

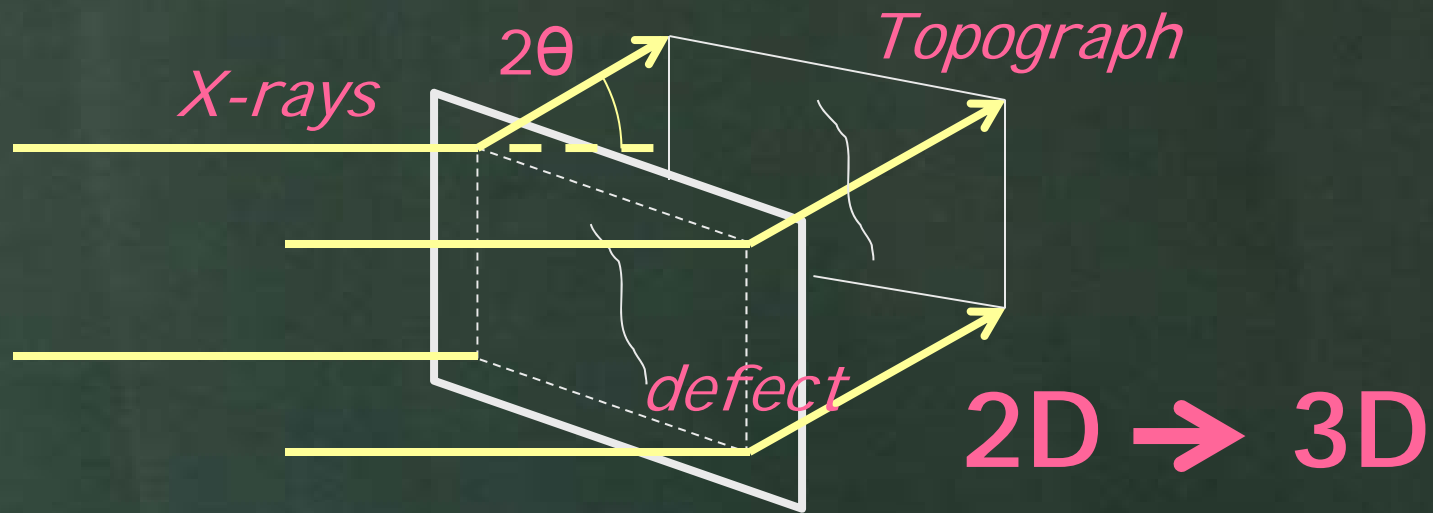
X線トポグラフィの 三次元化と応用

梶原堅太郎 (JASRI)、向出大平 (キヤノン)
飯田敏 (富山大)、川戸 清爾 (SAGA-LS)



X線トポグラフィ

- X線回折トポグラフィ
- イメージング技術
- 格子欠陥の可視化
- 非破壊評価

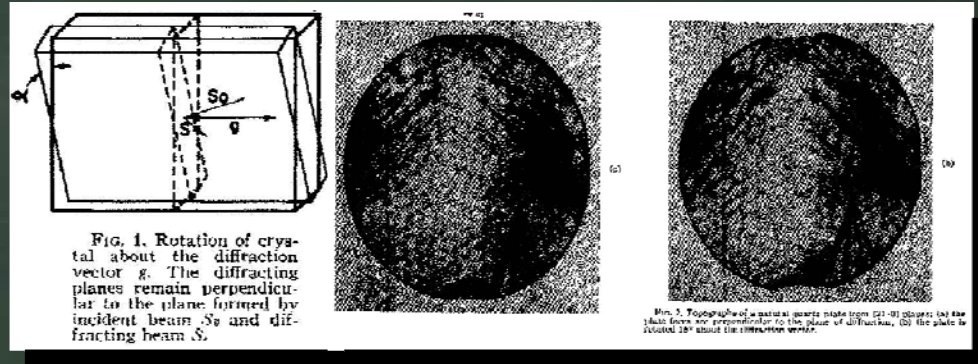


3D X-ray Topography

(1) Stereo-topography → Stereographic-viewer observation

Lang, 1959;

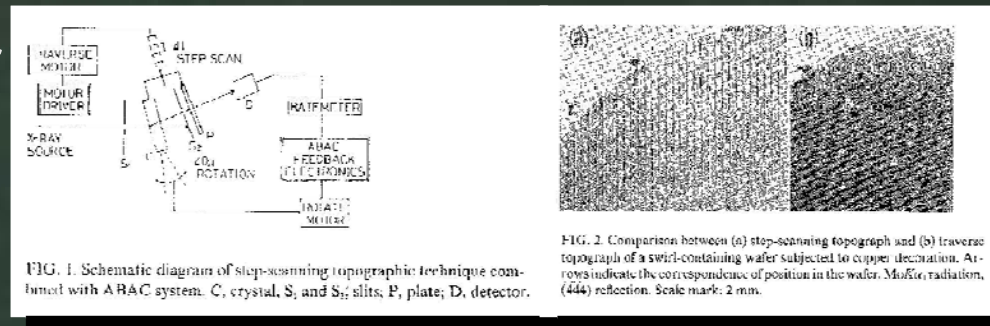
Haruta, 1965



(2) Step-scanned section topography

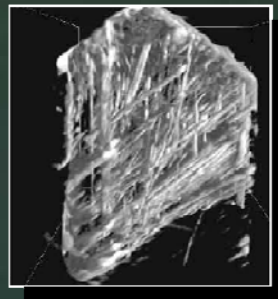
Andersen and Gerward, 1974;

Kawado and Aoyama, 1979



(3) Topo-tomography

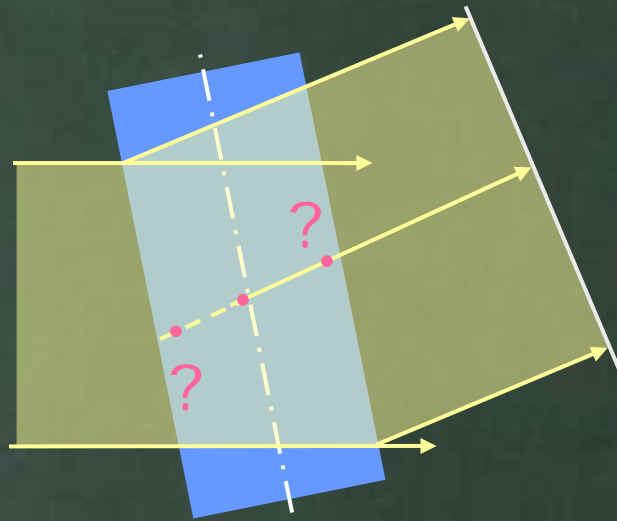
Ludwig et al., 2001



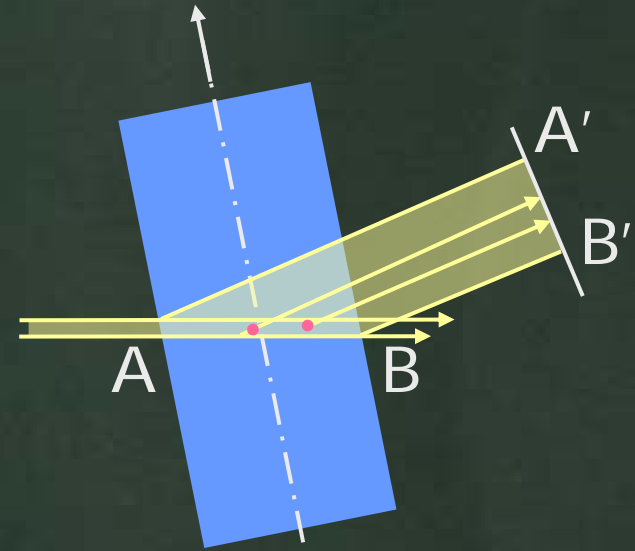
三次元化技術(1) セクショントポグラフィ

セクショントポグラフィ

- 断面の直接観察

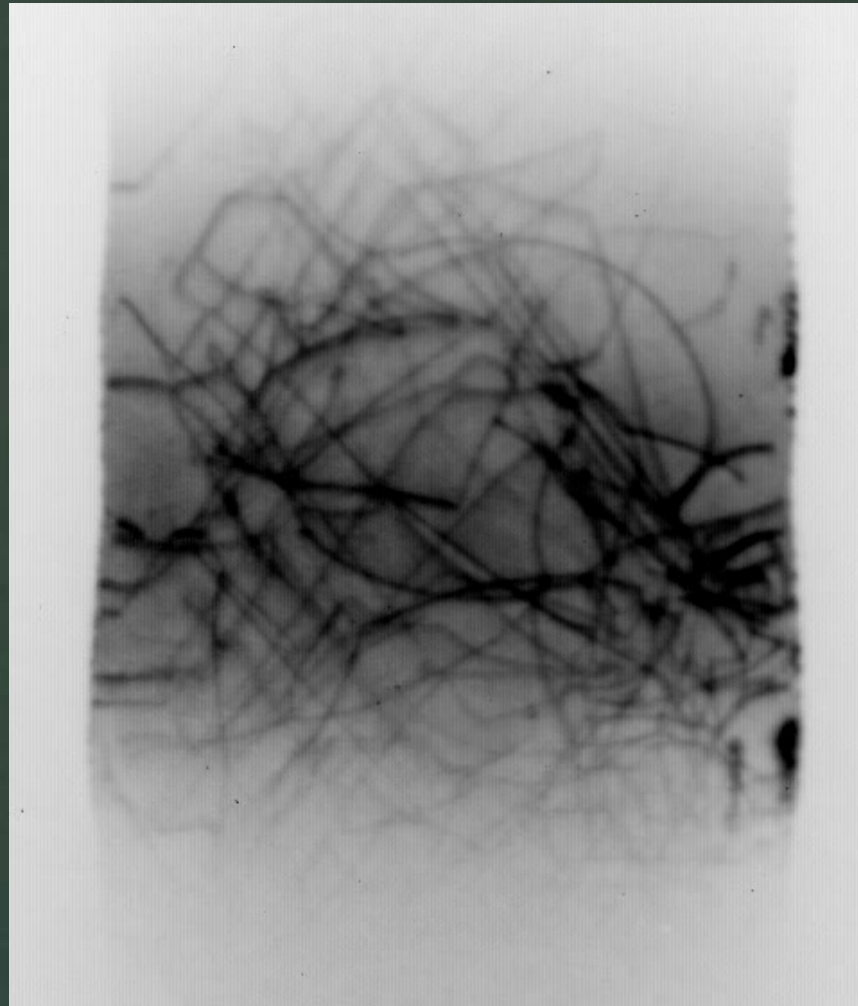


Topography
(large beam)



