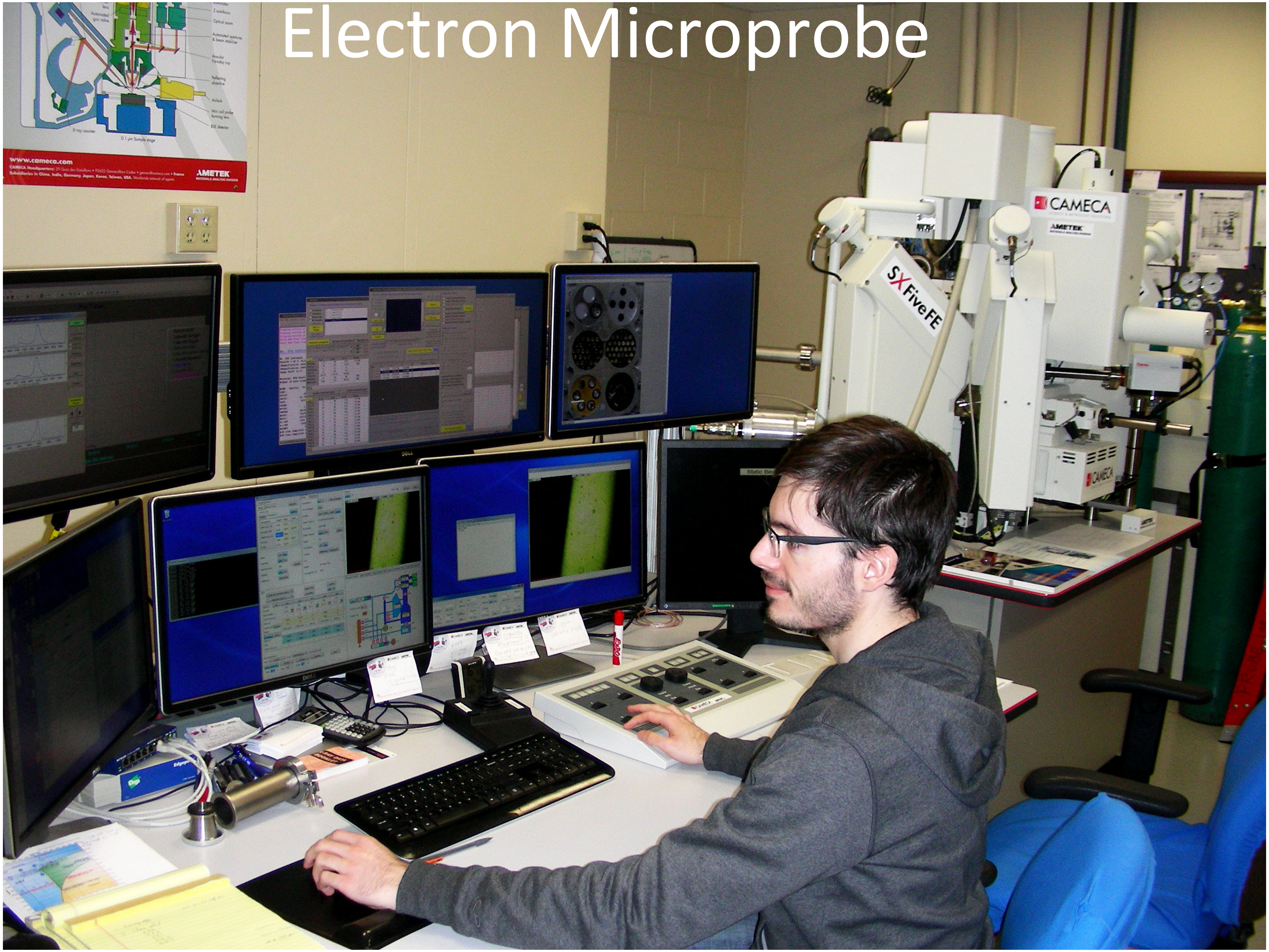
UW Madison Electron Microbeam Laboratory

- 2 CAMECA electron microprobes – only electron microprobes in Wisconsin = EPMA (electron probe microanalysis)
- Hitachi variable pressure SEM
- Located on 3rd floor of Weeks Hall
- Available to both educational and commercial researchers

