

# X-RAY DIFFRACTION SYNCHROTRON TOPOGRAPHY

MEHDI SAFA

Physics Department, ISFAHAN  
UNIVERSITY OF TECHNOLOGY

ISFAHAN - IRAN

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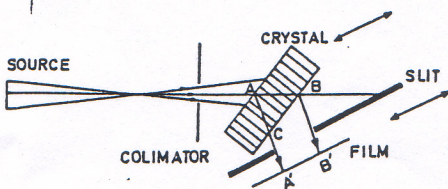


FIG.(2-3) Arrangements for Lang's transmission technique. projection topographs are obtained by scanning the crystal and film across the beam.

In the case of section topographs the crystal and the film are stationary and the beam is narrow.

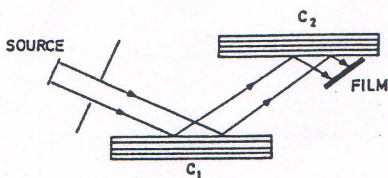


FIG.(2-5) The double crystal arrangement

In Lang Topography there are limitation:

- 1- photographic plate should be 1m after the slit to get 1 $\mu$ m Resolution
- 2- Adjustment
- 3- Long exposure time

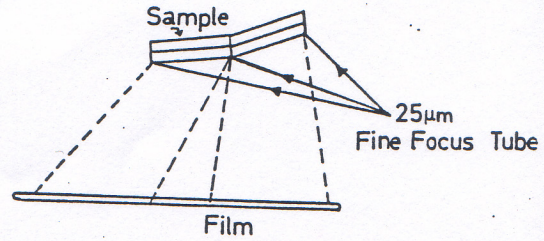


Fig.(2-1) Schematic arrangement for the Schulz technique. (1954)

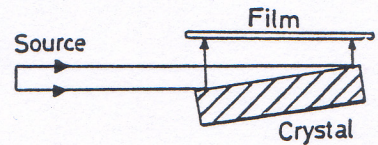


Fig.(2-2) Schematic arrangement of the Berg-Barrett technique.

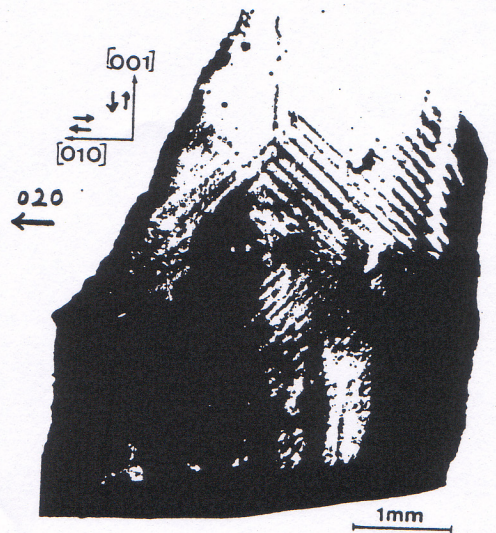


Fig.(6-1) Lang topograph of a crystal of  $\text{KNiF}_3$  at  $T=100\text{K}$ .  $\text{MoK}\alpha_1$ , 020 reflection, recorded on 50 $\mu\text{m}$  Ilford L4 Nuclear Emulsion, exposure time 2h. Antiferromagnetic domains with walls in  $\langle 110 \rangle$  directions are visible.

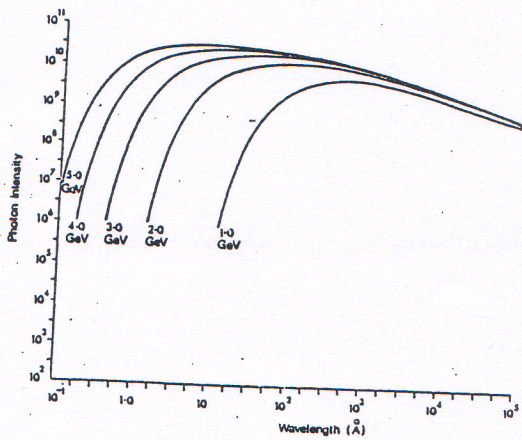
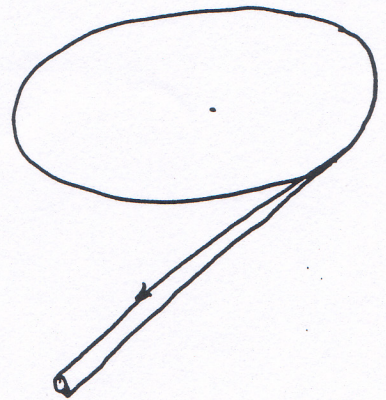