Section topography
(sheet-like beam)

セクショントポグラフィ



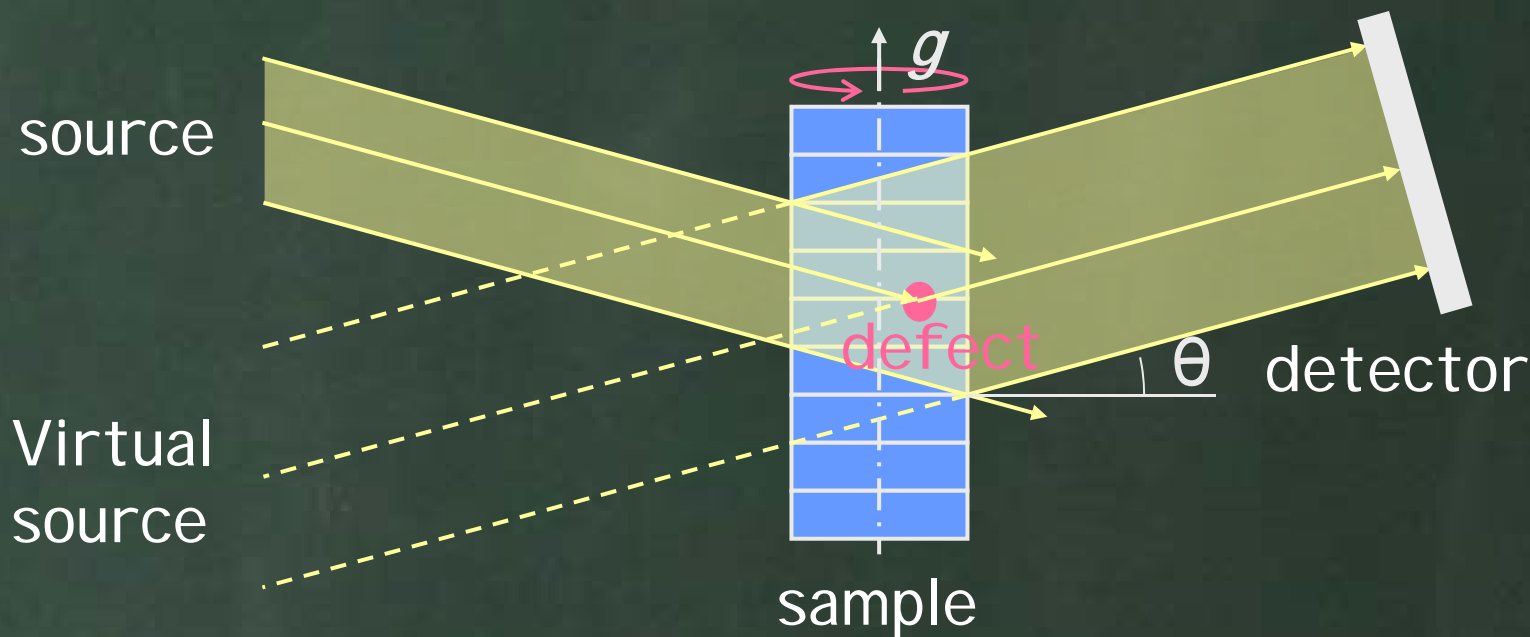
二次元投影像



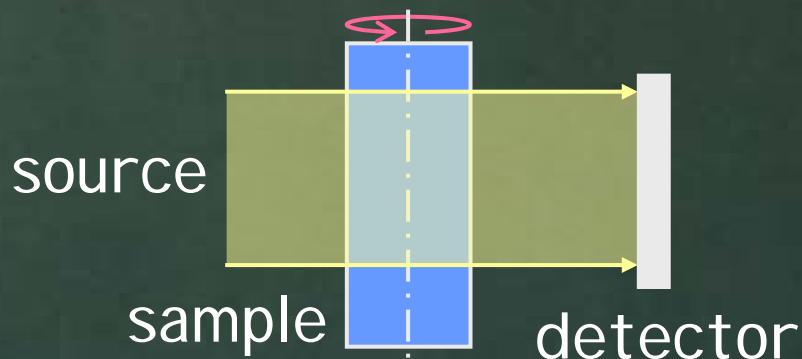
断面像

三次元化技術(2) トポトモグラフィ

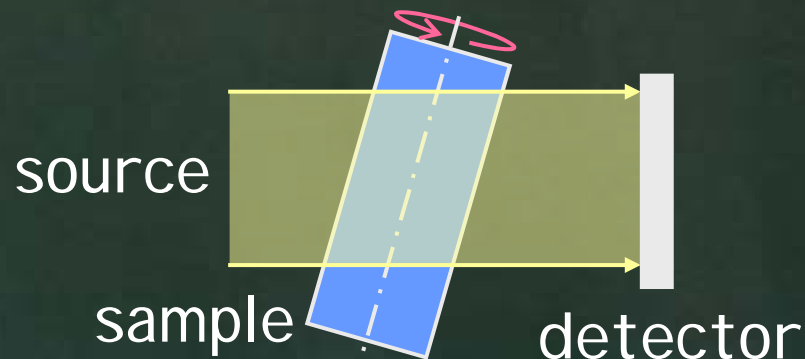
Topo-tomography



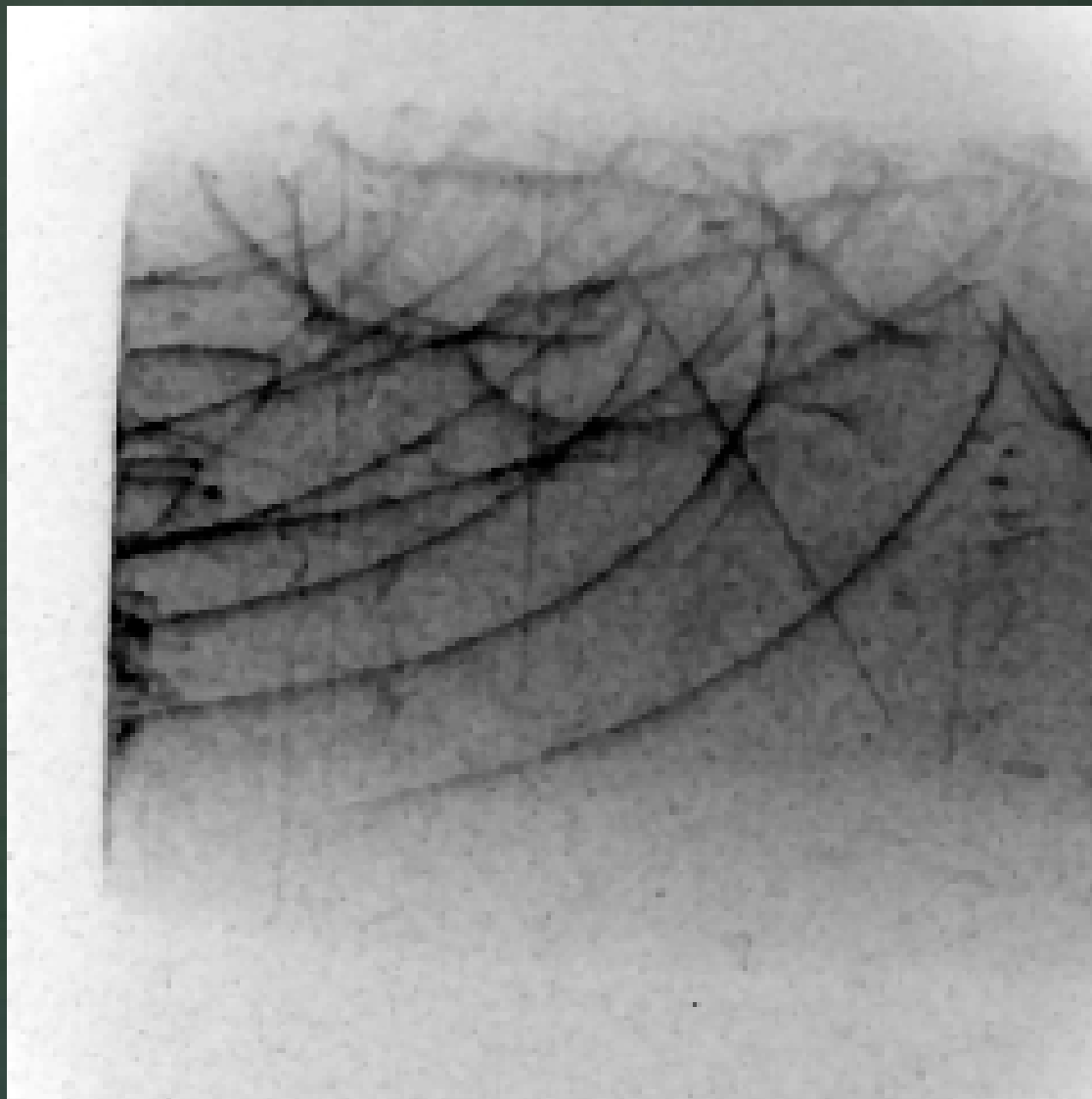
tomography



Laminography



トポトモグラフィ

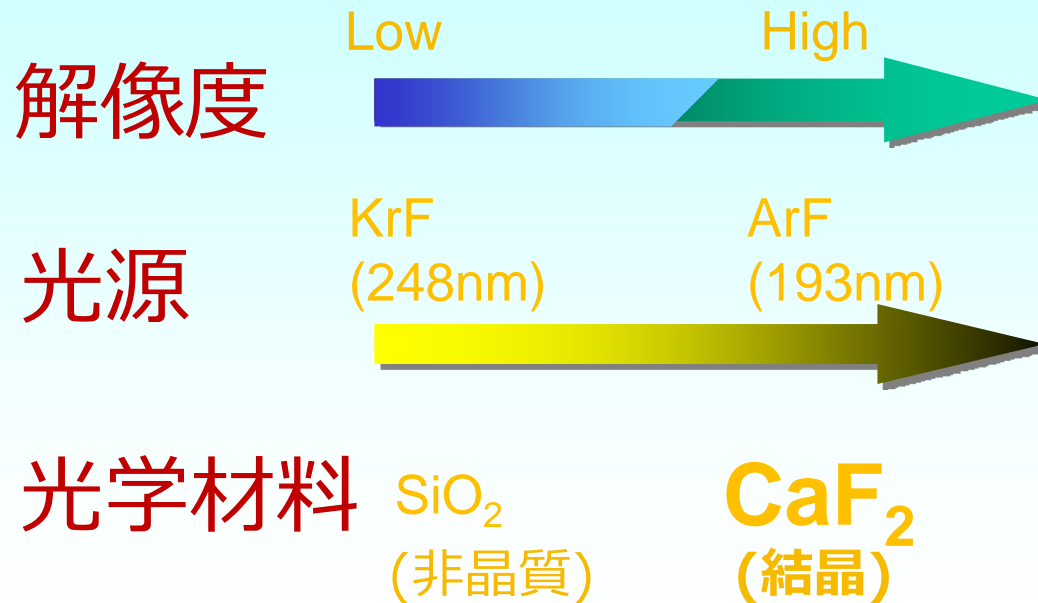


観察例(1) 蛍石

セクションポグラフィ

Introduction

光リソグラフィ

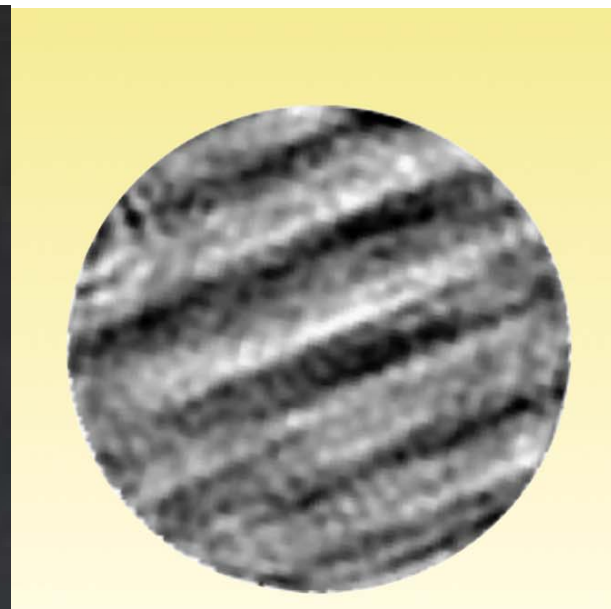


Introduction

課題 CaF_2 光学特性の不均一



螢石



屈折率分布

光学特性と結晶欠陥の相関を調べる

Experimental



Experimental system (BL28B2 at SPring-8)

SR



Sample



- Size of the beam:

12mm, 30mm (horizontal) × 0.1mm (vertical)

- Detector:

- CCD-based beam monitor

C4880-10-14A, Hamamatsu Photonics K.K. pixel size: 11.66 μ m, FOV: 11.66 × 11.87mm²

- Flat panel imager

C7942MOD, Hamamatsu Photonics K.K. pixel size: 50 μ m, FOV: 112 × 117mm²

- Absorber: Al (7mm thickness)

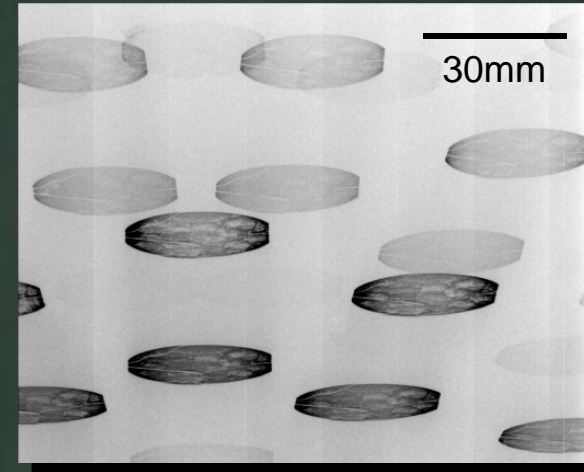
- Step size in vertical direction: 0.1mm

- Exposure time per step: 1 -15 sec

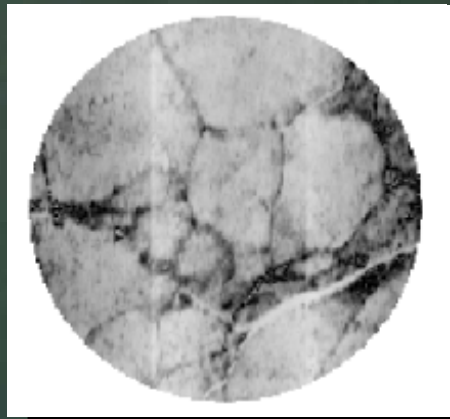
Results and discussion



Sample photograph
(30mm in diameter and 30mm thickness.)

