The Abdus Salam
International Centre for Theoretical Physics

United Nations
Educational, Scientific and
Cultural Organization

IAEA
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1959-20

Workshop on Supersolid 2008

18 - 22 August 2008

Pressure relaxation on annealing in solid helium

N. Mulders
University of Delaware, USA

X-ray scattering experiments on Solid helium

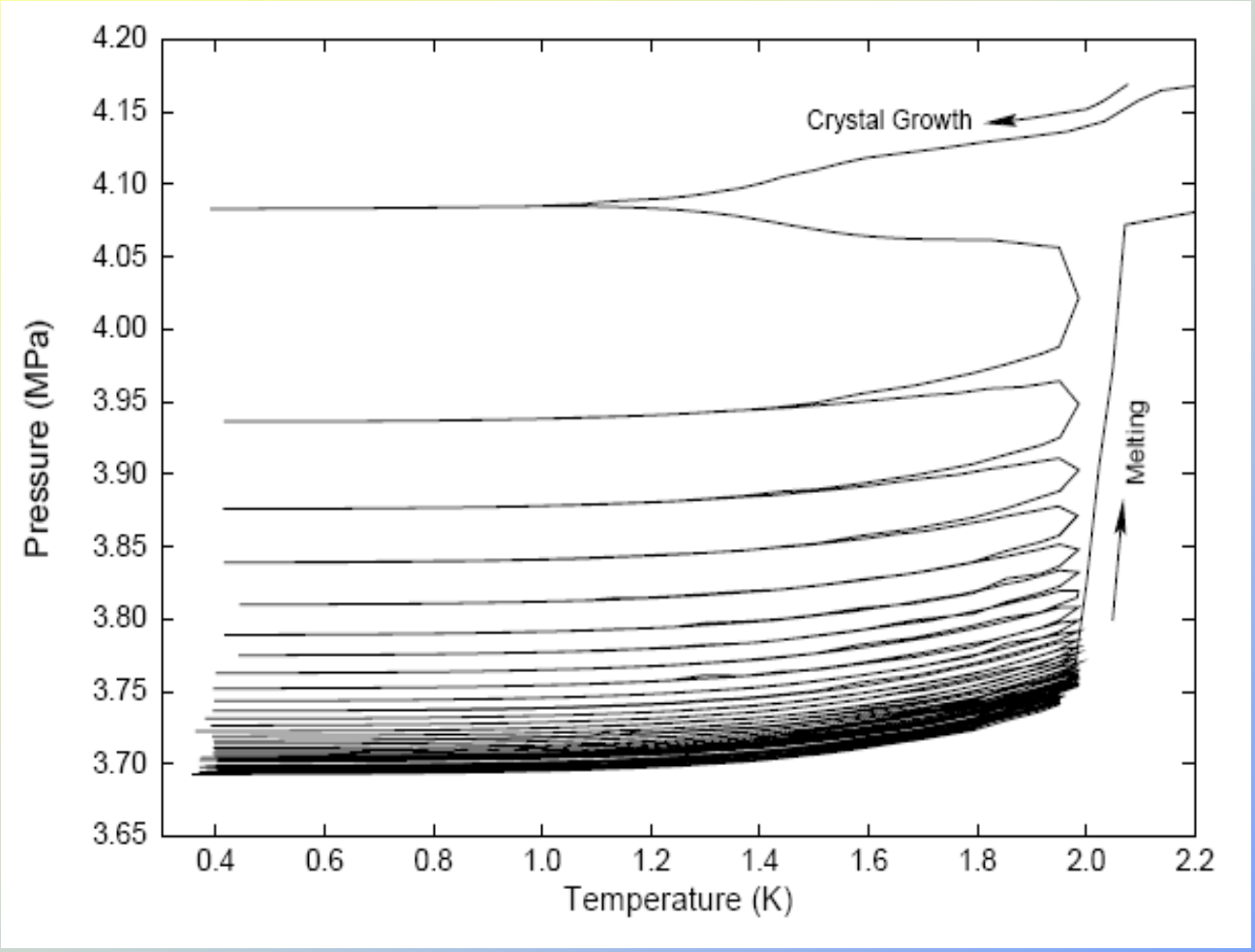
Norbert Mulders, University of Delaware

Larry Lurio, Northern Illinois U.

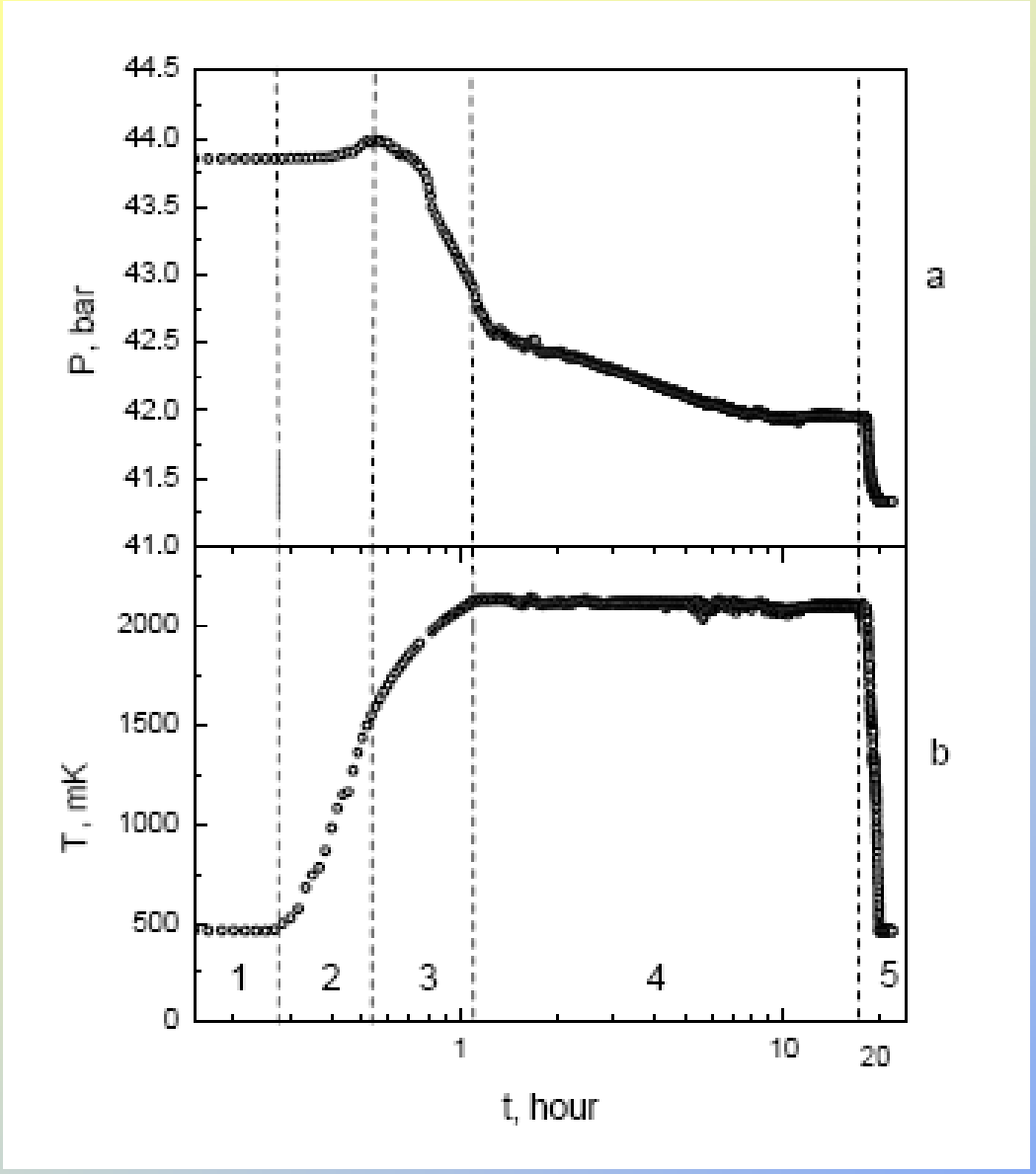
Clem Burns, Western Michigan U.

Josh West & Moses Chan, Penn State U.

Pressure drops on annealing



Shah/Simmons



Grigor'ev et al.

What can we learn?

- We know the compressibility of the "clean" solid.
- From ΔP we can find ΔV, the extra volume that became available on annealing.
- If we know the "volume per defect" we can find the number of defects

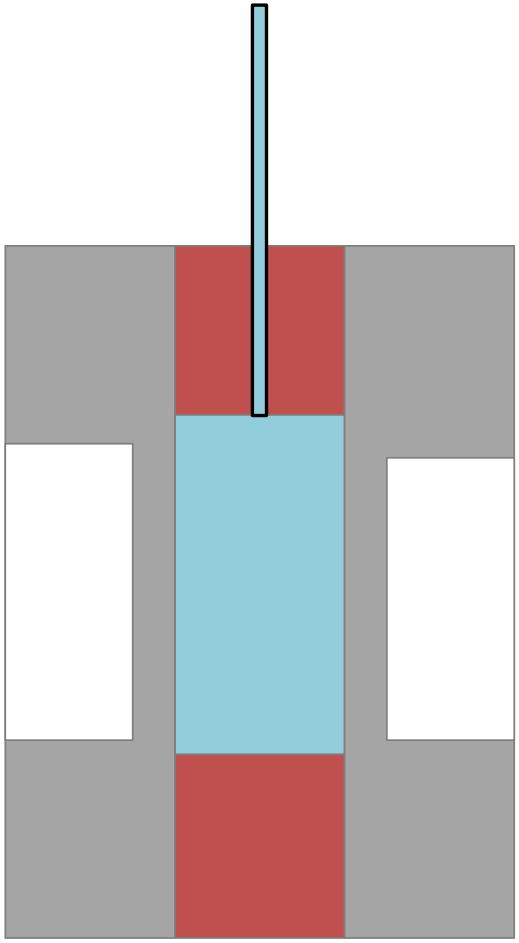
For example:

- Assume there is a glassy component with a density about 10% lower than the crystal.

