Characterizing Recrystallization Kinetics with Diffraction

Andrew Ying[1], Jean Jordan-Sweet[2], I.C. Noyan[1]
Christian Witt[3], Robert Rosenberg[4]

1. Columbia University, Dept of Applied Physics and Applied Math
2. IBM, X20A - NSLS, Brookhaven National Lab
3. GlobalFoundries, Yorktown Research Center
4. IBM, Yorktown Research Center

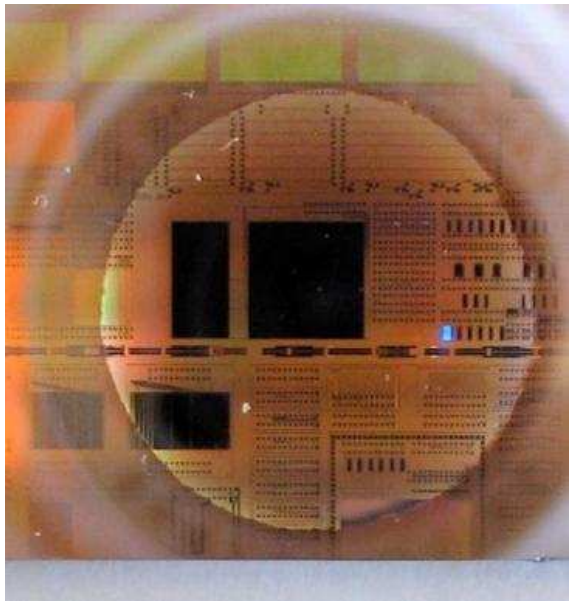
Purpose and Overview

- Our goal is to determine the recrystallization kinetics of metal films during annealing:
  - How fast do grains grow under various conditions?
  - What is the driving force?
  - Can we write a physics-based model for such growth?
  - We have tried both laboratory and synchrotron x-ray diffraction measurements.
  - While we have some success, more intense sources are needed for measuring small, buried, volumes.

SAMPLES

- Cu electroplated samples on PVD Cu seed.
- Plated Cu thickness is between .2 -1 microns.
- **Plating conditions:**
  - 0.4 V insertion voltage
  - 10 sec. trench-fill @ 5.6 mA, (~0.6 V)
  - 30 sec. overburden plating @56 mA (~1.2 V)

Plating over both blanket areas and over trenches.

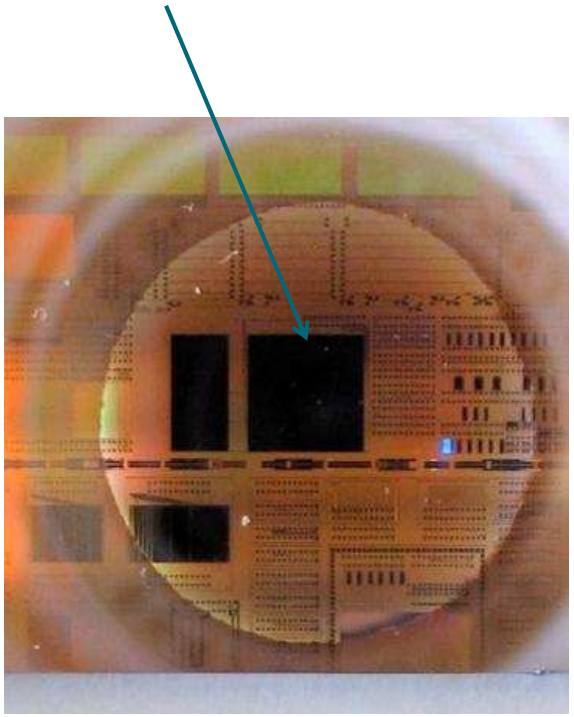


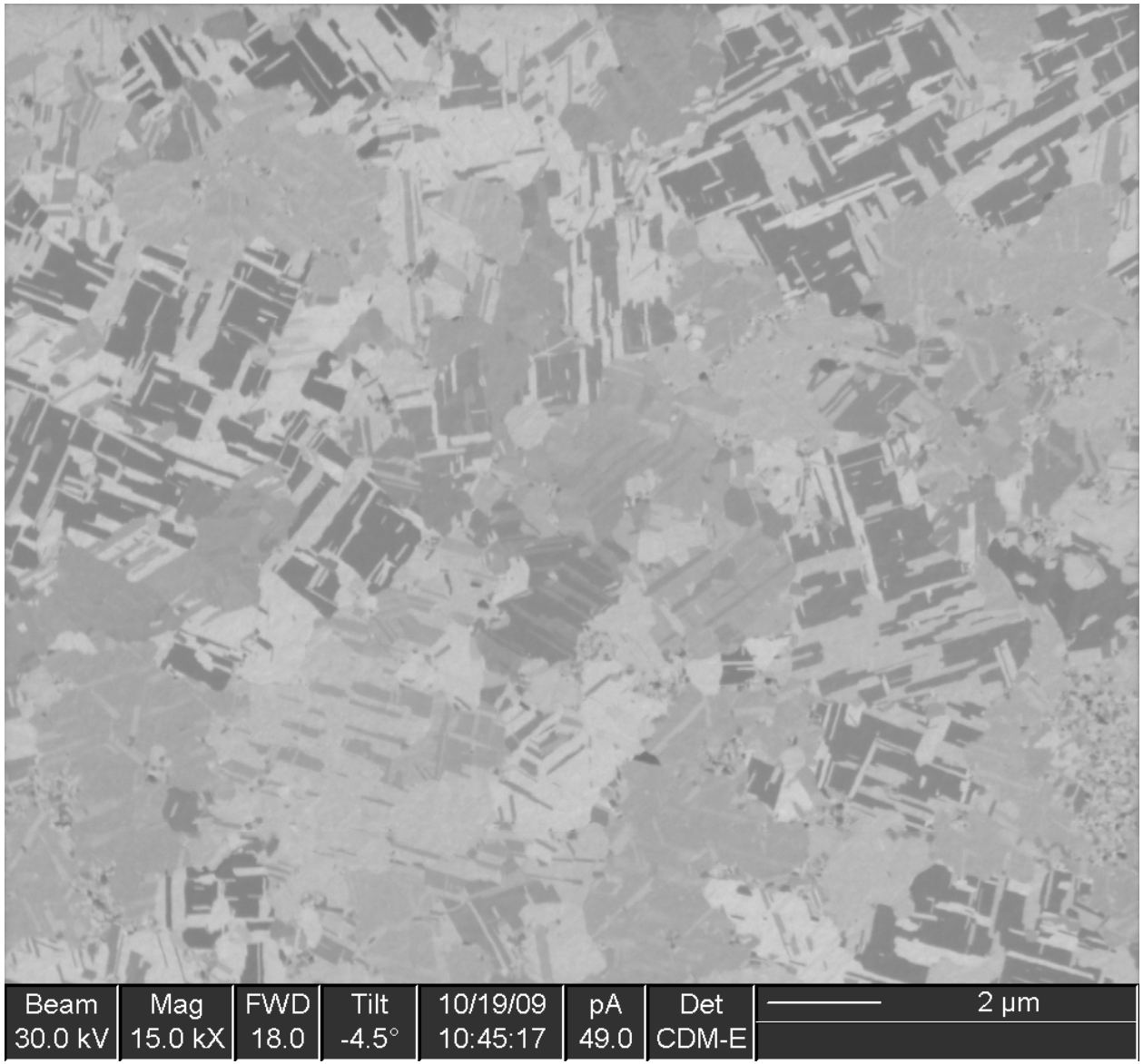
1-D detector Laboratory X-ray Experiments