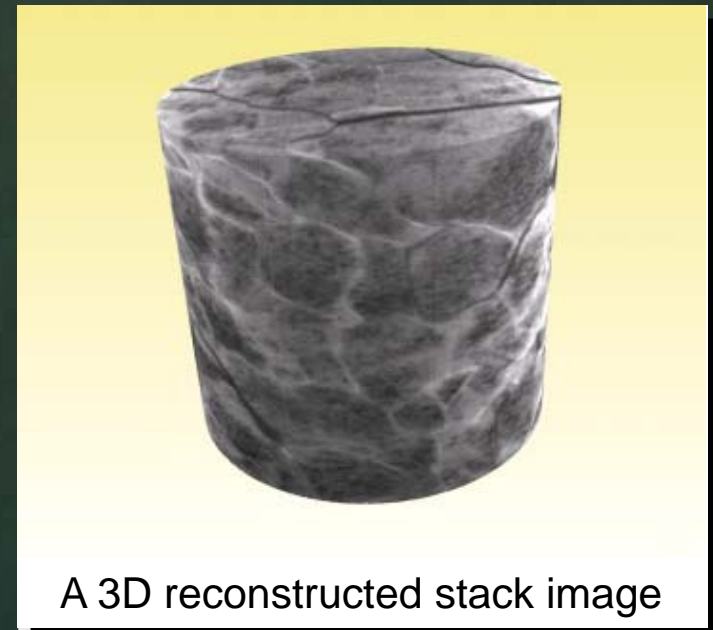


Laue diffraction patterns measured
by the flat panel detector

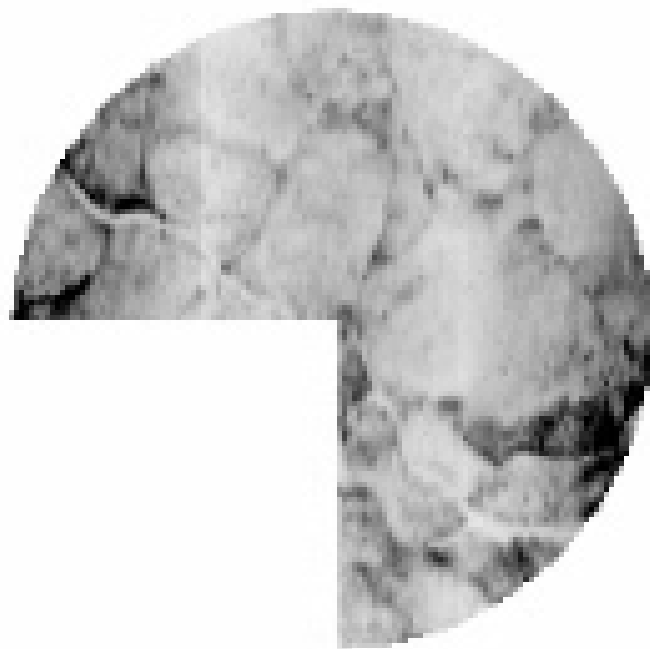


A image of section topography of
a cross section of the sample

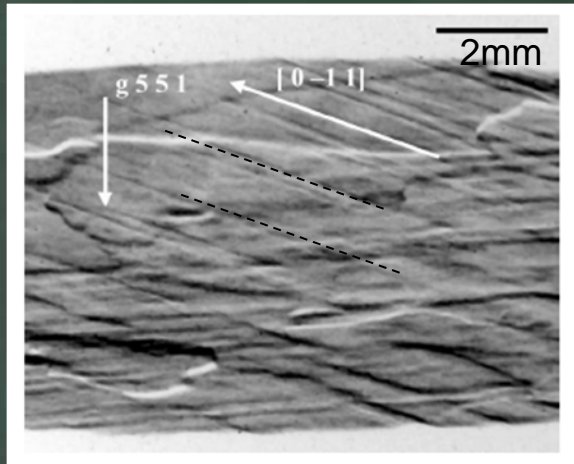
→
stacking



A 3D reconstructed stack image



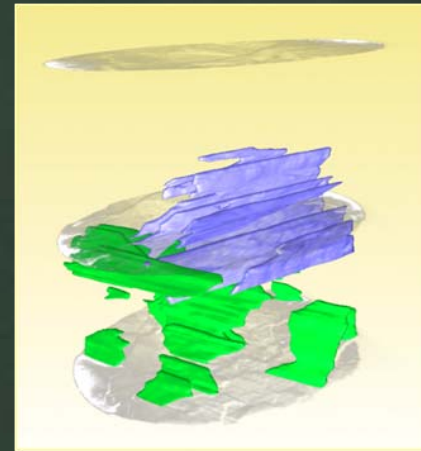
Results and discussion



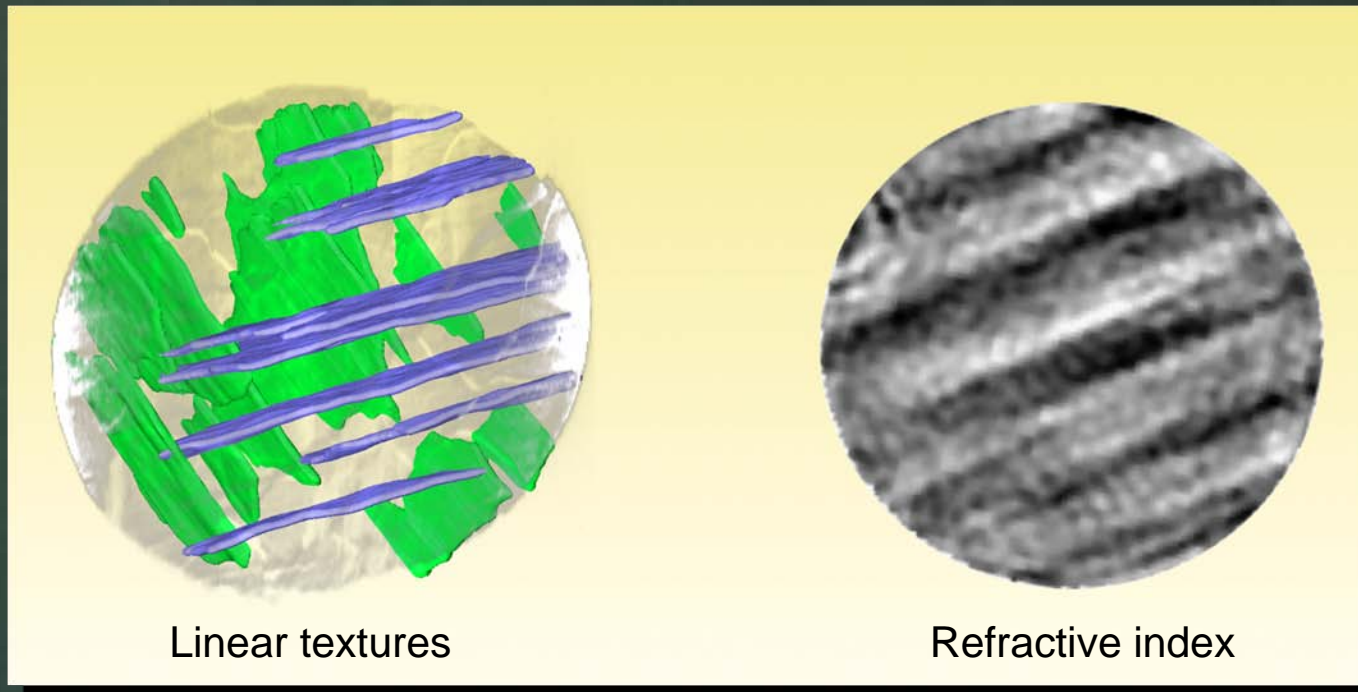
Linear contrast



A image of section topography of a cross-section of the sample



Reconstructed 3D image of some linear textures



Linear textures

Refractive index

観察例(2)

CZ-Si

トポ-トモグラフィ + 白色トポグラフィ
セクショントポグラフィ

Introduction

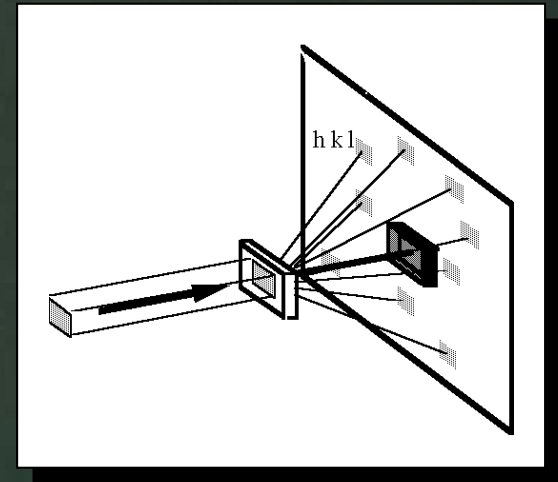
- Topo-tomographic technique
 - 3D distribution (shape and position) of dislocation

+

- White X-ray topography
 - Burgers vector of dislocation

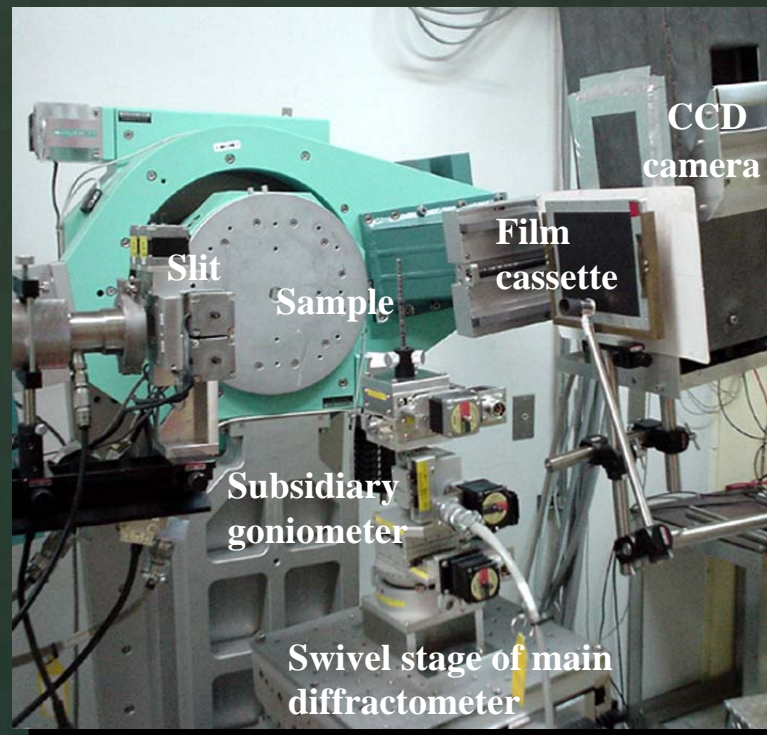
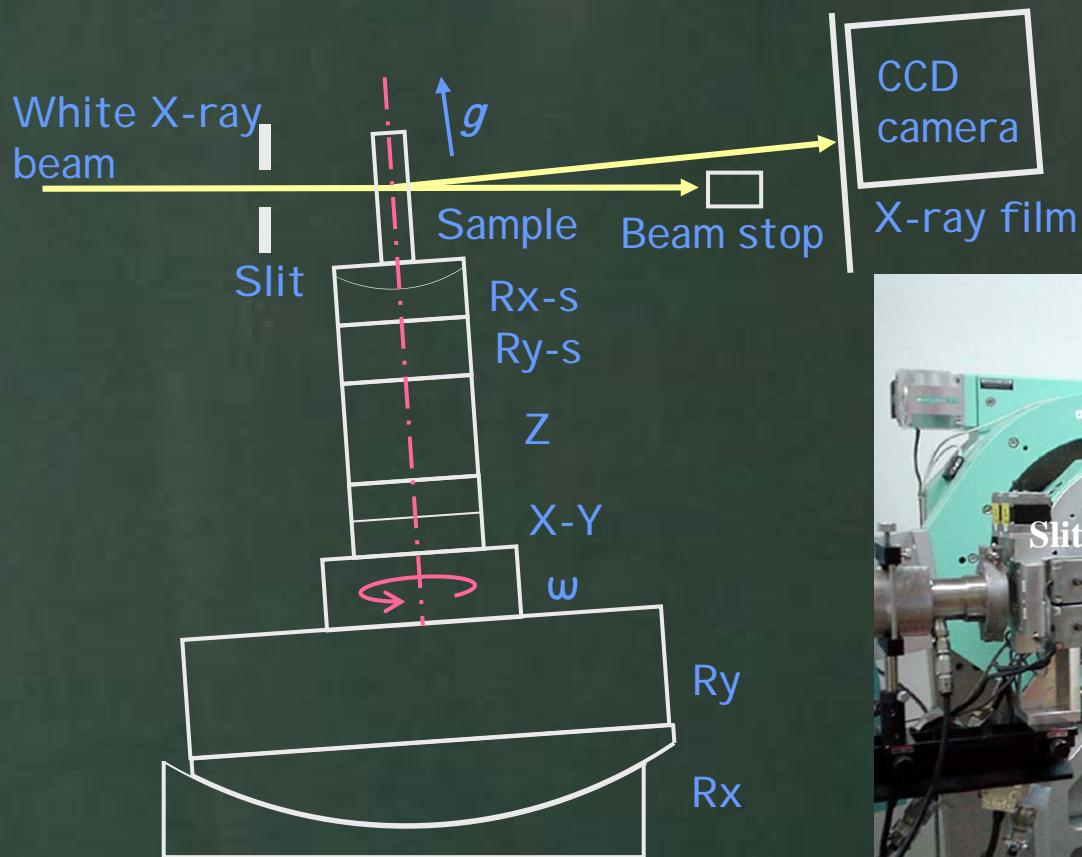
↓

- **Determination of character of individual dislocations**



Experimental arrangement for synchrotron white X-ray topography combined with a topotomographic technique

BL28B2, SPring-8 (Side view)

