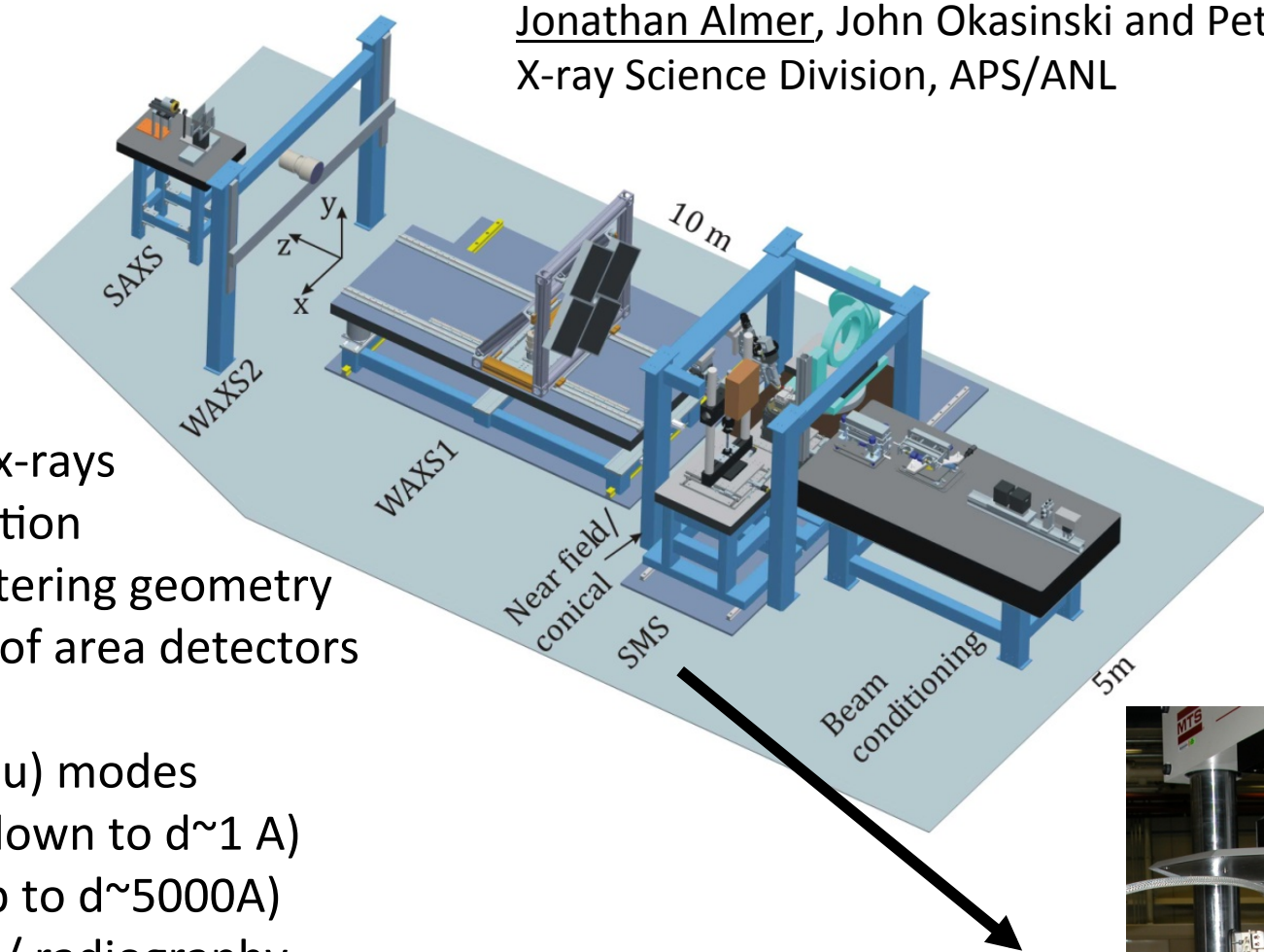
HE-SAXS/WAXS at 1-ID

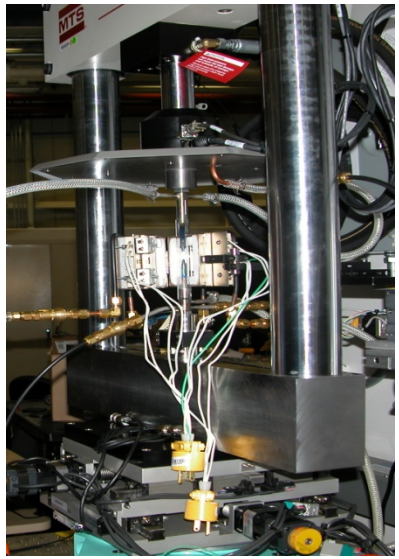
Jonathan Almer, John Okasinski and Peter Kenesei
X-ray Science Division, APS/ANL



50<E<120keV x-rays
- high penetration
- forward scattering geometry
- efficient use of area detectors

Multiple (in-situ) modes
- WAXS (down to d~1 A)
- SAXS (up to d~5000A)
- Imaging / radiography
- Flourescence

Transverse beamsize down to~1x5 um^2 using lenses
Typical SAXS/WAXS images in 1s



