Elements of the cosmos: what STEM-EDXS can tell us about the history of materials of the Universe

Guest speaker: Rhonda Stroud

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Moderator:

Dr. Igor Nemeth
Application Scientist EDS,
Bruker Nano Analytics

Guest Speaker

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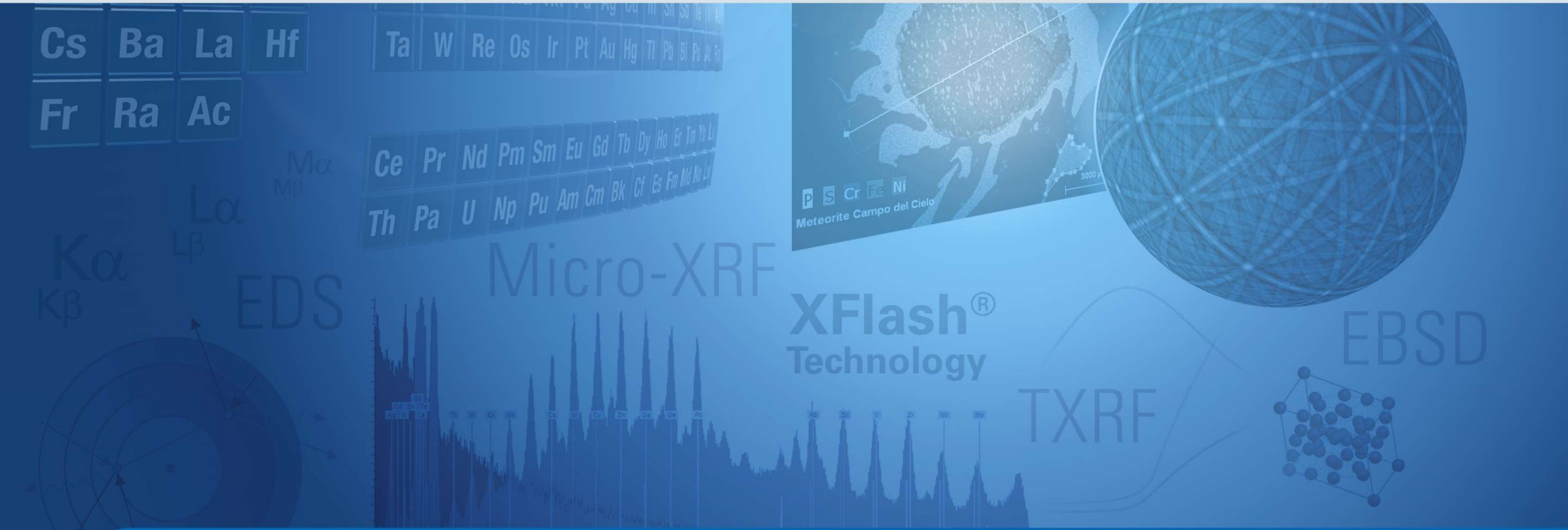
Research Physicist,
US Naval Research Laboratory

Dr. Meiken Falke

Global Product Manager EDS/TEM,
Bruker Nano Analytics

Bruker Nano GmbH,
Instrumentation for Materials Analysis

Meiken Falke et al.



Bruker Nano GmbH,
Instrumentation for Materials Analysis

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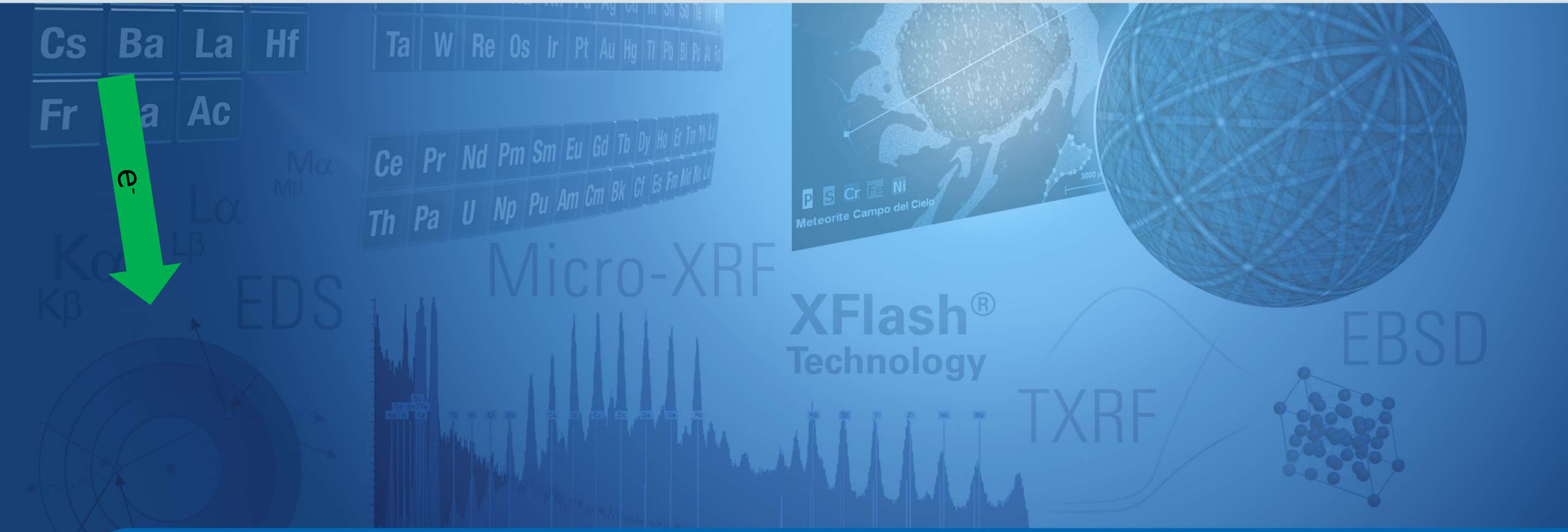

Bruker Nano GmbH,
Instrumentation for Materials Analysis

Meiken Falke et al.

Bruker Nano GmbH,
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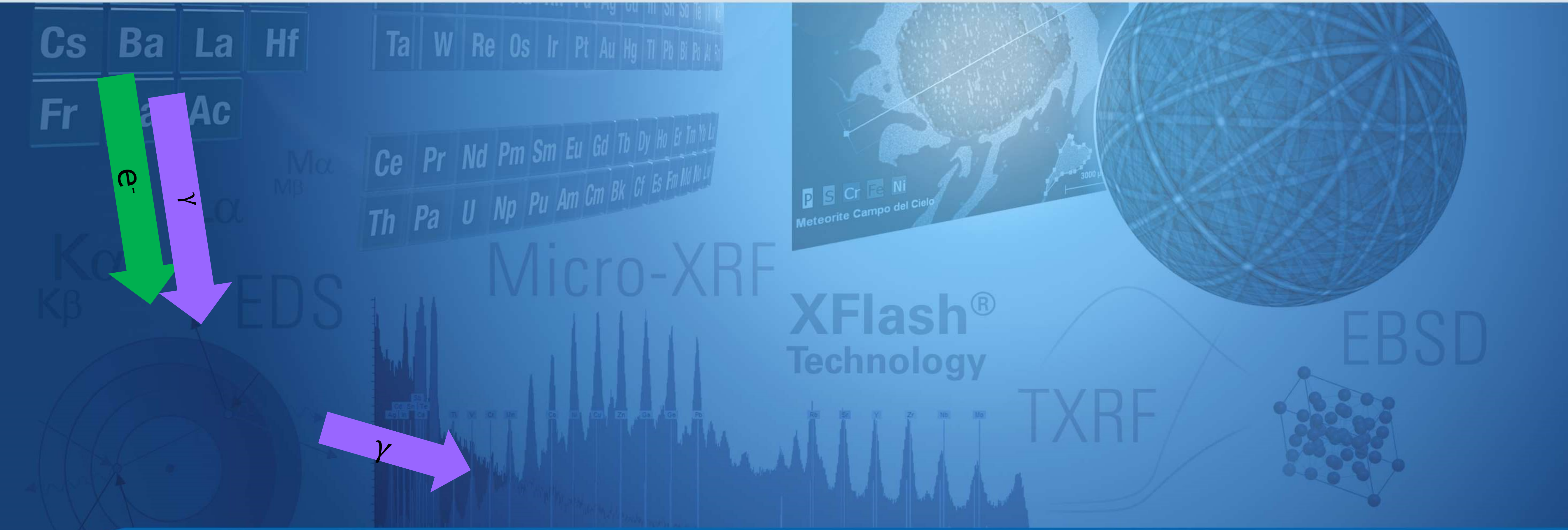