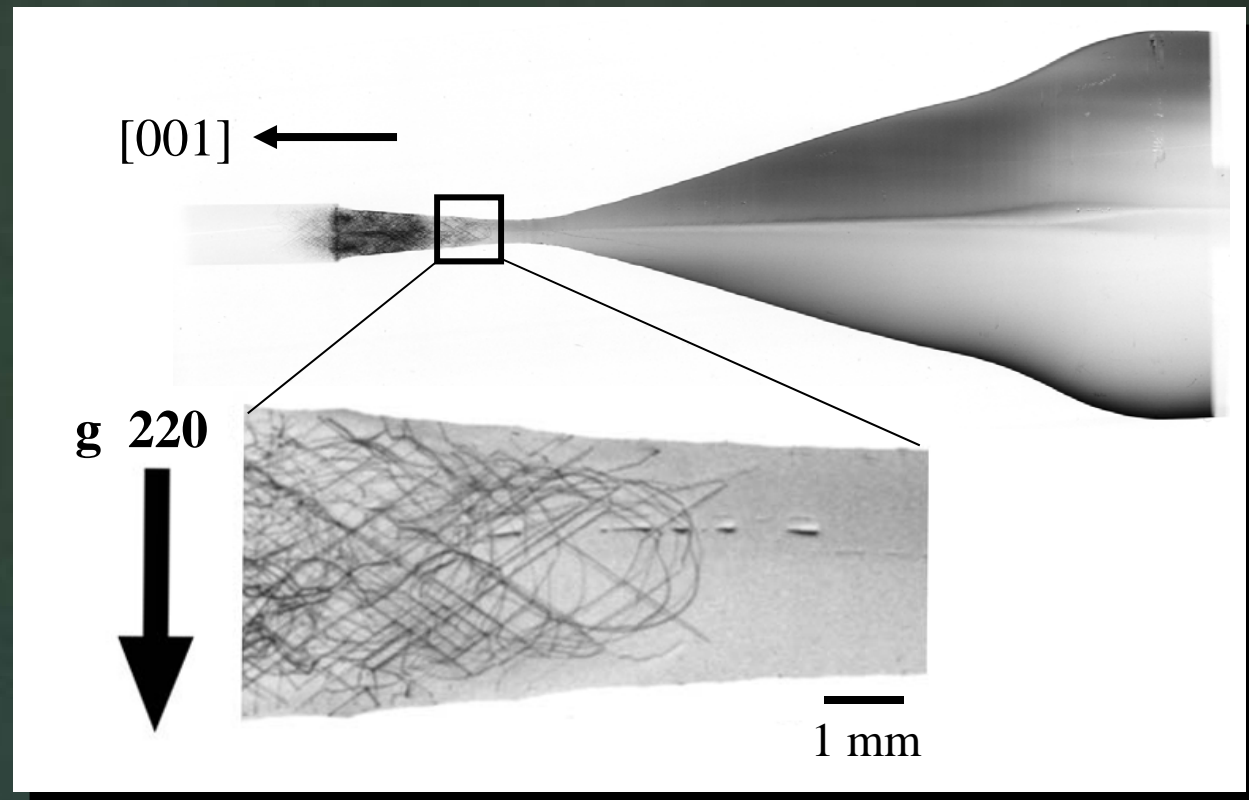


Preliminary observation using monochromatic X-ray topography

BL20B2, SPring-8



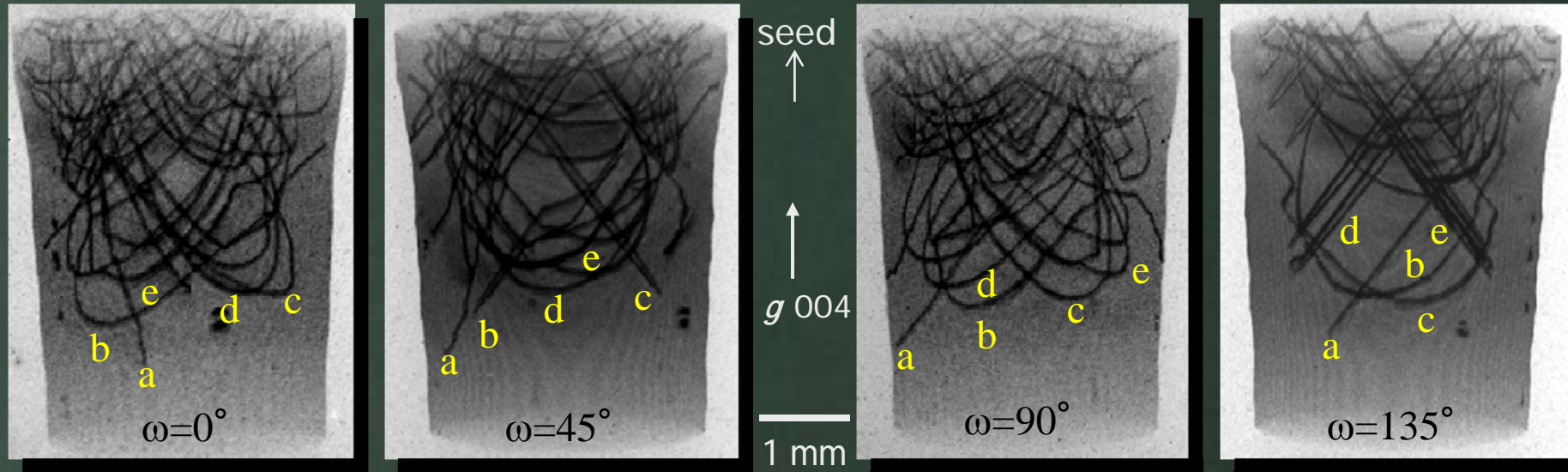
Dislocations propagated from the seed-grown crystal interface and then eliminated in the thin neck region.



