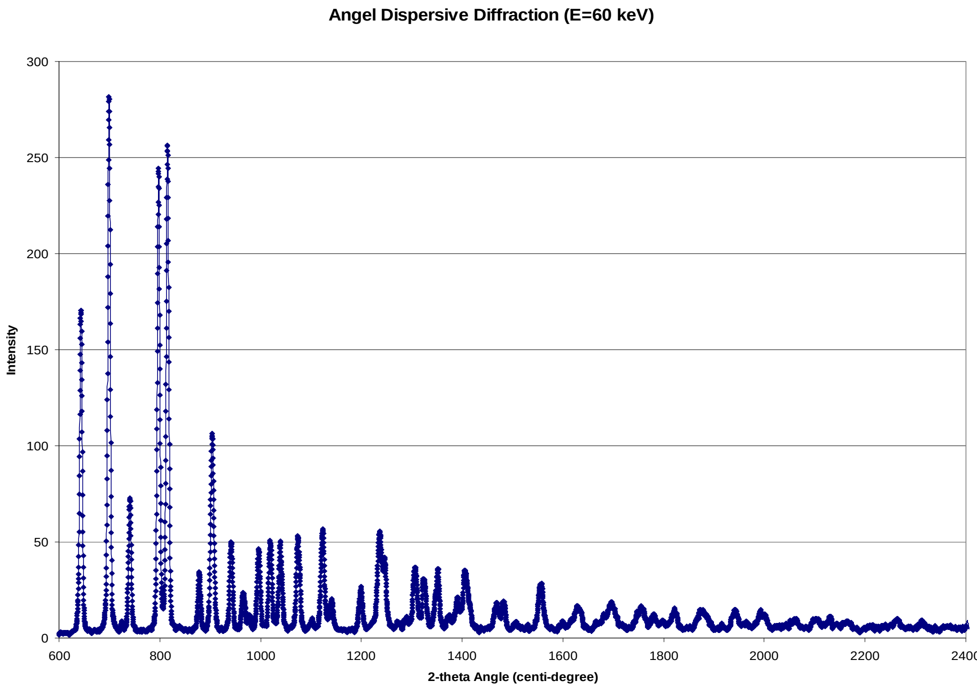
Intensity as a function of energy and 2-theta angle


Energy Dispersive Diffraction (2-theta @ 8˚)
Intensity
2000
1800
1600
1400
1200
1000
800
600
400
200
0
0
10
20
30
40
50
60
70
80
90
Energy (keV)