Bruker Nano GmbH,
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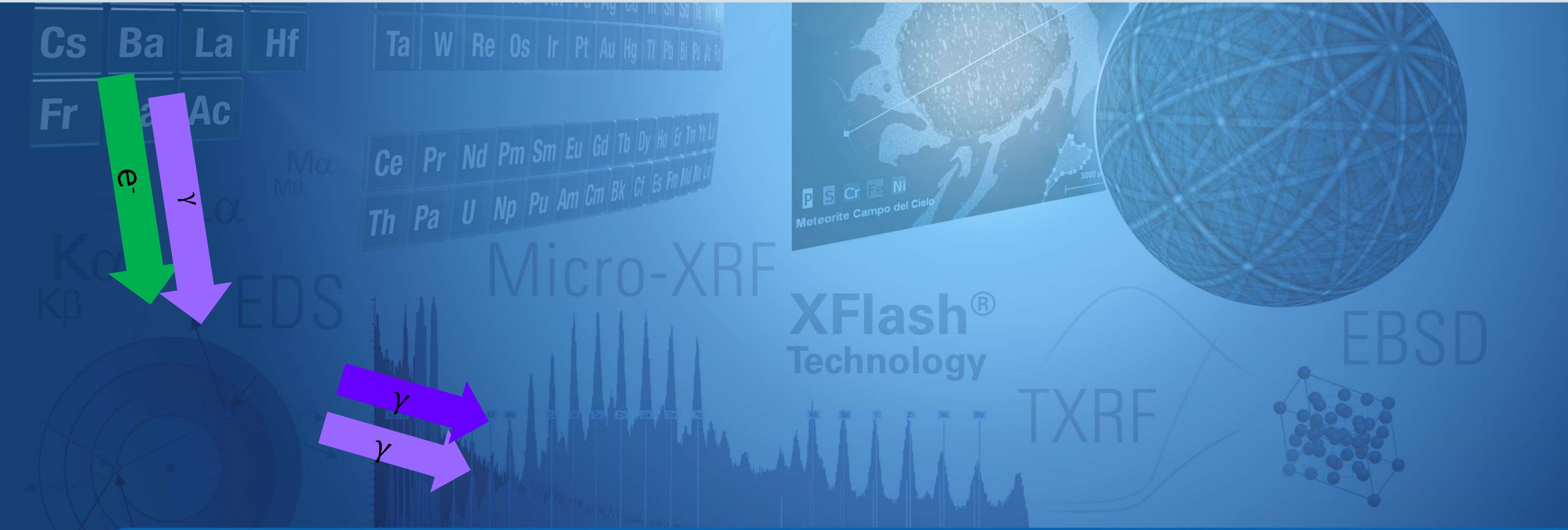

Bruker Nano GmbH,
Instrumentation for Materials Analysis

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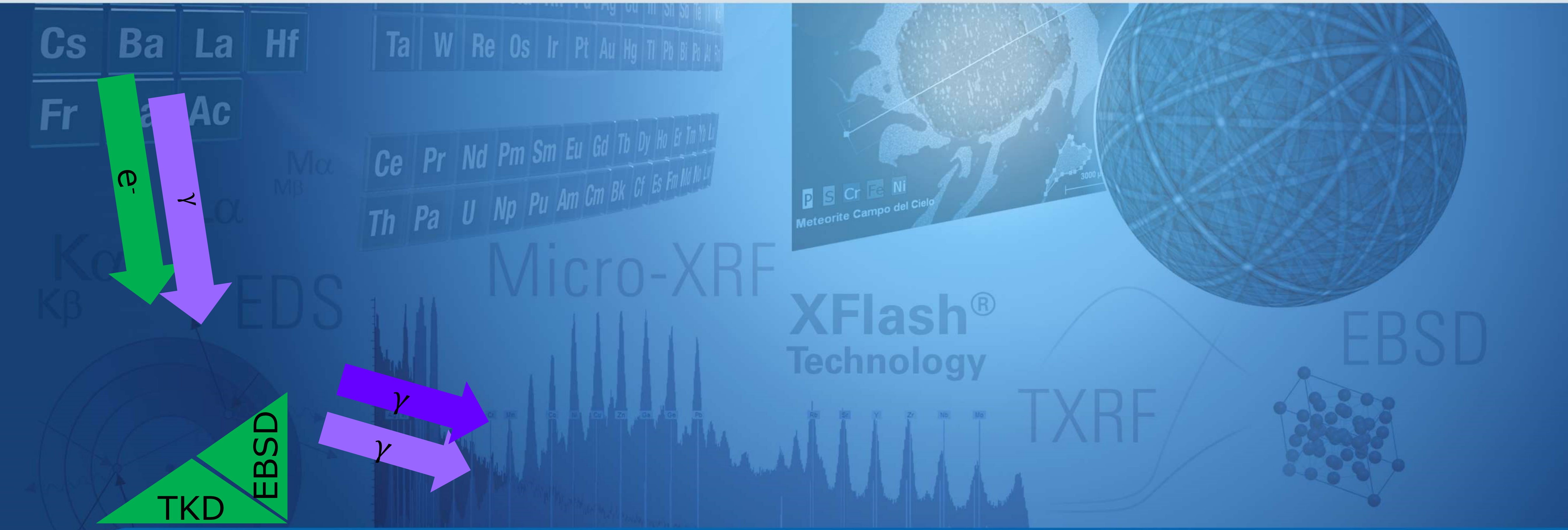
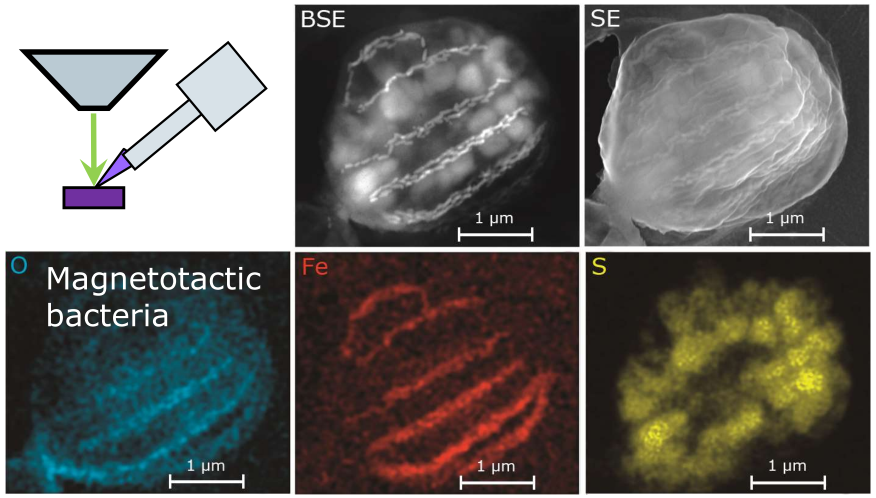
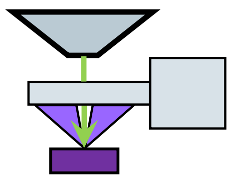


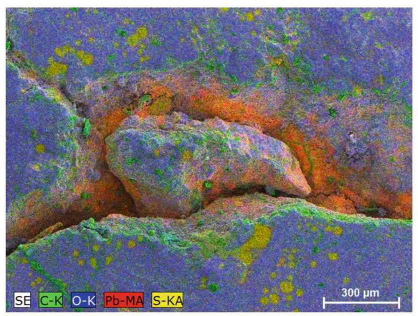
Bruker Nano GmbH,
Instrumentation for Materials Analysis (mm to µm)

Single/multiple EDS



Annular EDS, Flat QUAD

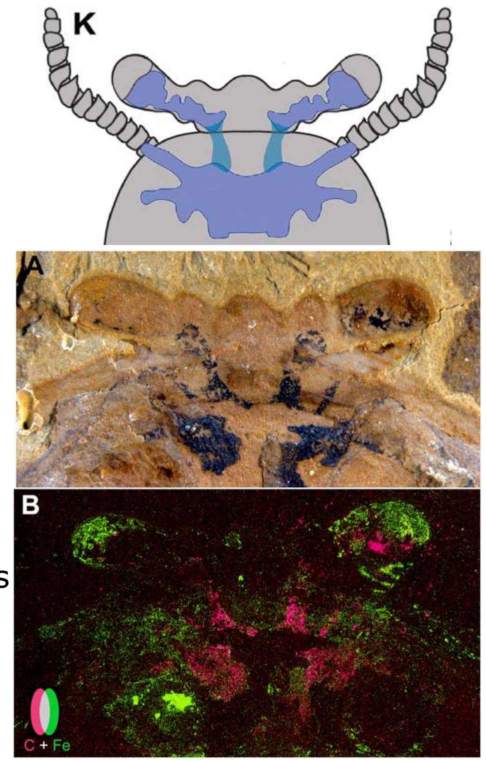




Composite net intensity map of a Mocs meteorite specimen: a cracked surface with lead deposits (red) within the cavities