- The pressure drops 4 bar on annealing

→ 10% of the solid was in the glassy state before we annealed it

Pressure cell

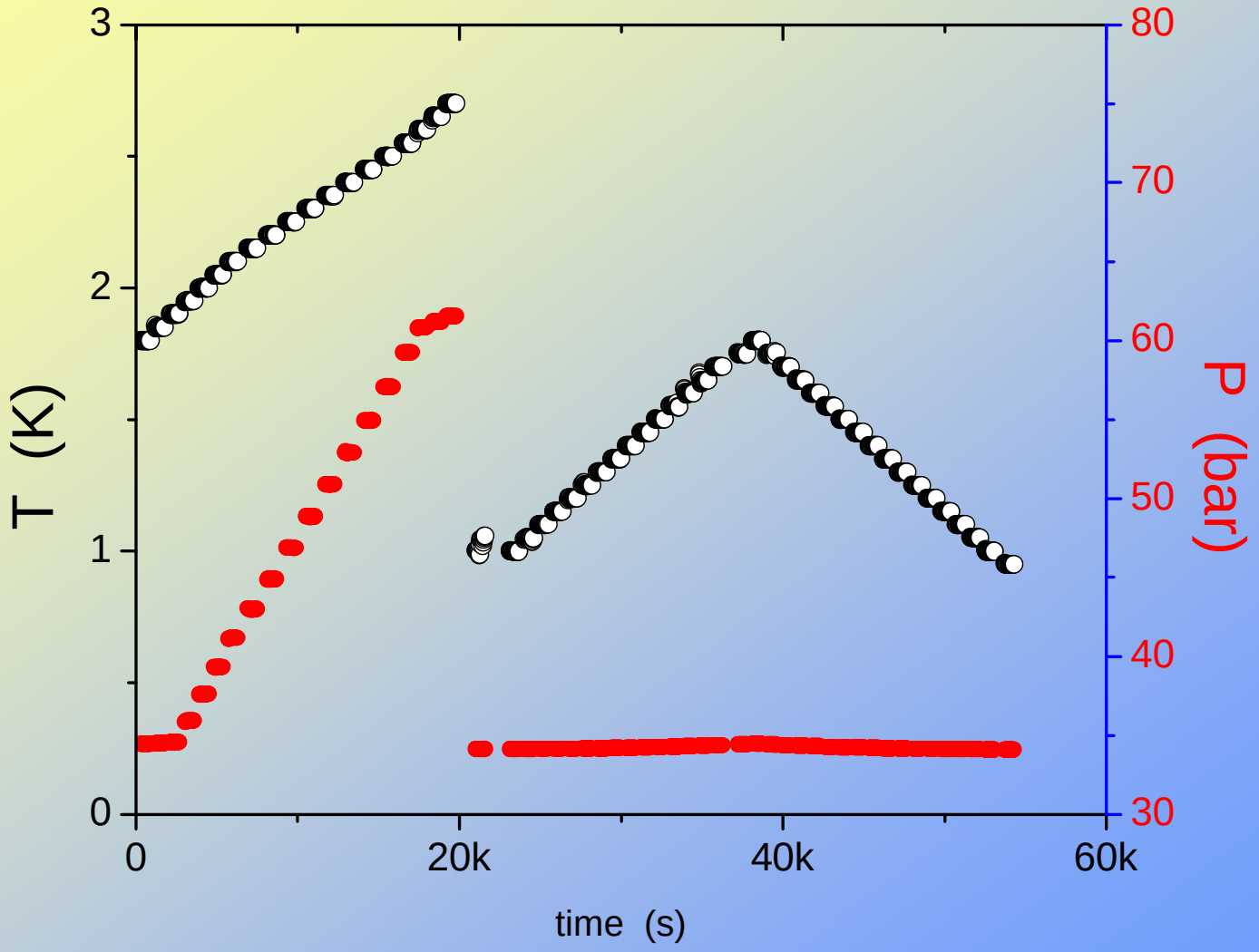


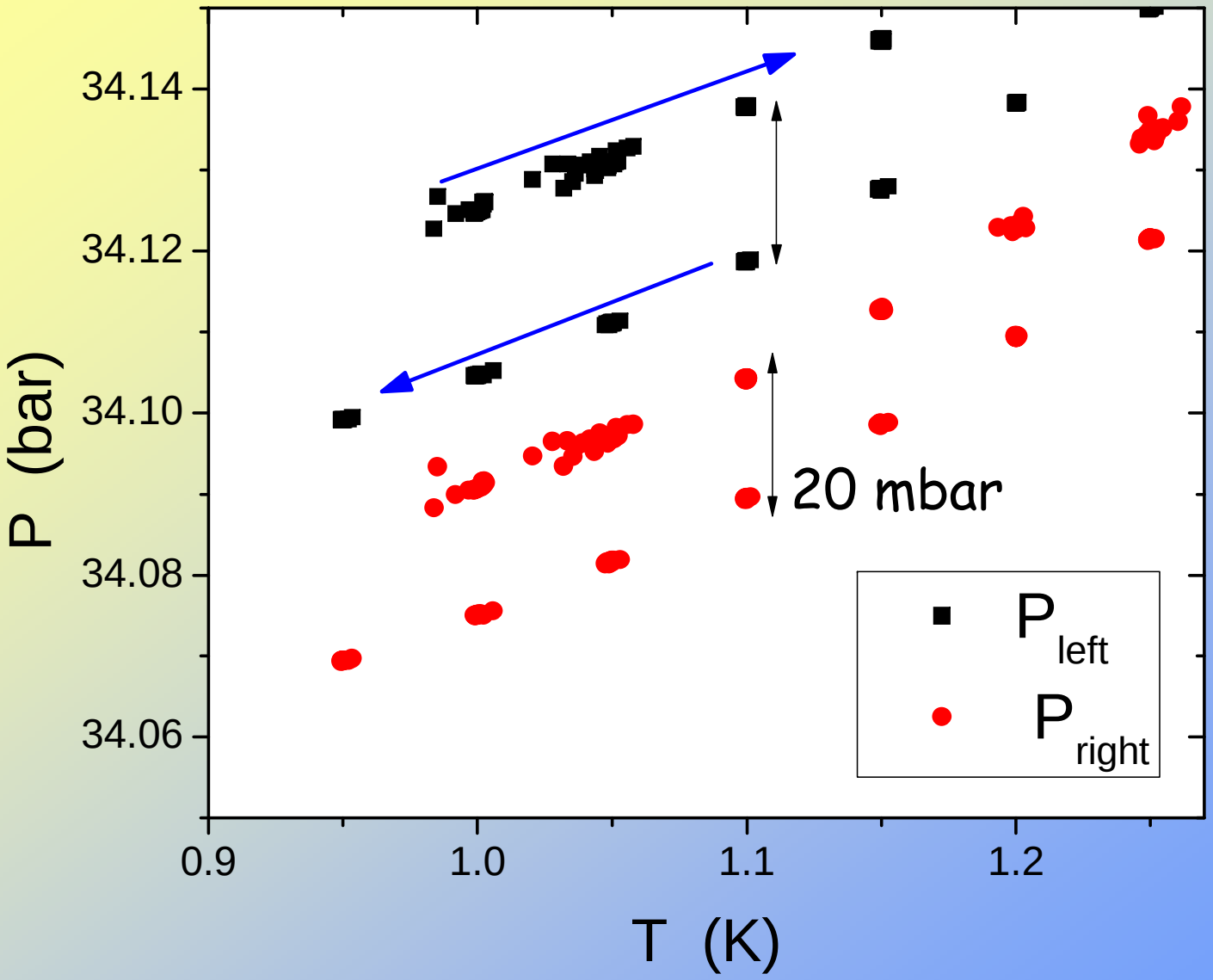
Pressure cell

15 mm diameter
8 mm width

Two transducers
facing each other

Temperrature "quench"



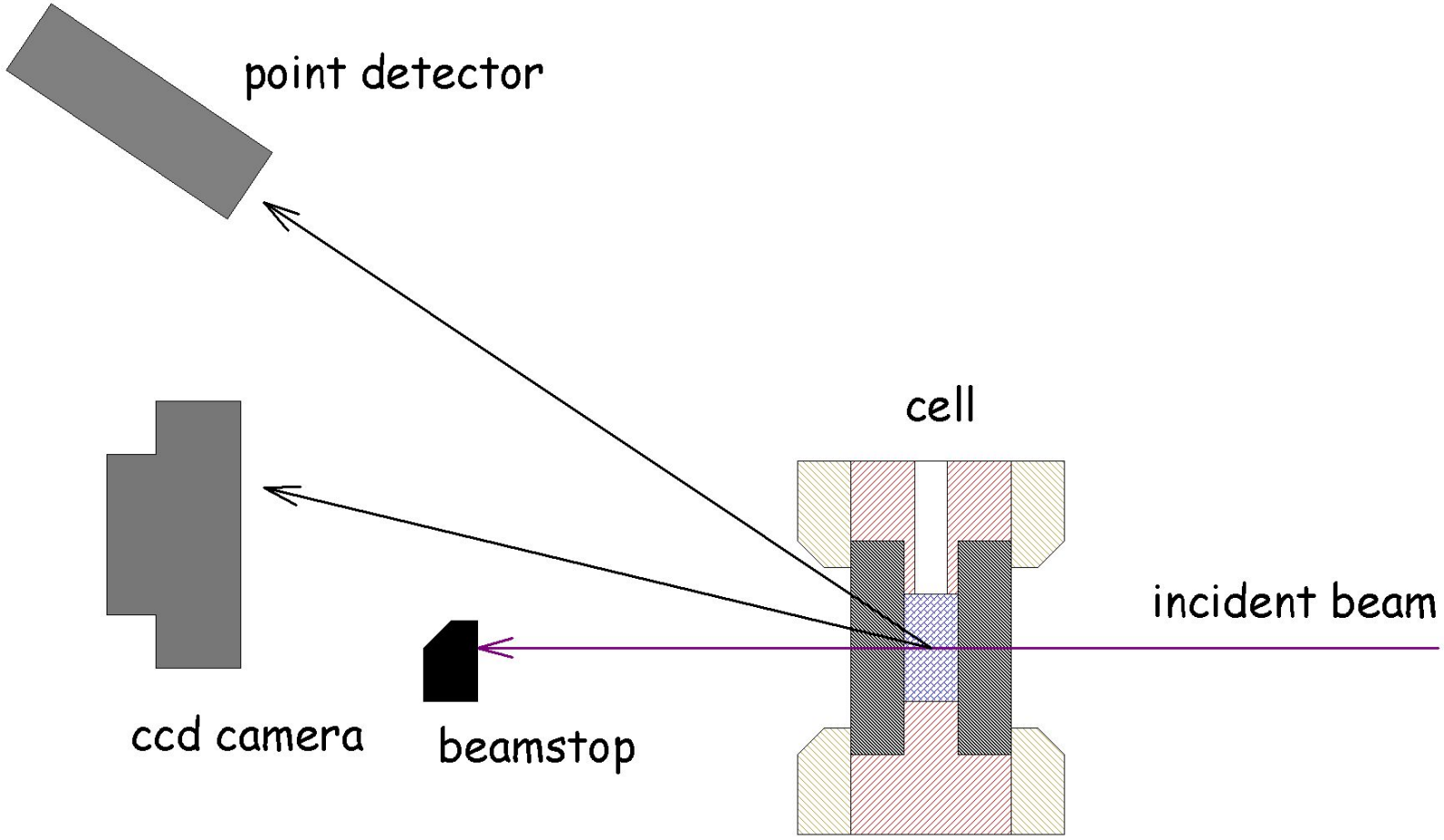

34.14
34.12
34.10
34.08
34.06
P (bar)
0.9
1.0
1.1
1.2
T (K)
20 mbar
P left
P right