Internal strain / microstructure in bulk systems

Damage and strain in engineering materials

- Alloy systems and composites
- SAXS/WAXS: Void nucleation (SAXS) and localized strain (WAXS) in steels (nuclear industry)

Strain in amorphous materials: T. Hufnagel et al, Phy. Rev. B 73 (2006)

Biomechanics of bone and teeth

- SAXS- interfibril strain (d~67nm); WAXS: intrafibril strain
- Ongoing collaboration with S. Stock and D. Dunand (NU)

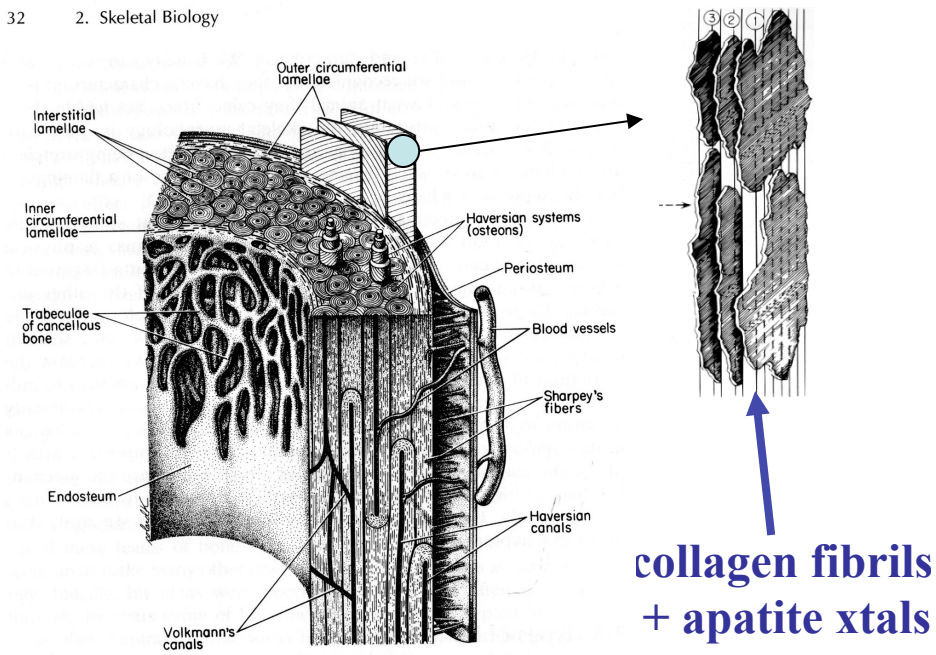
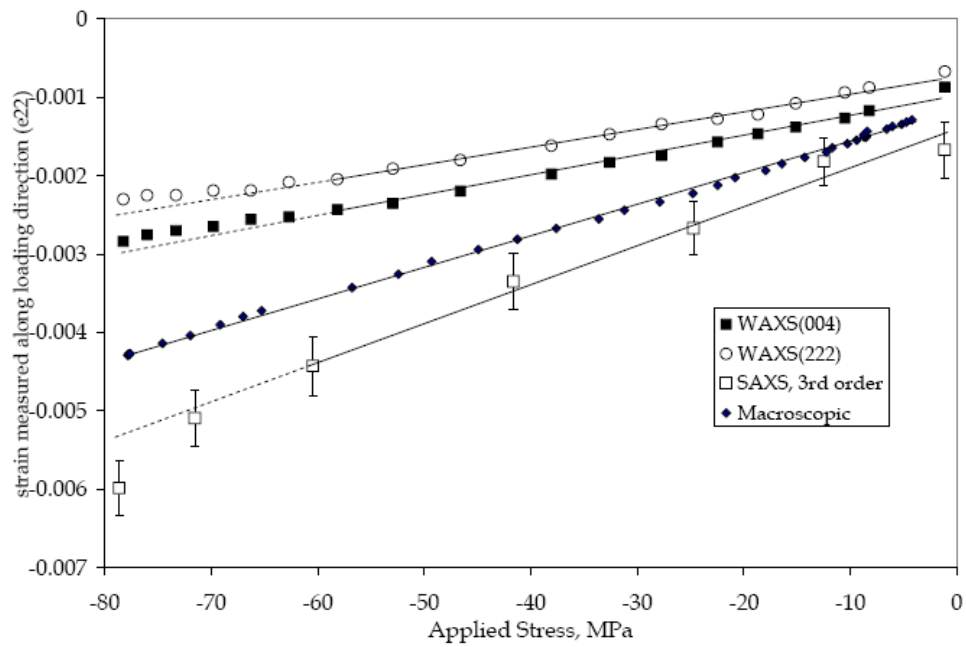