Electron Microprobe



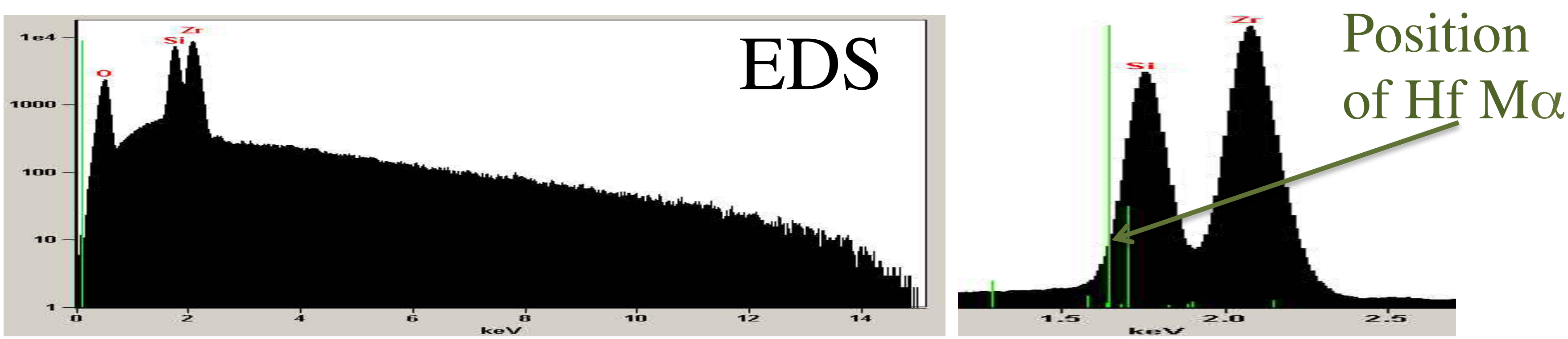
CAMECA SXFive-FE Probe installed in November 2014 in Room 312 Weeks Hall

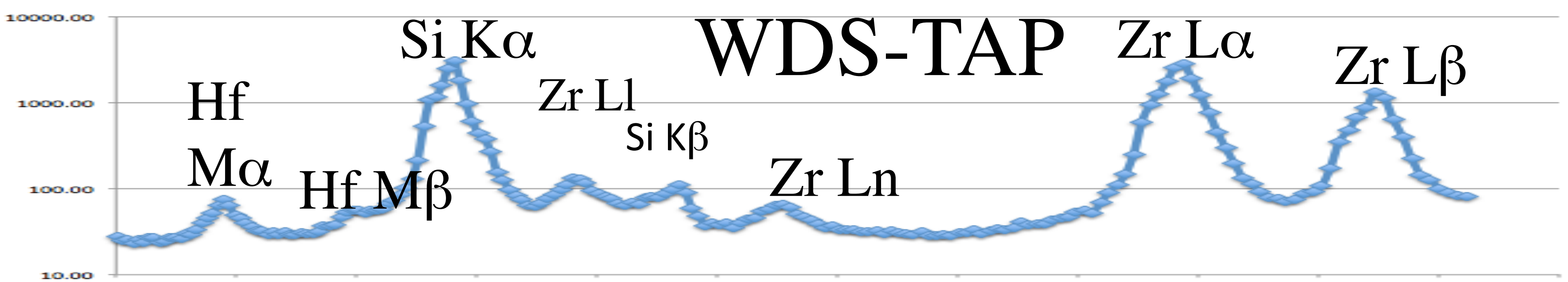
Electron Microprobe

- Field emission electron source
- “WDS”: wavelength dispersive spectrometry
- 5 high resolution crystal spectrometers ($n\lambda=2d\sin\theta$)
- Optimal spectral resolution to resolve peak overlaps
- Choice of 14 crystals to measure all elements B to U
- 6 large area crystals for enhanced intensities for trace elements
- Minimum detection limits to tens of ppms
- Quality control with a range of analytical reference materials
- Ability to operate at low keV to achieve sub-micron lateral analytical resolution
- Ability to determine thin film layer composition (using special software)

WDS and EDS: similar but key differences

- Many people are familiar with SEM with EDX/EDS (energy dispersive X-ray spectrometry)
- WDS can resolve many spectral interferences, such as shown here for (~1 wt%) Hf Mα adjacent to Si Kα:





