In situ diffraction and imaging studies of transient and irreversible phenomena in materials

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Effects of the MBA lattice in the high energy regime (60-100 keV) as I understand them

- ~2x increase in flux
- ~100x increase in brightness (mostly due to a reduction in horizontal beam size)
- Significant gain in coherent fraction/flux
- Possible changes in bunch structure (duration and spacing)

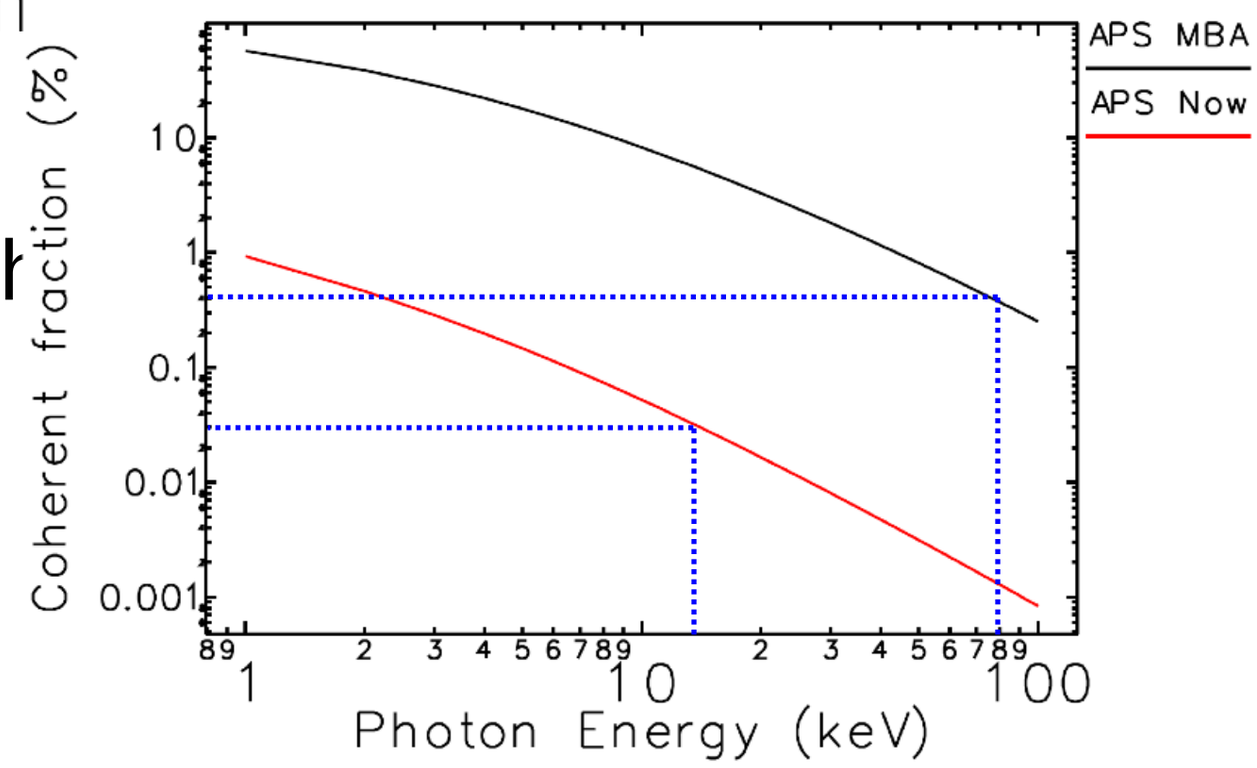
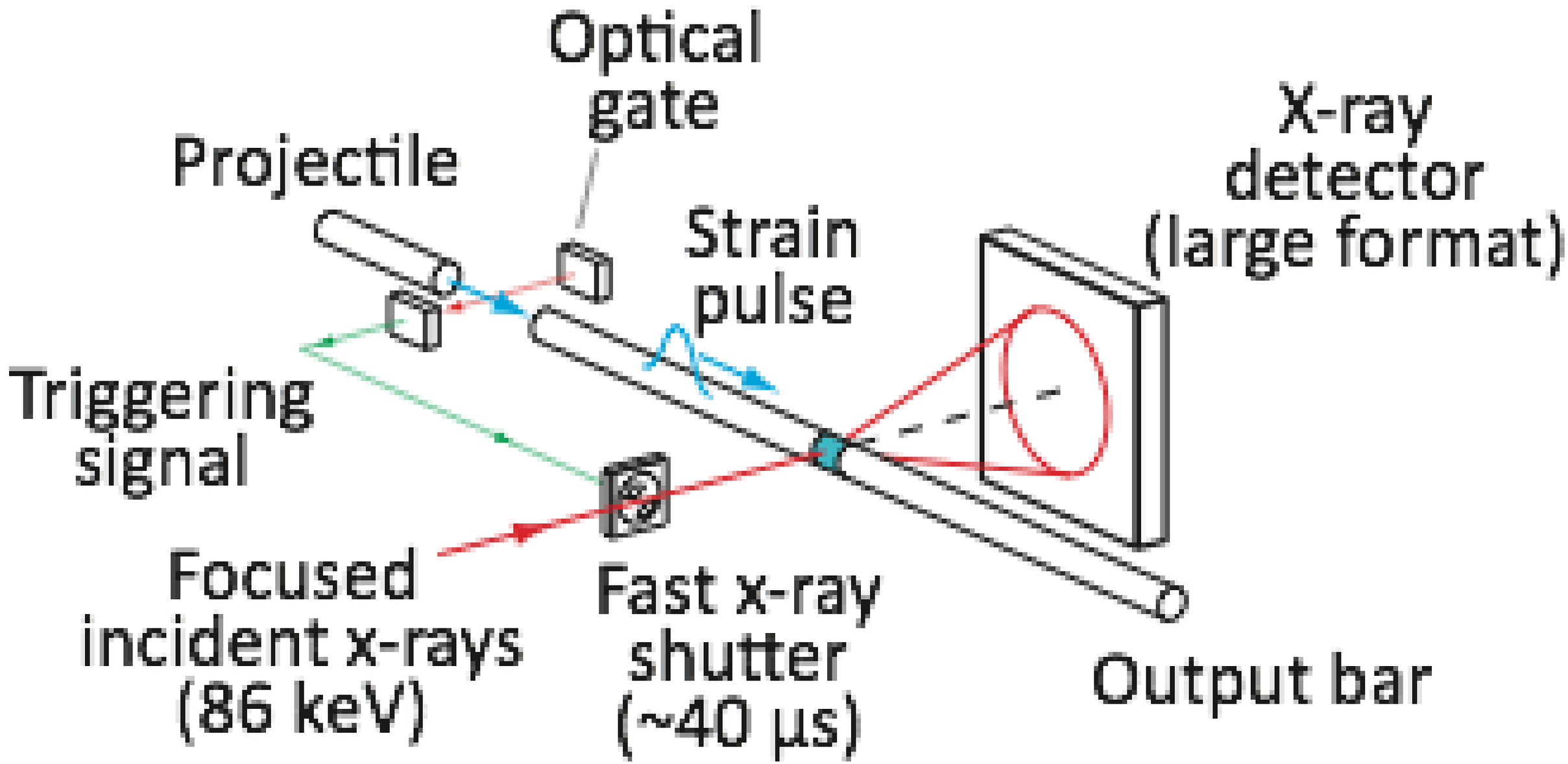
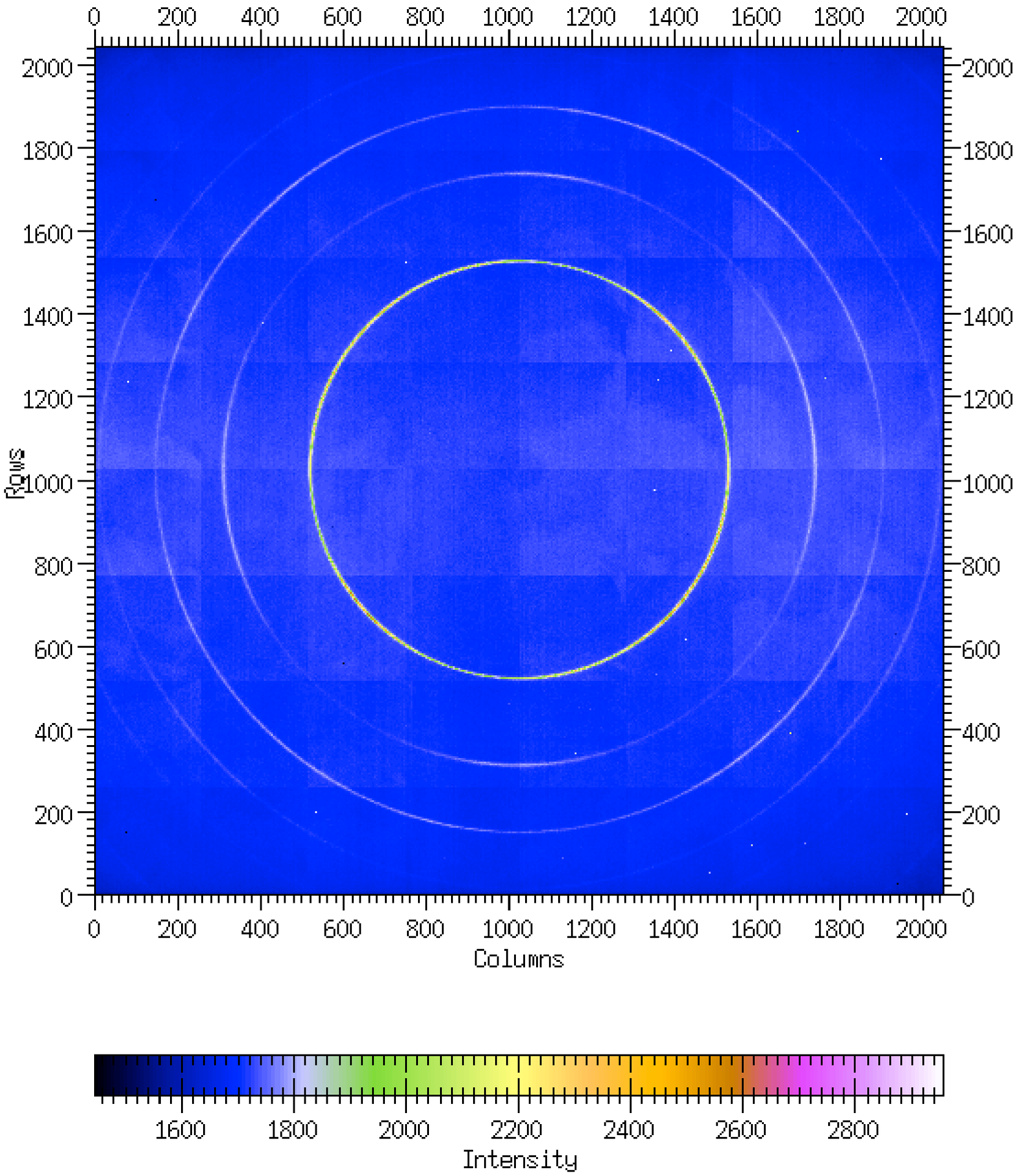