FIGURE 2.1. Sketch of some important features of a typical long bone. (After Benninghoff, 1949.)



In-situ synthesis of nano-particles for Li-ion batteries

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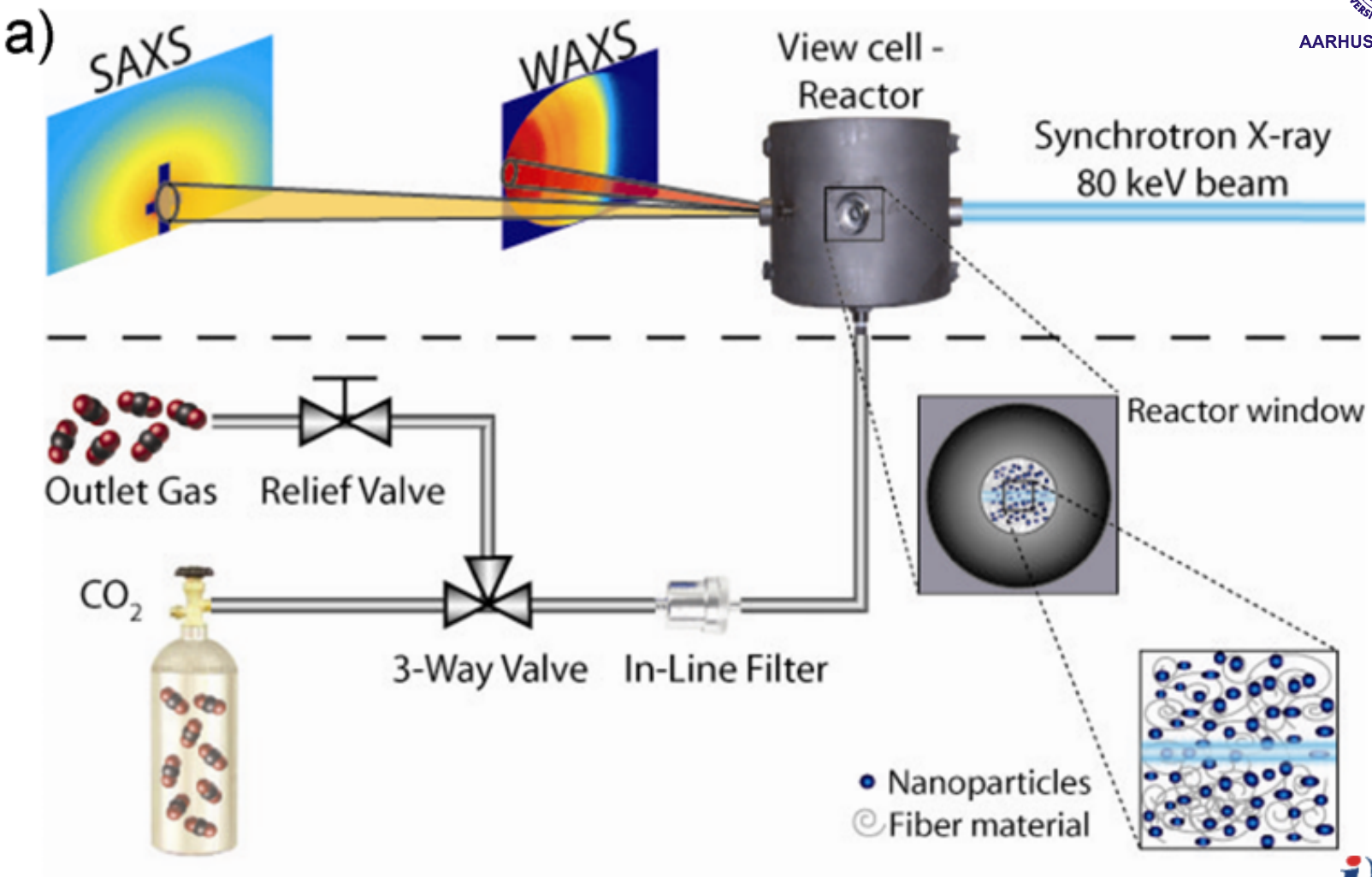