- Radial ($2\theta/\theta$) diffraction profiles on Siemens D500 and D5000 machines.
- We only mapped the 111 reflection region vs. time, 37-46° 2θ.
- Large beam spot (1 cm x 4 mm)
- 45kV/35 mA (IBM); 40 kV/20 mA (Columbia)

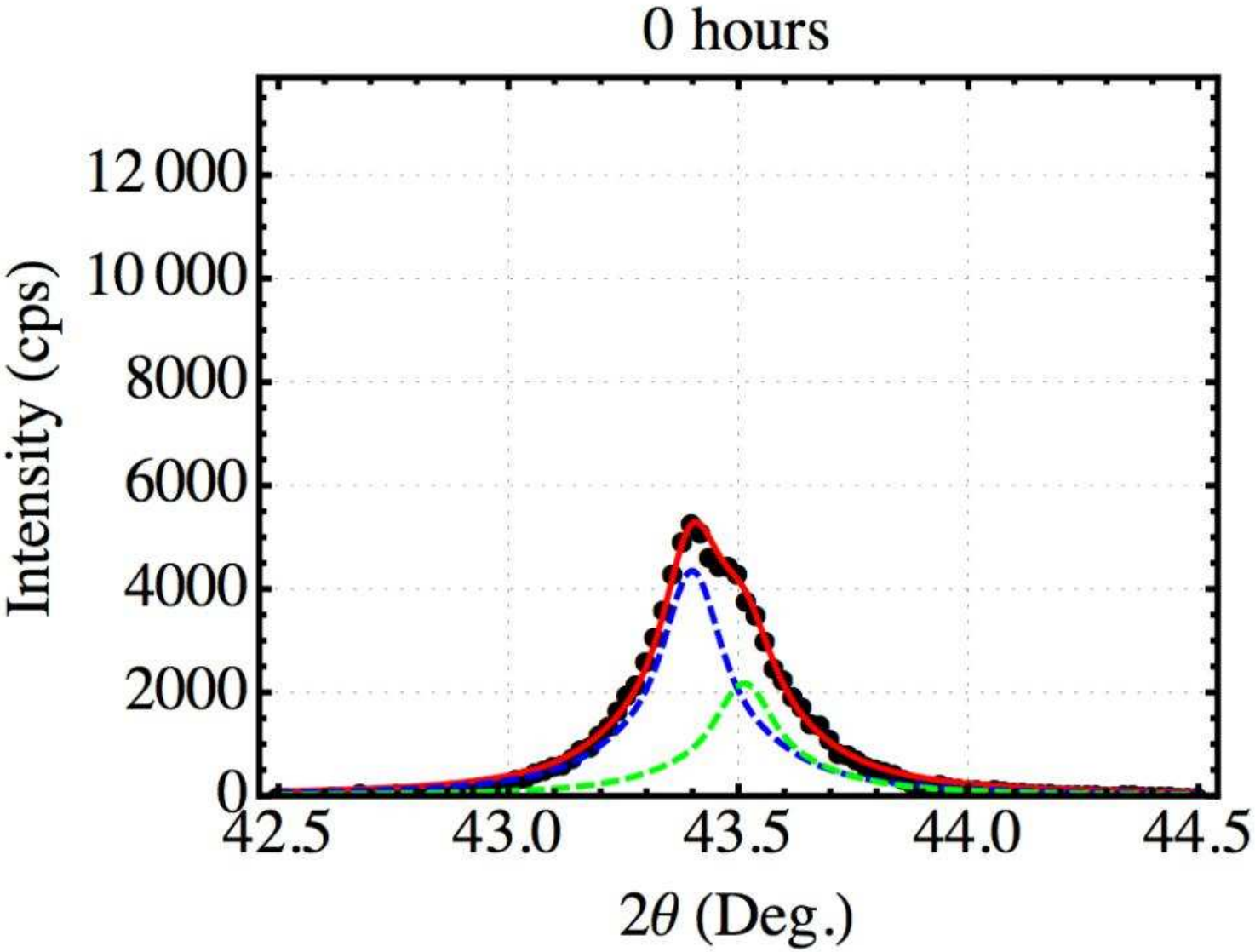
Sample: plated, quenched in liquid. N_2, kept at this temperature for 20 (96) hr, and then x-rayed.