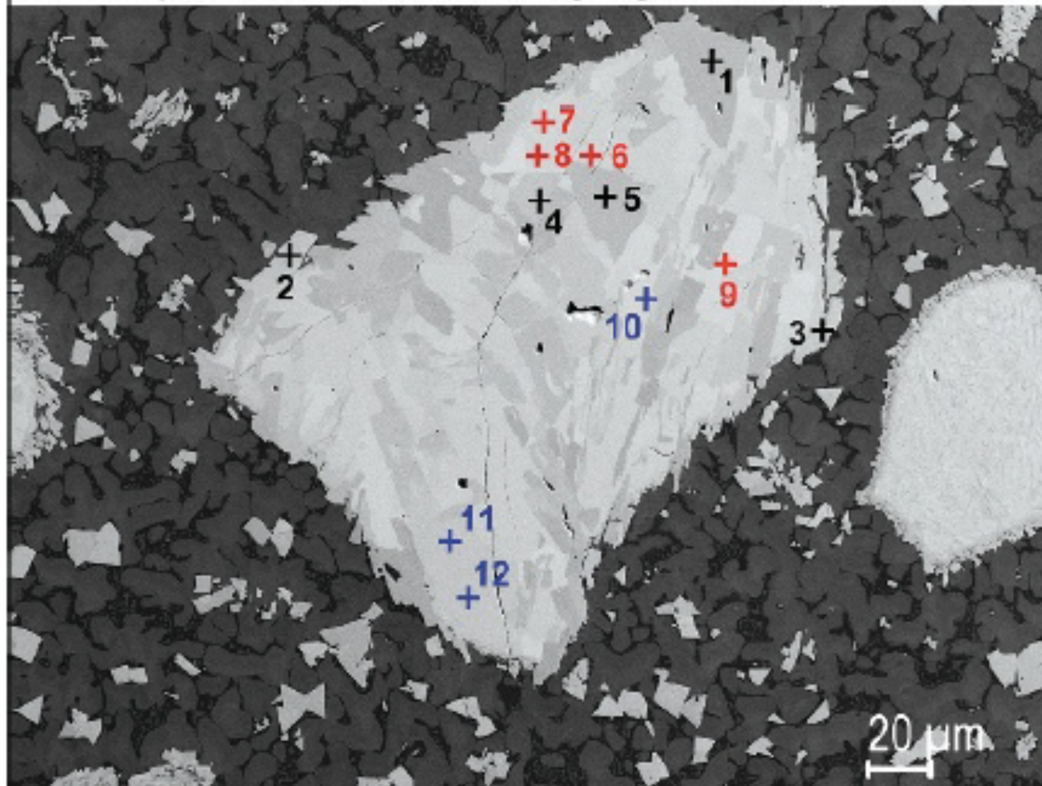
Highly accurate analyses (standards-based)

For example:

- Ability to accurately determine light element compositions (e.g., C), such as shown here (also B, not shown)
- Ability to identify hard-to-distinguish WC_{1-x} phase

Yu, P., Chai, X., Landwehr, D., & Kou, S., 2016, Ni-WC Hardfacing by Gas Metal Arc Welding. WELDING JOURNAL, 95(12).

Composition measurements by high-resolution EPMA



80 – 100 nm electron beam (very small)

WC_{1-x} close to W_3C_2

#9	phase	C (at%)	W (at%)	Others
1	WC	49.47	49.73	-
2	WC	51.72	49.42	-
3	WC	48.18	48.07	-
4	WC	52.14	44.79	-
5	WC	51.66	47.73	-
6	WC_{1-x}	39.06	59.37	-
7	WC_{1-x}	40.34	59.72	-
8	WC_{1-x}	39.07	64.18	-
9	WC_{1-x}	38.69	58.29	-
10	W_2C	32.23	69.22	-
11	W_2C	33.42	60.74	-
12	W_2C	34.98	63.92	-