Advanced Photon Source

iNANO

SCF Technologies

In-situ synthesis of $LiCoO_2$ nano particles for Li-ion batteries.

- Supercritical synthesis of $LiCoO_2$ collected at 400°C, 300 bar.
- SAXS morphology and size information on the nano-scale.
- WAXS structure on the atomic scale.

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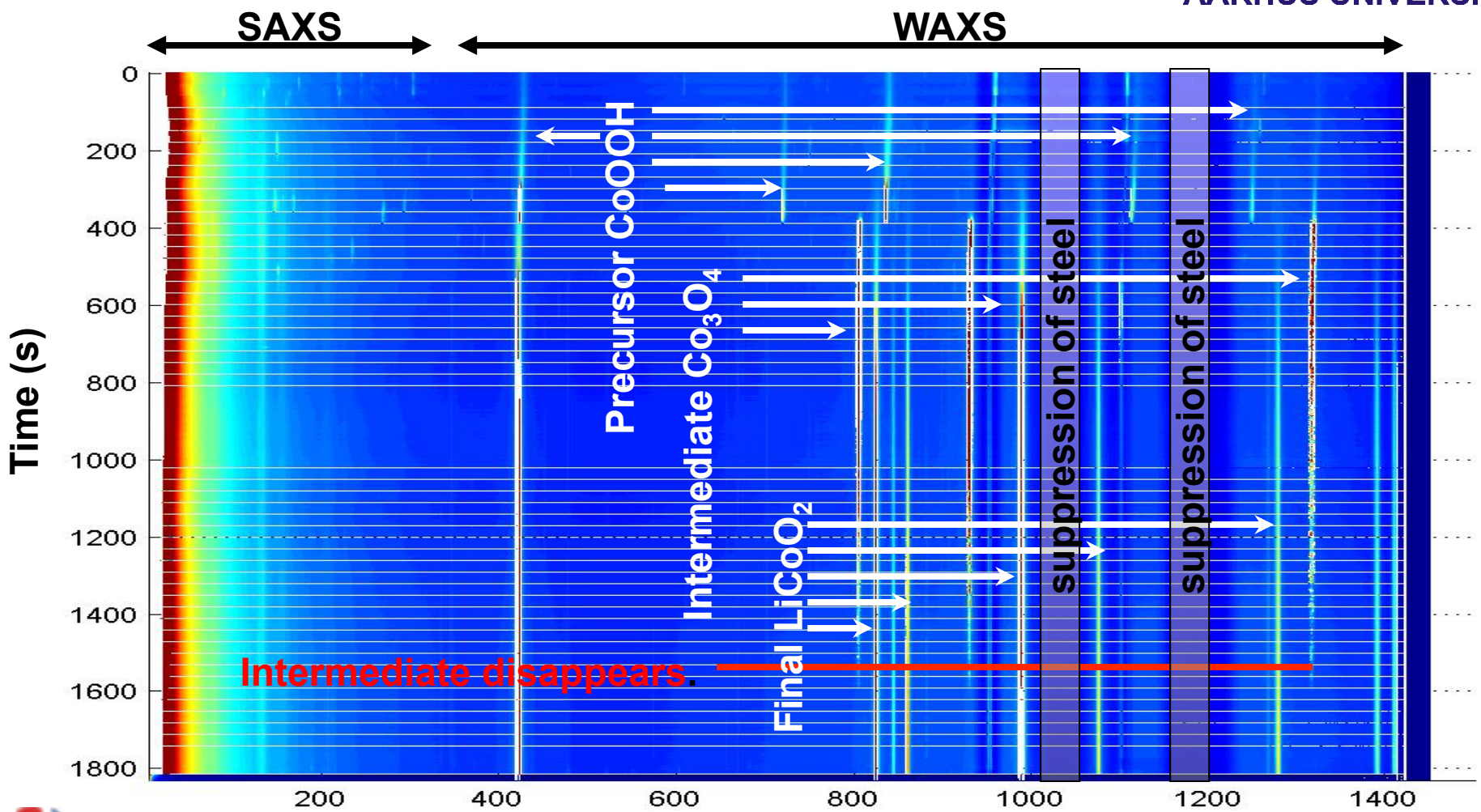