Microstructure of
Sample 2.)
FEI FIB200TEM
Dense area

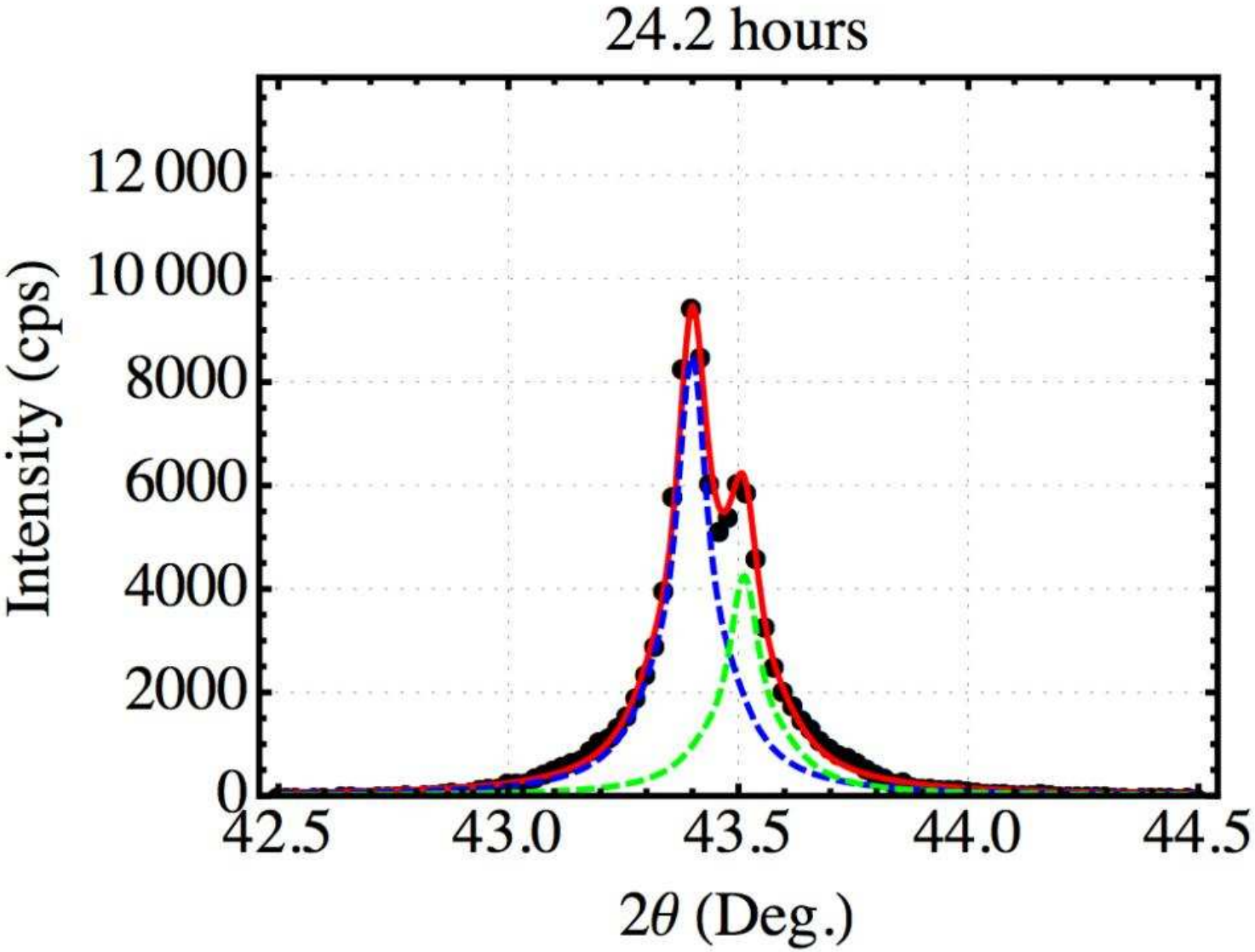




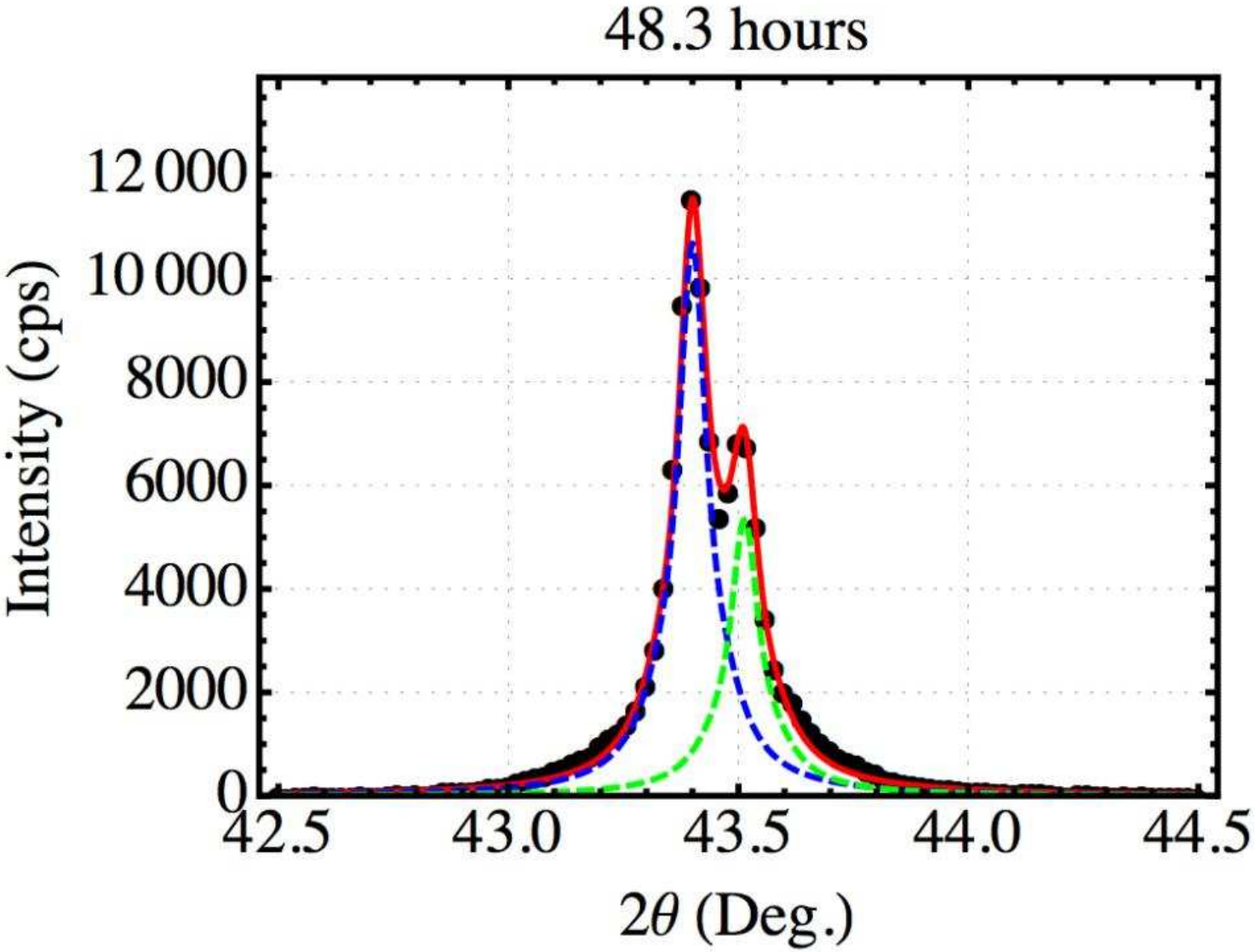
SAMPLE _2: Plated, quenched in liquid. N_2, stored for ~96 hr, and then x-rayed.