Annular EDS, FlatQUAD

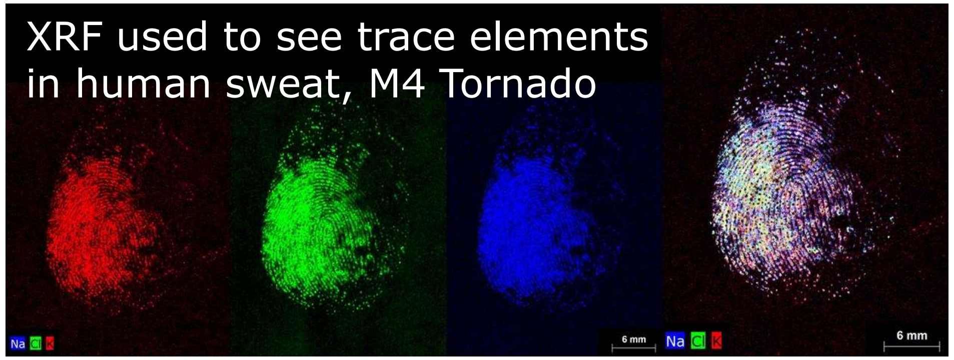
Ma et al., 2015, Current Biology 25, 2969–2975
http://dx.doi.org/10.1016/j.cub.2015.09.063
Open access under the CC BY-NCND license: http://creativecommons.org/licenses/by-nc-nd/4.0/



K: Interpretive drawing of a Cambrian arthropod fossil

A: Carbon-traces in direct illumination

B: EDS of **C**, **Fe**

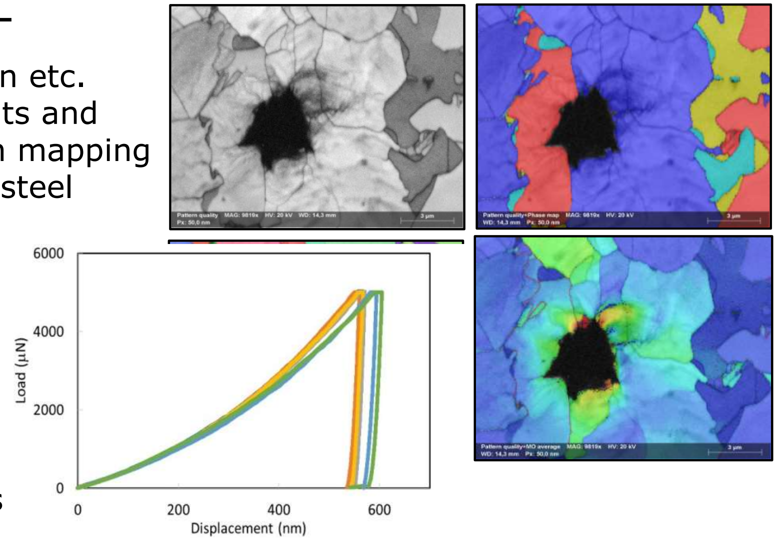


EBSD +

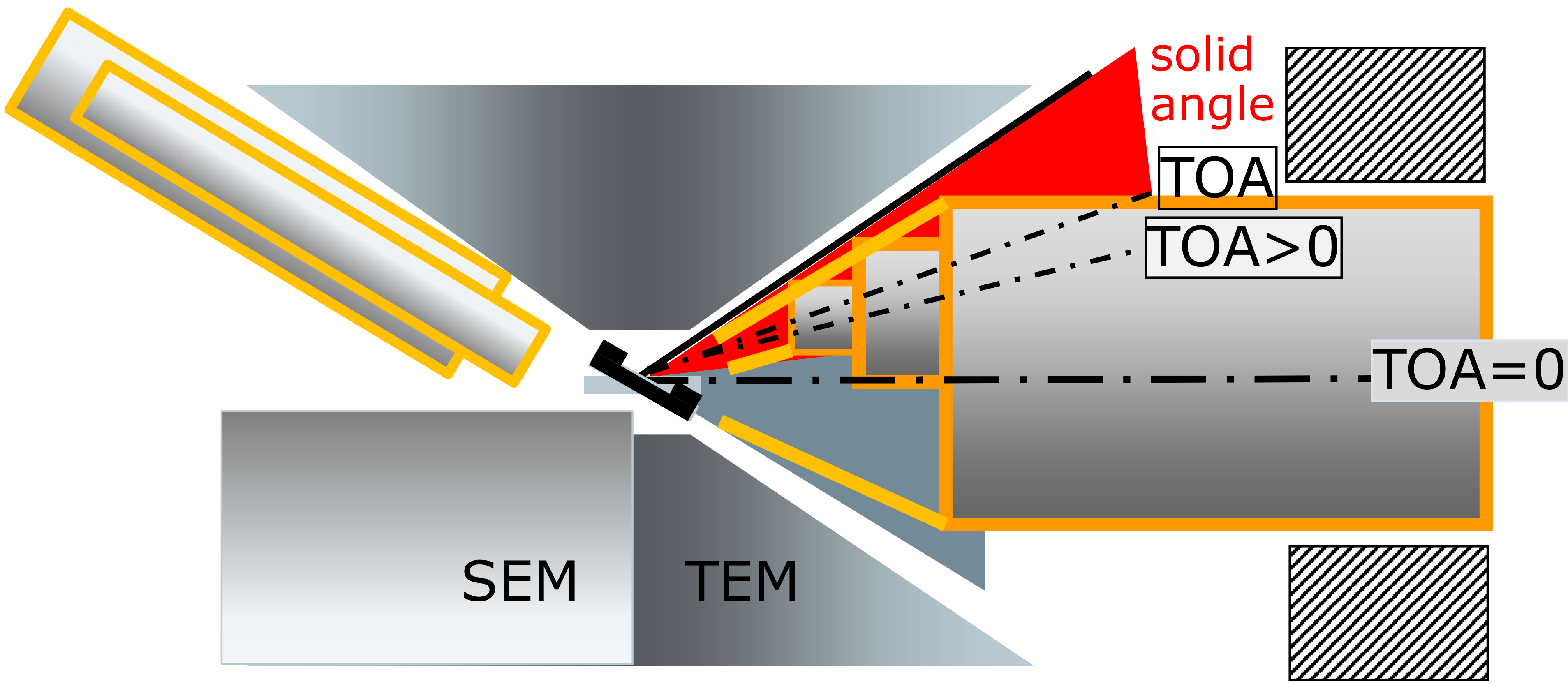
Indentation etc. experiments and orientation mapping In Duplex steel

TKD

Available as well on/off axis



Geometric constraints in SEM and TEM:
aiming for the nm scale and below,
Solid and take-off angle are important to consider!



Windowless high collection angle EDS with 100mm² oval silicon drift detector and All advantages of the ESPRIT analysis software

BRUKER

XFlash®6-100 oval for SEM

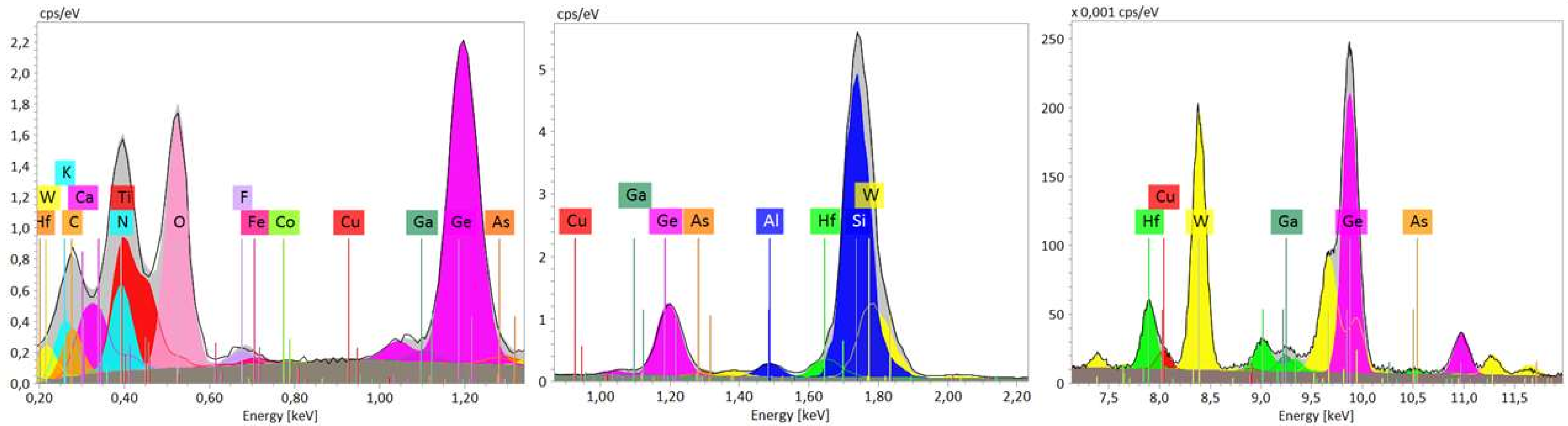
- Compatible to all types of SEM, FIB-SEM incl. STEM in SEM (T-SEM)
- Accelerating voltage up to 30 kV
- Optimized for each microscope geometry
- Solid angle of up to 0.4 sr