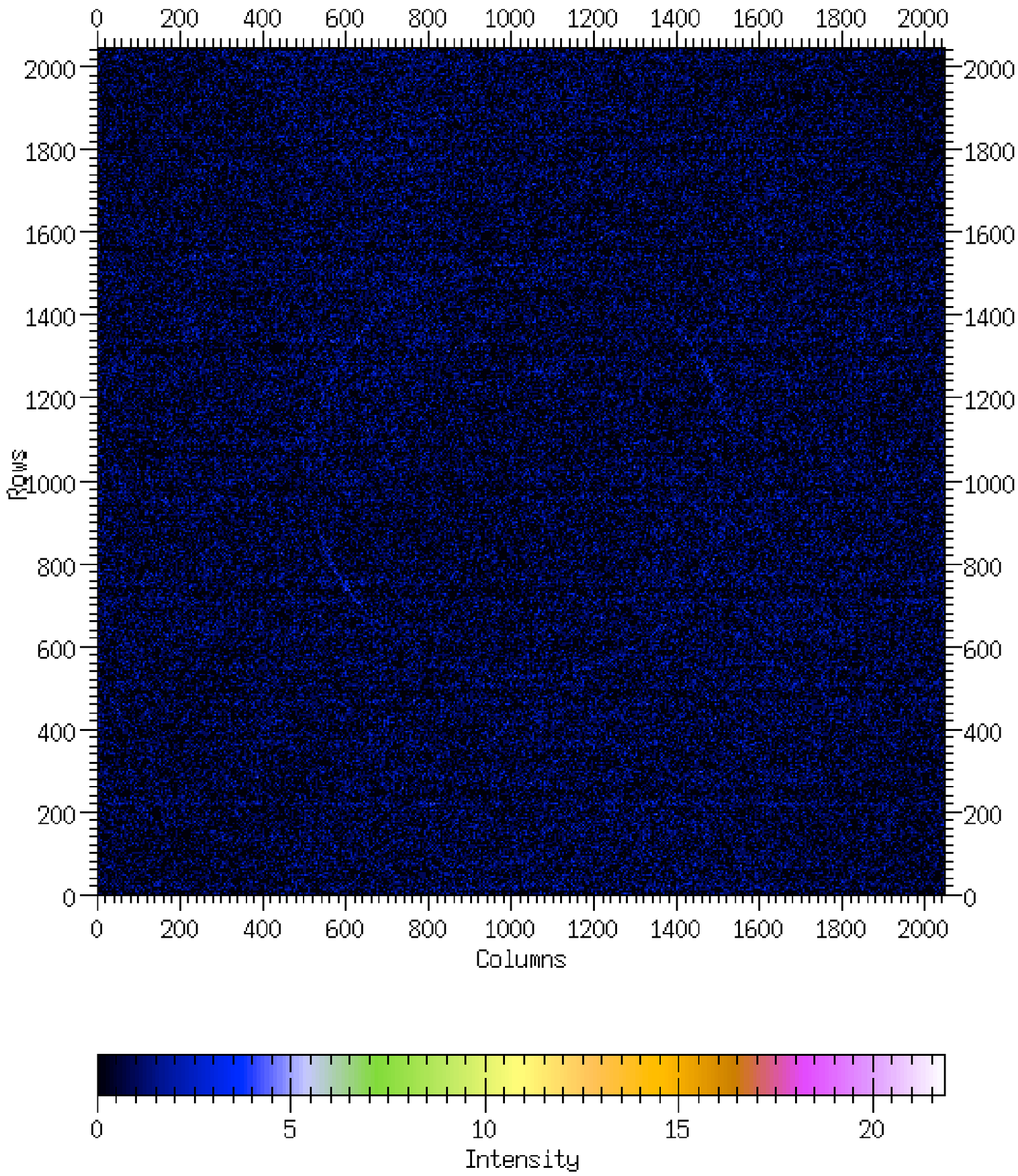
Figure from Jonathan Lang

Diffraction during dynamic loading





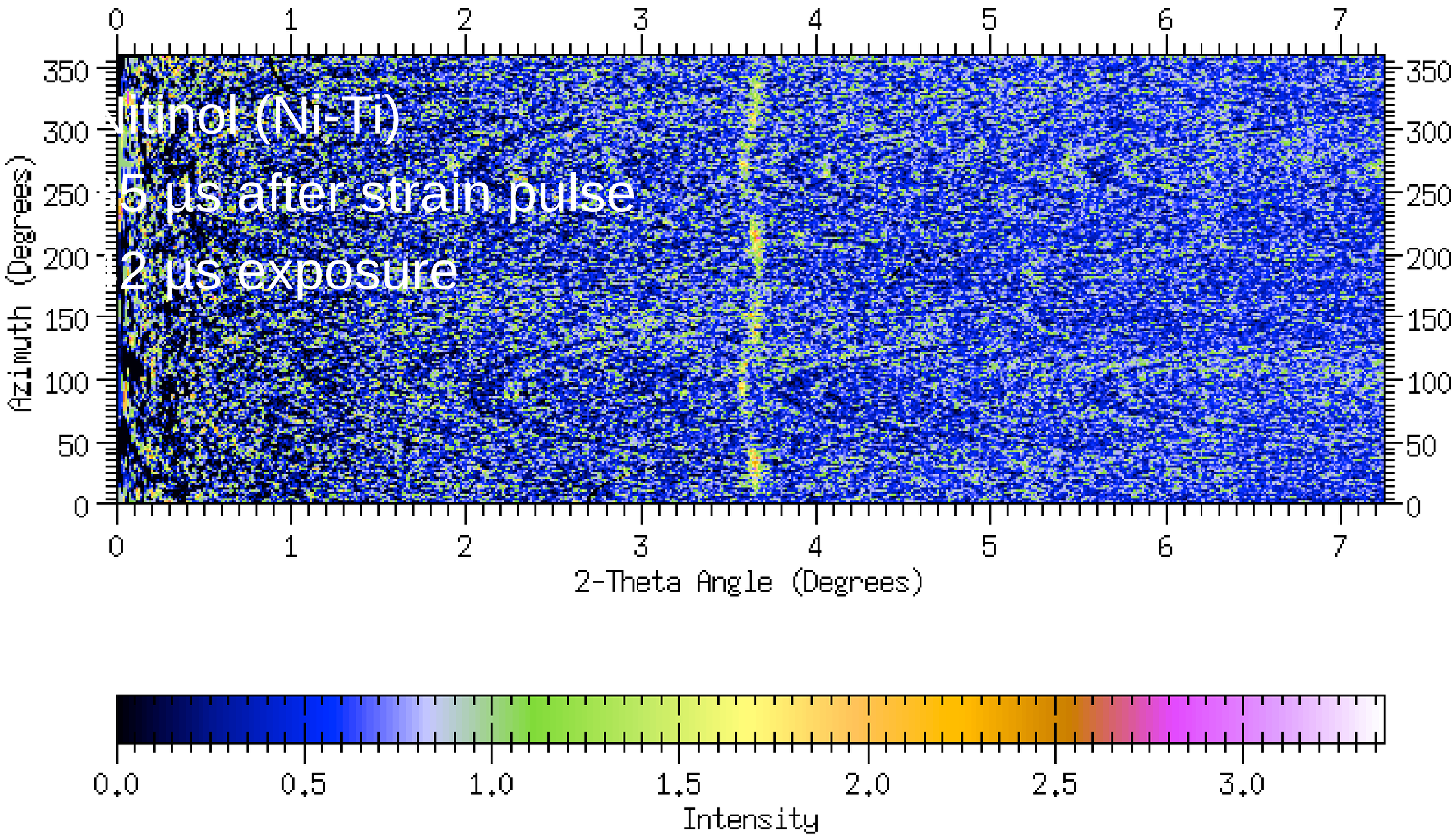
- Nitinol (Ni-Ti)
- No applied load
- APS 1-ID
- 86 keV
- 5 ms (single shot)