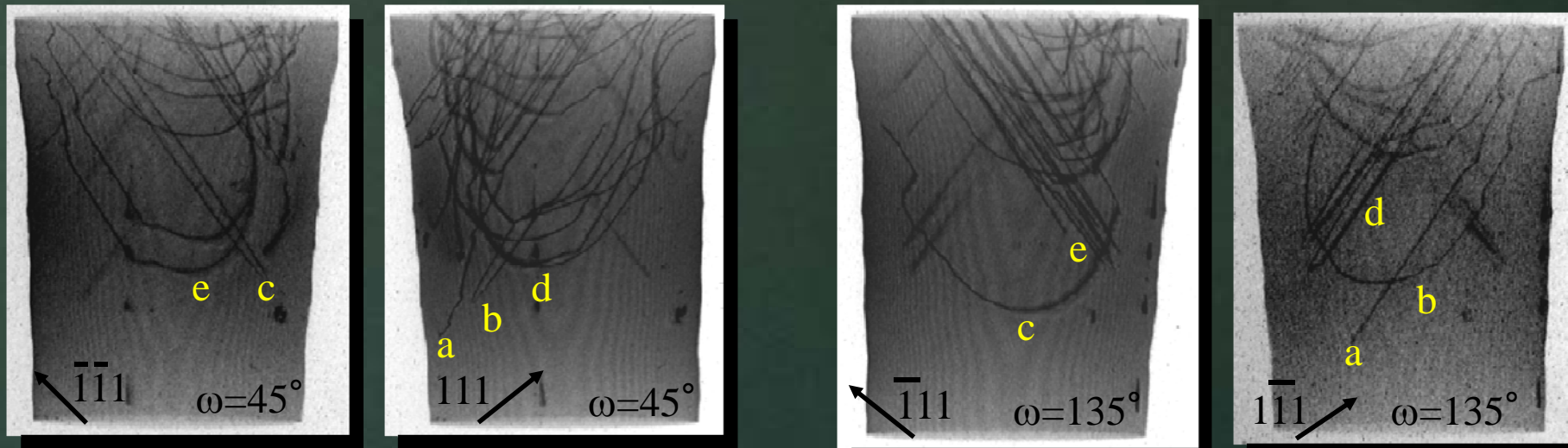
Result

BL28B2, SPring-8

Topo-tomography

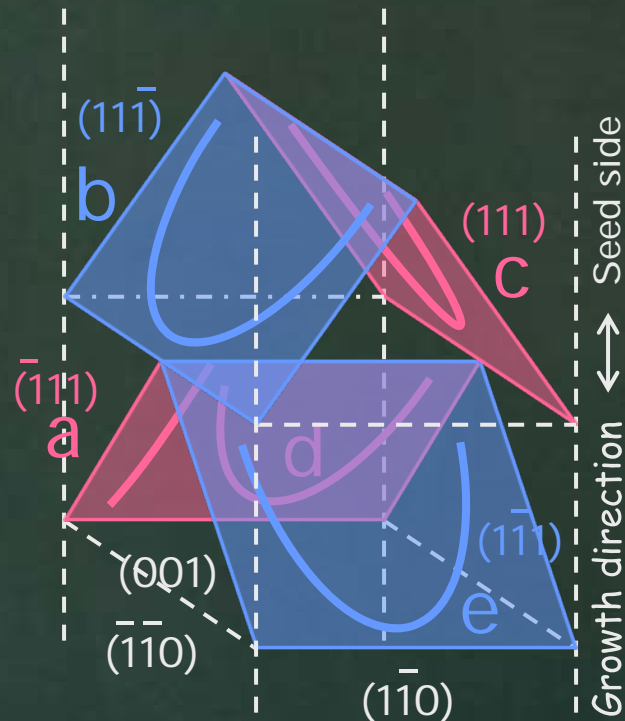


White X-ray topography



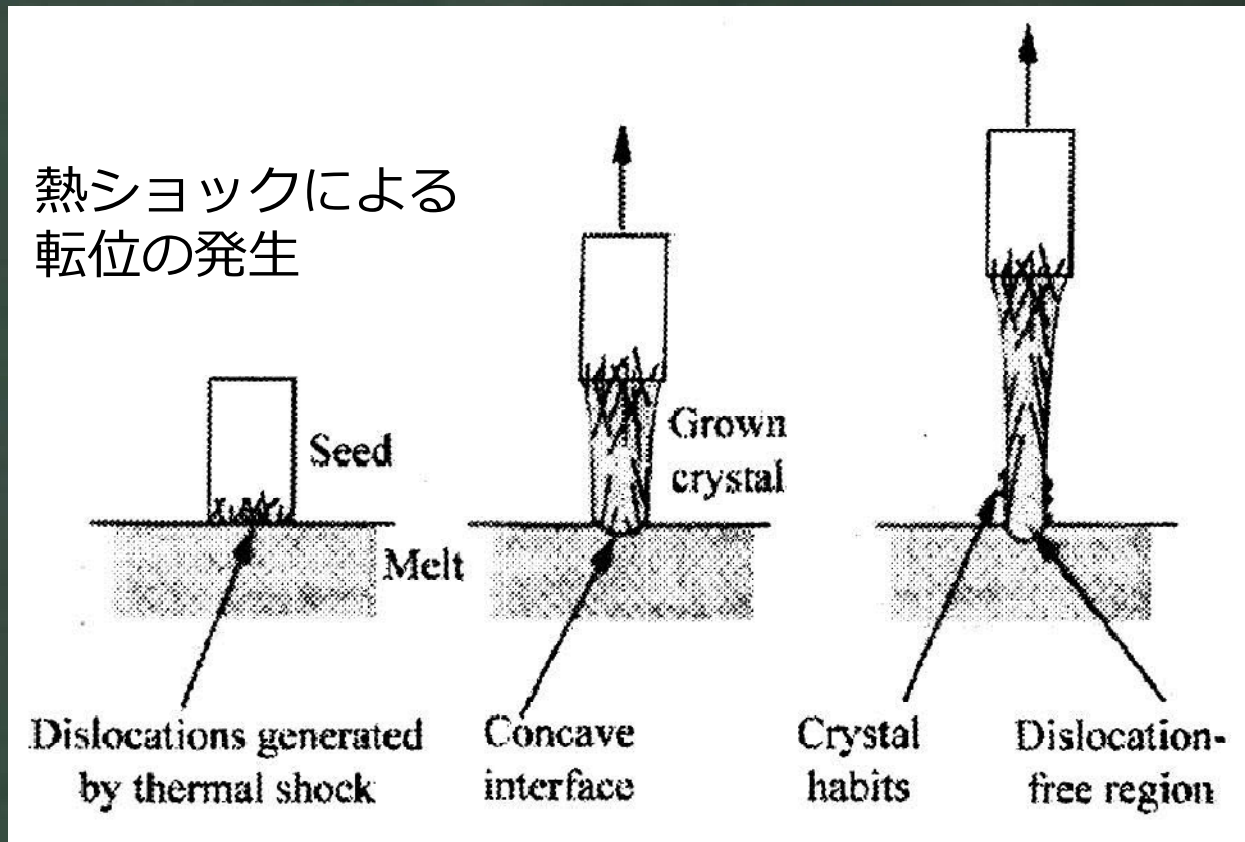
Summary

Dislocation image	shape	Laue spot invisible or weak	Burgers vector	Glide plane
a	straight	$\bar{1}\bar{1}1$ $\bar{1}11$	$\mathbf{a}/2[101]$	$(\bar{1}11)$
b	curved	$\bar{1}\bar{1}1$ $\bar{1}11$	$\mathbf{a}/2[101]$	$(11\bar{1})$
c	curved	111 $1\bar{1}1$	$\mathbf{a}/2[10\bar{1}]$	(111)