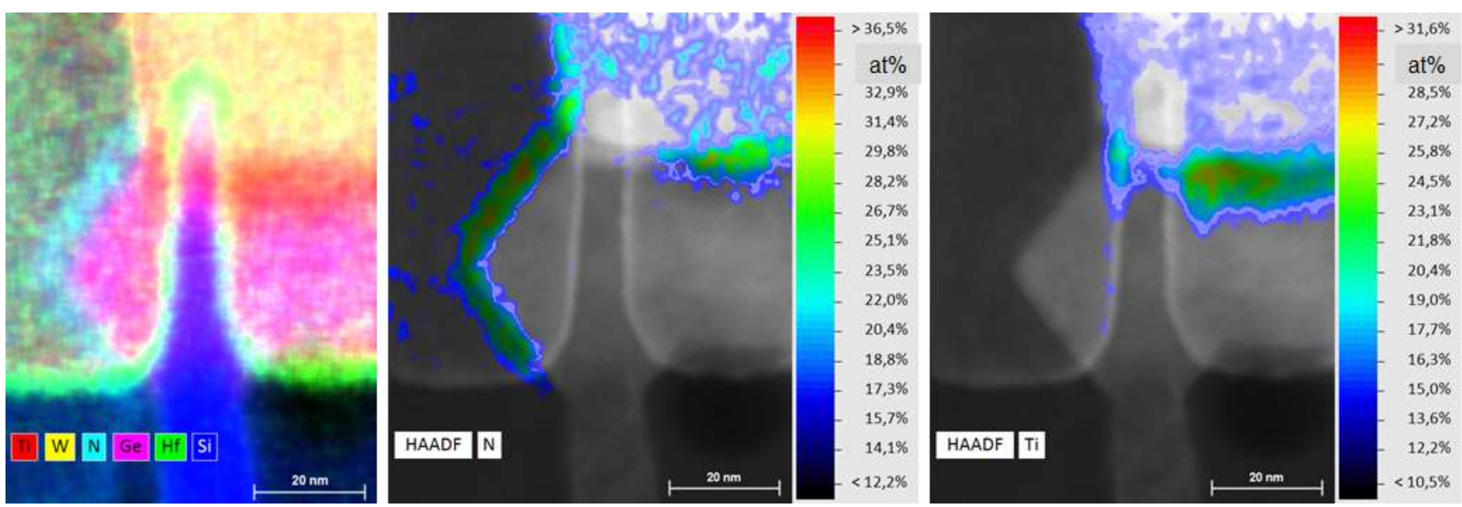
XFlash®6T-100 oval for STEM

- Tailored to individual pole piece geometry
- Accelerating voltage up 300 kV, UHV compatible
- Solid angle up to 0.7 sr or e.g. 0.4 sr
- Take-off angle up to 13.4°, adaptions vary

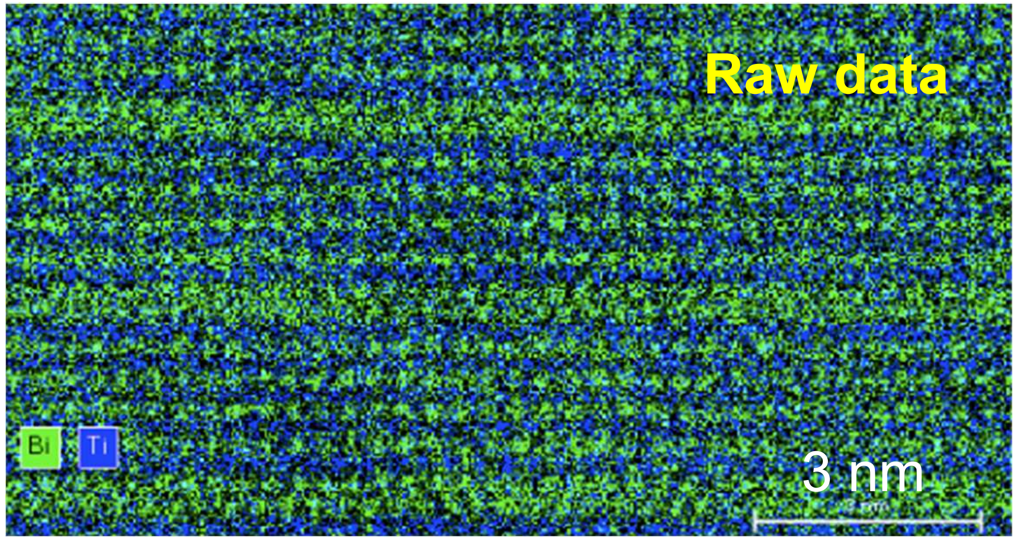
Example of STEM EDS using XFlash®6T-100 oval on TFS Titan: Quantitative Element Mapping of Semiconductor Nanostructures

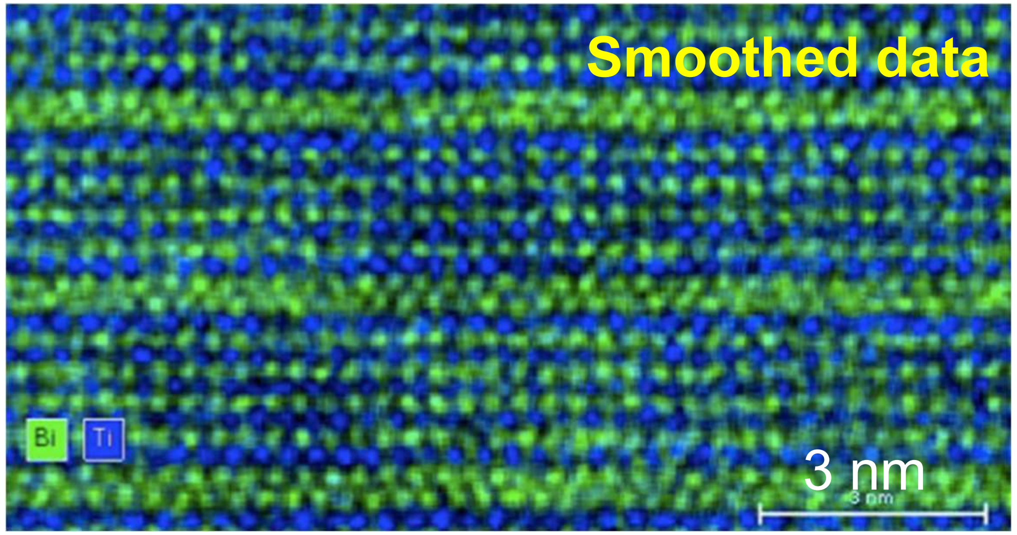
Deconvolution and Quantification Result





Example of STEM EDS using XFlash®6T-100 oval Nion UltraSTEM: Element Mapping of Multiferroic $Bi_6Ti_xFe_yMn_zO_{18}$





TCD (Trinity College Dublin)
Nion UltraSTEM200XE 200 kV,
Dedicated STEM, CFEG;
$\Omega \sim 0.7$sr, TOA > 13°

432x225 pixels,
4.1 msec/pix => 400 sec for map.
No drift correction.
Bi = green, Ti = blue.

courtesy Lynette Keeney, Clive Downing and Valeria Nicolosi. TCD, Ireland.

Specimen:
$Bi_6Ti_xFe_yMn_zO_{18}$
See:
*"Direct atomic scale determination of magnetic ion partition in a room temperature multiferroic material" Scientific Reports **7**, Article number: 1737 (2017) open access*

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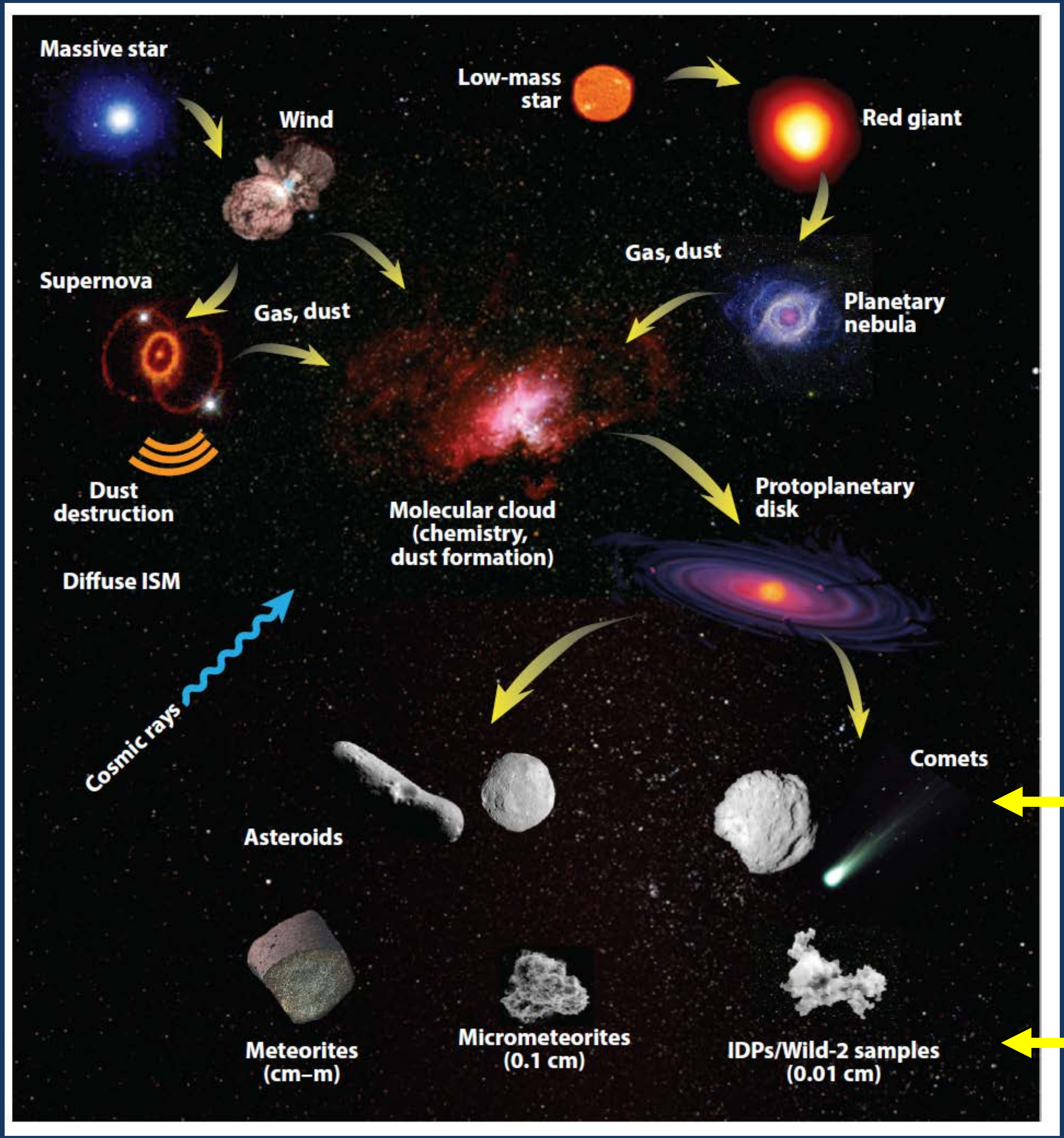
Rhonda Stroud