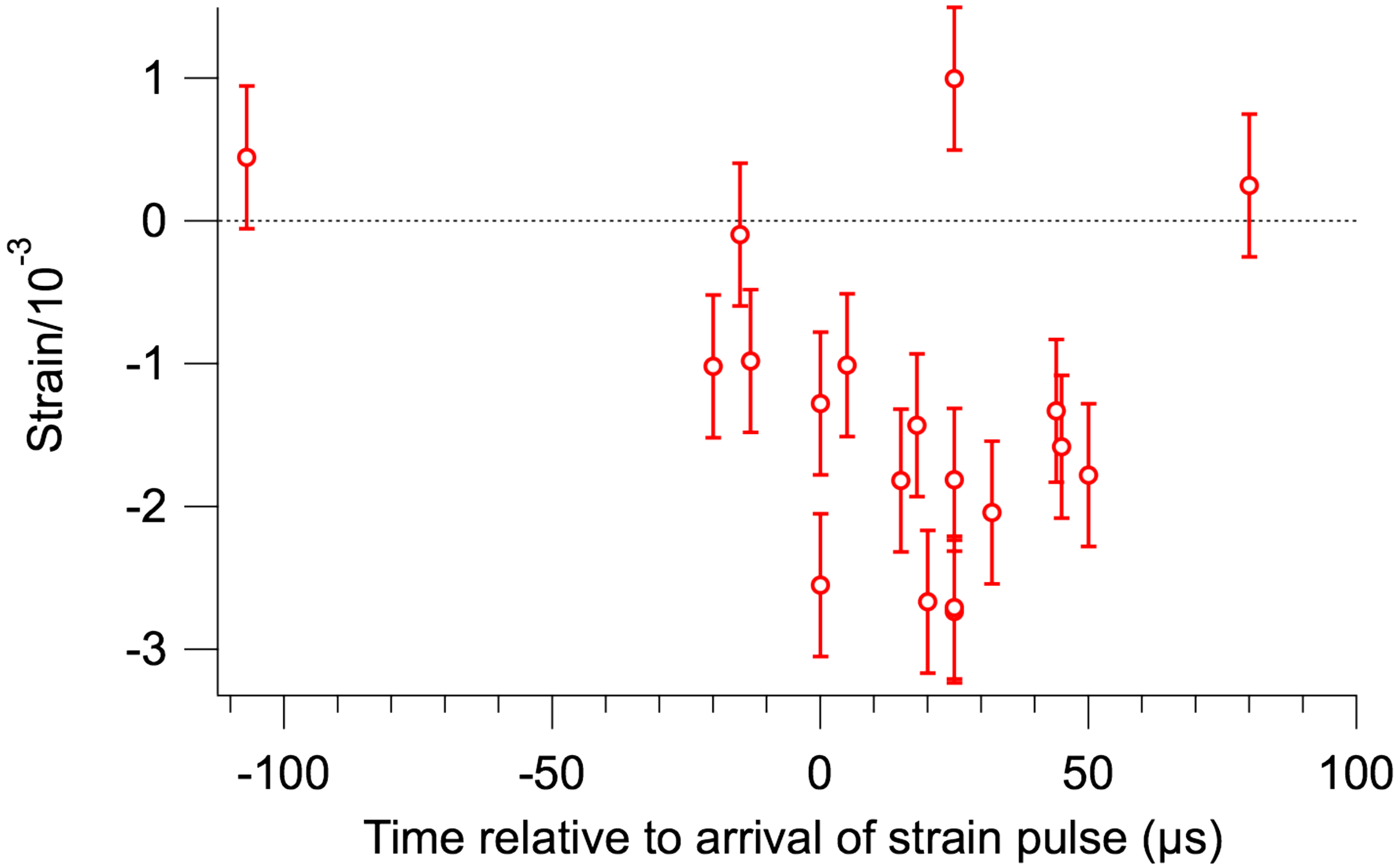


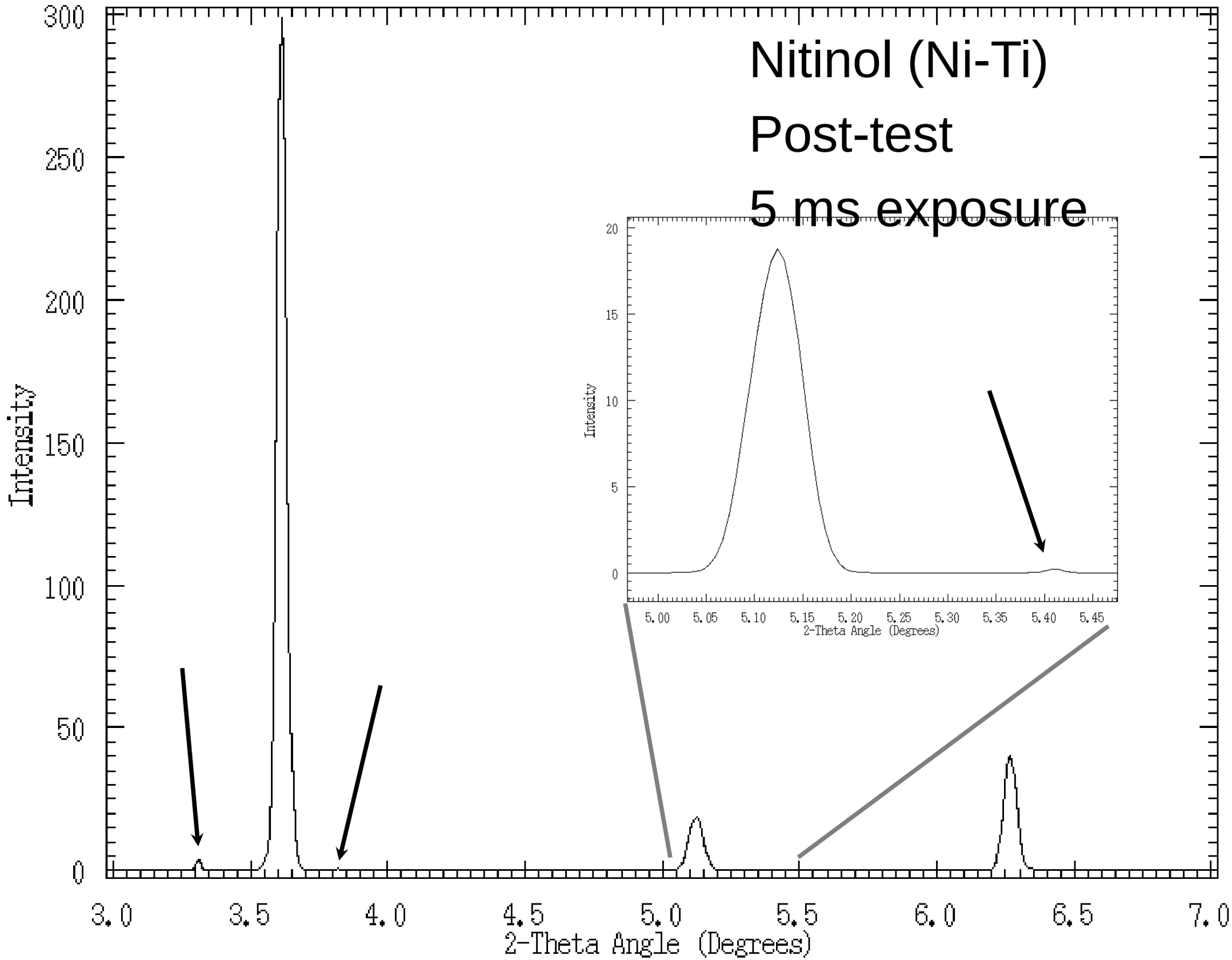
Nitinol (Ni-Ti)

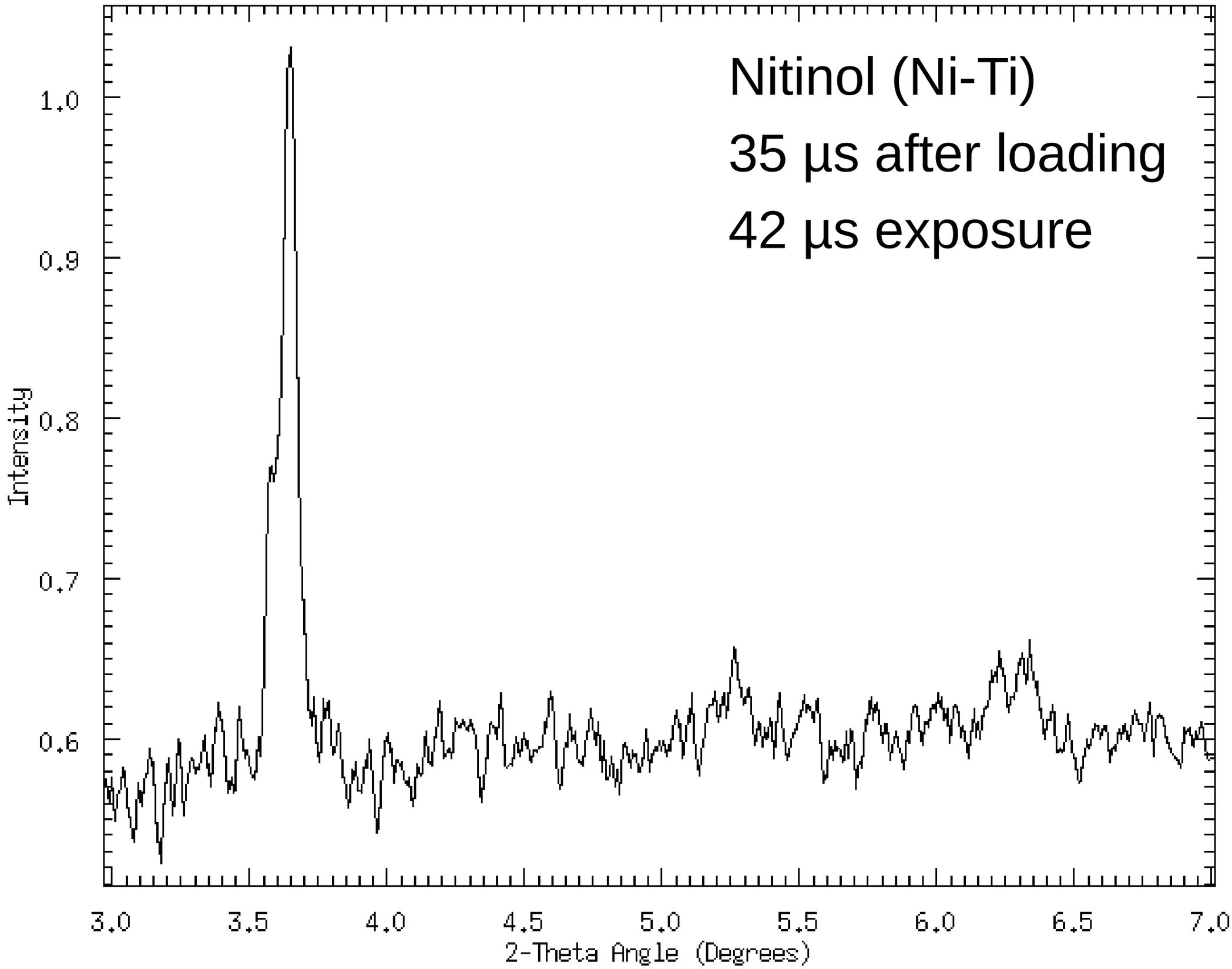
35 µs after arrival of strain pulse

42 µs exposure


Nitinol (Ni-Ti)
5 µs after strain pulse
2 µs exposure
Azimuth (Degrees)
2-Theta Angle (Degrees)
Intensity
0
1
2
3
4
5
6
7
0
50
100
150
200
250
300
350
0.0
0.5
1.0
1.5
2.0
2.5
3.0


1
0
-1
-2
-3
Strain/10^-3
-100
-50
0
50
100
Time relative to arrival of strain pulse (μs)


Nitinol (Ni-Ti)
Post-test
5 ms exposure
Intensity
2-Theta Angle (Degrees)
300
250
200
150
100
50
0
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
20
15
10
5
0
5.00
5.05
5.10
5.15
5.20
5.25
5.30
5.35
5.40
5.45


Nitinol (Ni-Ti)
35 µs after loading
42 µs exposure
Intensity
1.0
0.9
0.8
0.7
0.6
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
2-Theta Angle (Degrees)

What would help this experiment?