Variable Pressure SEM: a unique analytical instrument

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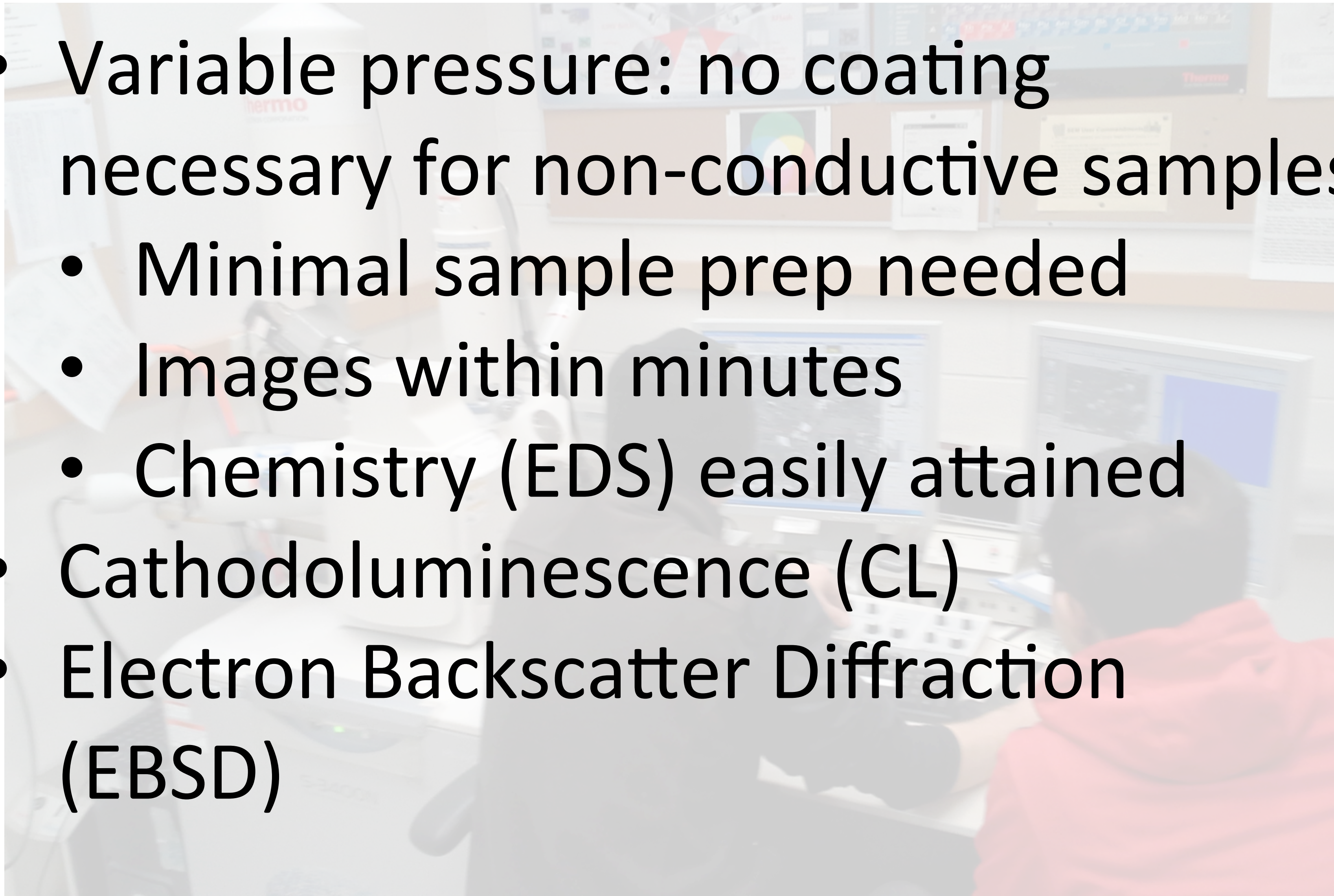
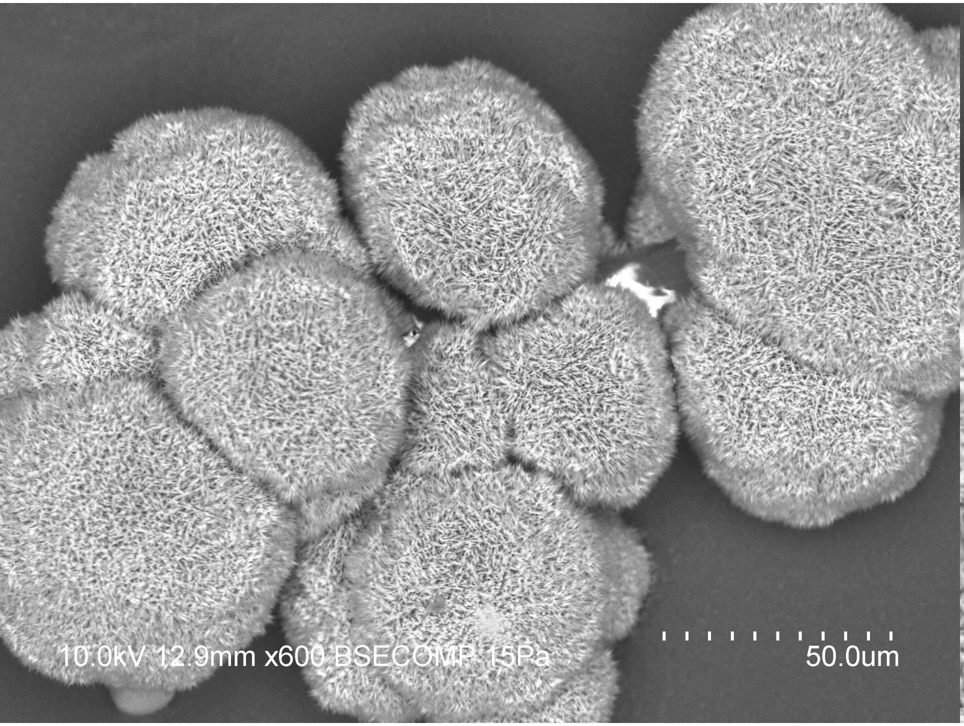
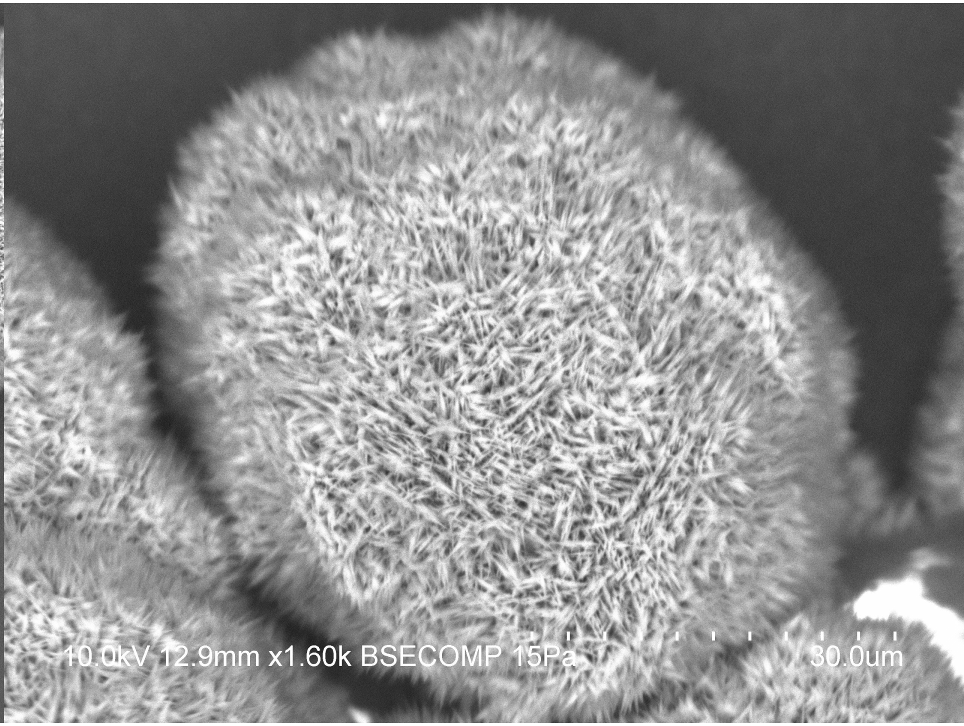