d	curved	$\bar{1}\bar{1}1$ $\bar{1}11$	$\mathbf{a}/2[101]$	$(\bar{1}11)$
e	curved	111 $1\bar{1}1$	$\mathbf{a}/2[10\bar{1}]$	$(1\bar{1}1)$



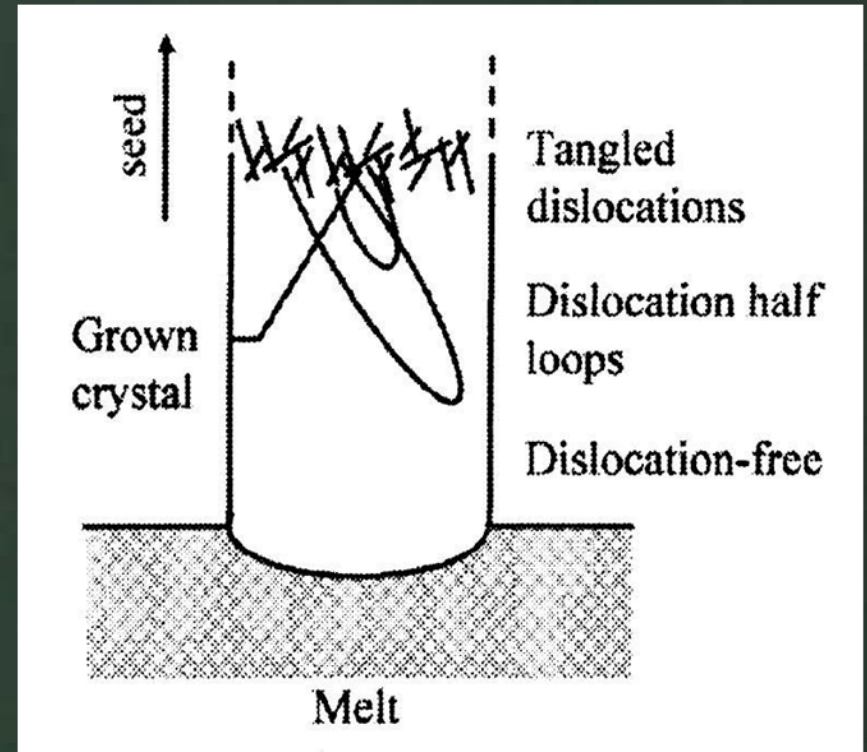
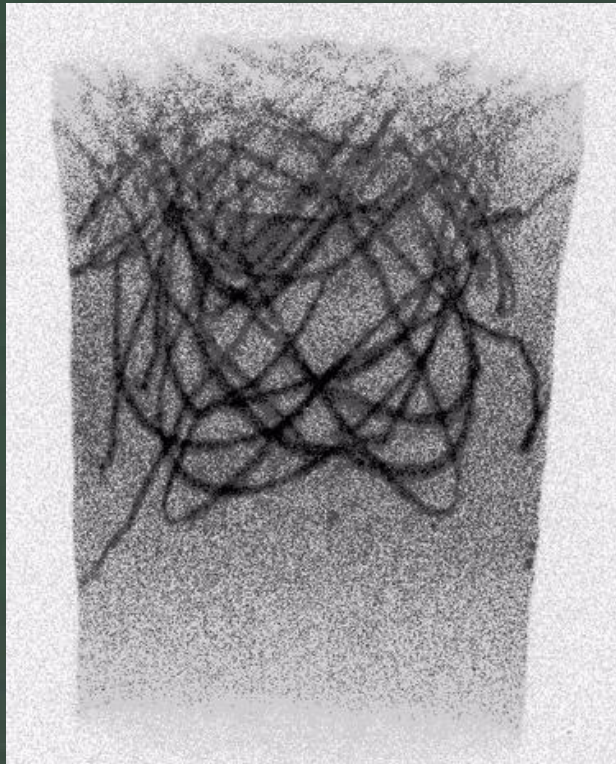
考察

CZ法における転位消滅過程の従来説



転位は結晶の細径化によって、**結晶の外へ抜ける**

考察

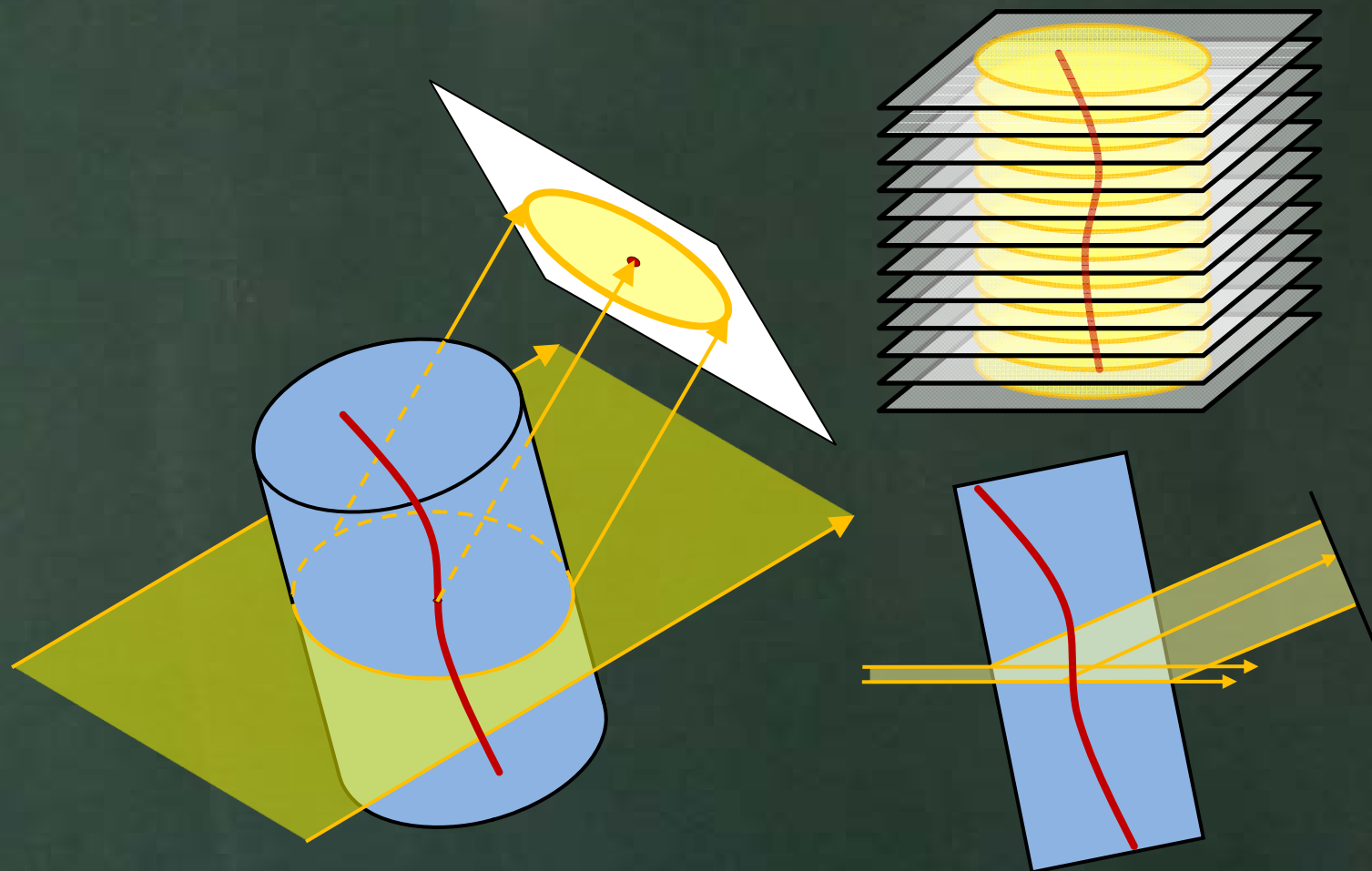


転位消滅の最終段階では、転位は成長方向に凸の半ループ状で、結晶の外には抜けず、**結晶の内部で止まっている**

- S. Kawado, et. al., J. Synchrotron Radiat. **11**, 304 (2004)
- S. Kawado, et. al., J. Phys. D: Appl. Phys. **38**, A17 (2005)