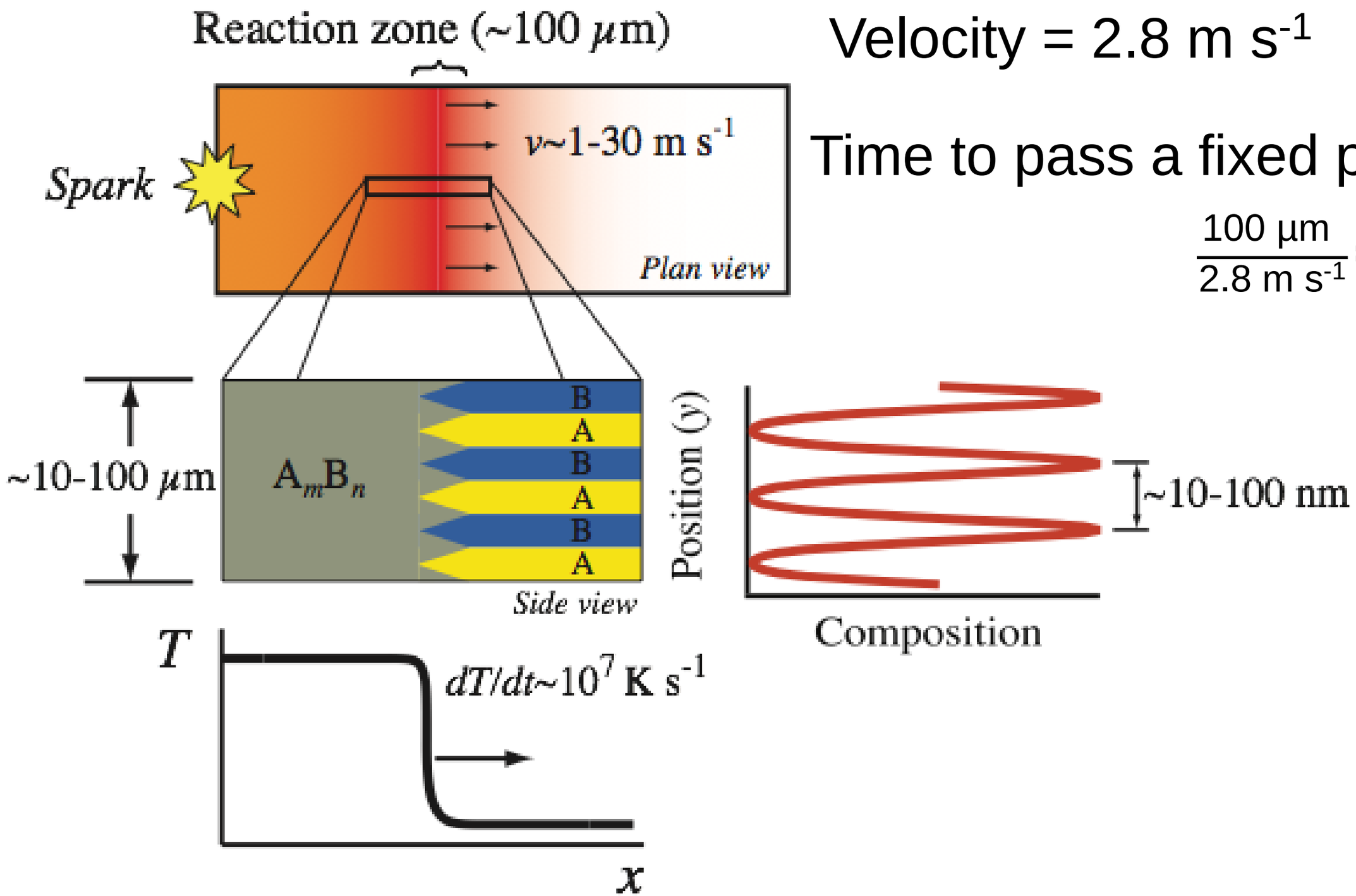
- More flux → better signal-to-noise ratio
- Smaller x-ray beam does not help much and may hurt (due to powder averaging)
- Better (i.e. faster) detectors would help a lot

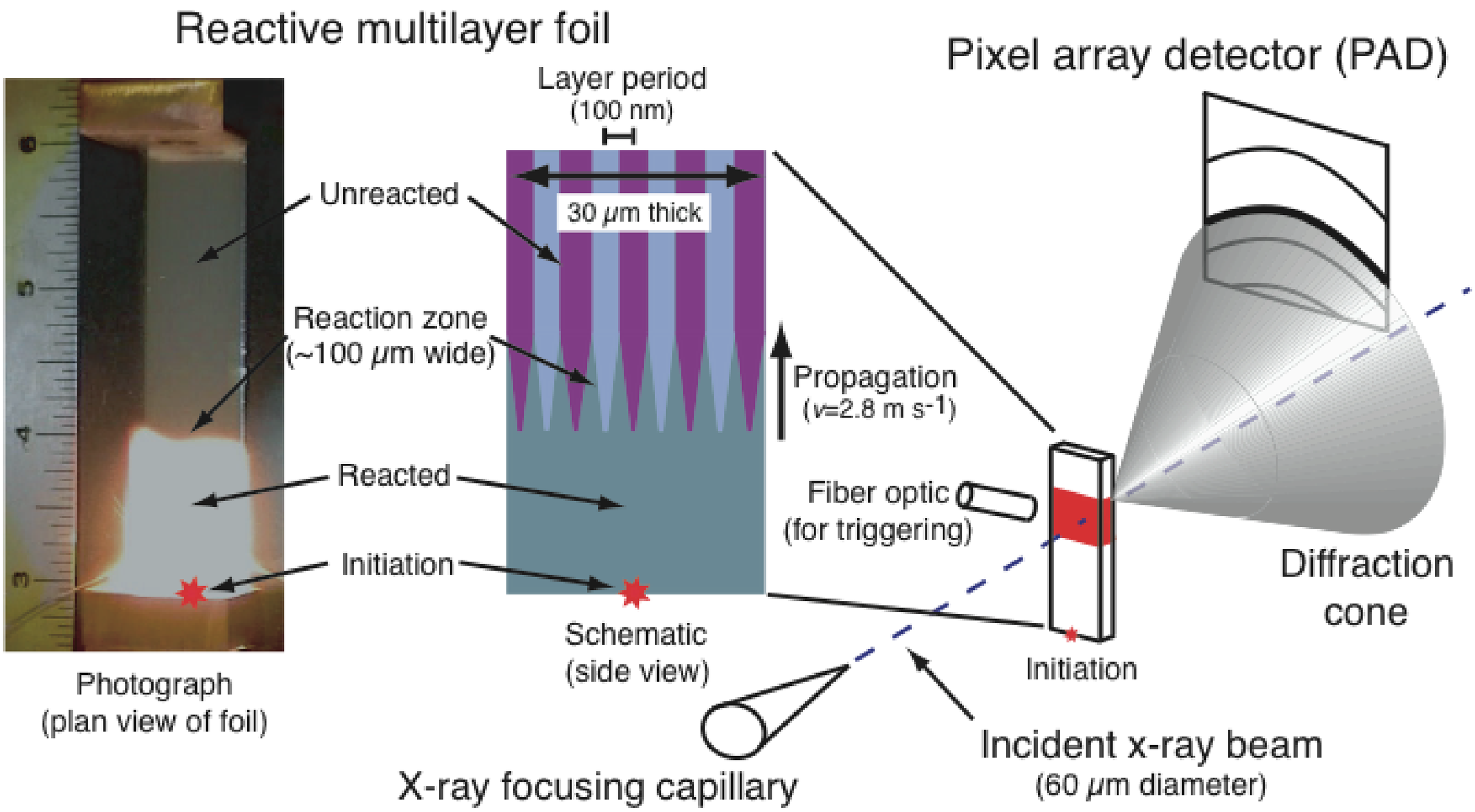
Diffraction during rapid phase transformation

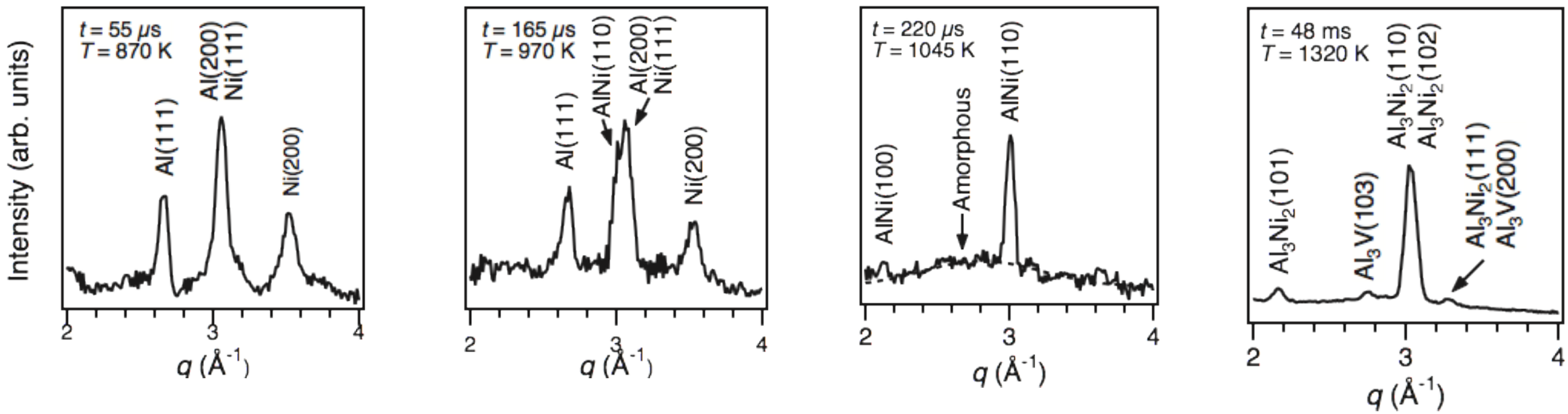
Reaction zone width: ~100 μm
Velocity = 2.8 m s^{-1}