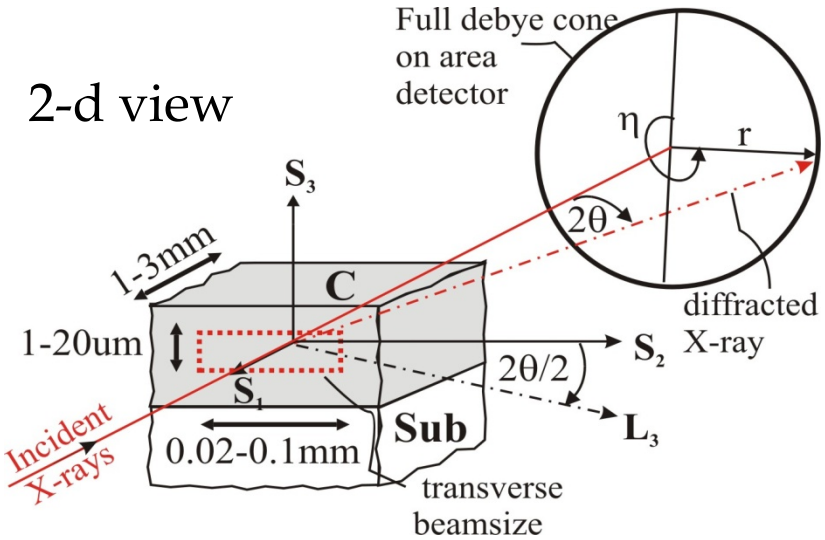


iNANO

SCF Technologies

Layered systems

2-d view



Key features

• Vertical resolution of ~1um with sufficiently small divergence (~100urad)

<u>Direct depth resolution</u> (cf. cumulative in reflection geometry)

• Small bragg angles –> measure 2 of 3 principal sample directions +

• Coatings typically isotropic in-plane -> <u>unique information in single exposure</u>