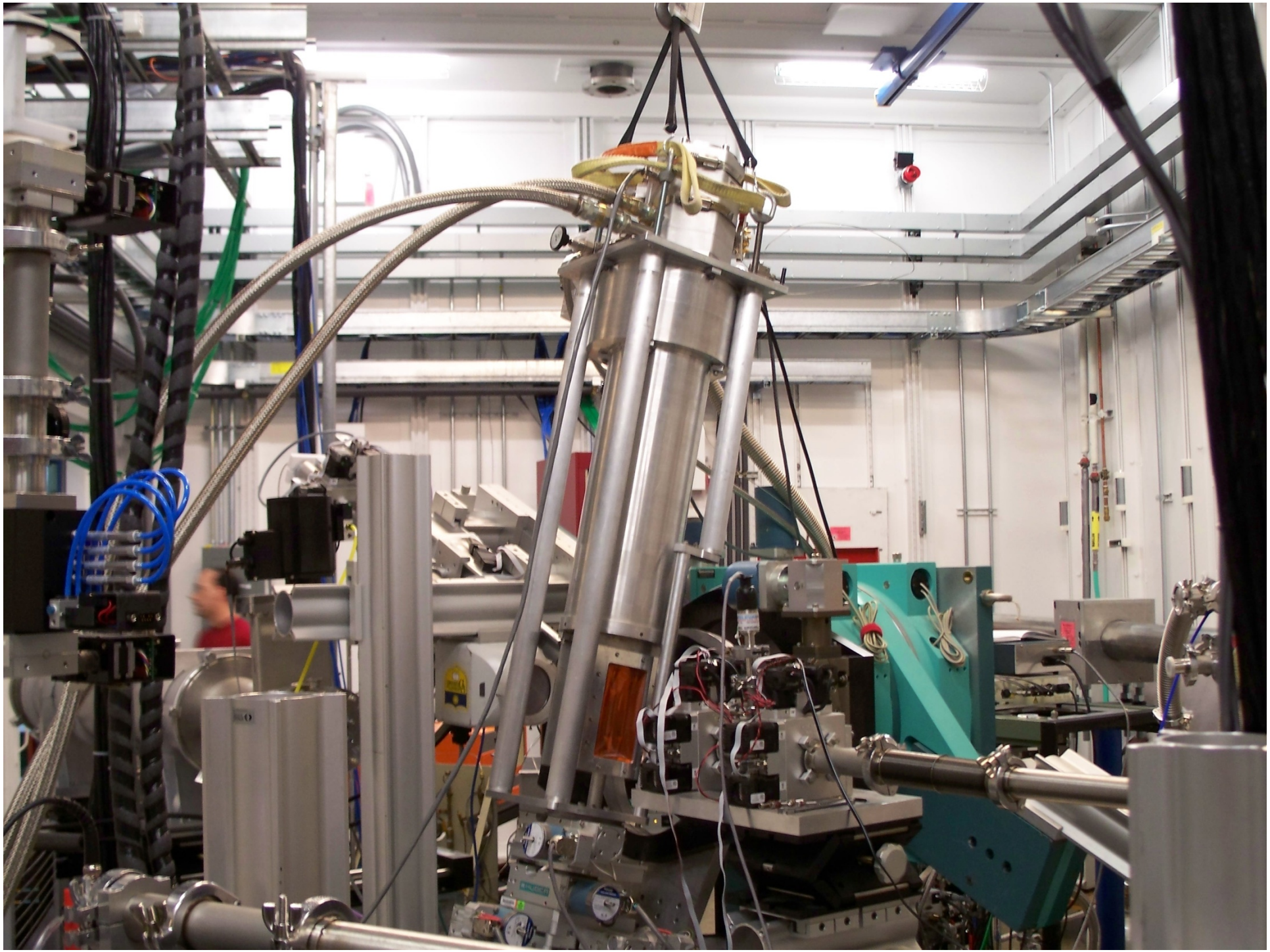
What is in the cell ?

- It cannot be a perfect crystal.
- It is a glass
- It's all in the grain boundaries
- Dislocations, dislocations !
- Liquid "droplets", "channels" etc.


Advanced Photon Source
Argonne National Laboratory
UNI-CAT
Sector 33
Argonne Central Campus
Central Lab/Office Bldg.
Argonne Guest House
APS Conference Center
Center for
anoscale Materials
Booster/Injector
Rf/Extraction Bldg.
Linac
Storage Ring
Experiment Hall
IMM-CAT
Sector 8
Lab/Office
Module

X-ray diffraction set-up





"All these experiments take a long time."

H. Kojima

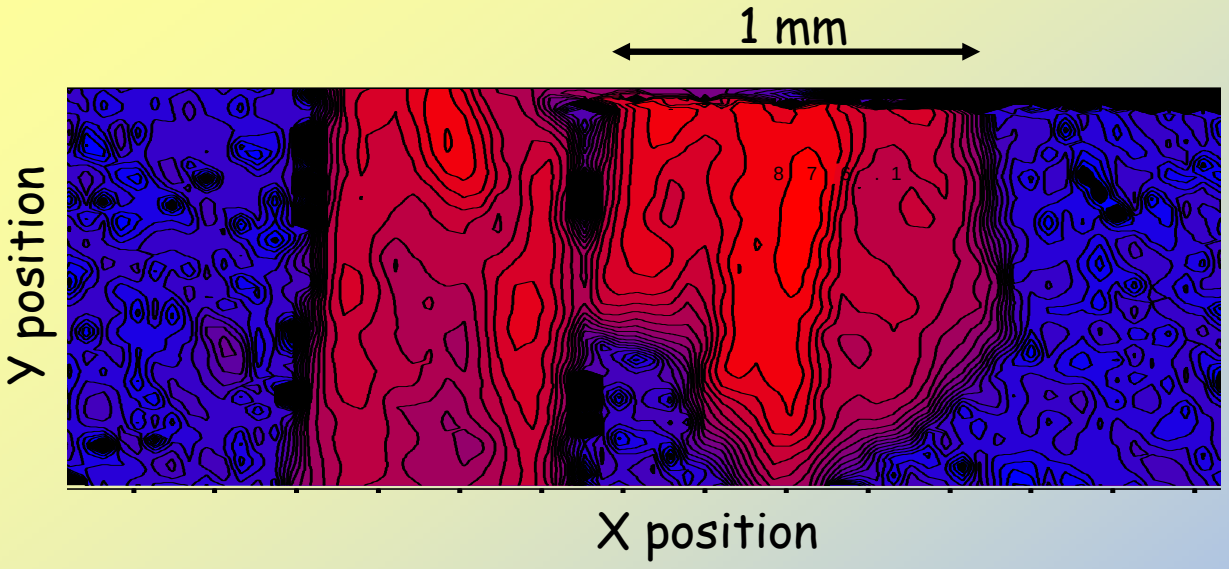
"Let me get this right. We must do a low temperature experiment *and* an x-ray experiment, and we have five days to do it?"