U.S. NAVAL RESEARCH LABORATORY



Nittler and Ciesla (2016)

Solar systems origins written in gas and dust

Fossils of Early Solar System

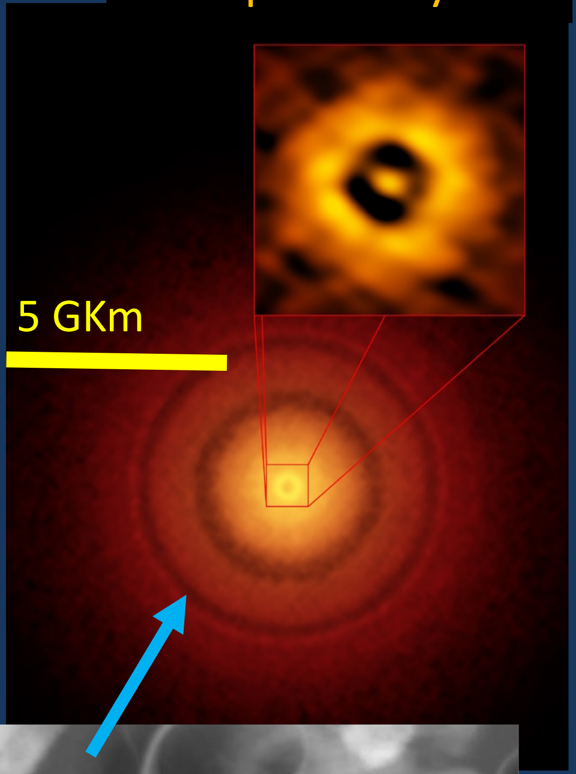
Laboratory Samples

Secrets of the Early Solar System *to be* Revealed by Laboratory Analysis

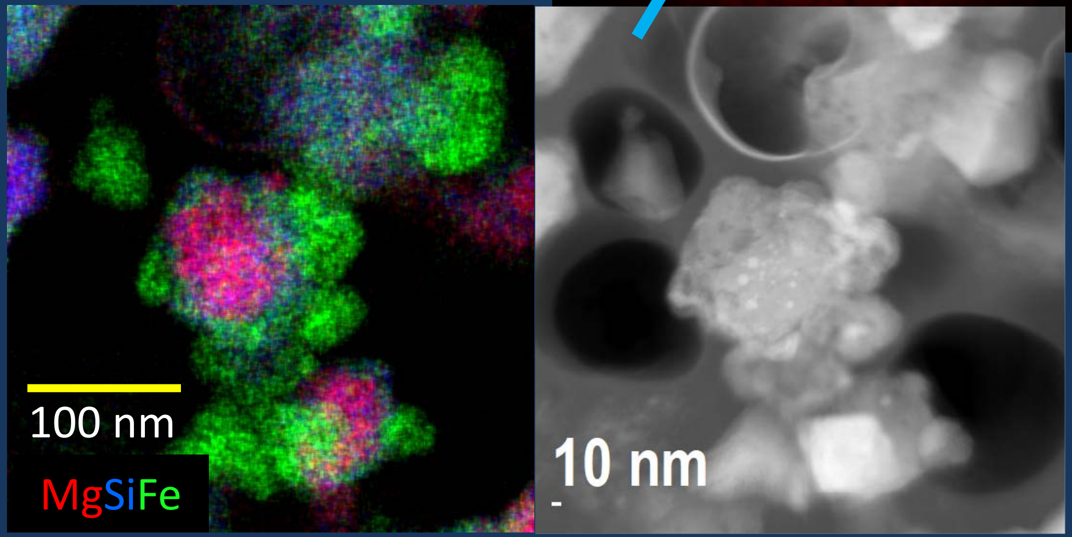
- **Essential Science**
  - **What were the original ingredients or our solar system?**
  - **Why life on Earth? Where else?**
  - **Are there technologically useful materials in the cosmos?**

- **Coordinated Microanalysis provides answers**
  - **SEM + SIMS + FIB + STEM + XANES**

ALMA Image of Protoplanetary disk



Cometary building block found in a meteorite



Nittler et al.
Nature Astronomy (2019)

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- Essential Science
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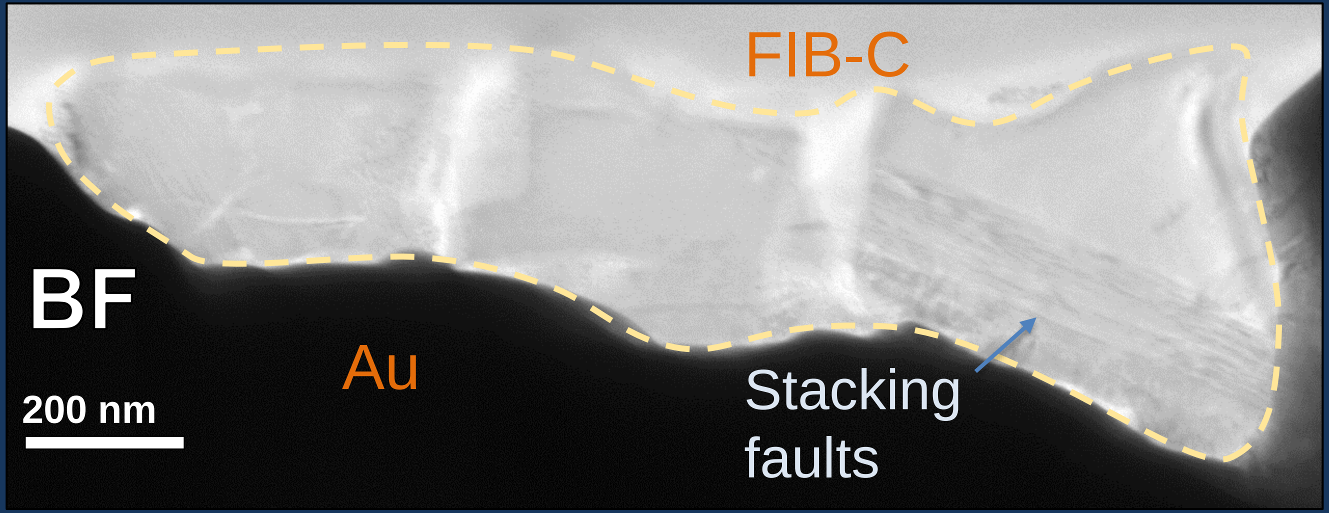
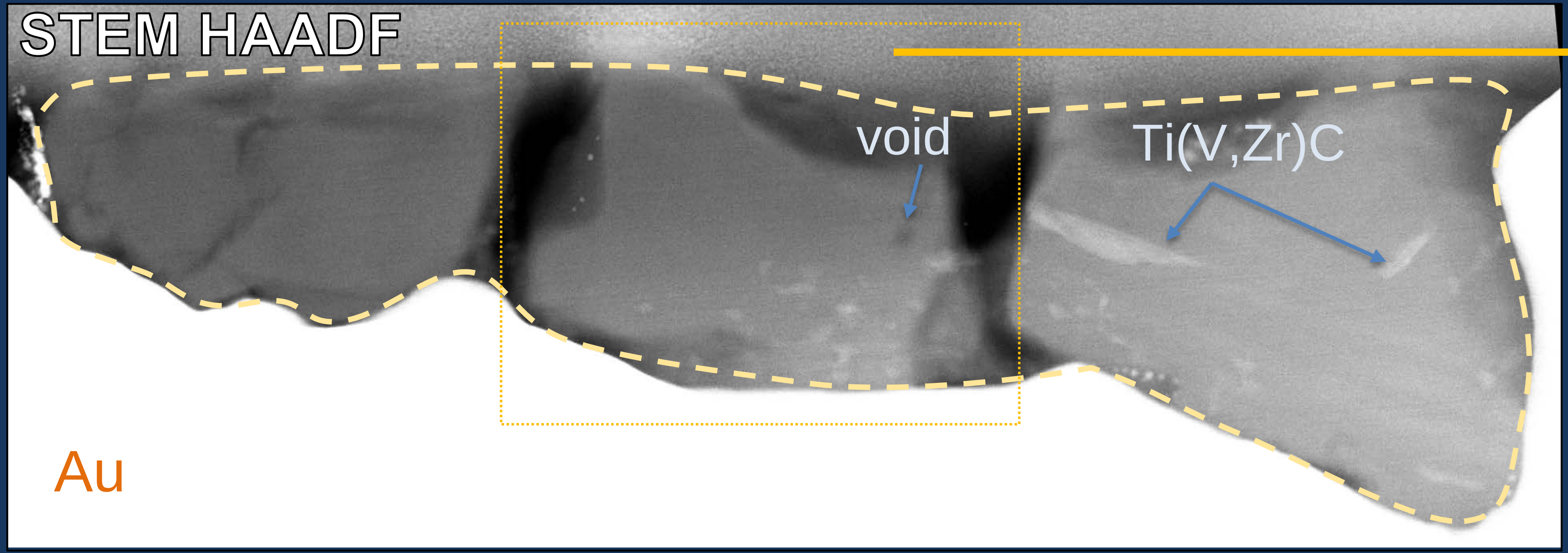
Nion UltraSTEM 200-X
0.7 sr Bruker SDD Xflash 100
Gatan Enfinium ER