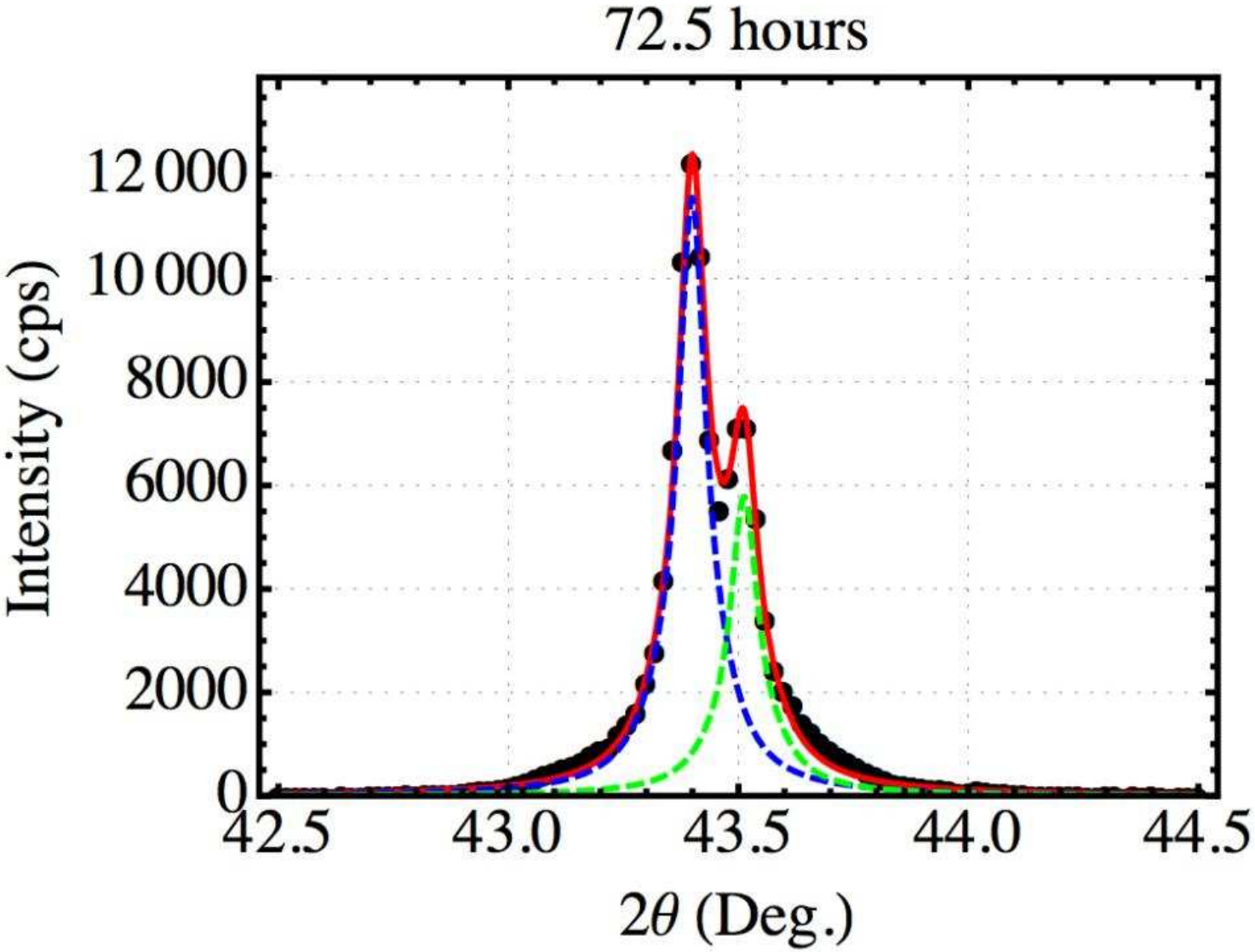
SAMPLE _5: Plated, quenched in liquid. N_2, stored for ~96 hr, and then x-rayed.



SAMPLE _5: Plated, quenched in liquid. N_2, stored for ~96 hr, and then x-rayed.