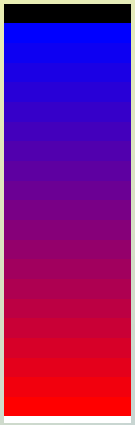
J. T. West

I. Experiments on crystals

All crystals were grown at constant pressure,

P = 60 bar, T_M = 2.6 K

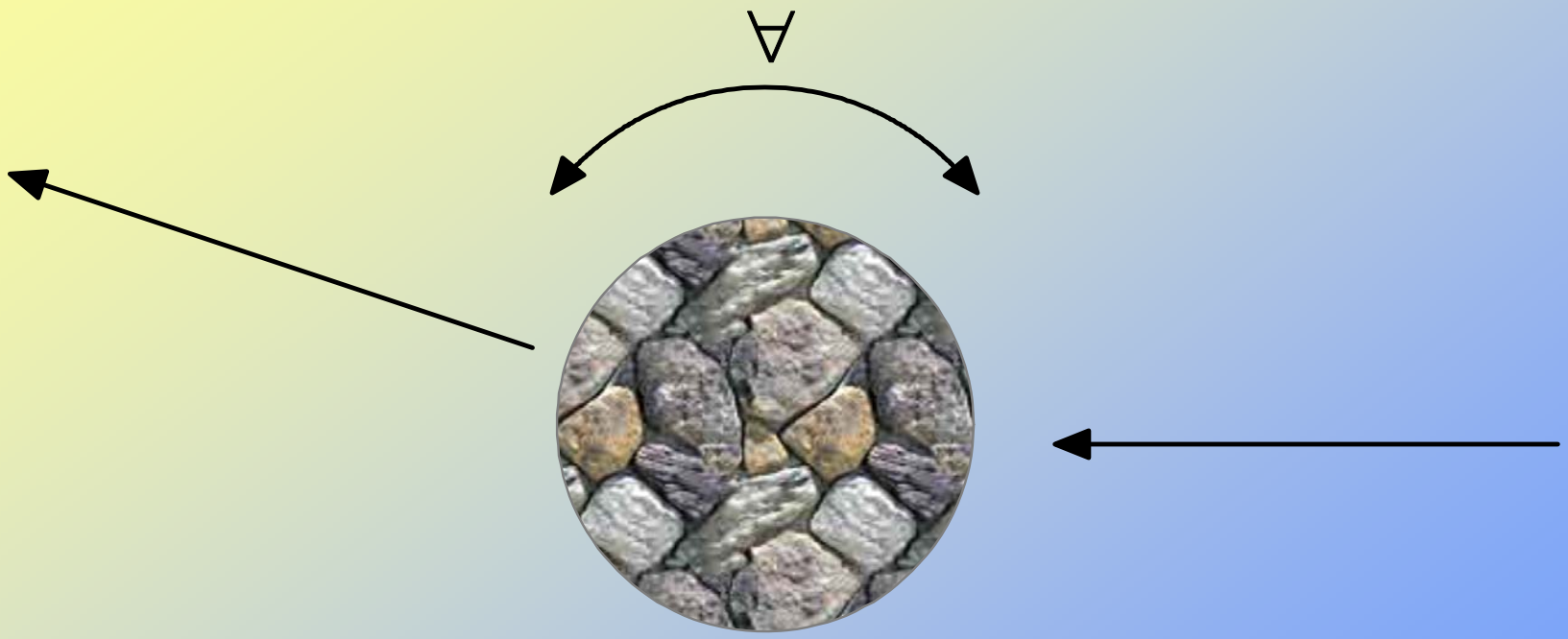




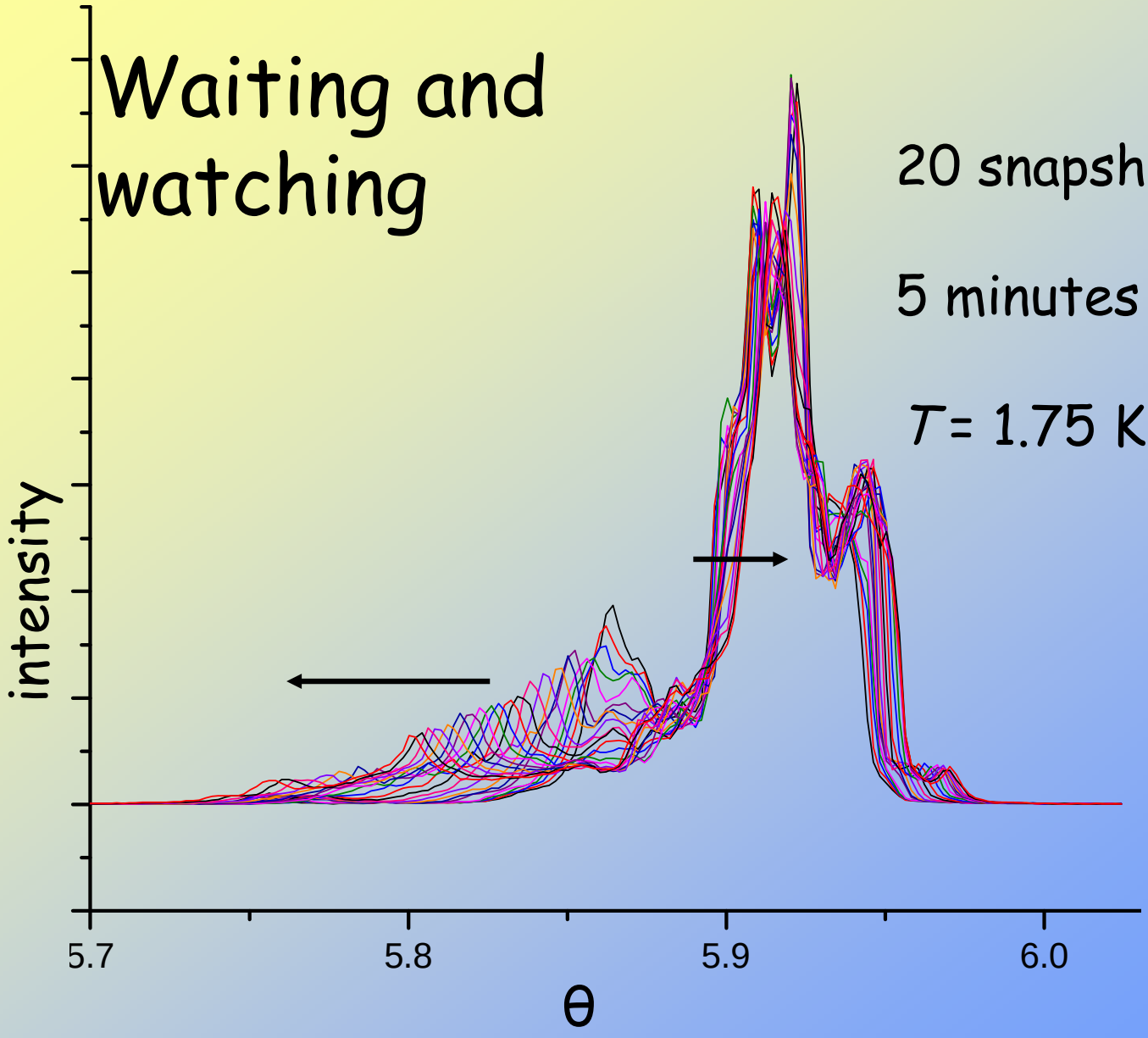
Mapping a crystal

Typically, crystals span the tube diameter and are a few mm wide.

Looking at "strain"