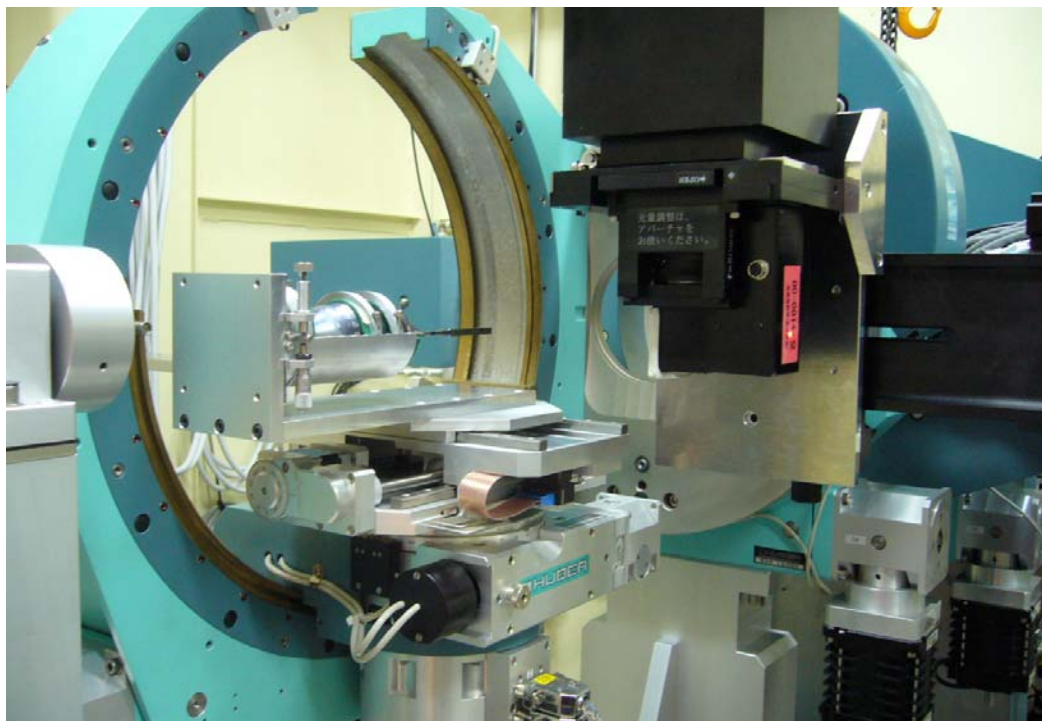
研究の背景

- CZ 法育成シリコン単結晶
 - Dash's necking による無転位化
 - 単結晶インゴットの大型化
 - インゴットの重量 > ネック部の耐荷重
- 転位消滅の過程
 - 従来説
 - 新事実

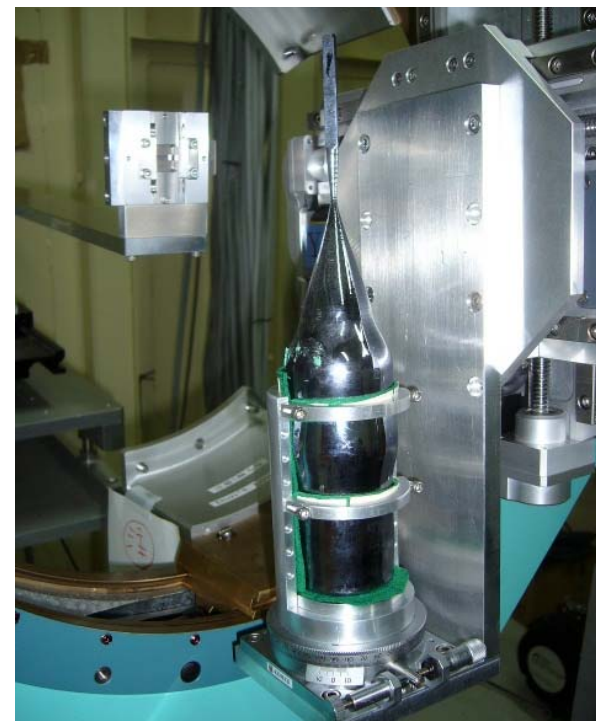


実験

- SPring-8, BL19B2
- 単色X線 (35keV)
- 004回折、400回折(複数のX線入射方向)
- 二次元デジタル画像検出器 (X線CCDカメラ)



試料結晶：横置き

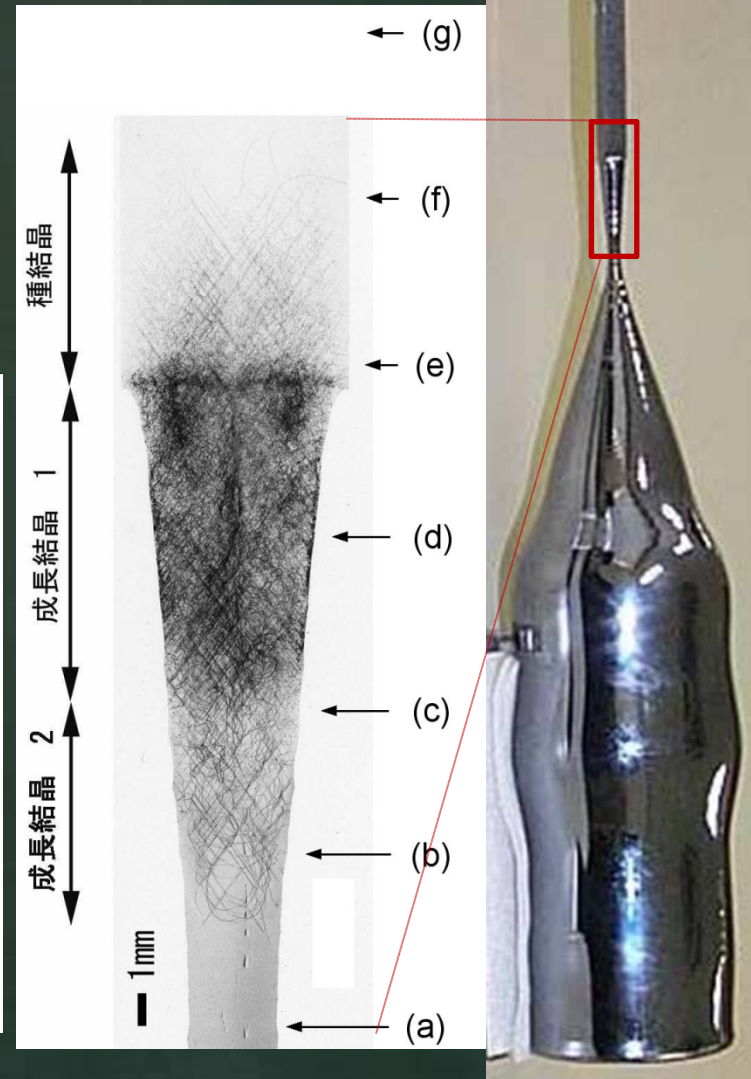
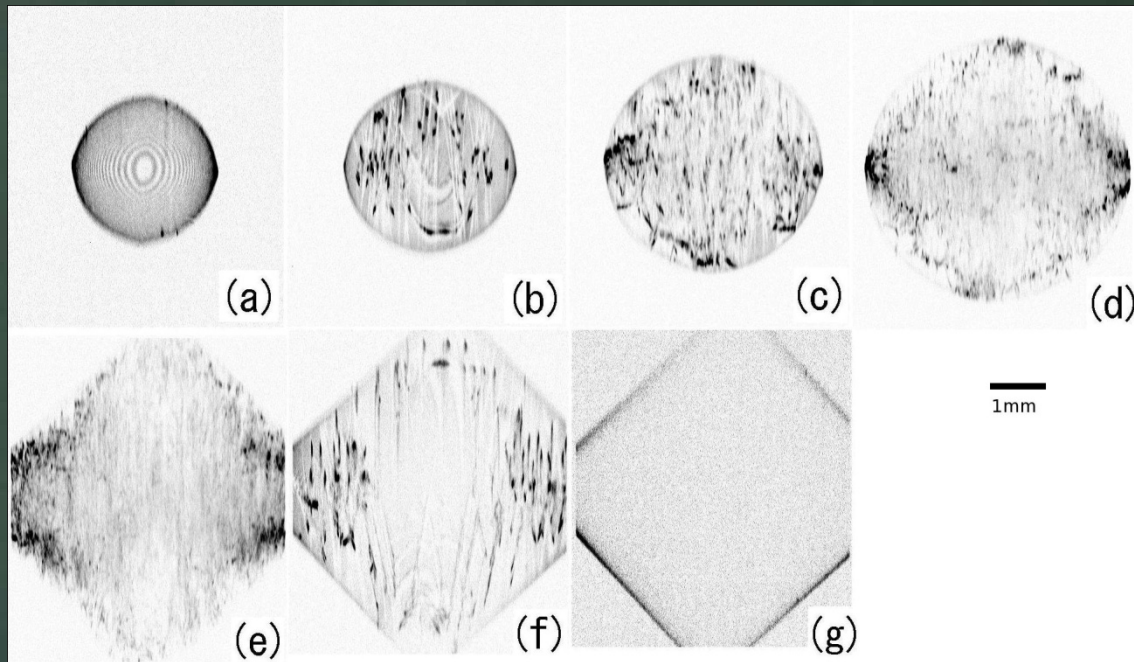


試料結晶：縦置き

実験結果

断面トポグラフ

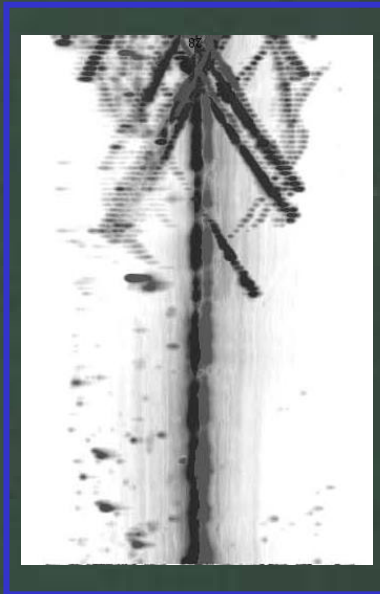
- SPring-8, BL19B2
- 単色X線 (35keV)
- 004回折
- X線CCDカメラ