- Variable pressure: no coating necessary for non-conductive samples
  - Minimal sample prep needed
  - Images within minutes
  - Chemistry (EDS) easily attained
- Cathodoluminescence (CL)
- Electron Backscatter Diffraction (EBSD)

Examples of non-conductive samples easily imaged with Hitachi S3400 VP-SEM





Our sample is aragonite ($CaCO_3$) spherulites -- spherical aggregates of polycrystals, where aragonite fibers grow along the radial direction of the sphere.

–Chang-Yu Sun, Gilbert Group, Physics Department, UW Madison

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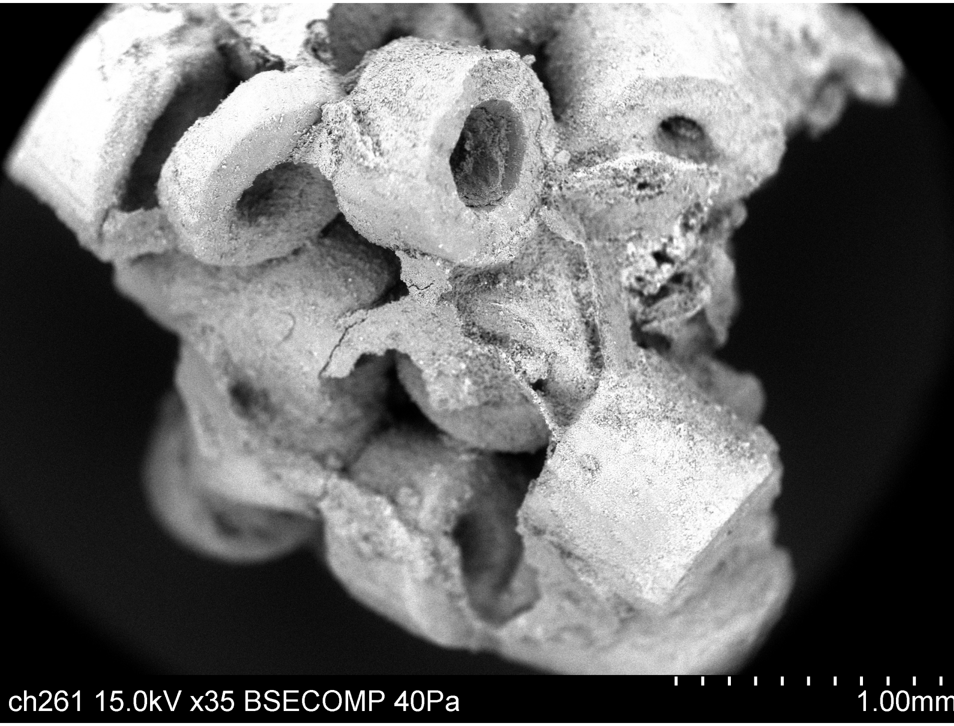
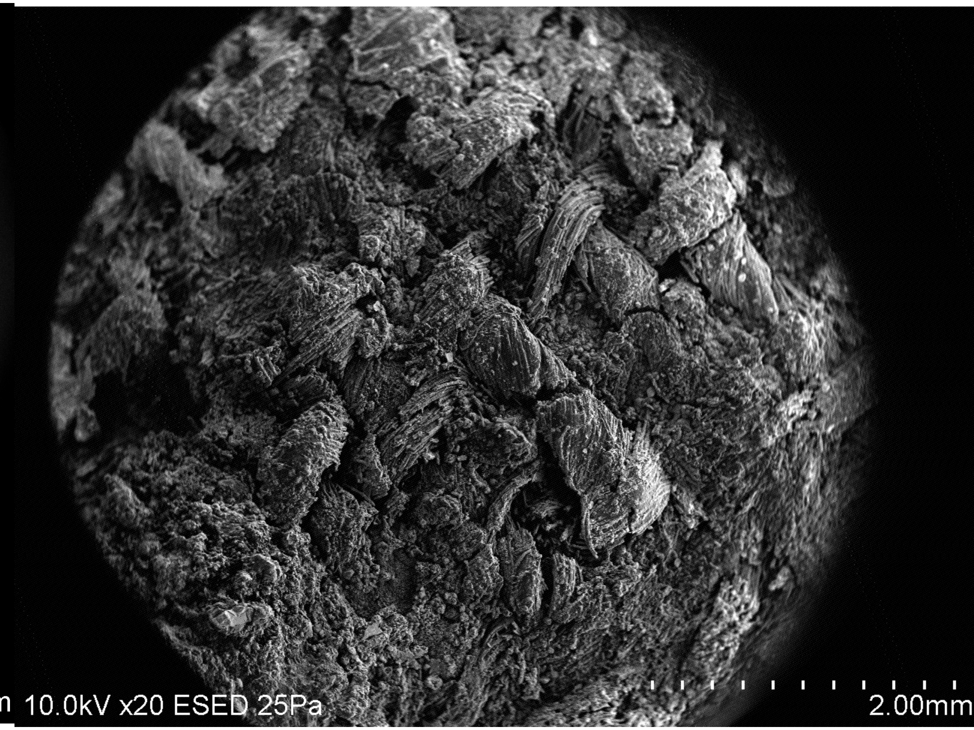