Waiting and watching

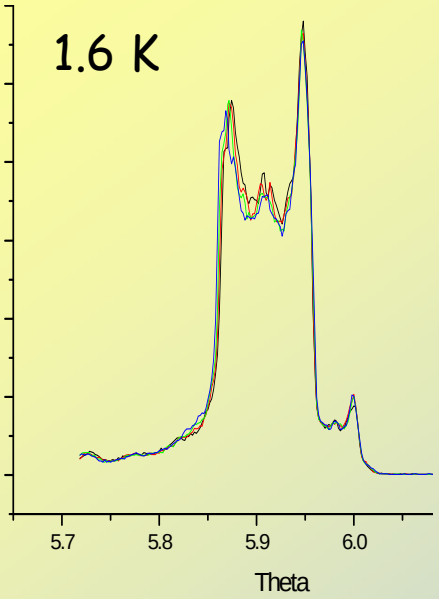


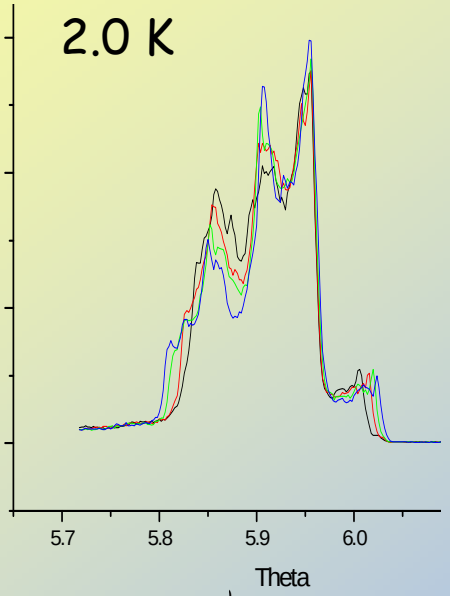
20 snapshots

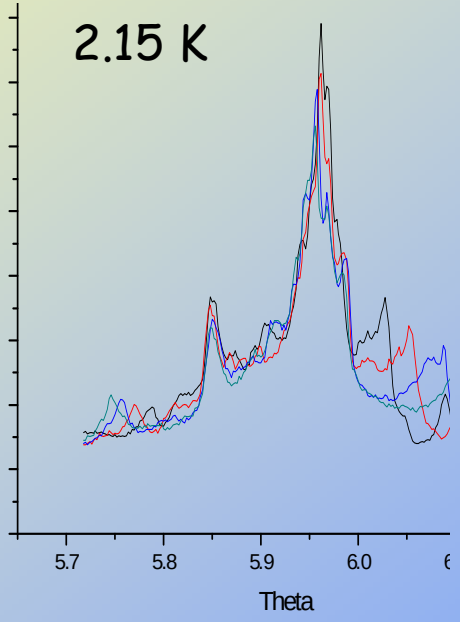
5 minutes apart

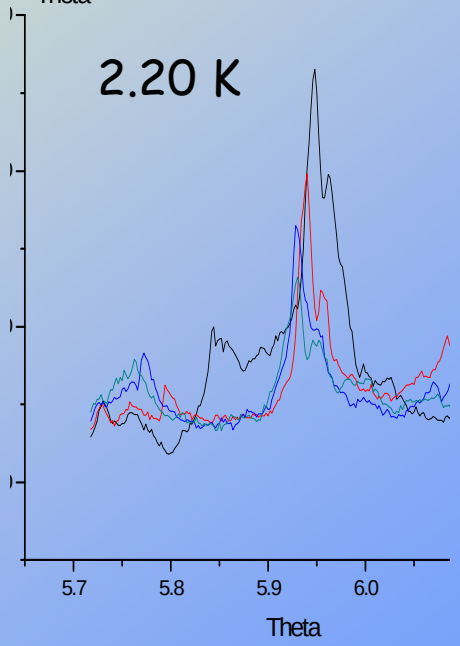
T = 1.75 K

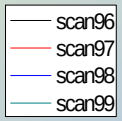
Crystal in motion



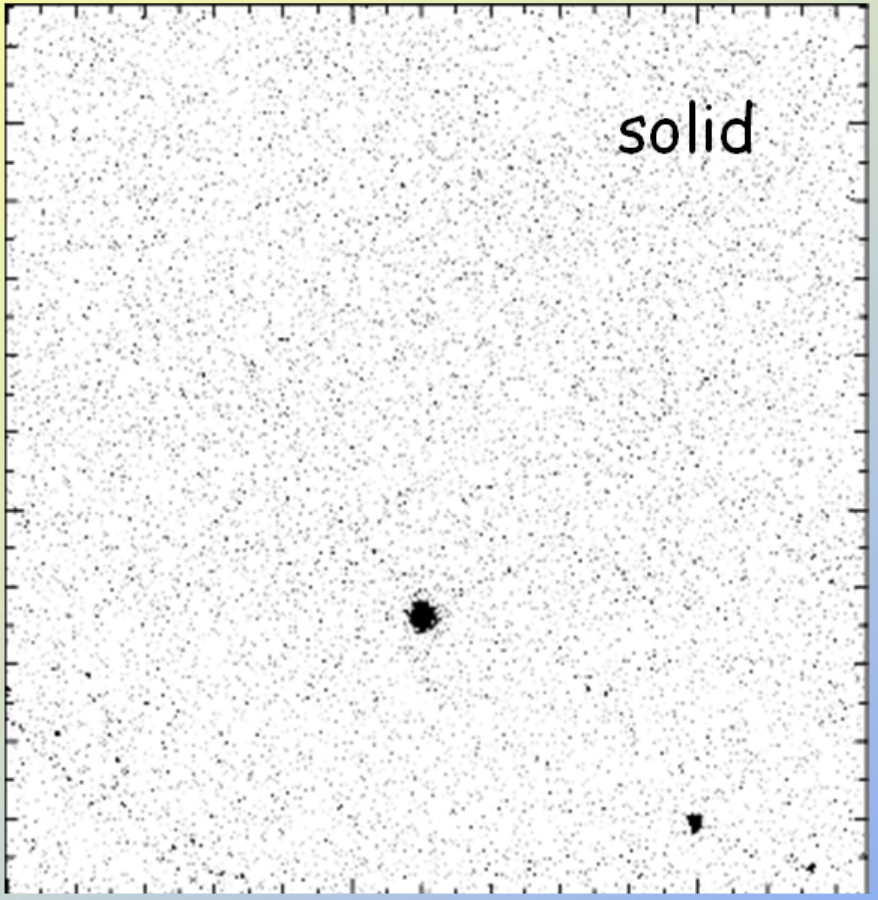


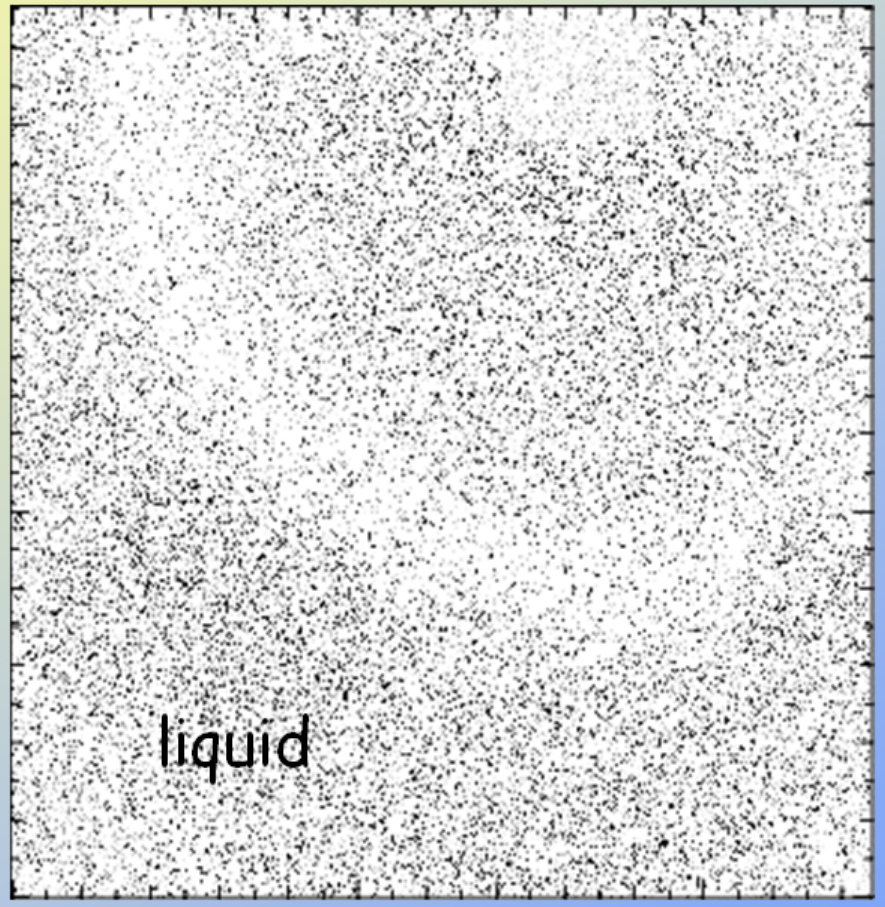






And gone !


solid


liquid

Conclusions I

- Constant pressure crystals are fairly large but severely strained.

- Some mobility 1.75 K < T < 2.15 K (60 bar, T_M = 2.6K),

  rapid motion at larger temperature

II. Solid helium in aerogel

Motivation?

R&R: NCRIf → 20%

"due to disorder"

Logical step: Build in disorder

But also

1. Shear modulus and NCRIf track

2. Shear modulus drops because dislocation lines become mobile ?

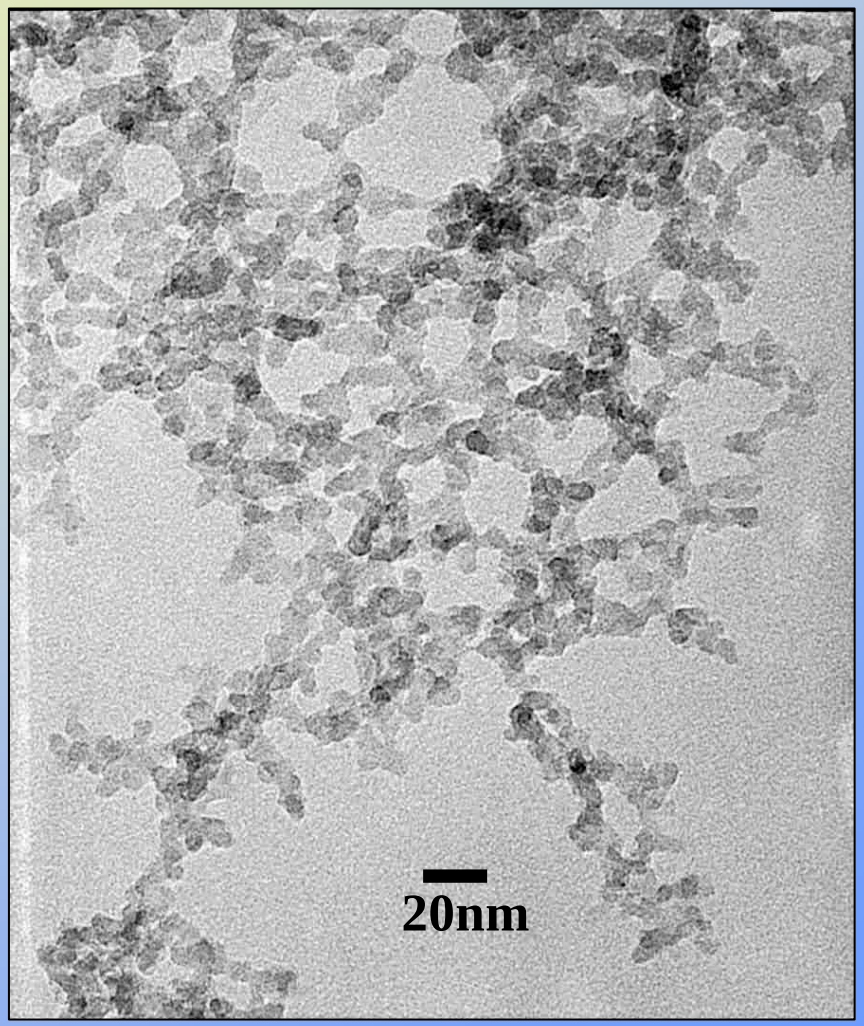
→ Pin the dislocations and both shear modulus and NCRIf will remain high up to some high temperature ???

Aerogels ???