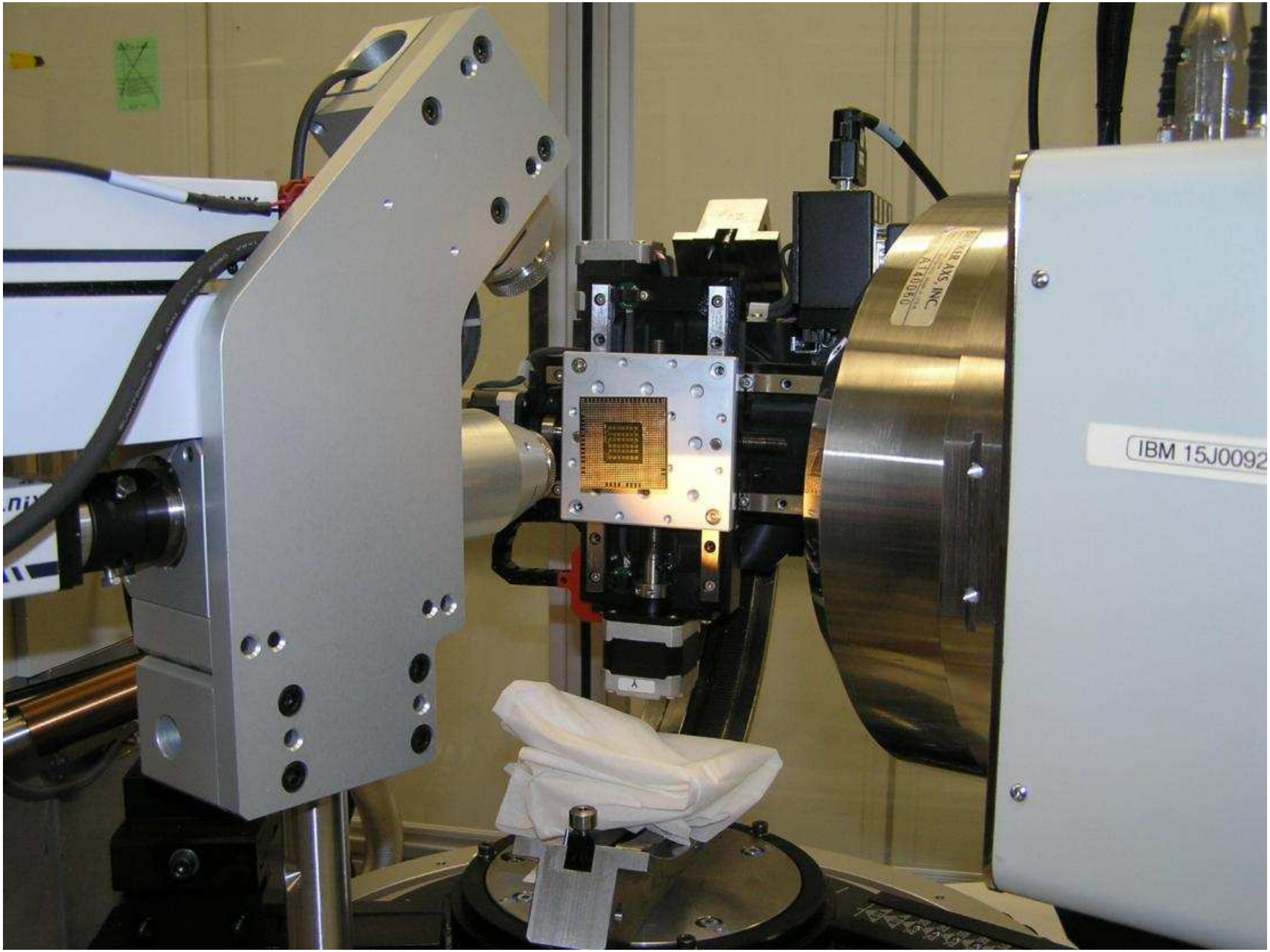
SAMPLE _5: Plated, quenched in liquid. N_2, stored for ~96 hr, and then x-rayed.