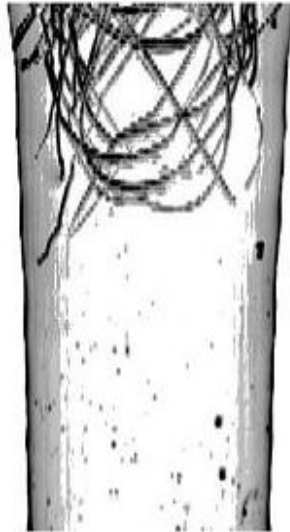
実験結果

成長結晶部の転位

転位は{111}面上に乗っている



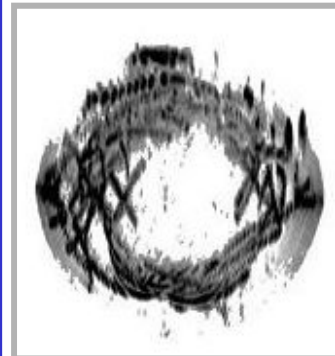
90°



0°



60°



90°



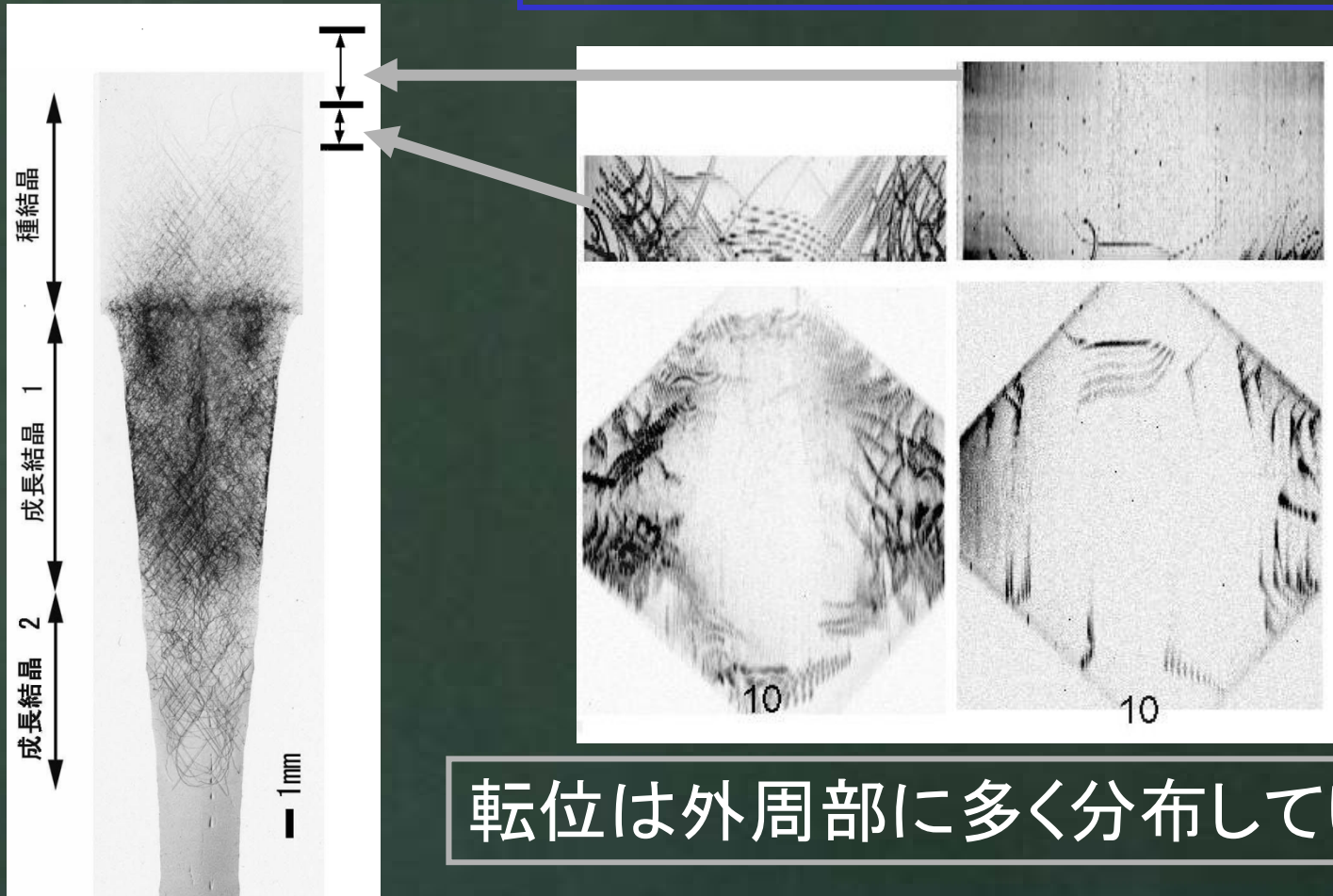
120°

転位は外周部に多く分布している

実験結果

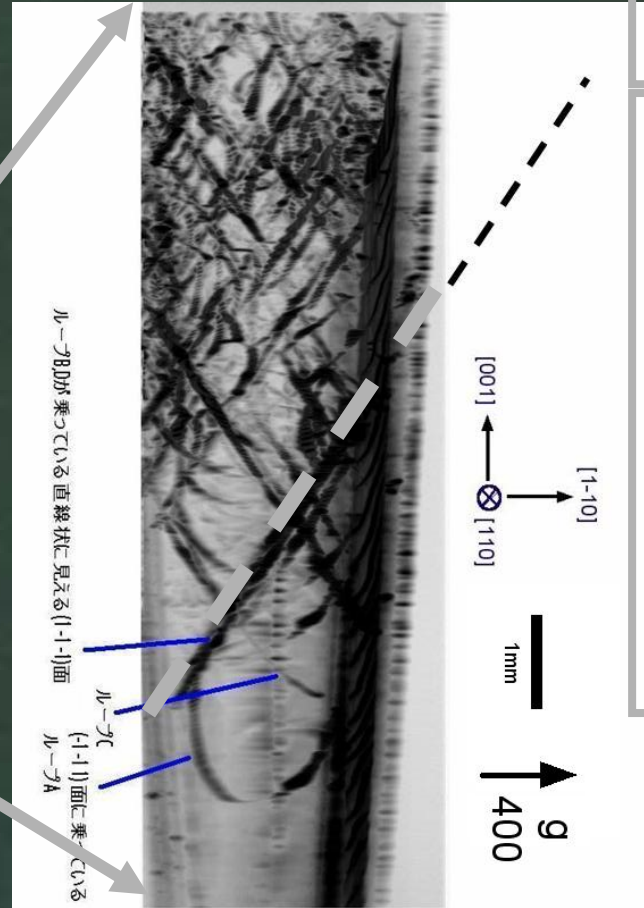
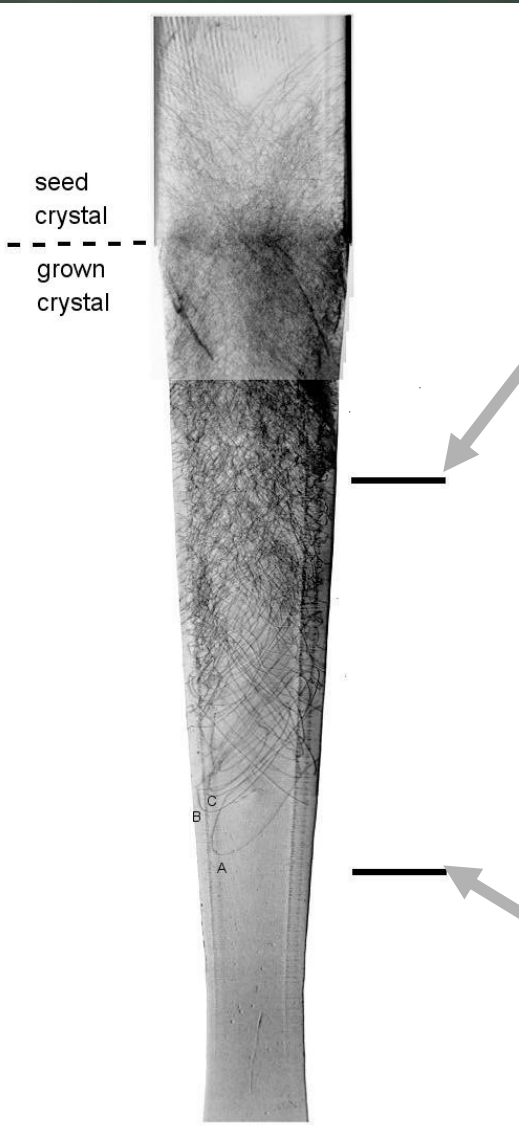
種結晶部の転位

転位は{111}面上に乗っている

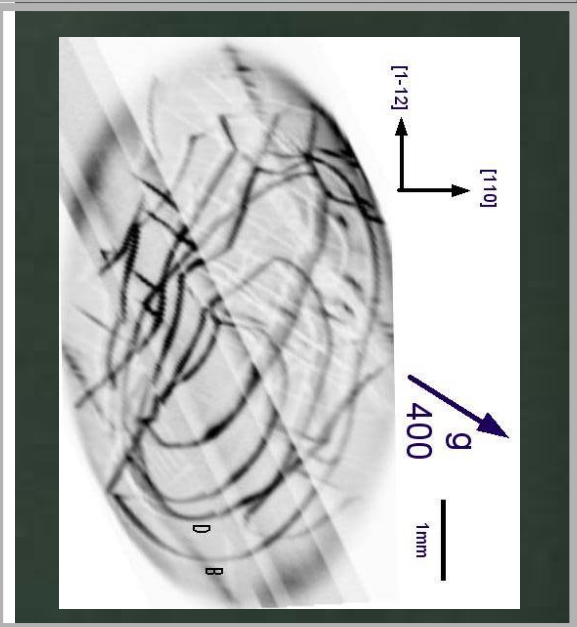


転位は外周部に多く分布している

実験結果：半ループ状転位の発生源



転位B&D 切断面



まとめ

- X線トポグラフィの三次元化
 - セクショントポグラフィ(断面の直接観察)
 - トポトモグラフィ
- 観察例
 - 蛍石
 - 光学特性と格子欠陥の分布の対応
 - CZシリコン
 - 転位線の形状、分布、バーガースベクトルの決定
 - (転位消滅メカニズムの調査中)