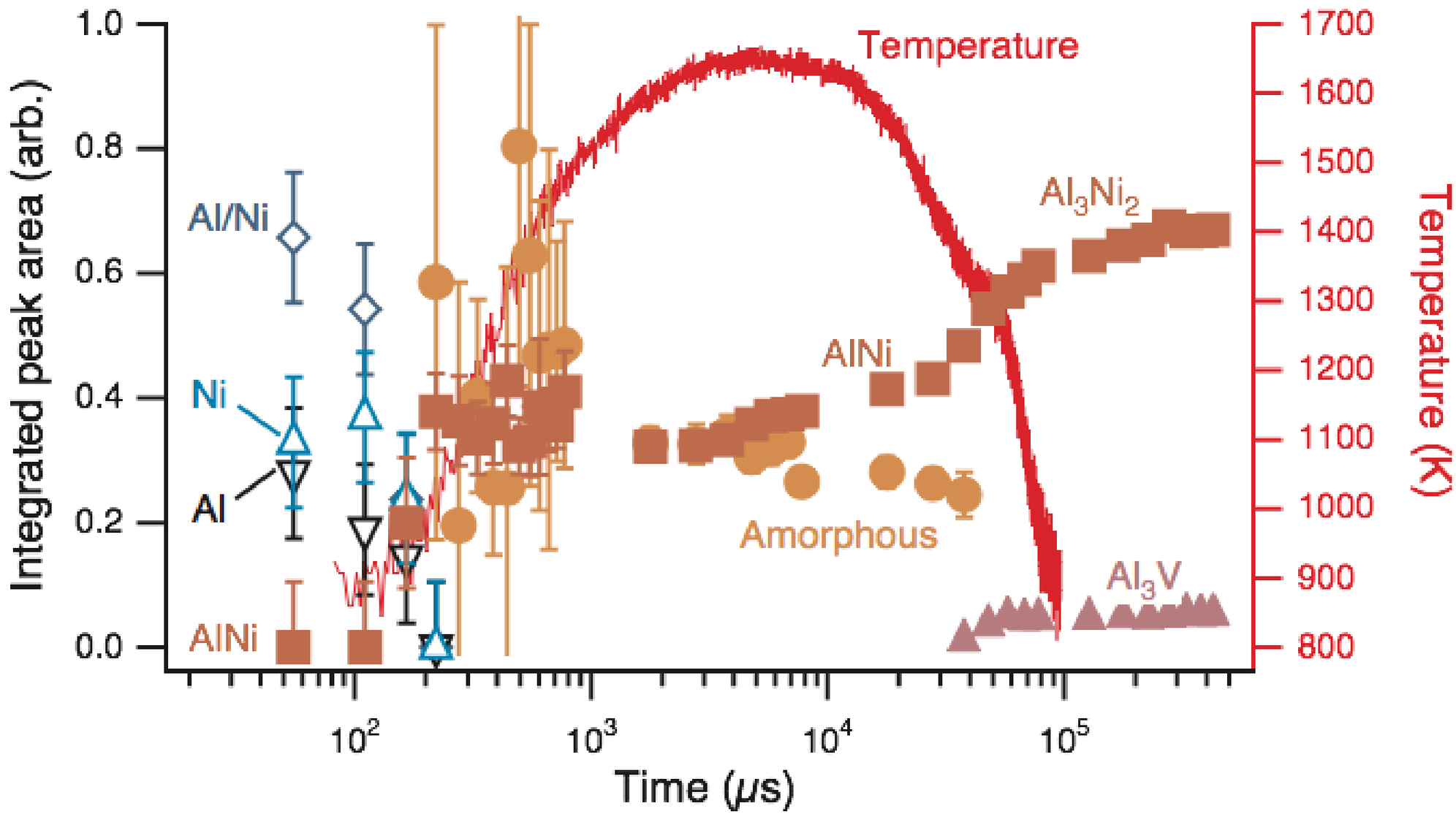
Time to pass a fixed point:

$$\frac{100\ \mu m}{2.8\ m\ s^{-1}} \approx 36\ \mu s$$

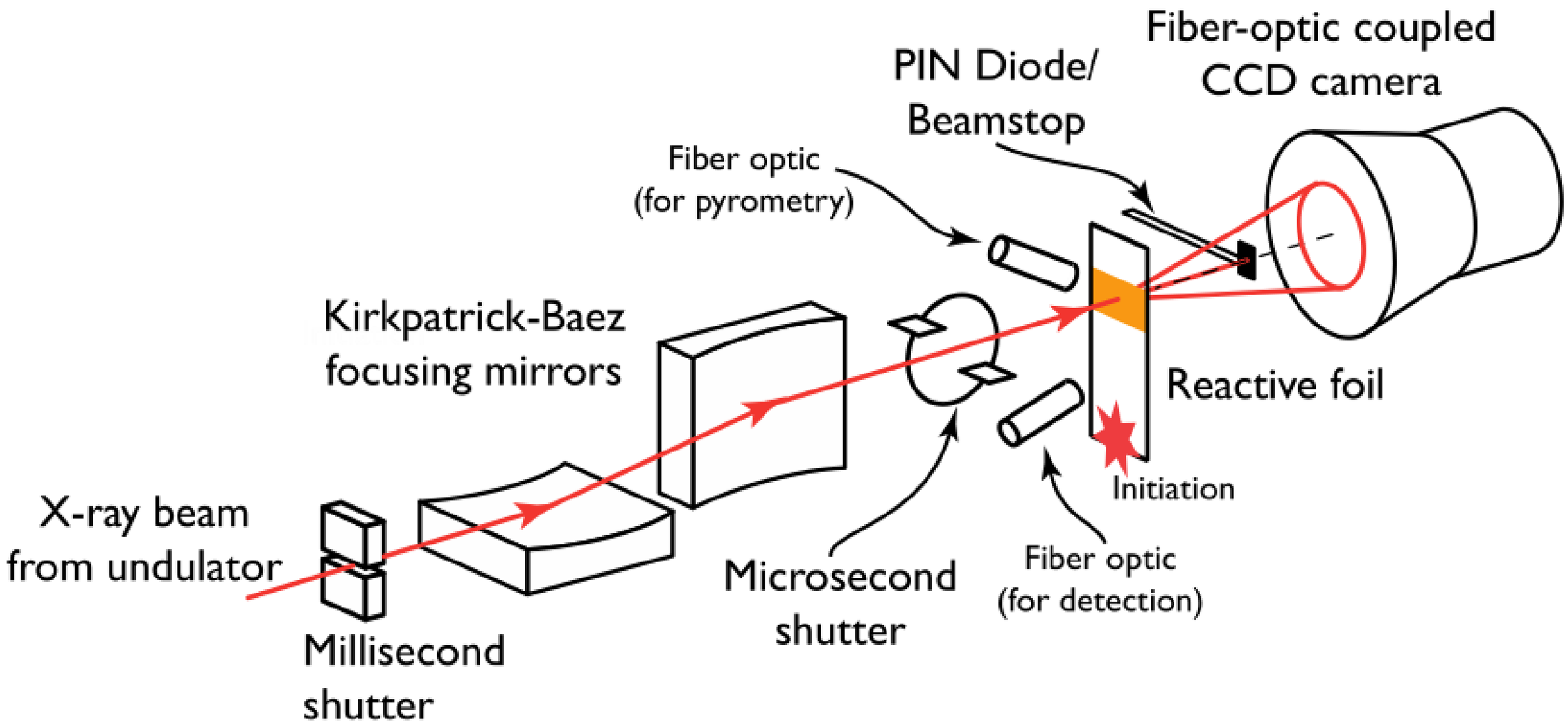



Reactive multilayer foil
Layer period
(100 nm)
Pixel array detector (PAD)
Unreacted
30 μm thick
Reaction zone
(~100 μm wide)
Propagation
(v=2.8 m s-1)
Reacted
Fiber optic
(for triggering)
Initiation
Diffraction
cone
Schematic
(side view)
Initiation
Photograph
(plan view of foil)
Incident x-ray beam
(60 μm diameter)
X-ray focusing capillary