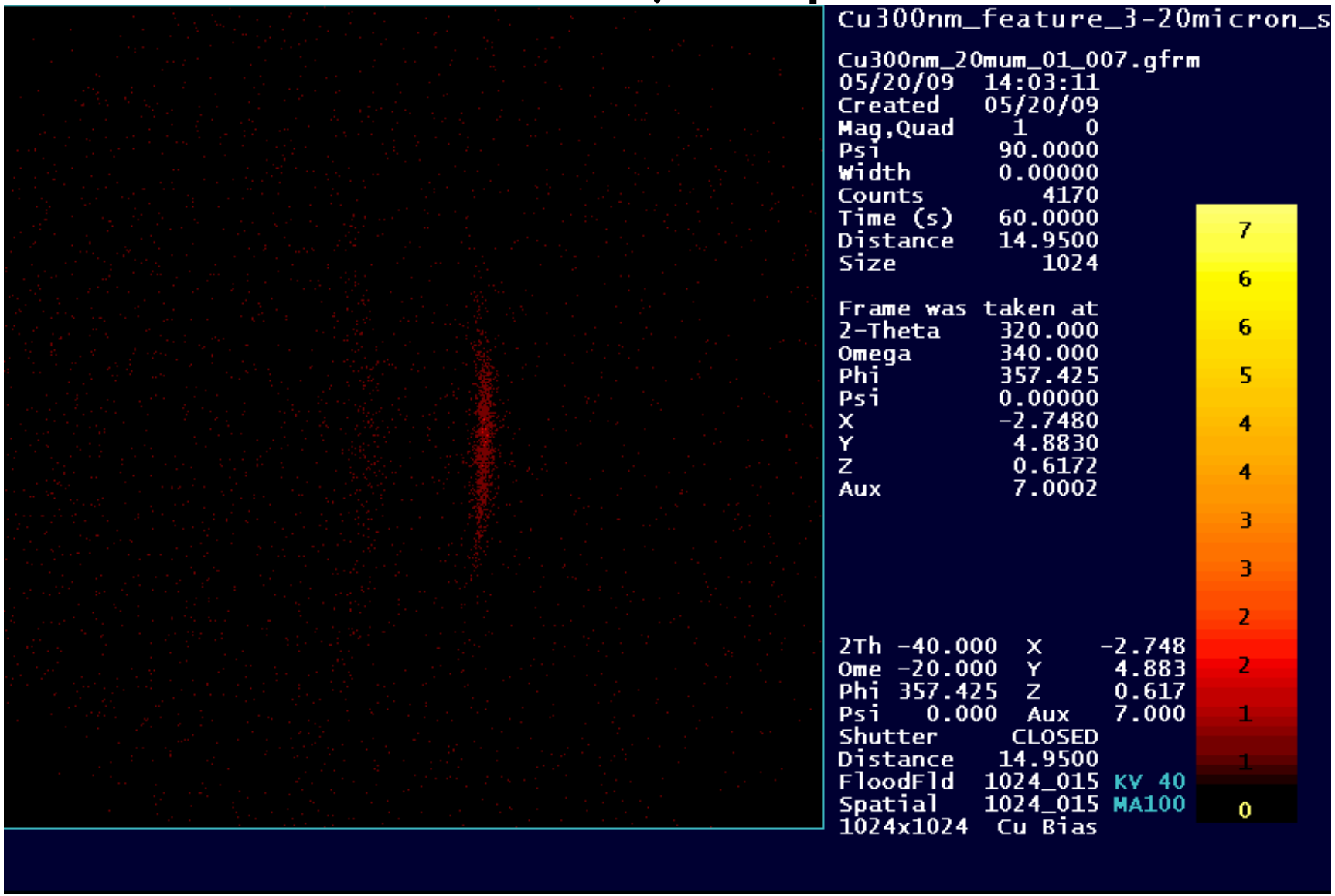
BRUKER GADDS SYSTEM

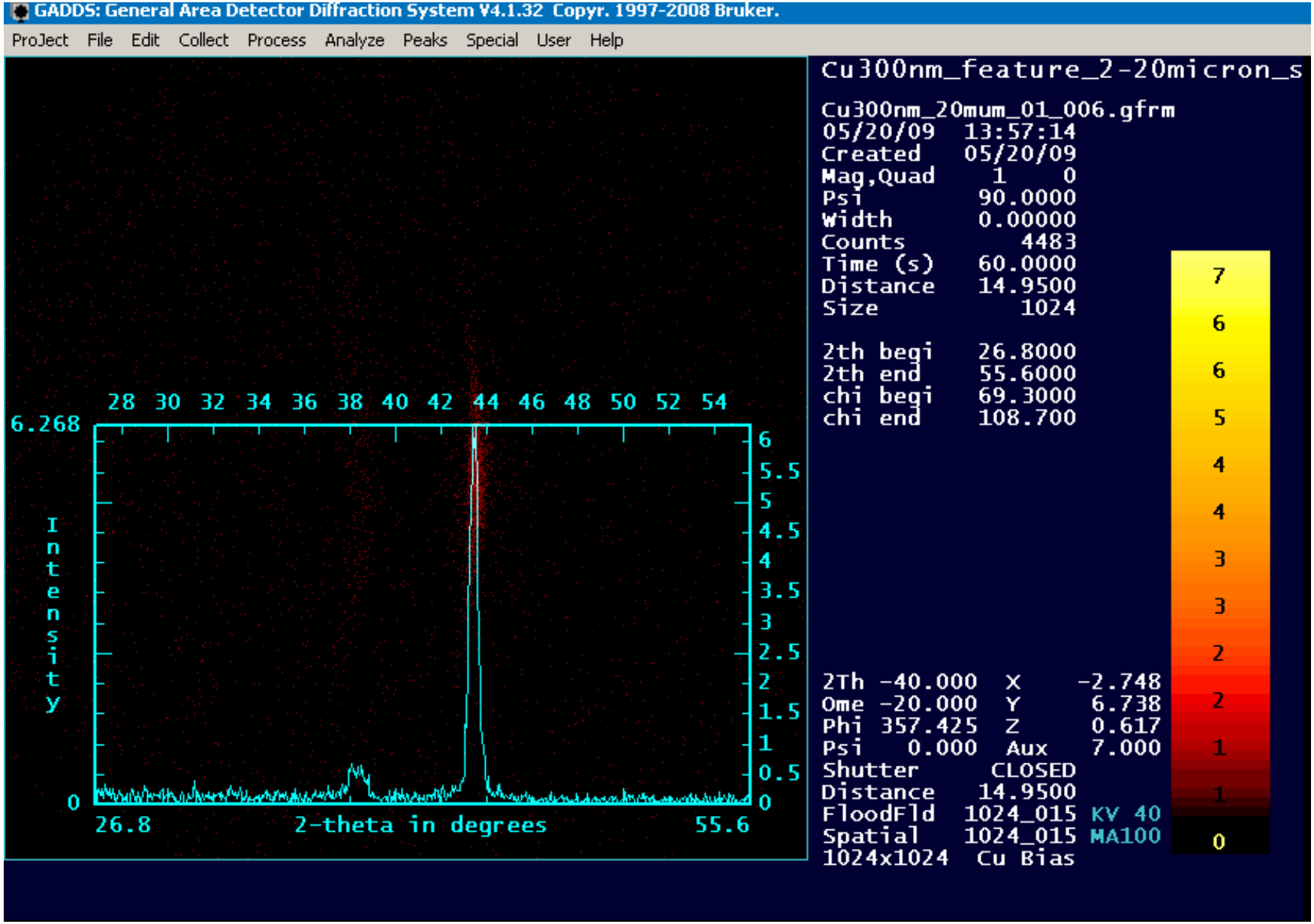
- Monochromatic radiation
- 300 μm and 20 μm capillary optics,
- GADDS Area detector (wire prop. Counter)
- Rigaku rotating anode at 5 kW.