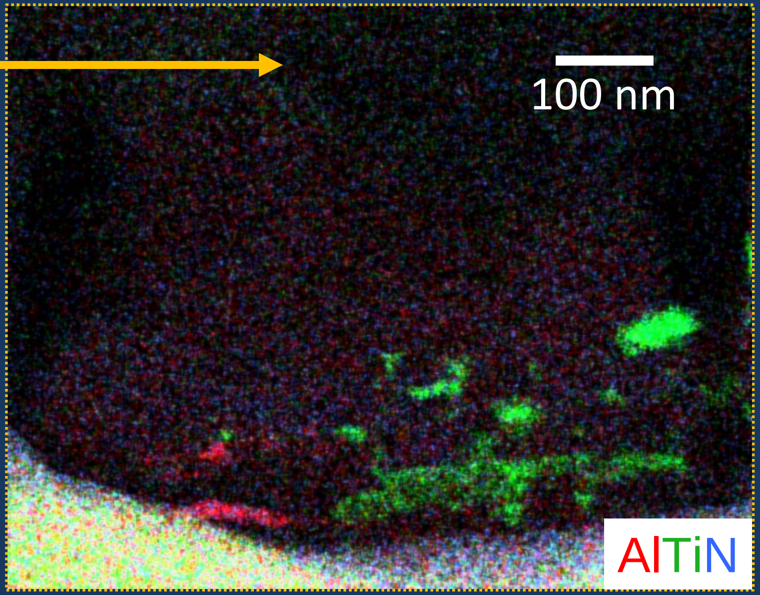
STEM-EDS of SiC from an AGB Star *Singerling et al., in review*



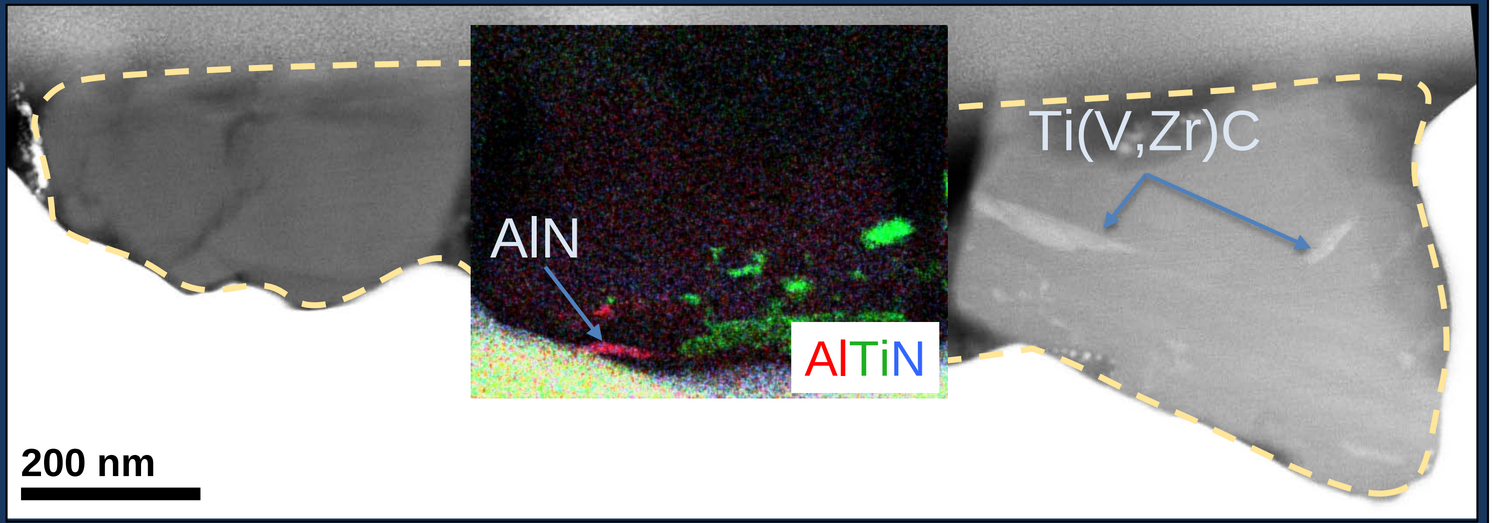
Internal structure reveals complex condensation history from > 4.5Gy ago