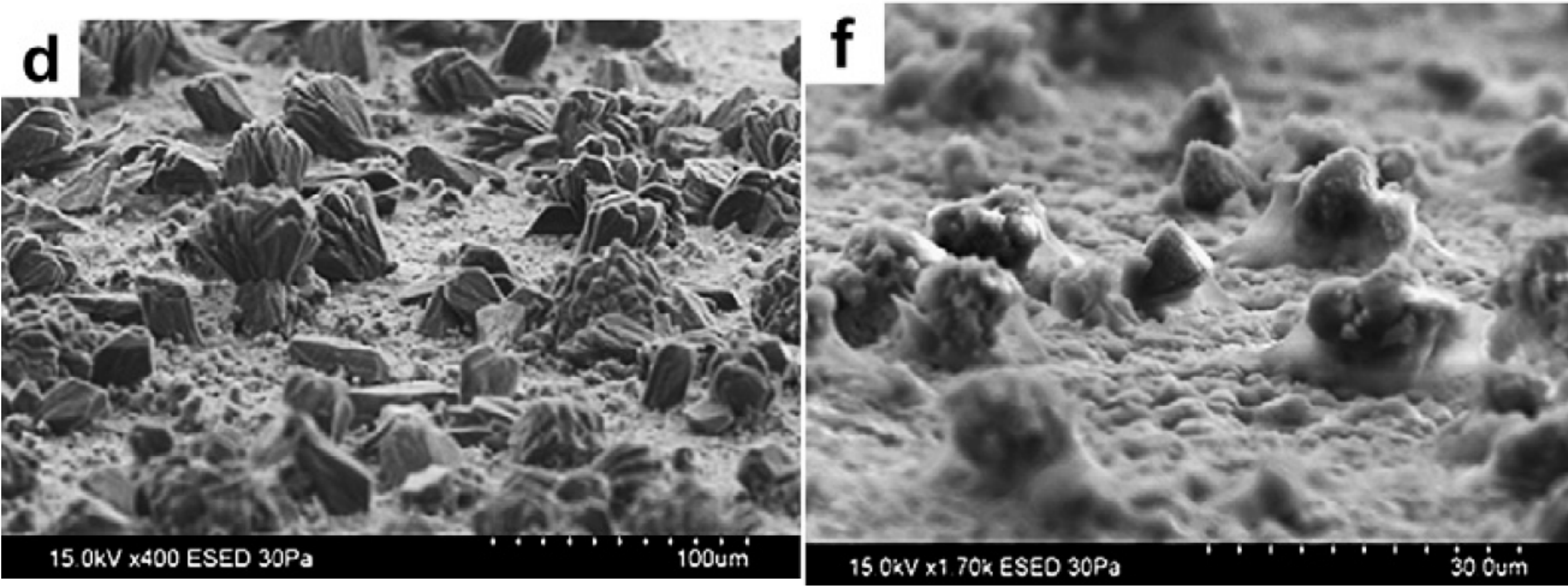


From Mark Kenoyer, Anthropology:

Steatite microbeads (steatite fired at over 1000°C → enstatite) From Chanhudaro, Pakistan, circa 2450-2000 BC

Woven silk from Harappa 2450 BC. The silk fibers have been preserved by copper salts from the corrosion of a bronze tool.