Fig. (3-1) Synchrotron radiation spectra from NINA. Photons/s/mA beam/mrad horizontally within a 0.1% bandwidth. (After Poole 1975)

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### Synchrotron topography

synchrotron Radiation characteristics may be schematically listed as follows:

- 1 - Synchrotron Sources deliver a continuous spectrum.
- 2 - Beam divergence is small and of the order of  $10^{-5}$  to  $10^{-4}$  Rad.
- 3 - The X-ray beam is highly Polarized in the plane of the orbit.
- 4 - The beam is characterized by its time structure, It consists of pulses with a high frequency repetition rate.

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$$I_{hkl} \sim P(\lambda) \cdot F^2 \frac{\lambda^3}{hkl \sin^2 \theta} e^{-kt}$$

$P(\lambda)$  = photon flux reaching the sample

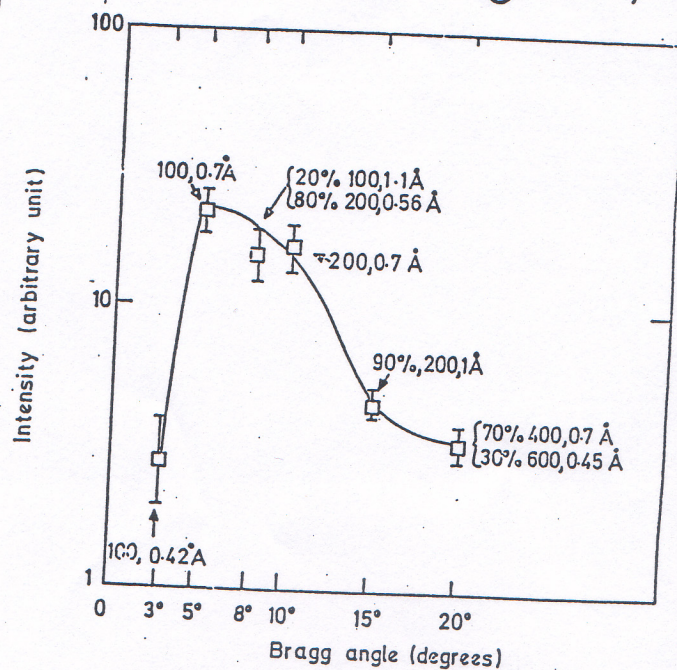


Fig. (3-3) Intensities in transmission of the beams diffracted from {100} planes of  $\text{KNiF}_3$  as a function of the Bragg angle, using synchrotron radiation at 5 GeV. electron energy.

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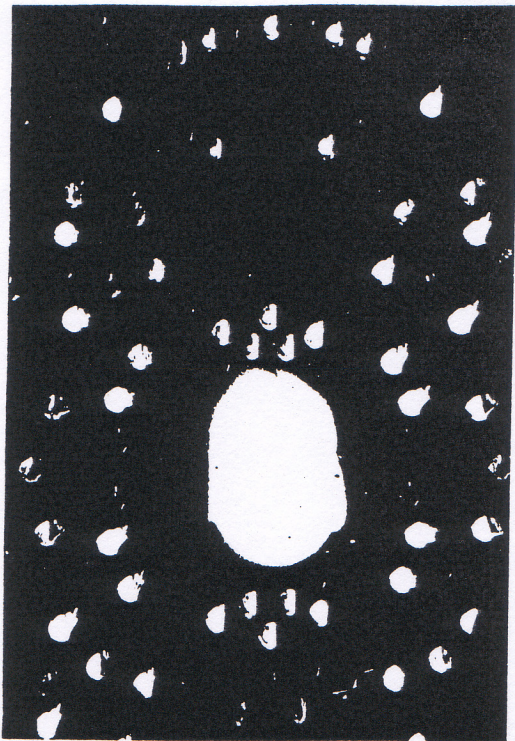


Fig.(3-2) Laue pattern of a  $DyPO_4$  crystal taken with synchrotron radiation. Each "spot" is an image of the crystal within which contrast from imperfections can be seen. 5 GeV, 10mA, 50s exposure,  $L_4$  10 $\mu$ m nuclear emulsion.