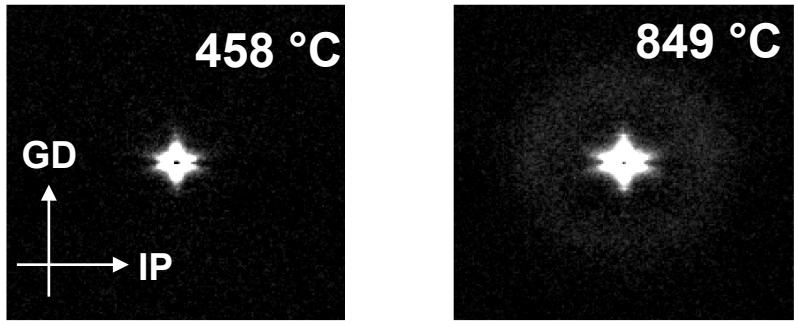
• <u>SAXS/WAXS on same sample volume</u>

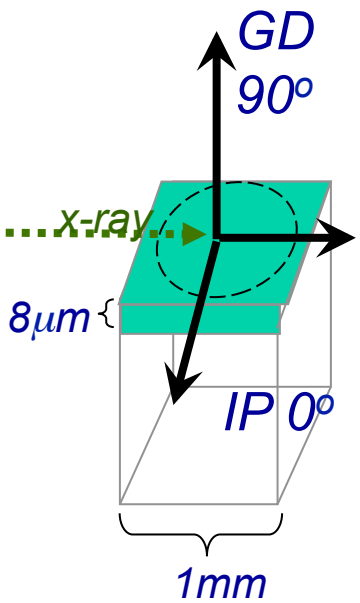
- **Thermal and environmental barrier coatings**
  - Phase stability and strain evolution in Ta-0 EBCs
    - C. Weyant et al, J. Am. Cer. Soc. 83 (2005) and 89(2006).
  - Depth-resolved strain/porosity of EB-PVD TBCs
    - A. Kulkarni et al, J. Am. Cer. Soc 87 (2004).
  - In-situ oxidation studies on TBC bondcoat
    - J. Almer et al, Mat. Sci. Forum 490 (2005)
  - Depth-resolved strain in plasma-sprayed TBCs
    - J. Thornton et al, J. Am. Cer. Soc. 88 (2005).
- **Metal-nitride coatings and multilayers**
  - Strain, texture and phase-decomposition
    - J. Almer et al, J. App. Physics 94 (2003).
    - M. Oden et al, APL (2009).
    - C. Aydiner et al, J. App. Physics 102 (2007).
- **Solid-oxide fuel cells**
  - Depth-resolved porosity and phase
    - A. Allen et al, Cer. Eng. 25(3) (2004).
  - Cr-poisoning mechanism
    - D.J. Liu et al, APL (2009).

Decomposition of arc-evaporated c-$Ti_{0.50}Al_{0.50}N$ thin films during annealing

In situ SAXS/WAXS shows formation of c-TiN and c-AlN during spinodal decomposition & coarsening.