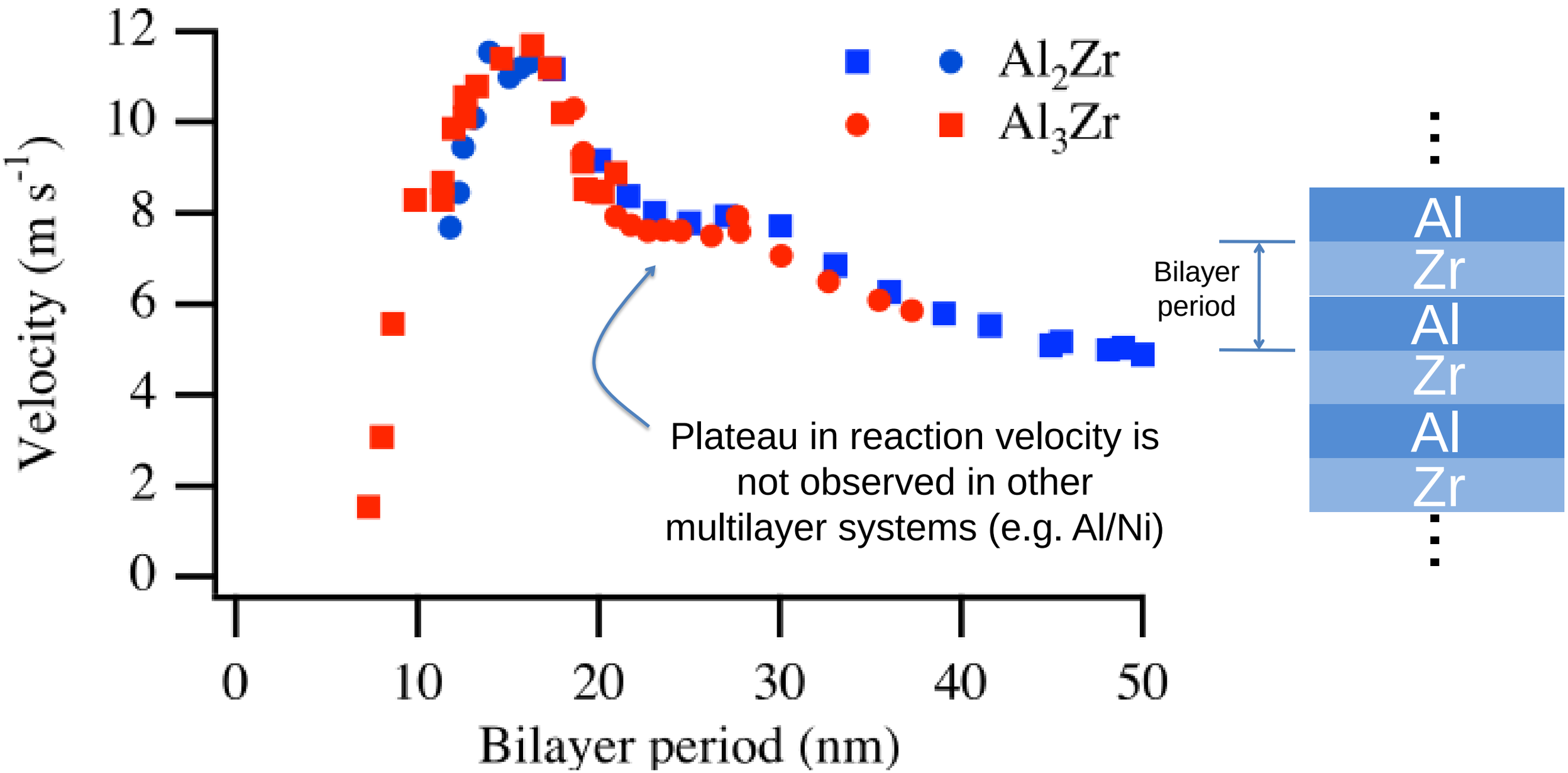
J. C. Trenkle *et al., Appl. Phys. Lett.* **93**, 081093 (2008)



- Spatial resolution from focusing mirrors (9 μm × 7 μm beam)
- Temporal resolution from single x-ray pulses (15 μs), repeated on multiple specimens

S. T. Kelly *et al., J. Synch. Rad.* **18**, 464 (201

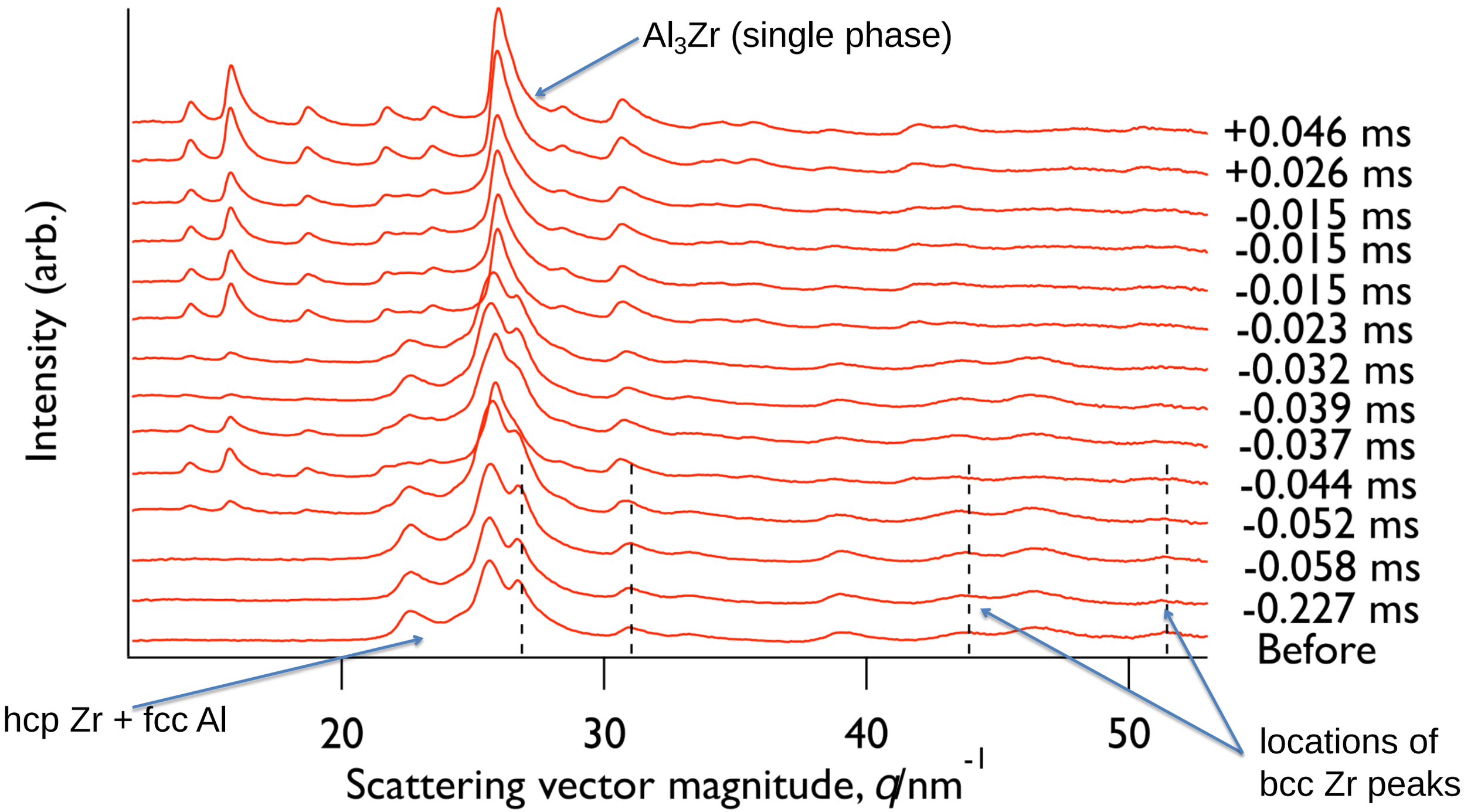
Self-propagating reactions in Al/Zr multilayers



S. C. Barron *et al.* (submitted to JAP)

Resolution limited by use of pink beam

- Al_3Zr multilayers, 90 nm bilayer
- Transformation to Al_3Zr complete in <30 μs