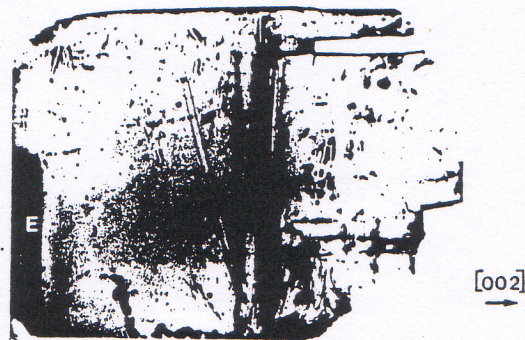
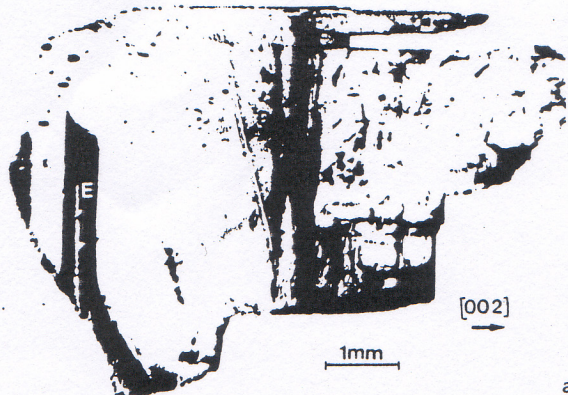


Fig.(3-4) Transmission topographs of a 200 $\mu$ m thick (100) plate of  $KCoF_3$  crystal. (a) Lang topograph, 002 reflection,  $AgK\alpha_1$  radiation ( $\lambda=0.56\text{\AA}$ ), 3h exposure. (b) Synchrotron topograph, mainly 002 reflection,  $\theta_B=7^\circ$ , 5 GeV, 10mA, 20s exposure,  $L_4$  25 $\mu$ m nuclear emulsion.

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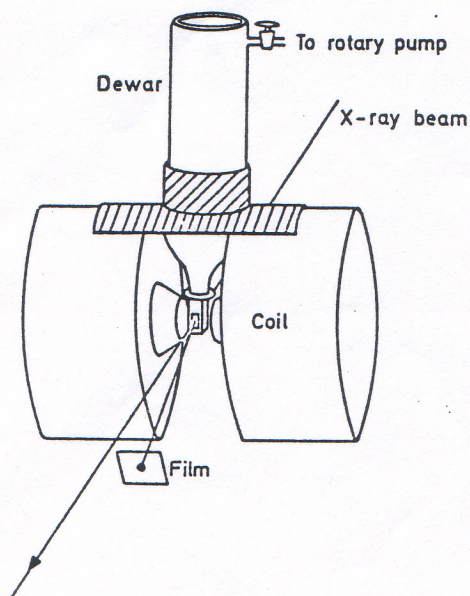


Fig.(5-2) Schematic diagram of the cold-finger cryostat mounted directly on the electromagnet coils C for synchrotron topography.

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### Applications

white beam x-ray topography has been already utilized in a number of fields of scientific activity. Most studies rely on high source intensity as well as on the fact that recording several Laue spots simultaneously may allow for quick defect identification.

Some experimental studies are listed:

- 1 - Recrystallization of polycrystals
- 2 - Generation and movement of Dislocations
- 3 - plastic deformation of single crystals
- 4 - domain wall movement in magnetic and antiferromagnetic crystals