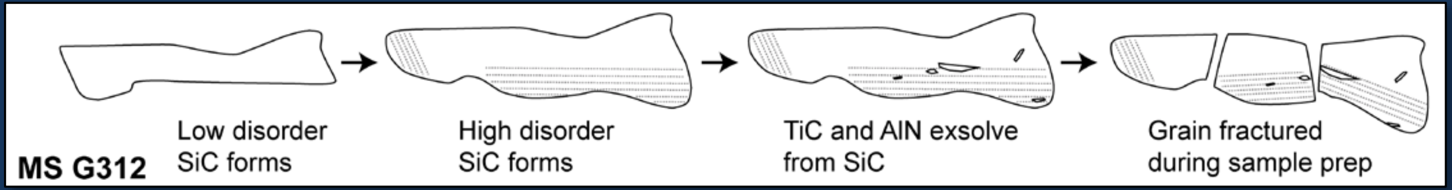


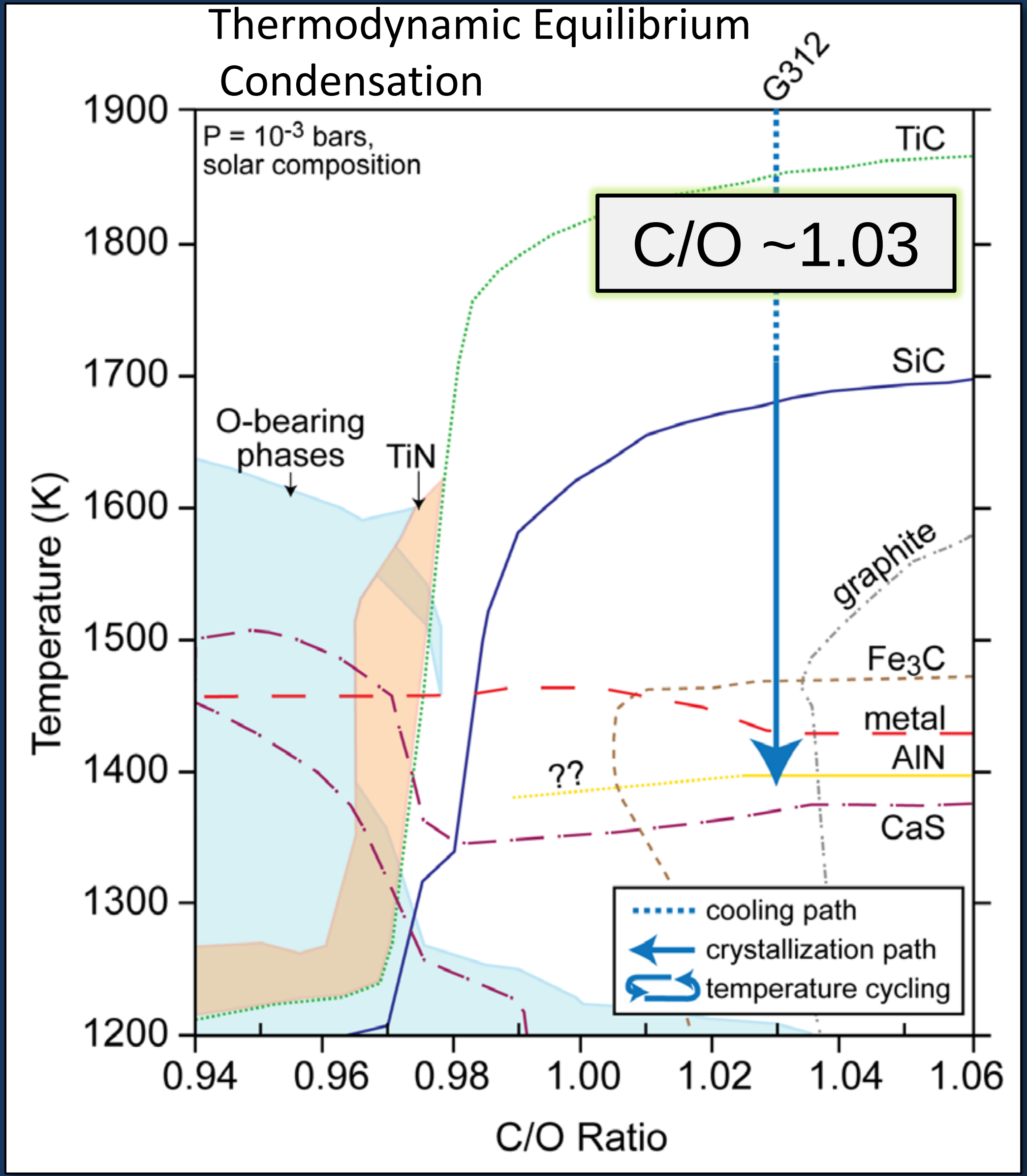
RGB Al-Ti-N Map



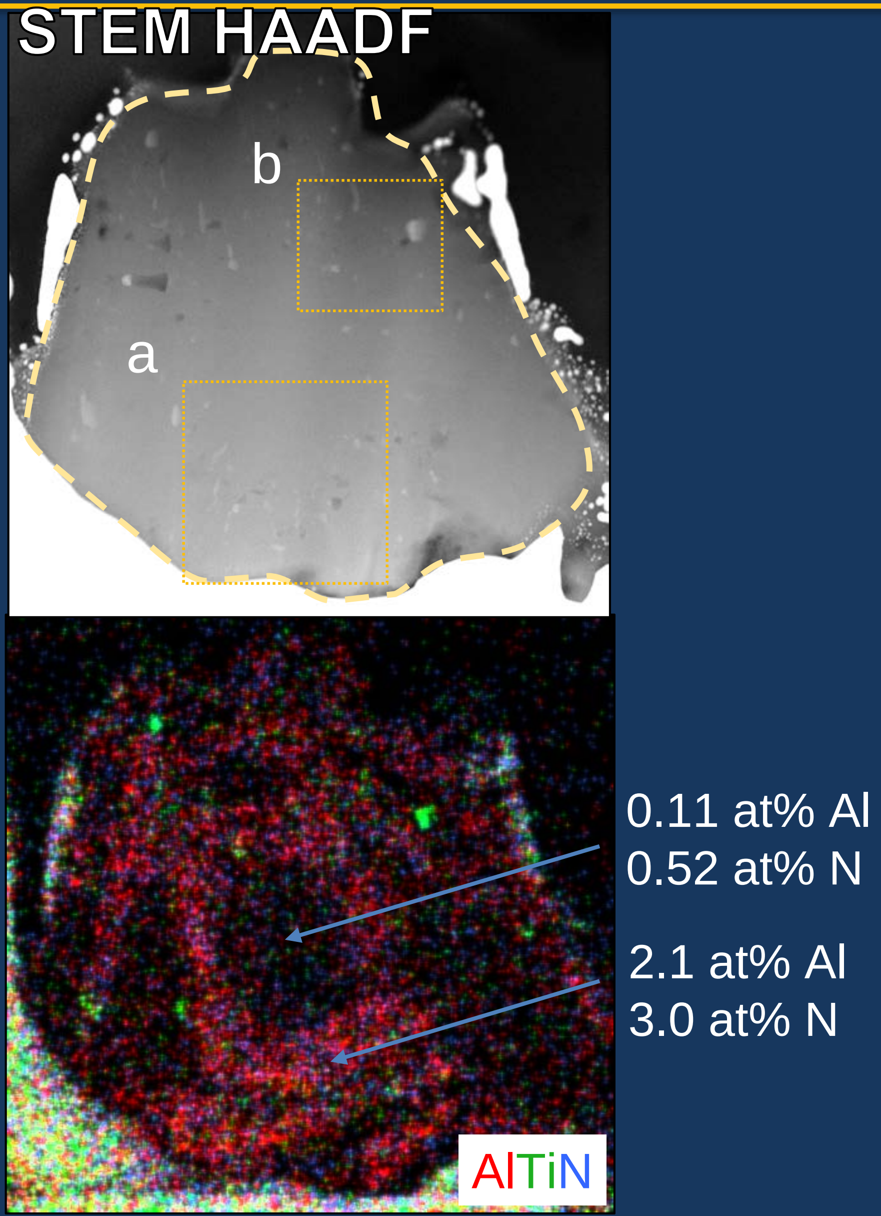
Implications for Circumstellar Conditions