Examples of non-conductive samples easily imaged with Hitachi S3400 VP-SEM

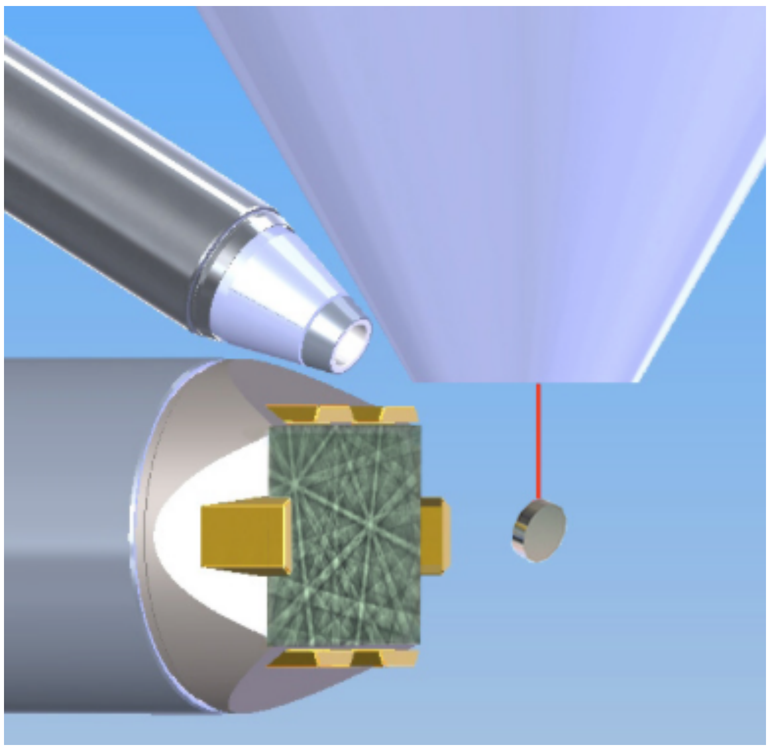


Experiments growing stem cells upon pseudo-wollastonite ($CaSiO_3$) substrate after 12 days. Control=d. Calcite crystals grow up from substrate, no stem cells present. f=high seeding density (30,000 cells/ cm^2). Cells now are present.

Zhang, N., Molenda, J. A., Fournelle, J. H., Murphy, W. L., & Sahai, N. (2010). Effects of pseudowollastonite ($CaSiO_3$) bioceramic on in vitro activity of human mesenchymal stem cells. *Biomaterials*, *31*(30), 7653-7665.

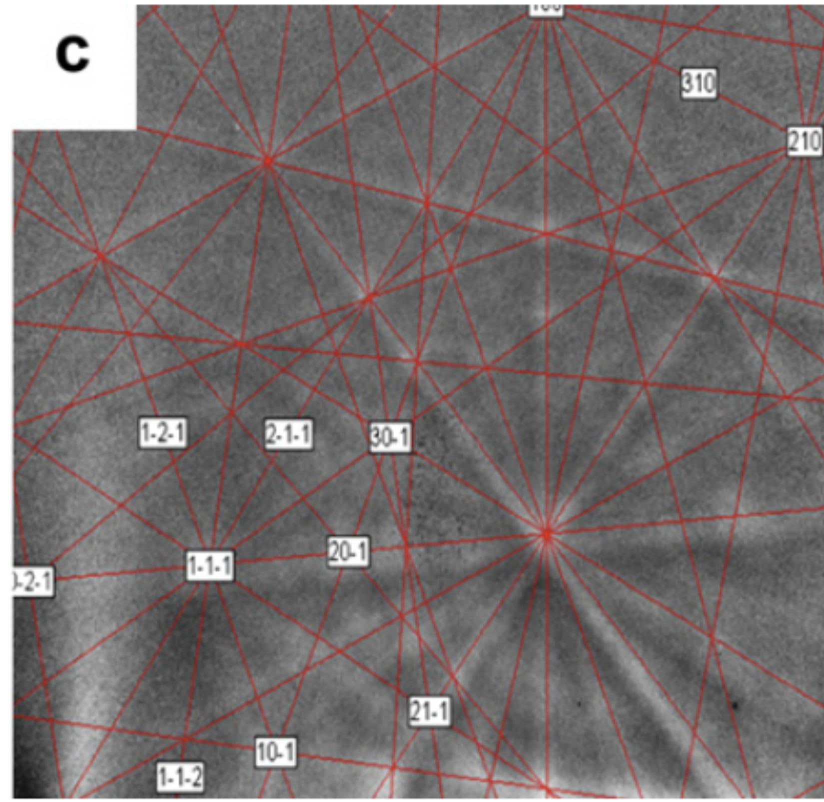
Examples of non-conductive samples easily imaged with Hitachi S3400 VP-SEM and crystal identification by EBSD

=Electron Backscatter Diffraction



Our Oxford EBSD system can easily index triclinic, monoclinic, orthorhombic, hexagonal, tetragonal as well as cubic patterns.