Very porous silica glass,
90% --- 99% open space

Large surface area,
1000 m^2/g

→ Small primary particles,
2 – 5 nm diameter



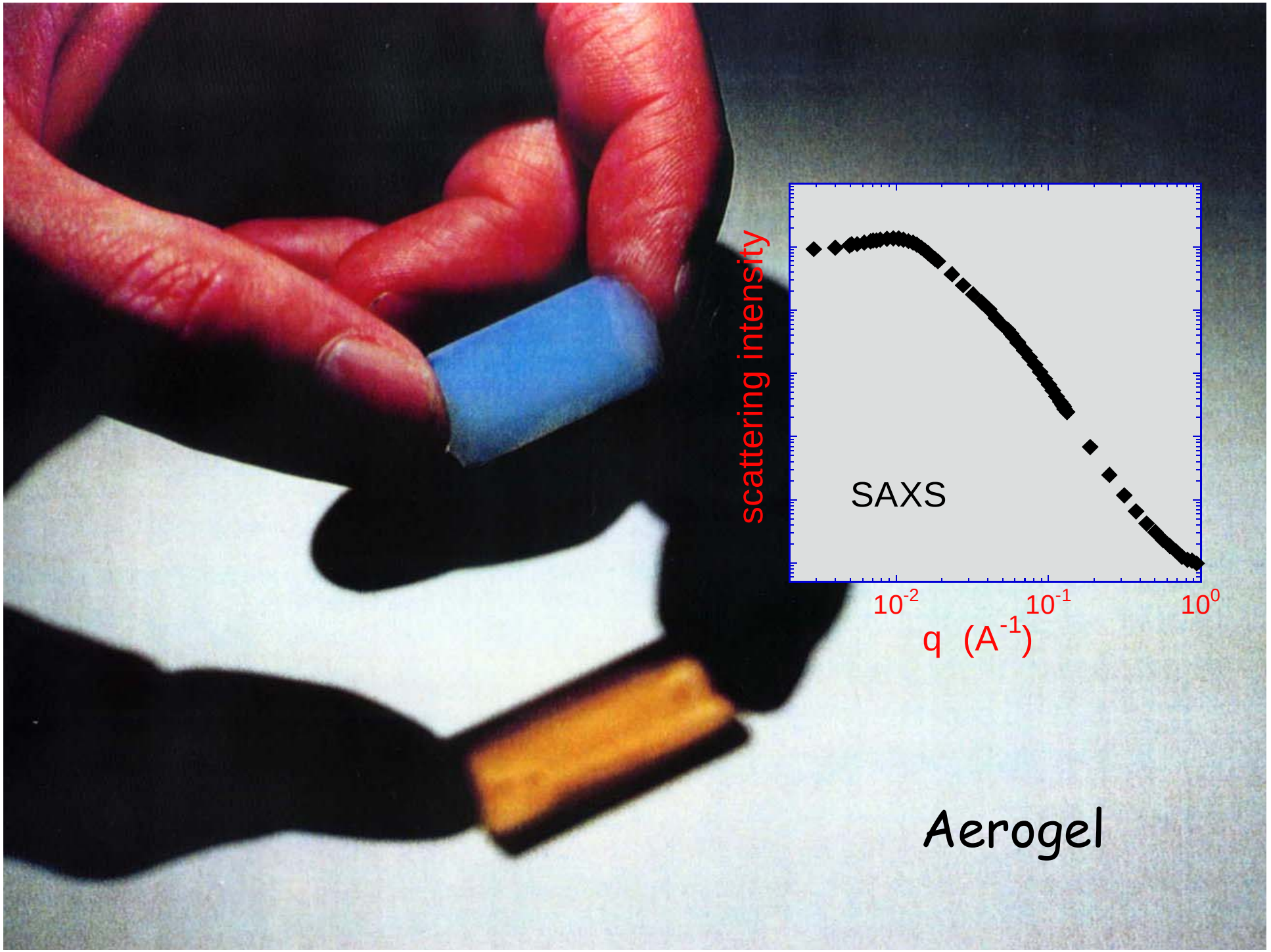
TEM picture of 5% dense silica aerogel

Herman and co-workers,
U. Alberta

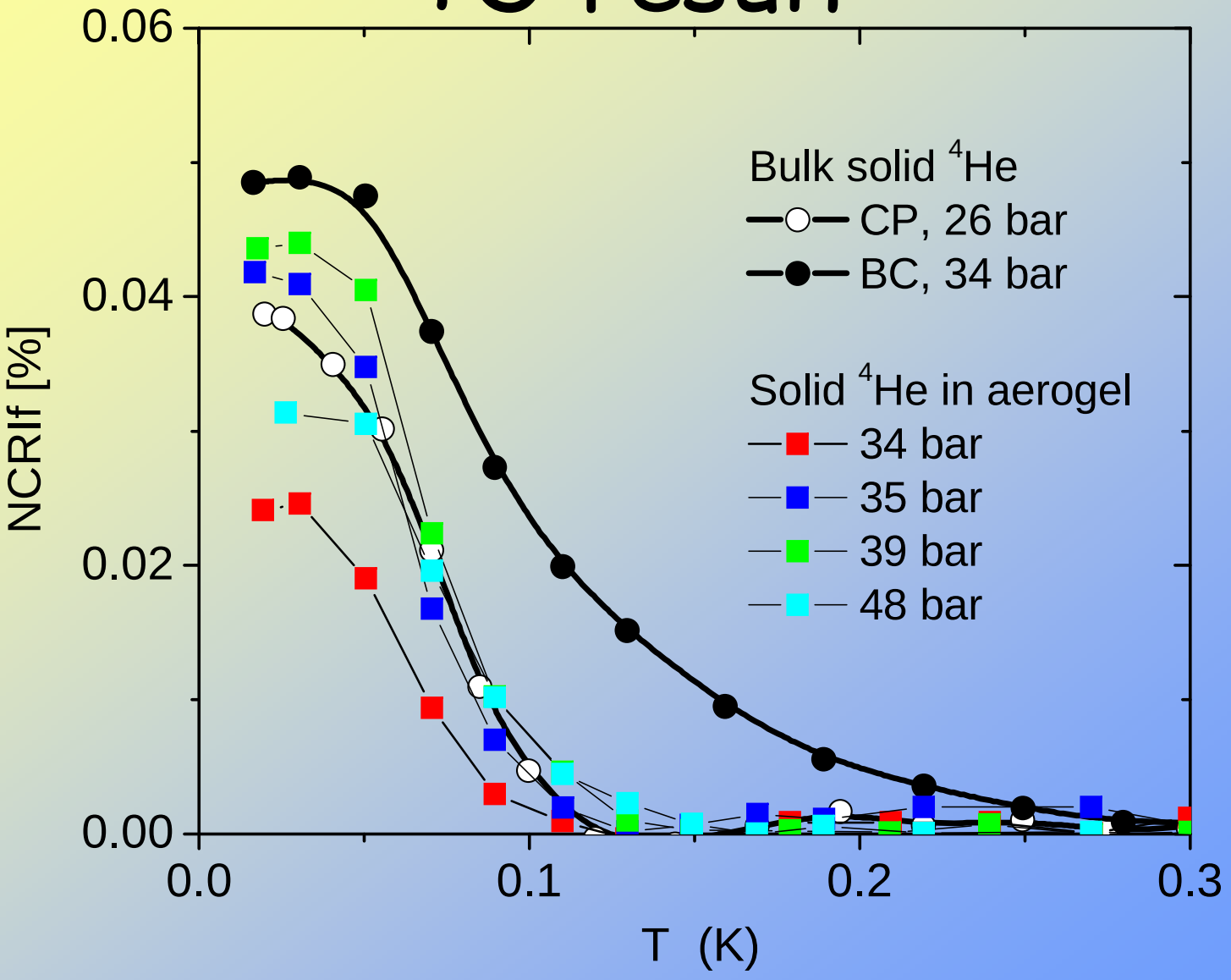
The large surface-to-volume ratio, 100 m^2/cm^3 → ~3% of the solid helium is within 3 Å of a silica surface.

No helium is more than ~200 Å away from silica.

The mean free path, the mean distance between pinning sites ~500 Å.


scattering intensity
SAXS
10^{-2}
10^{-1}
10^{0}
q (A^{-1})
Aerogel

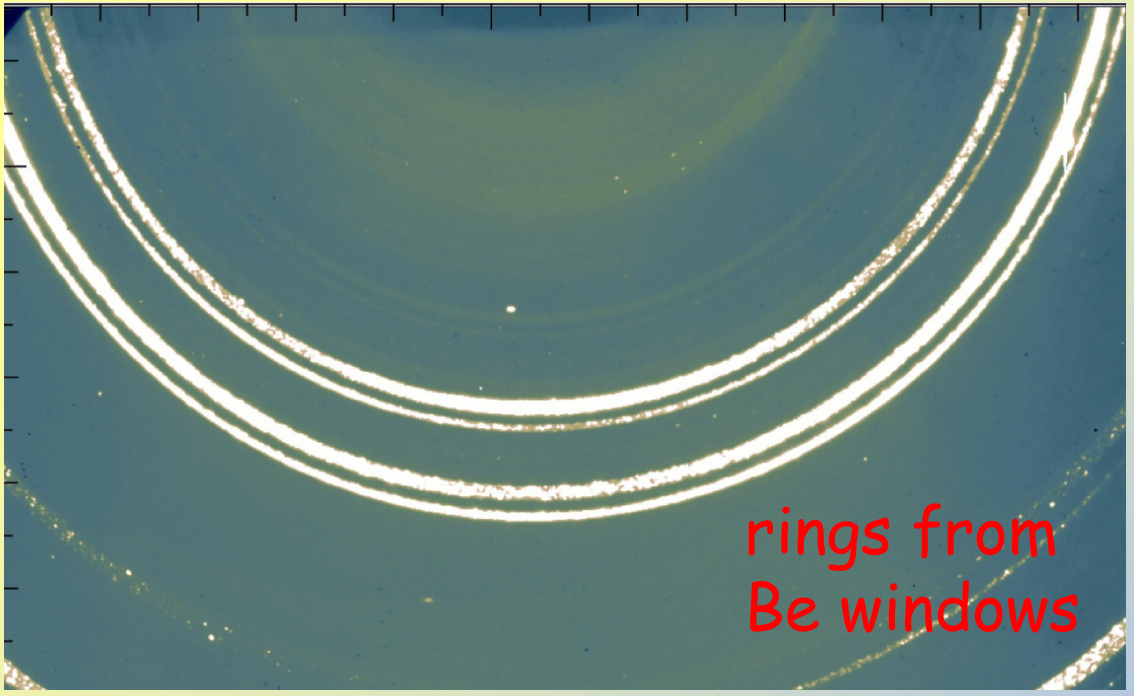
TO result


0.06
0.04
0.02
0.00
NCRIf [%]
0.0
0.1
0.2
0.3
T (K)
Bulk solid ^{4}He
CP, 26 bar
BC, 34 bar
Solid ^{4}He in aerogel
34 bar
35 bar
39 bar
48 bar

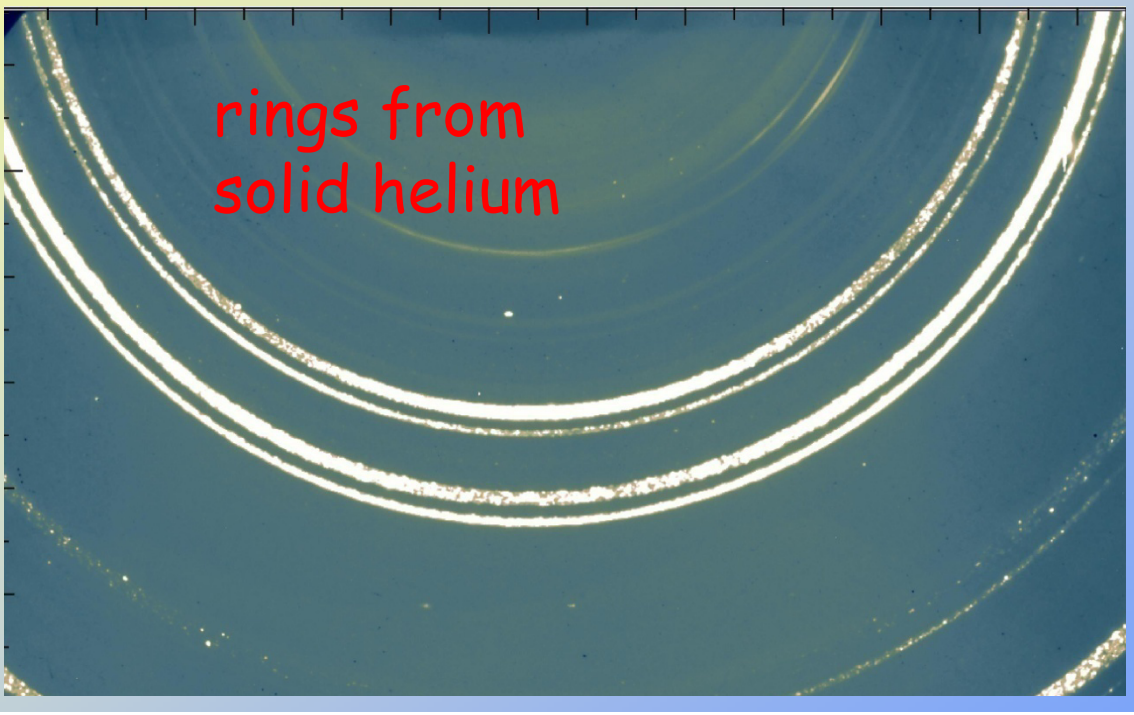
We observe a transition similar to the good/pure samples !