Explain "self-hardening" mechanism at elevated temps





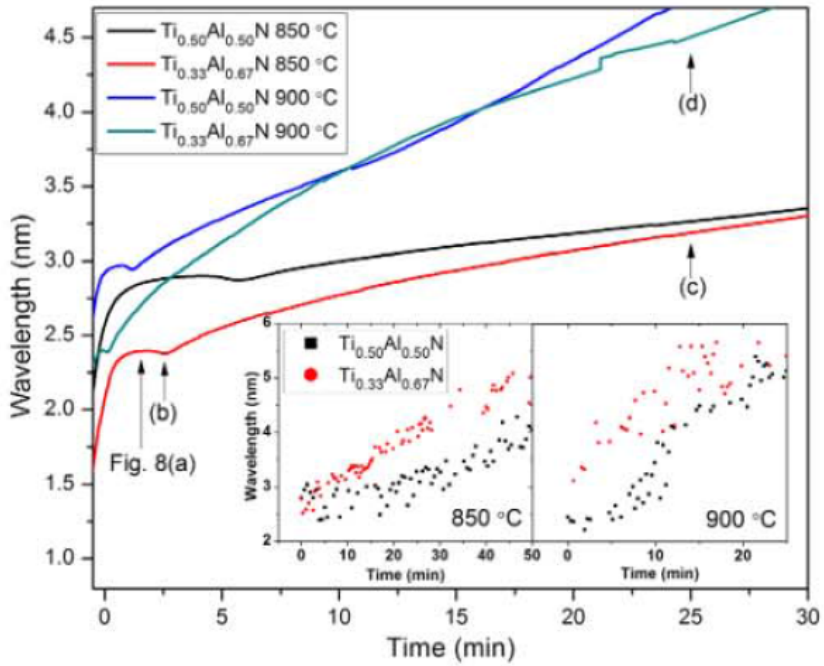
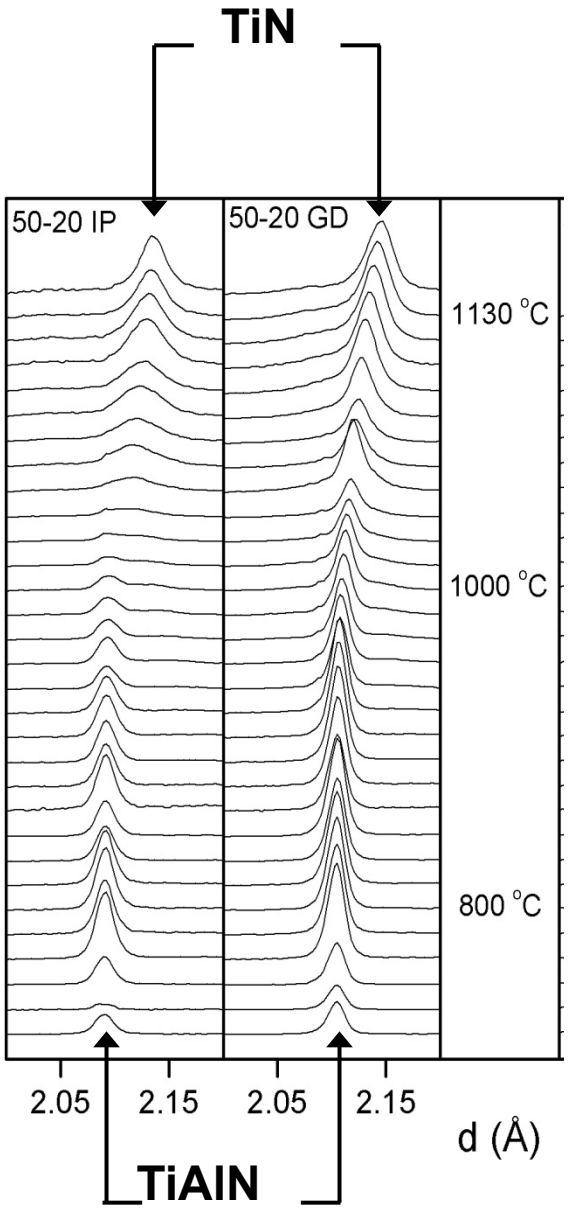
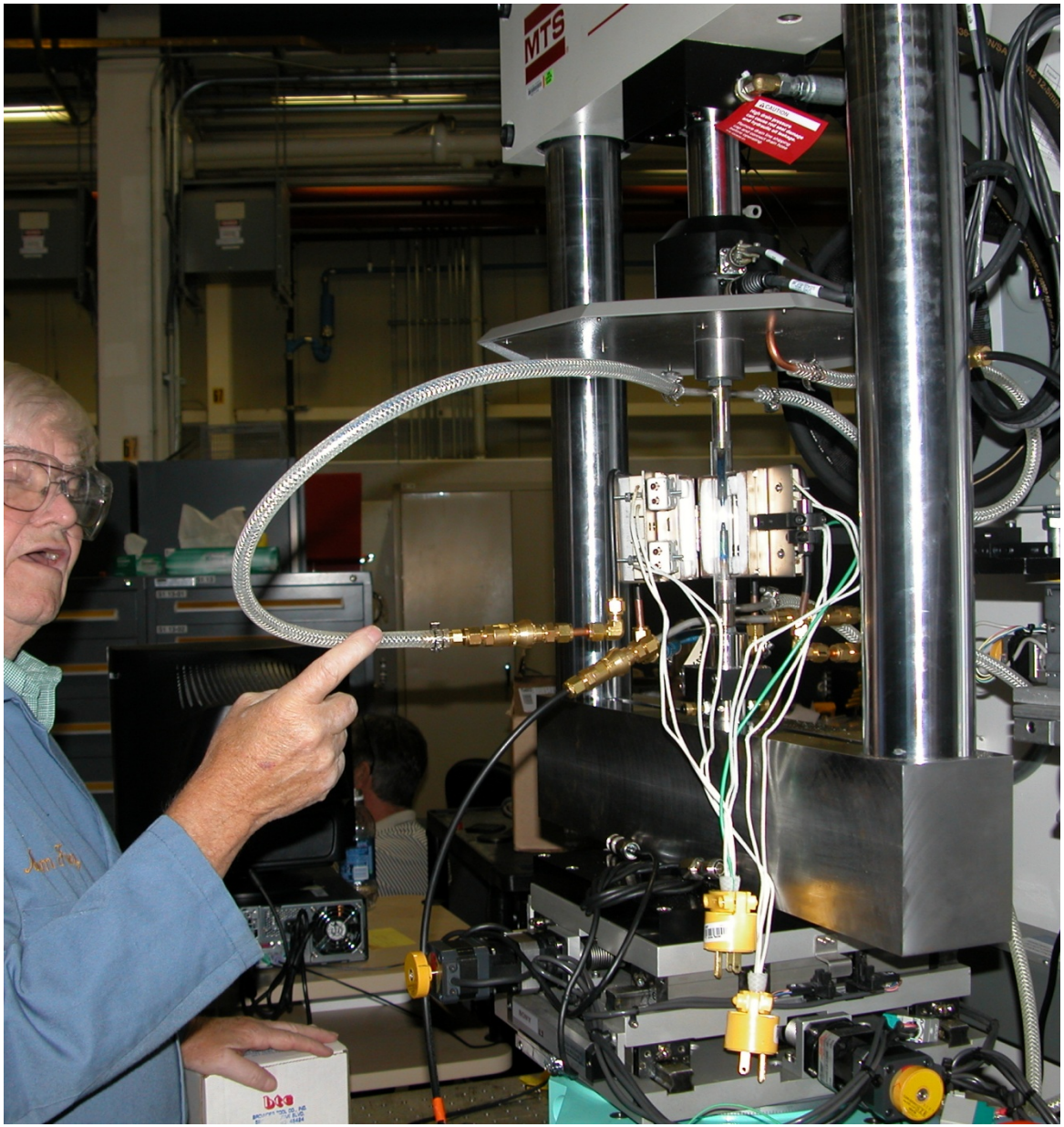


FIG. 7. Simulated compositional wavelength versus annealing time of $Ti_{0.33}Al_{0.67}N$ and $Ti_{0.50}Al_{0.50}N$ at 850 and 900 °C. Time equal 0 is set at the time when the simulation reaches the maximum annealing temperature. Insets show SAXS-wavelength data from annealing at 850 °C (left) and 900 °C (right). The arrows in the figure indicate the times for the simulated microstructure shown in Figures 8(a)–8(d).



Knuttson et al, JAP 113 (2013)

In-situ thermo-mechanical device



Specification	Value
Loading mechanism	Hydraulics
Load rating	+- 15 kN
Stroke	100 mm
Loading modes	Tension, bending, compression (monotonic and fatigue)
Furnace	1250 C (inert possible)
Low-temperature	Liquid N_2 (inert environment)
Weight	200 kg
Degrees of freedom	4 translations, 1 rotation, 2 tilts

Custom-designed by Materials Testing Systems, user-collaborations on environments