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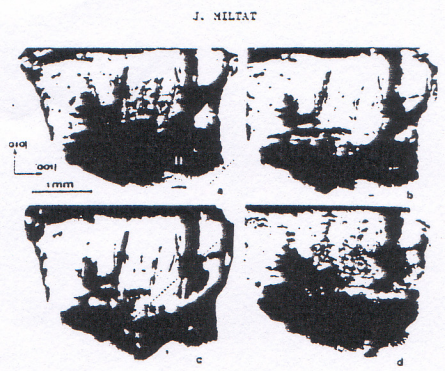
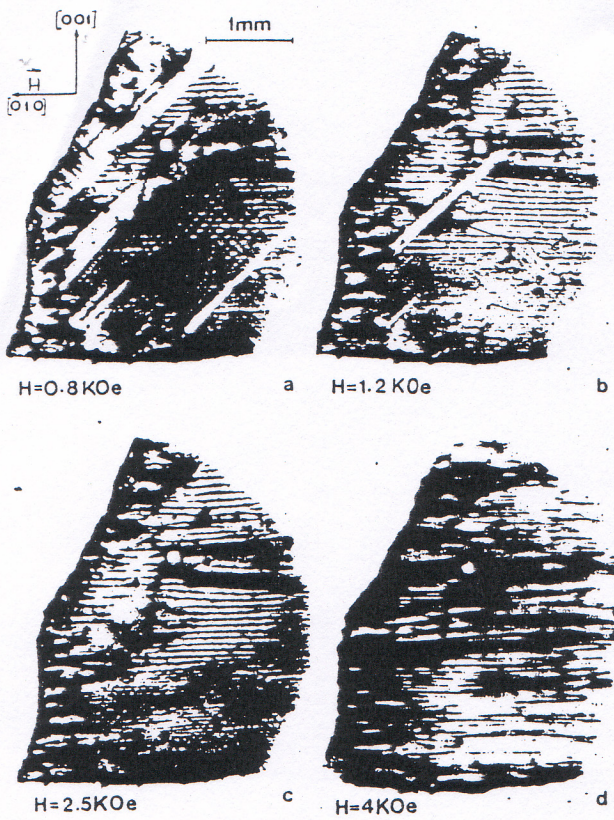


Figure 14. Movement of a domain wall in the antiferromagnet  $KCoF_3$  as a function of applied field (field along 001)  $T = 77K$  a)  $H = 0.22T$ ; b)  $H = 0.72T$ ; c)  $H = 0.94T$ ; d)  $H = 1.4T$  [44]

The physical nature of this restoring force is not yet entirely elucidated. Magnetoelastic coupling may, at least partially, account for it. Accordingly, wall motion should be reversible upon decreasing the field. Such a behaviour has been observed by Saha and Tanner. In experiments of this type, it is rather essential to avoid long term rearrangements of wall geometry. Therefore, short exposure times are necessary. Synchrotron based topography is, here again, a very valuable tool.

#### 16.6 Conclusion

Synchrotron white beam X-ray topography is a now well established topographic technique. More should be however known on defect contrast properties. Applications up to now have neglected the time structure of the beam. Direct stroboscopic experiments are certainly feasible, provided some kind of lattice imperfections can be moved at frequencies in the MHz range. Magnetic domain walls are obvious candidates.

Wall movement in the antiferromagnets  $KCoF_3$  and  $KNiF_3$ .

In domain studies, x-ray topography is a unique tool for a number of reasons. One of the most prominent features of the technique is that it enables one to observe both domain walls in magnetostrictive (or electrostrictive) materials and lattice imperfections in relatively massive samples. It is therefore particularly studied for the study of interactions between domain walls and lattice imperfections. We observe the movement of walls in  $KCoF_3$  and  $KNiF_3$  which are antiferromagnets.

A sequence of topographs recorded with increasing applied magnetic fields.

Domain 1 and 2 have spins parallel and antiparallel to  $[010]$  and  $[001]$  respectively. 15

The boundary between domains 1 and 2 in  $KCoF_3$  is the broad oblique black stripe on the topographs. When a field is applied along  $[001]$ , the boundary sweeps to the right since spins tend to align perpendicularly to the field. It was shown that wall displacement as a function of field agrees well with an energy model in which it is assumed that

- 1) Wall displacement may only occur if the field exceeds a given field, the coercive field,
- 2) Wall displacement is resisted by a restoring force proportional to displacement.

In the case of in situ recrystallization the exposure time should be short compared with grain boundary velocity. This requires high X-ray fluxes.

Synchrotron sources are therefore very adequate sources for these kinds of experiments and, once the grain growth has started, it is highly interesting not to stop phenomenon.

By means of such experiments gained informations are gained on:

- 1) the shape of growing grains whose boundaries were shown to follow low index crystallographic planes at the onset of grain growth,
- 2) the shape of moving boundaries,
- 3) the interaction of moving boundaries with obstacles and subsequent dislocation generation,
- 4) The evolution of growth defects on cooling the <sup>sample</sup>

CONCLUSION  
Synchrotron white beam X-ray topography is a well established topographic technique. More should be however known on defect contrast properties. Application should take into account the time structure of the beam. Direct stroboscopic experiments are certainly feasible, provided some kind of lattice imperfections may be moved at frequencies in the MHz range. With the use of image intensifier and video camera we can observe the motion of dislocations and domain walls dynamically.