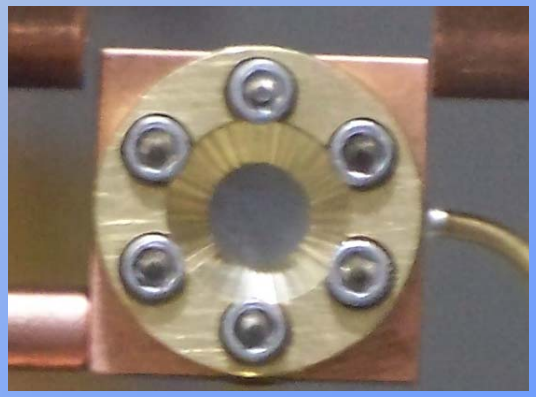
What sort of sample do we have ?

X-ray powder diffraction



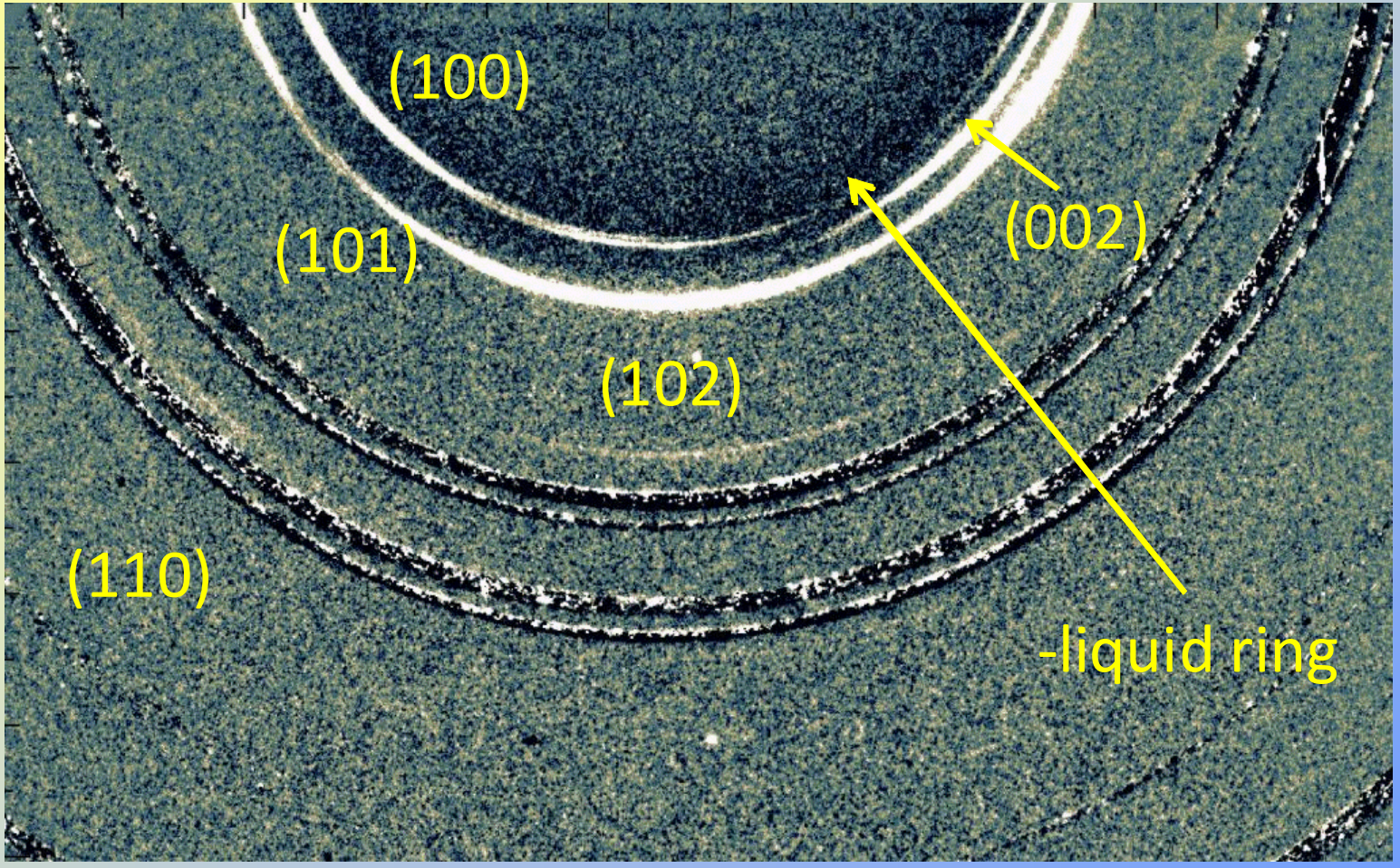
liquid





solid

Solid - liquid



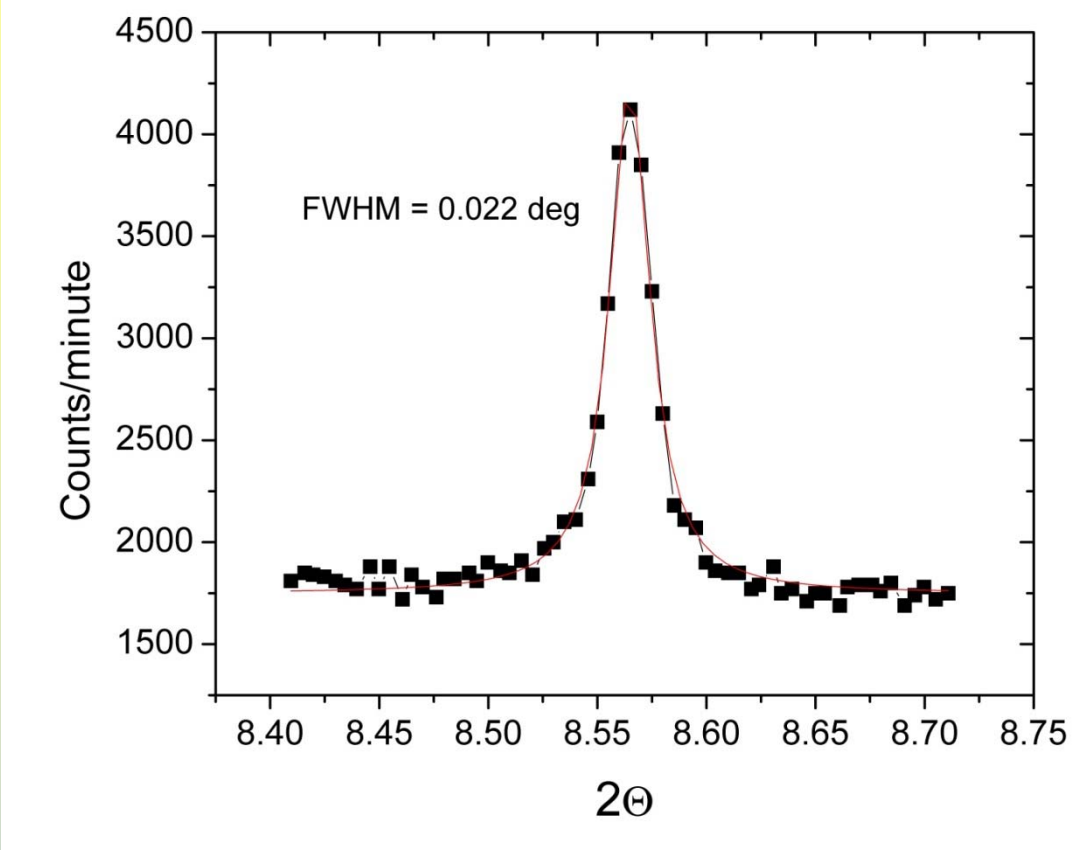
Clearly, we observe rings,
not isolated Bragg spots

→ we have a "powder"
not a single crystal

We can index the rings,
finding an HCP structure

Can we learn more?

From the diffraction peak width We can obtain the grain size:



$$D = \frac{S\lambda}{W_{FWHM}\cos\theta}$$

D ~ 1000Å

Connection with NCRIf ?