  - Experiments with assistance from C. Goldsmith and P. Dehaven (FSH)


IBM 15J0092

300 nm film, 20 μm spot, 60 sec.




GADDS: General Area Detector Diffraction System V4.1.32 Copyr. 1997-2008 Bruker.
Project File Edit Collect Process Analyze Peaks Special User Help
Cu300nm_feature_2-20micron_s
Cu300nm_20mum_01_006.gfrm
05/20/09 13:57:14
Created 05/20/09
Mag,Quad 1 0
Psi 90.0000
Width 0.00000
Counts 4483
Time (s) 60.0000
Distance 14.9500
Size 1024
2th begi 26.8000
2th end 55.6000
chi begi 69.3000
chi end 108.700
2Th -40.000 X -2.748
Ome -20.000 Y 6.738
Phi 357.425 Z 0.617
Psi 0.000 Aux 7.000
Shutter CLOSED
Distance 14.9500
FloodFld 1024_015 KV 40
Spatial 1024_015 MA100
1024x1024 Cu Bias
28 30 32 34 36 38 40 42 44 46 48 50 52 54
6.268
0
Intensity
26.8
2-theta in degrees
55.6
6 5.5 5 4.5 4 3.5 3 2.5 2 1.5 1 0.5 0
7 6 6 5 4 4 3 3 2 2 1 1 0

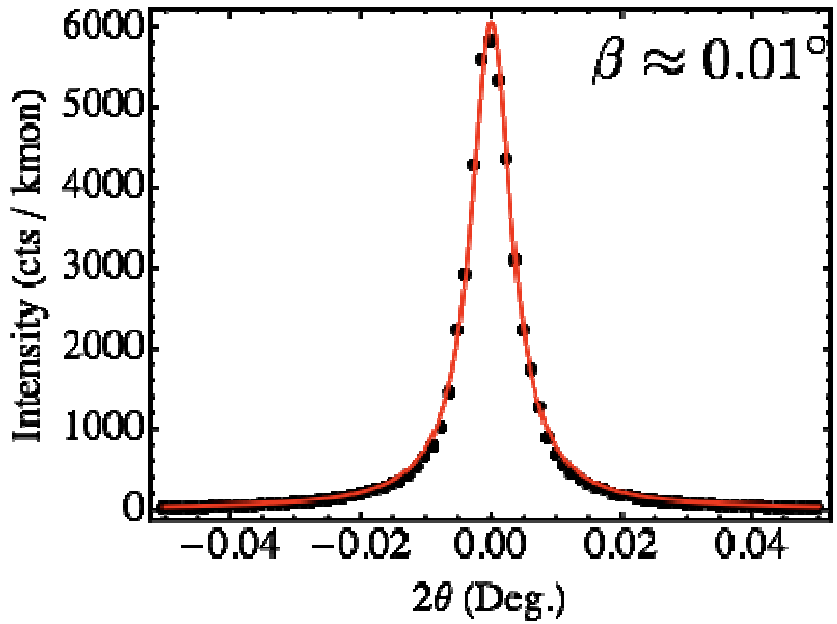
Laboratory Source Summary

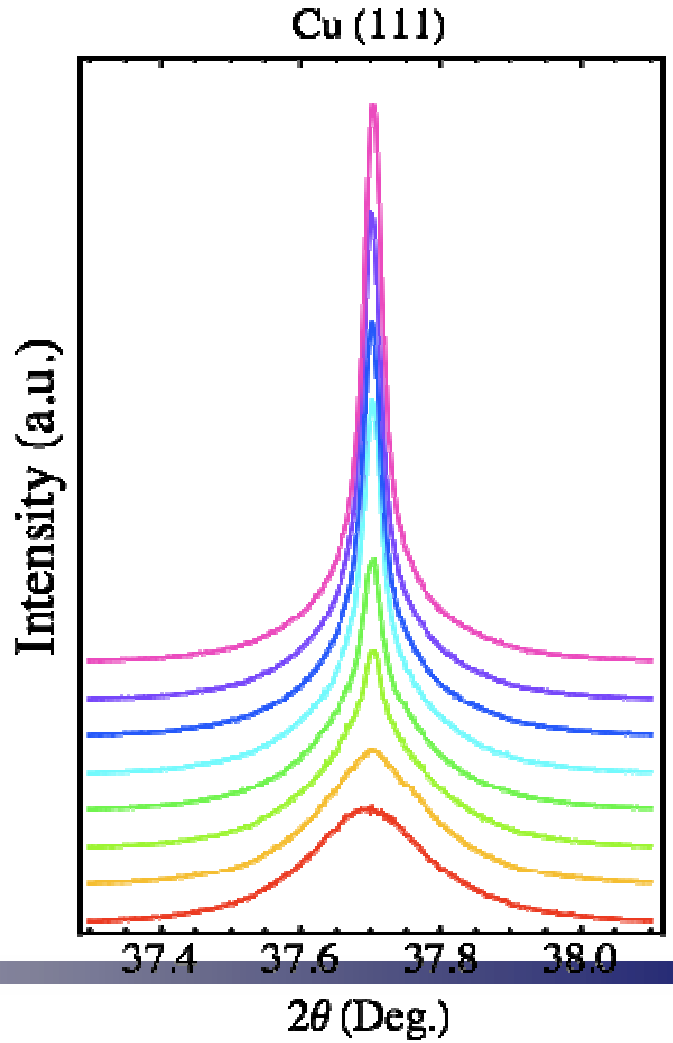
- We can obtain qualitative information.
- Instrument resolution is not sufficient for separating the recrystallized and initial grain profiles.
- The presence of $K\alpha 2$ is an added complication.
- Synchrotron measurements are needed.