Angle dispersive diffraction profiles at different energies





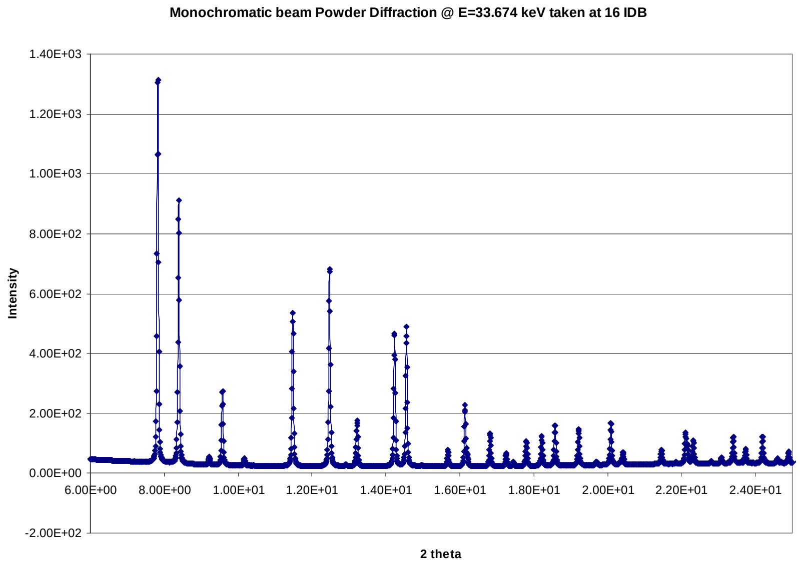


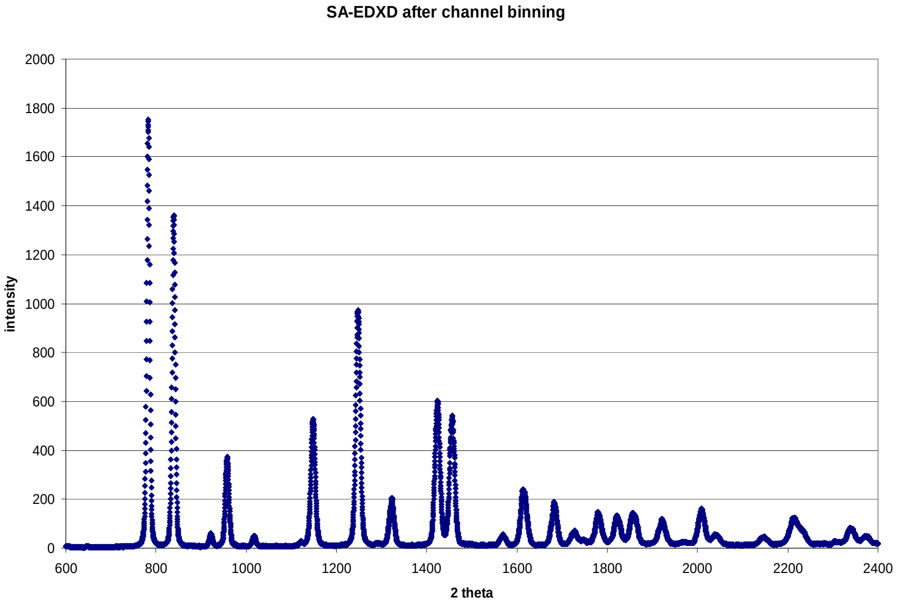
Diffraction relative intensity is modified as you change the ADX energy

Applying the energies close to element Absorption edges, one can get anomalous Scattering effects to get element specific Site occupation;

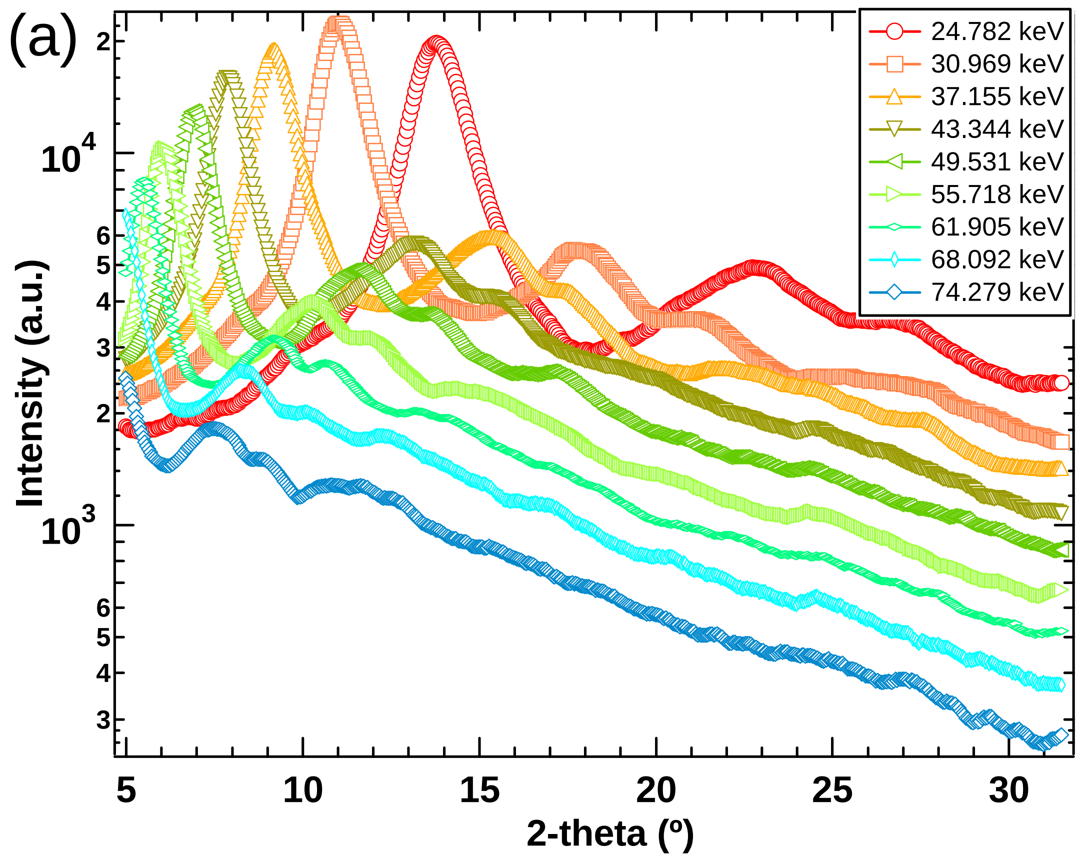
Diffraction from low-Z materials;