A crystallite size of 1000 Å

→ 30 m^2/cm^3 of grain boundaries

→ ~ 1% of the solid within 3 Å

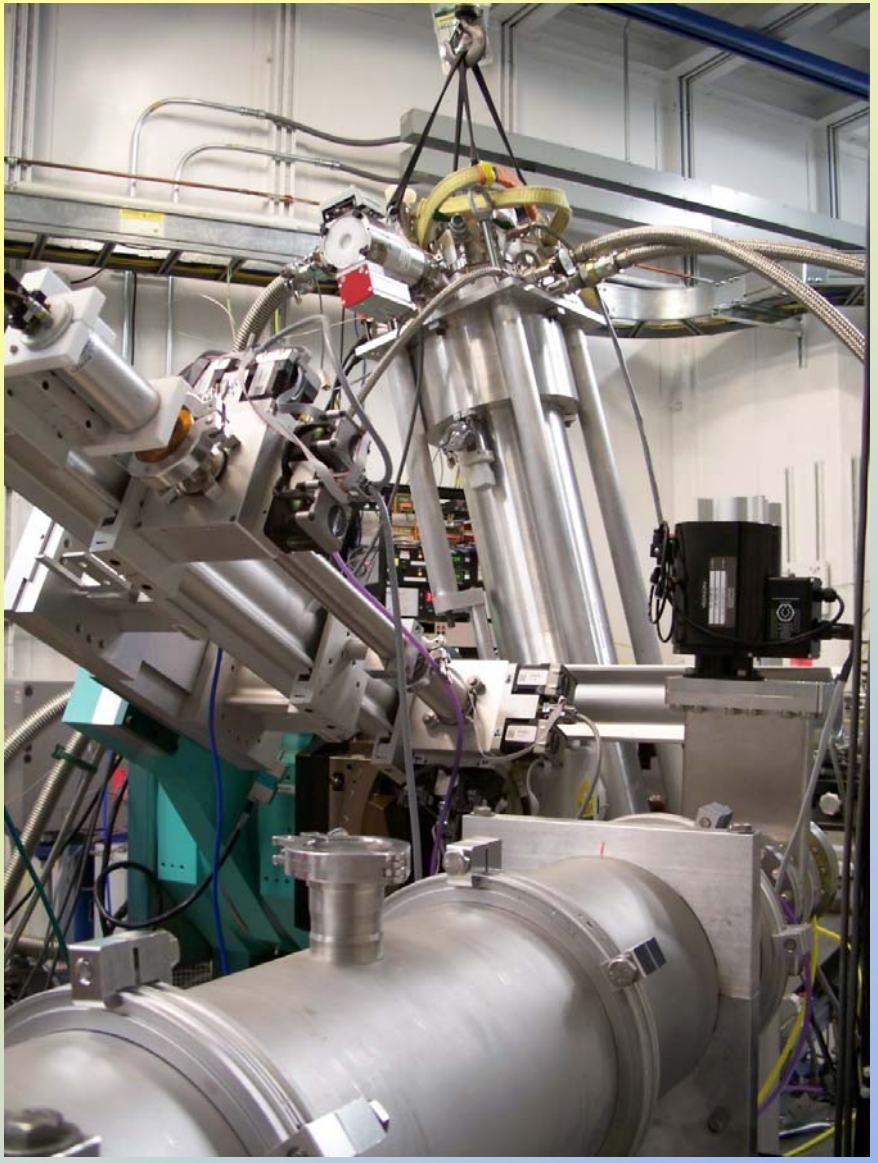
a much larger fraction than the actually observed NCRIf

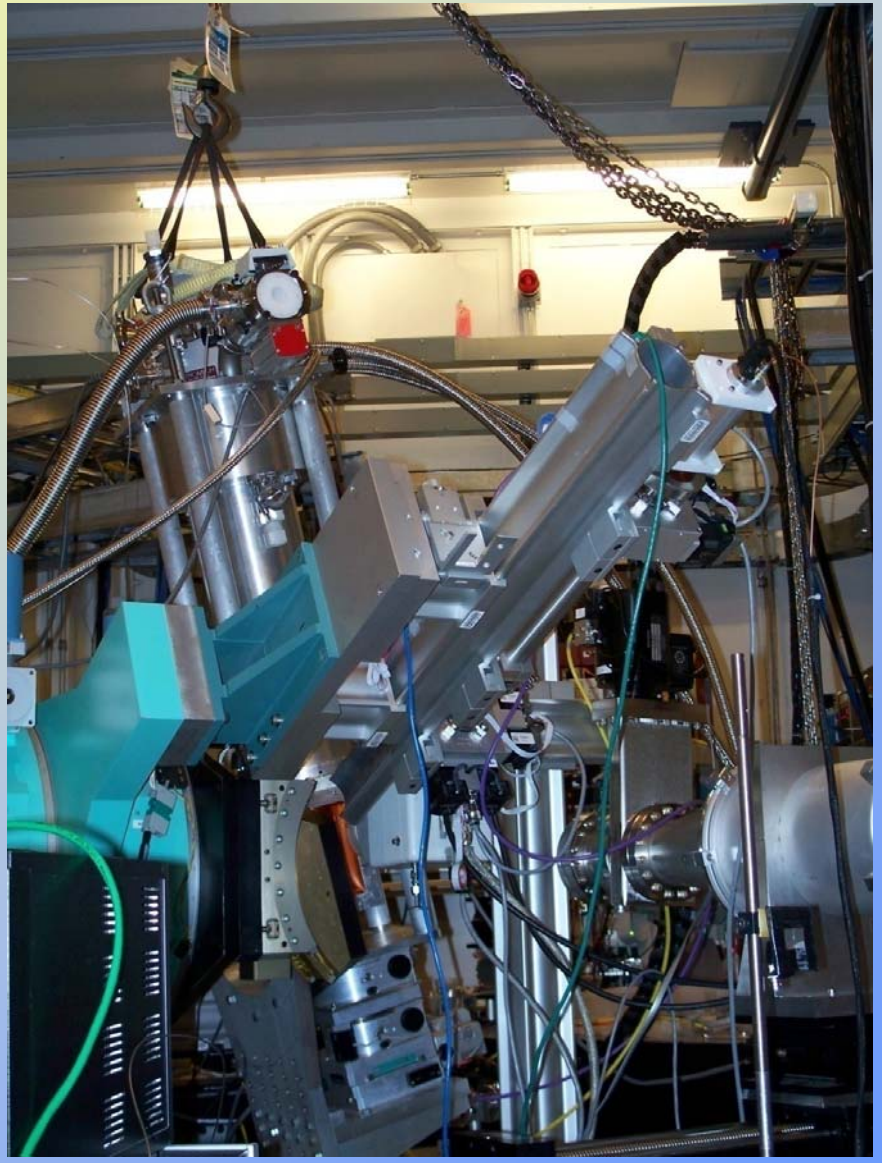
Conclusions II

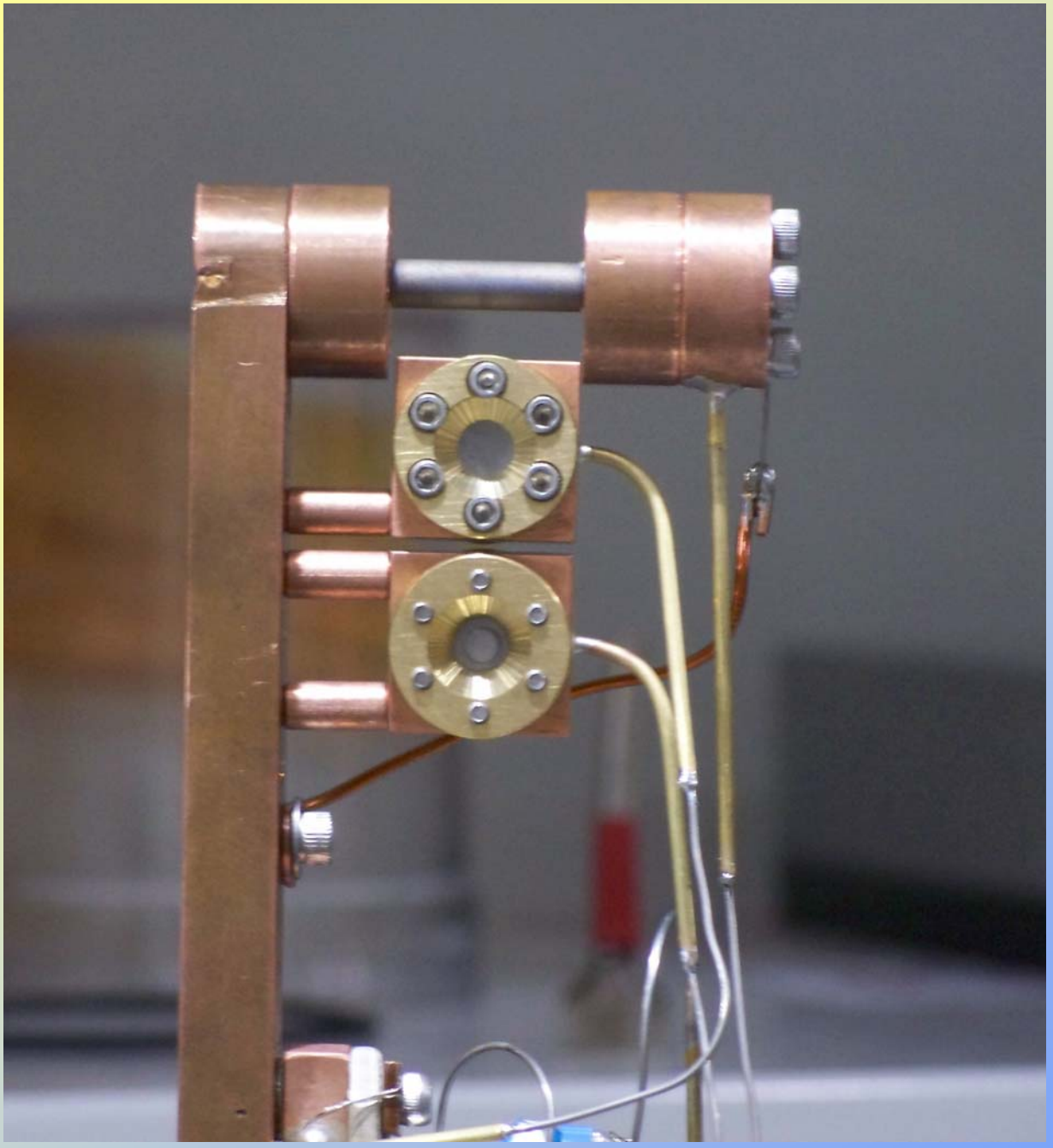
We have made very disordered samples, consisting of 1000 Å diameter HCP crystallites.

We observe a super solid transition similar to that in "high quality" samples.

???







Ambition

TO results

Josh West, PSU