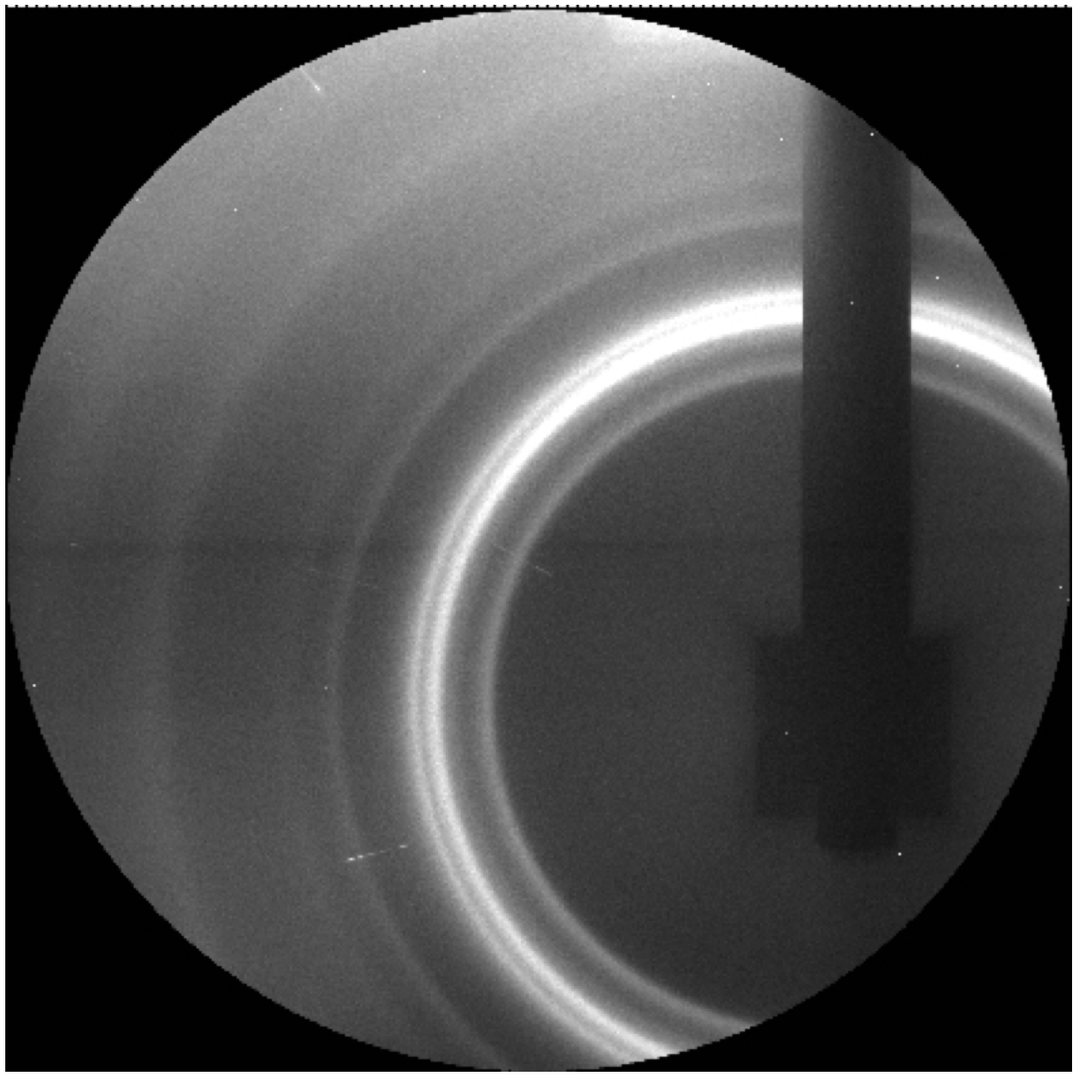


S. C. Barron *et al.* (submitted to JAP)

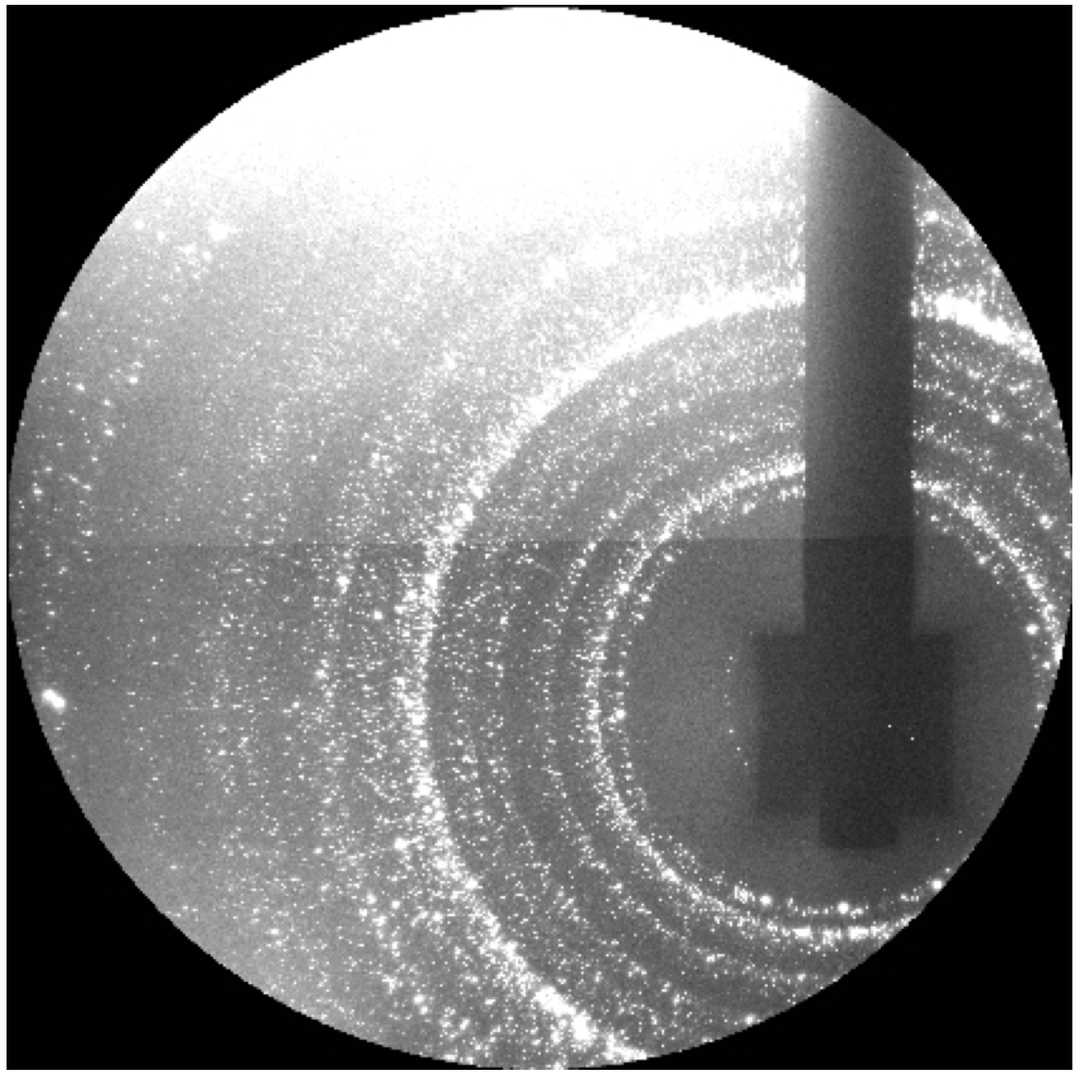
What would help this experiment?

- More flux → better signal-to-noise ratio, possibility for using monochromatic beam to improve resolution
- Smaller x-ray beam does not help much and may hurt (due to powder averaging)
- Better (i.e. faster) detectors would help a lot