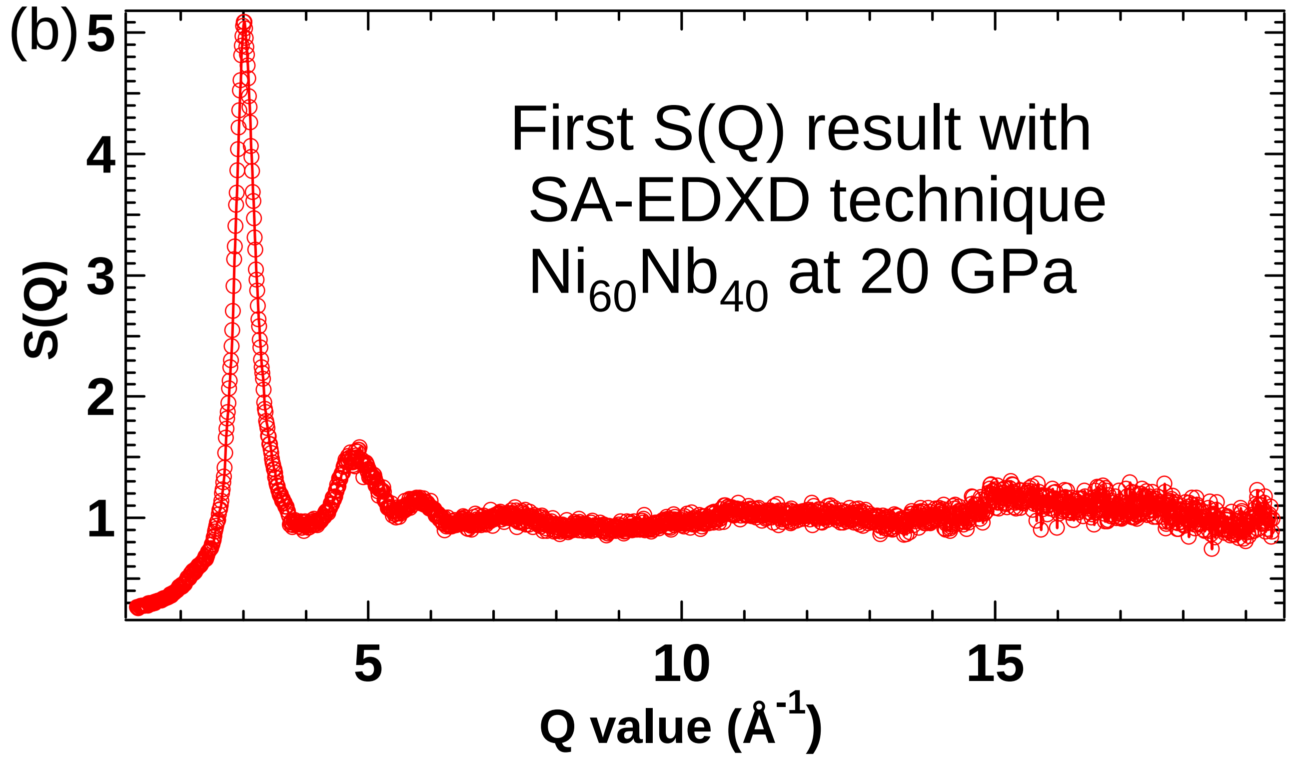
Comparison with regular angle-dispersive diffraction with monochromatic beam





Amorphous Ni-Nb BMG at 20 GPa in DAC




(b)
S(Q)
5
4
3
2
1
5
10
15
Q value (Å^{-1})
First S(Q) result with
SA-EDXD technique
$Ni_{60}Nb_{40}$ at 20 GPa

Comparison with regular monochromatic ADX

Advantage:

Multi energy data collected simultaneously, which could be used for anomalous diffraction methods;

High signal to noise ratio, which is specially useful for low-Z crystalline, liquid and amorphous studies;

Easy data interpretation;

No fluorescence background;

…

challenges:

Data collection time longer;

Stable experimental setup is required, including small sphere of confusion, steady incident intensity and spectrum, stable sample support…

For crystalline sample, fine powder is required to avoid the texture effects from the point detector;

Escape lines, can be removed effectively, but interference with diffraction lines;

…

Summary

A scanning angle EDXD method has been developed for structure study with DAC

A dedicated analysis software package has been written in IDL

Comparison with routine angle-dispersive diffraction with monochromatic beam shows similar angular resolution

Good signal to noise ratio makes it very promising to study low-scattering materials (low-Z, amorphous, liquid …)

Multiple energy data can be used for structure refinement and anomalous diffraction effects

Partial structure factor of amorphous structure is under development.