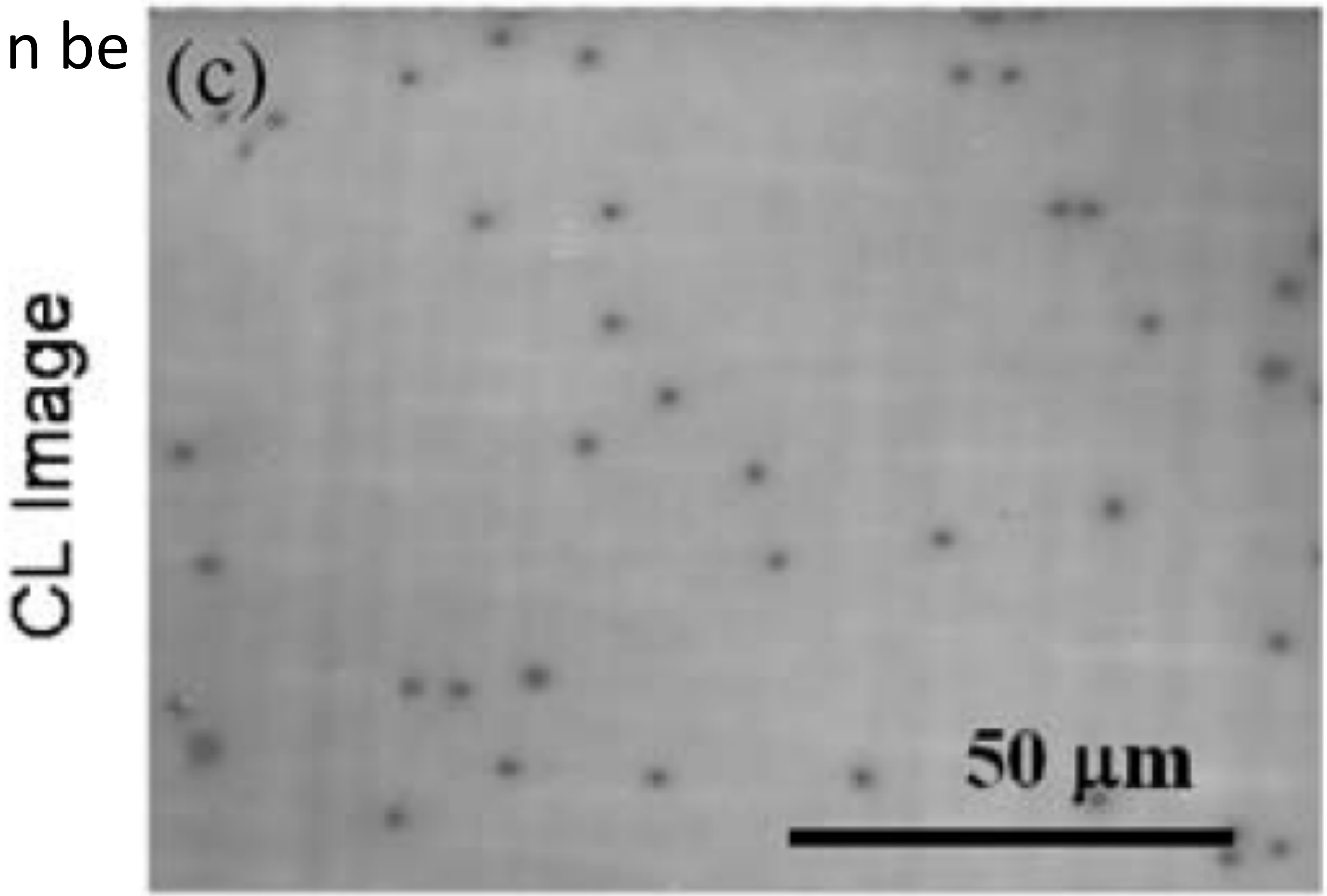
c



$CaCO_3$ determined to be calcite by EBSD

Zhang, N.,et al. (2010). *Biomaterials*, *31*(30), 7653-7665

Cathodoluminescence example: Tuning lattice mismatch in solar cells -- CL is emitted everywhere except where there are threading dislocations (black spots): the densities can be quantified.



Fetzer, C. M. et al, (2004, High-efficiency metamorphic GaInP/GaInAs/Ge solar cells grown by MOVPE. *Journal of crystal growth*, *261*(2), 341-348.