Synchrotron measurements

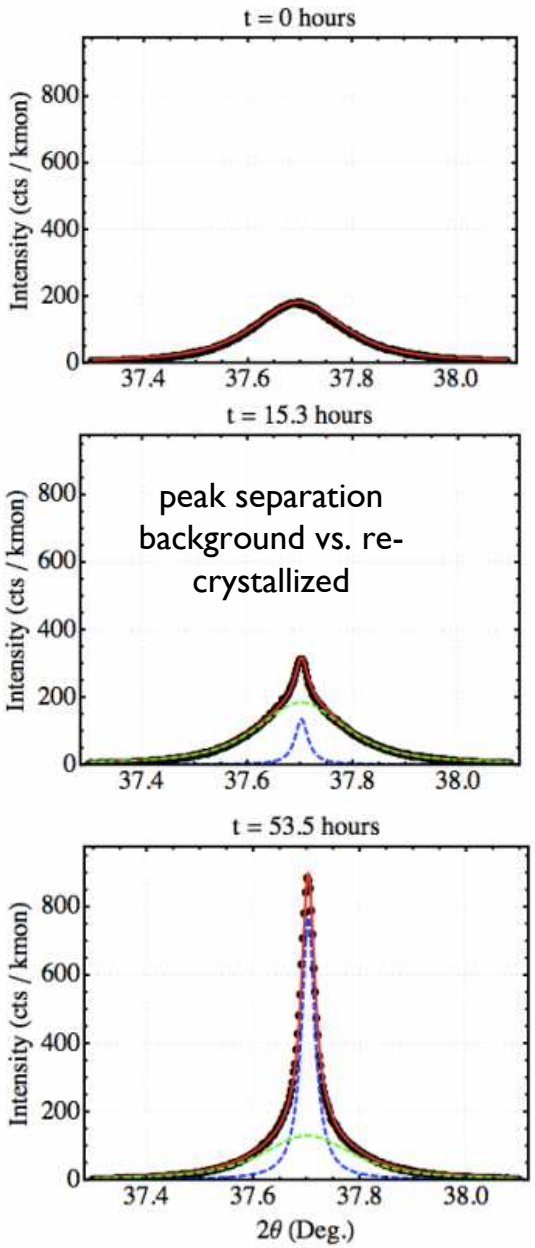
X-ray measurements performed at X20A beamline at NSLS.

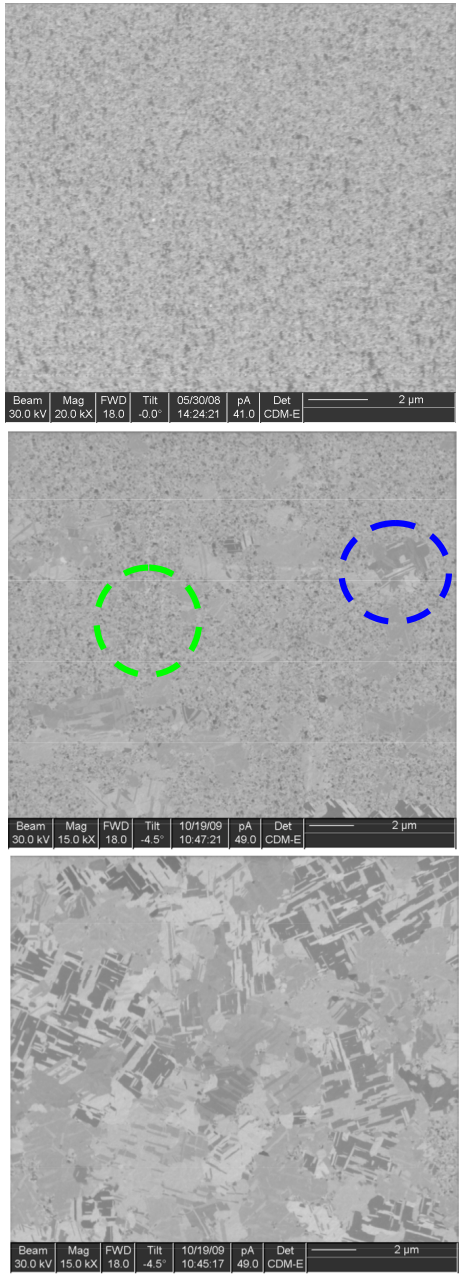
- High resolution XRD w/ analyzer crystal.
- Symmetric radial scans of 111, 200 and 311 peaks.





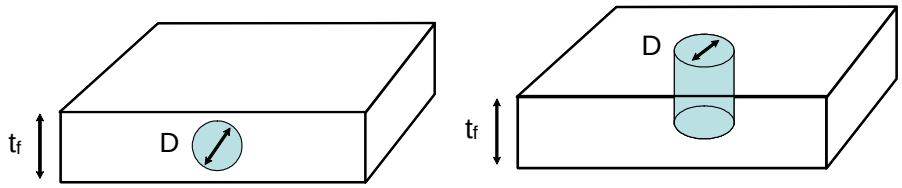
~8 hour interval between measurements shown.





Assume initial small grains are spherical, and grow in volume over time.

- **$T < \tau_1$: Stage I: incubation period**
- **$\tau_1 < T < \tau_2$: Stage II: vol. growth**
- **$\tau_2 < T$: Stage III: planar growth**



- **The grain growth in the overburden for the 111 and 200 type grains has two modes:**
  - Volumetric (spherical) growth,
  - Planar (circular) growth.
- **For our plating parameters (111 grains) :**
  - The spherical diameter growth rate is approximately linear with time, ~ 300 Å/ hr.
  - Planar growth shows exponential decay. The diameter growth rate is initially ~ 150 Å / hr.
- **The grains that form are almost perfect grains with little sub-grain boundaries or defects.**

Summary

Synchrotron measurements can be used to characterize recystallization kinetics.

For this purpose we need high-resolution XRD:

-The FWHM of the instrument has to be narrower than the FWHM of the fully transformed sample.

-The peak intensities from low volume –fraction populations are very weak. Synchrotron beams from ID beamlines are needed for good statistics.