Powder sampling is a problem with small beams

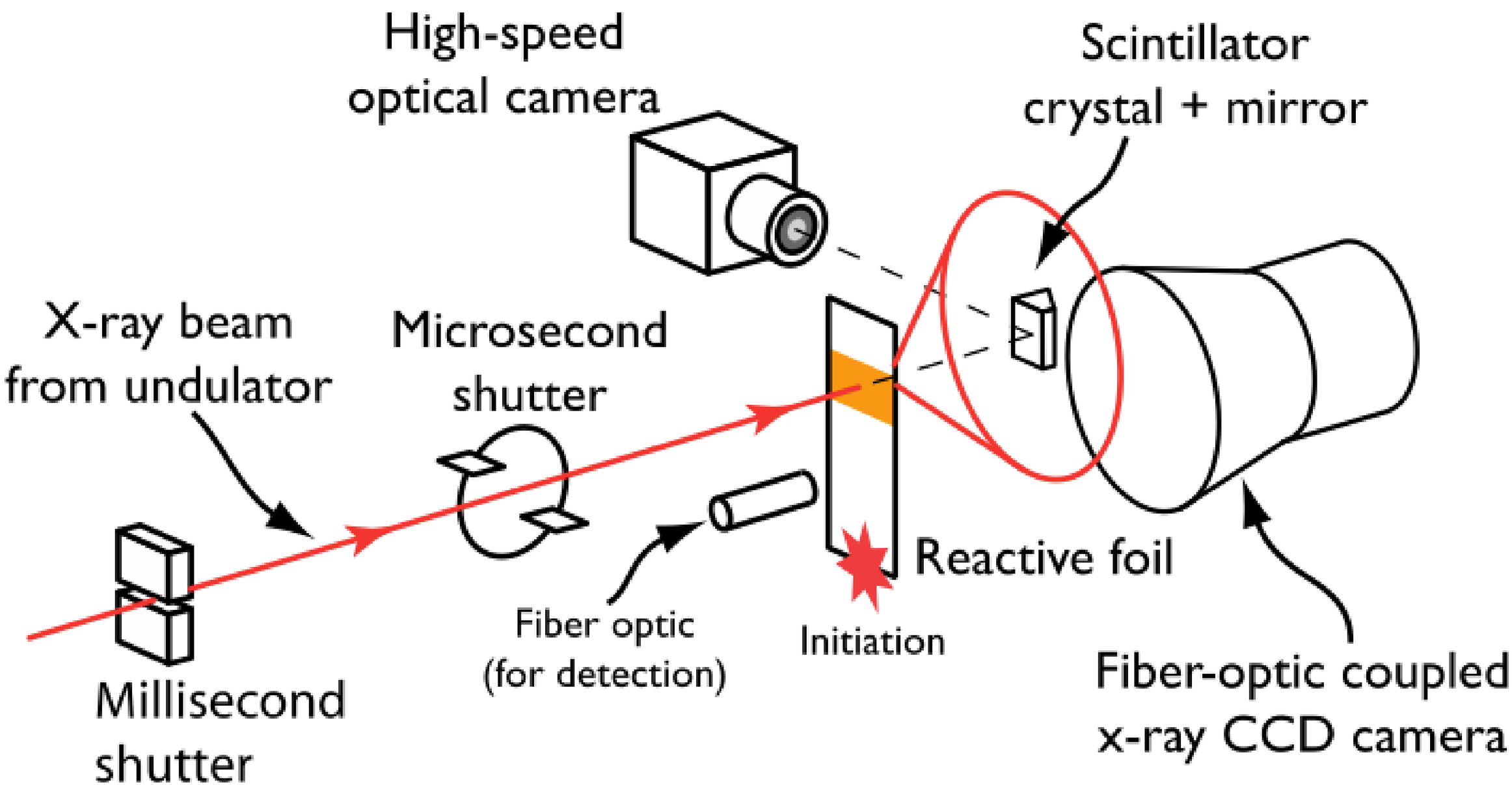


Before
Grain size ~20 nm



After
Grain size ~2 µm

Phase contrast imaging: Into real space



APS Sector 32 ID-B
12.4 keV, 5% bandwidth
~6×10^{14} ph/s

Phase contrast imaging: Into real space

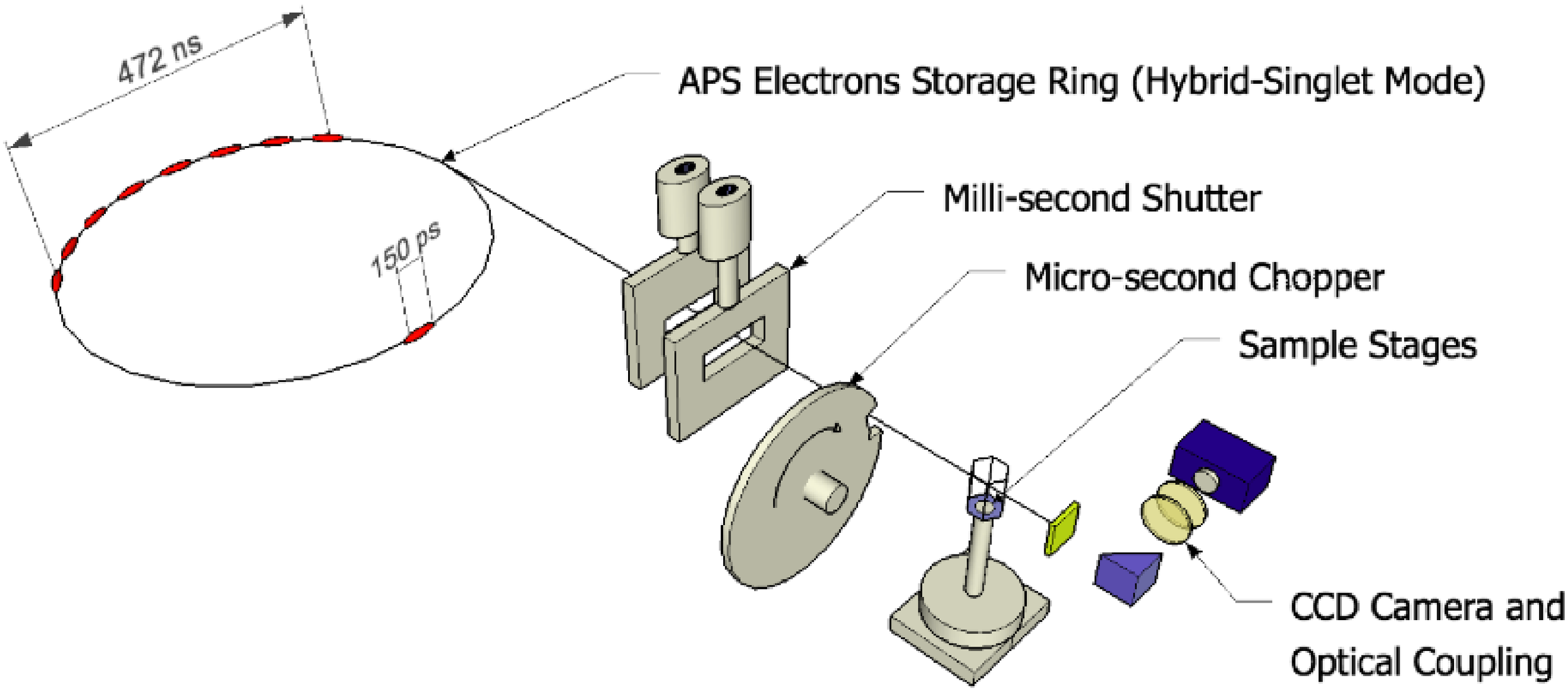
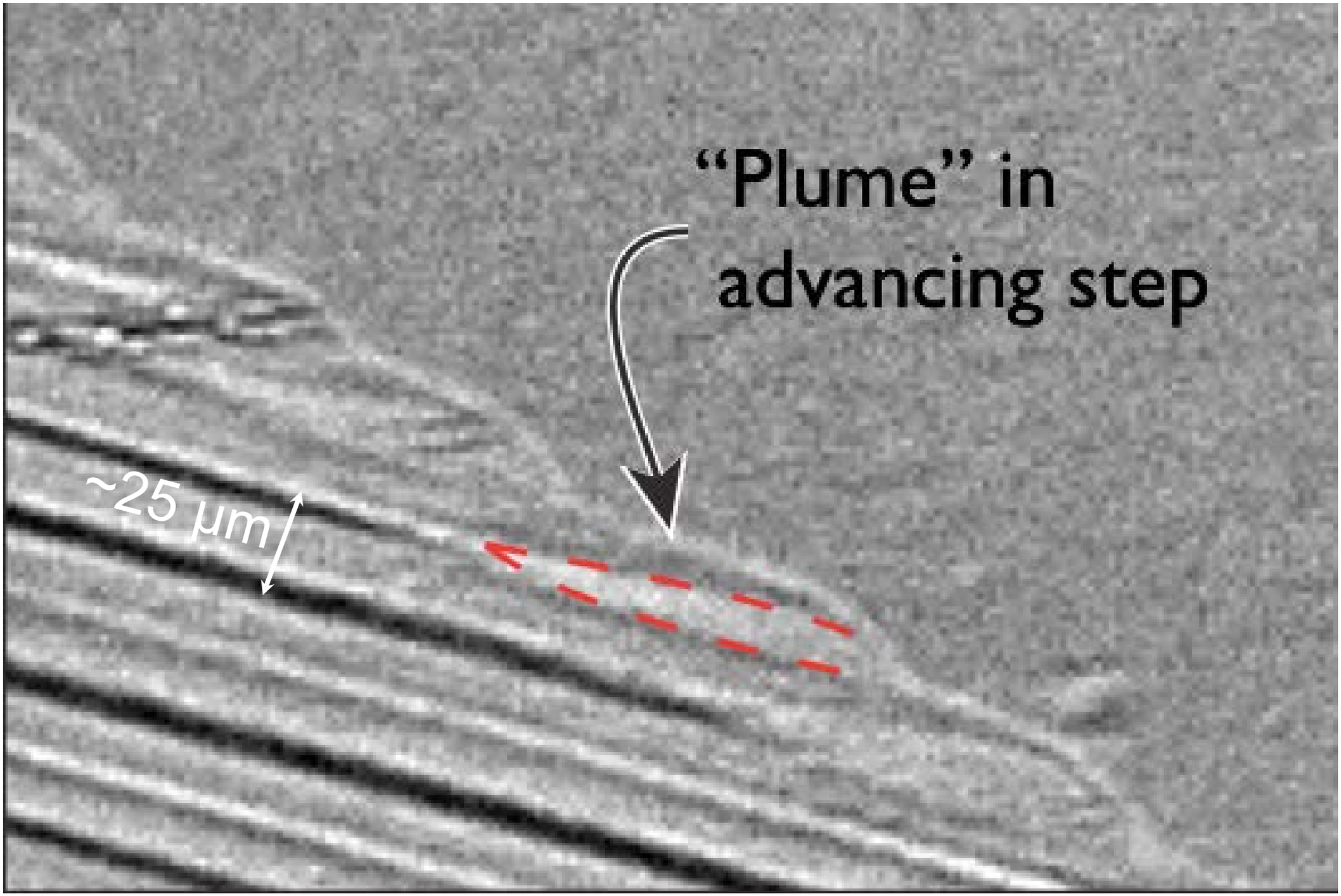


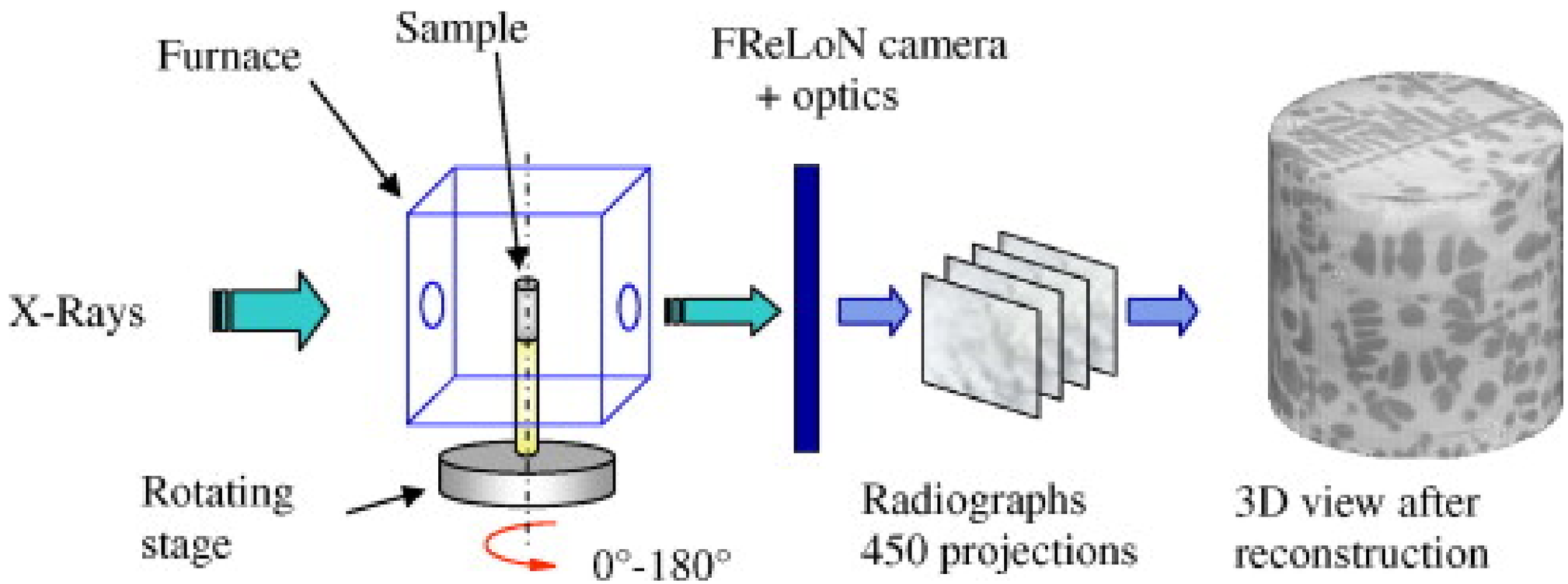
Image courtesy Kamel Fezzaa (APS/AN

AlZr 7.5 µs/frame, ~2µm per pixel

Internal morphology of advancing reaction front is visible with phase contrast imaging



Real time tomography during solidification



Al-10% Cu
Constant cooling at 3 K/min
50 ms per frame
Resolution 2.8 µm
Volume analyzed 0.3 mm

N. Limodin *et al. Acta Mater.* **57**, 2300 (2009)

Real time tomography during solidification

N. Limodin *et al. Acta Mater.* **57**, 2300 (2009)

Summary

- High x-ray energies are useful because they
  - Are highly penetrating, allowing studies of bulk materials/behavior
  - Compact a lot of reciprocal space into a small range of scattering angles—very helpful for *in situ* studies
- For time-resolved studies of irreversible phenomena in polycrystalline specimens, flux is the most important beam characteristic
- Brightness helps, too—it may allow improved spatial resolution and resolution of diffraction peaks (monochromatic v. pink beam)
- Phase-contrast imaging benefits from improved coherency
- Many experiments are limited by detector technology