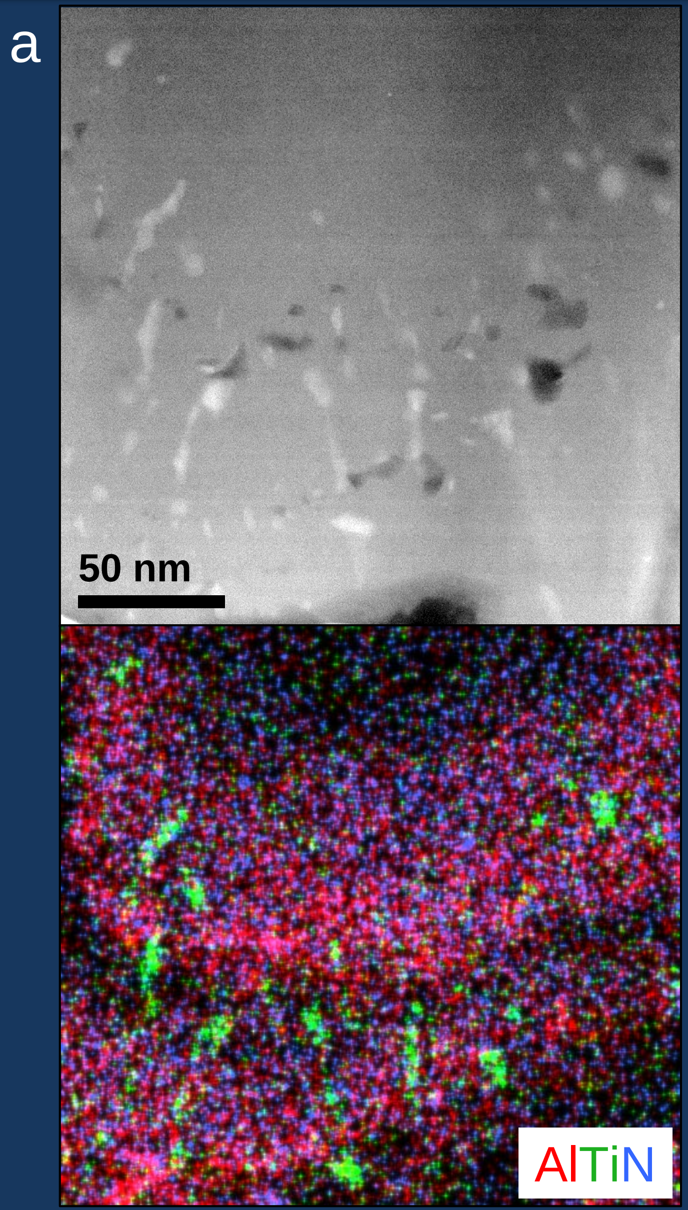


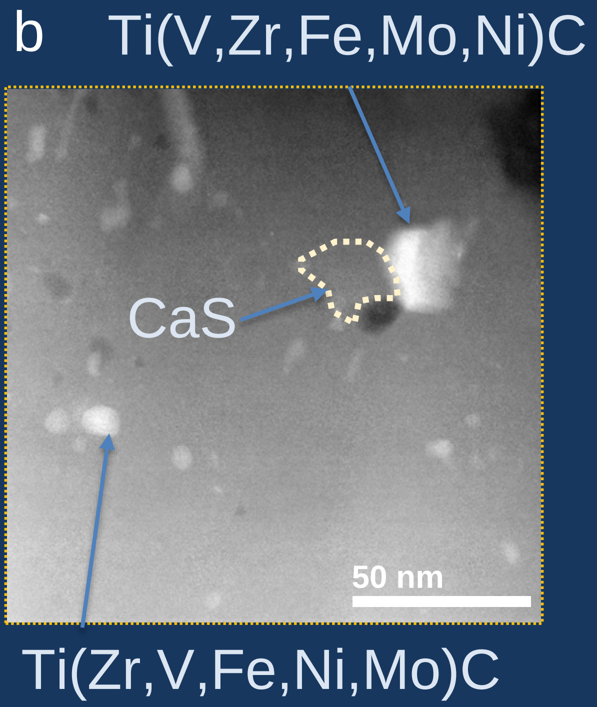




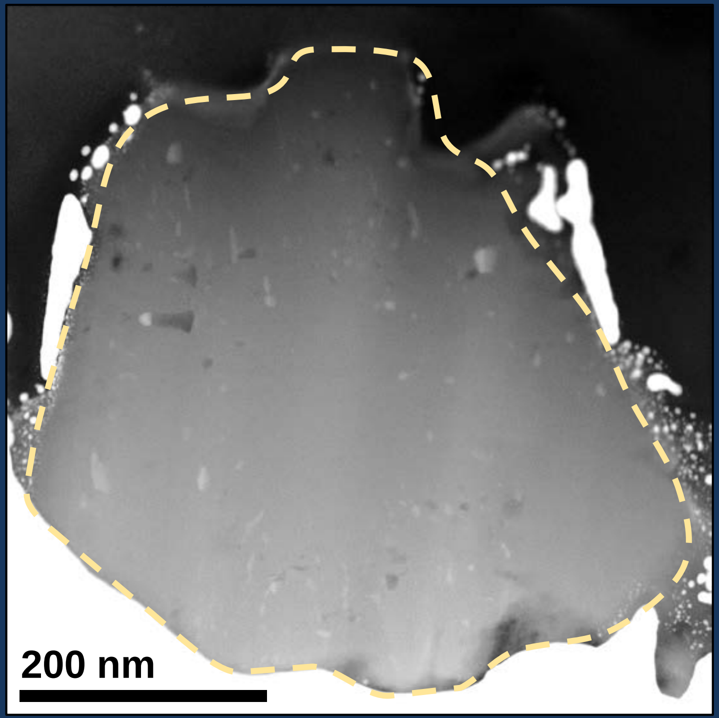
SiC Stardust from another AGB star

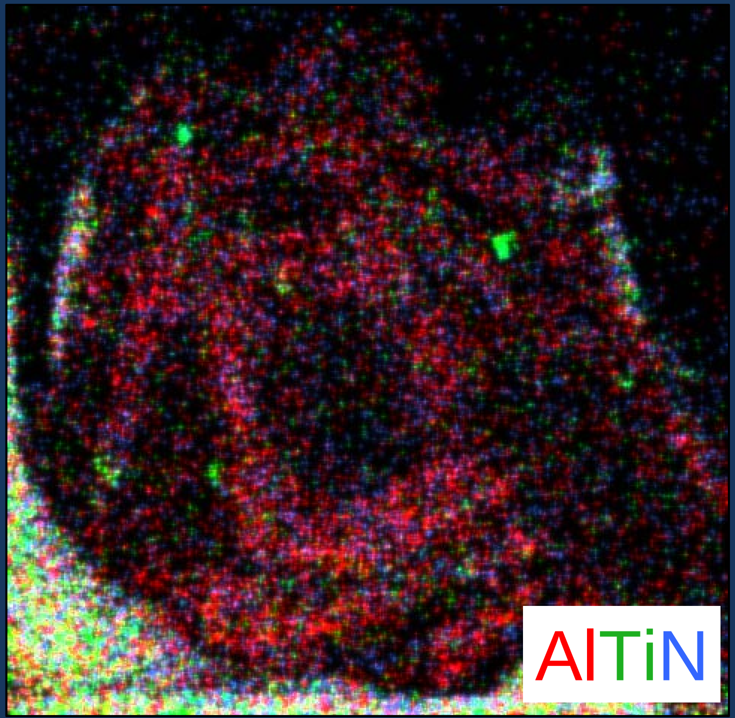


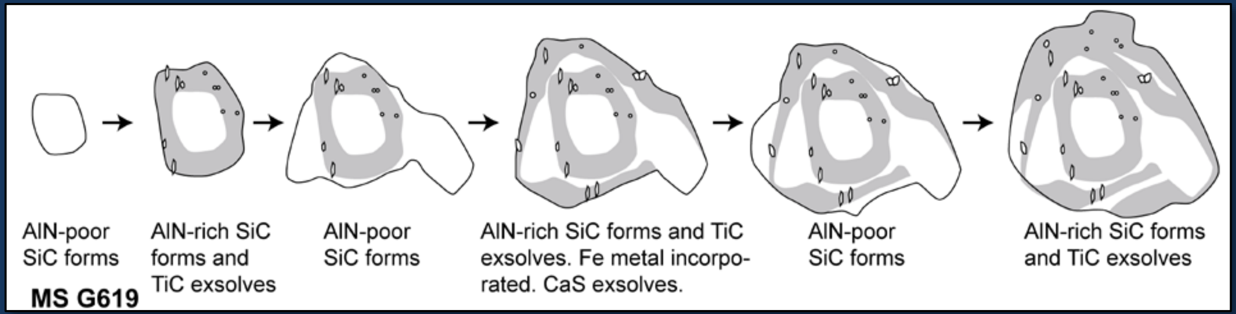


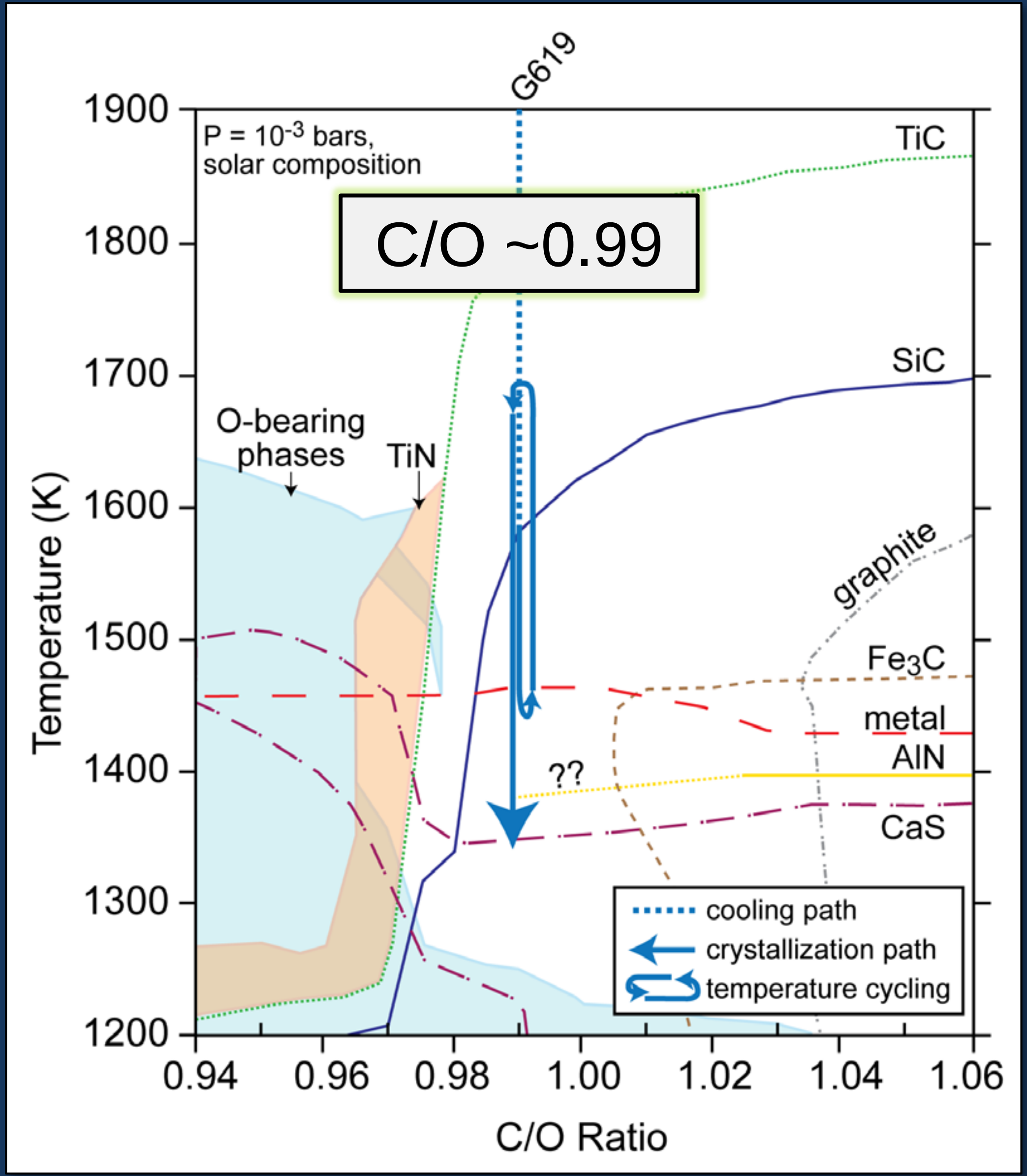


Different Circumstellar Conditions





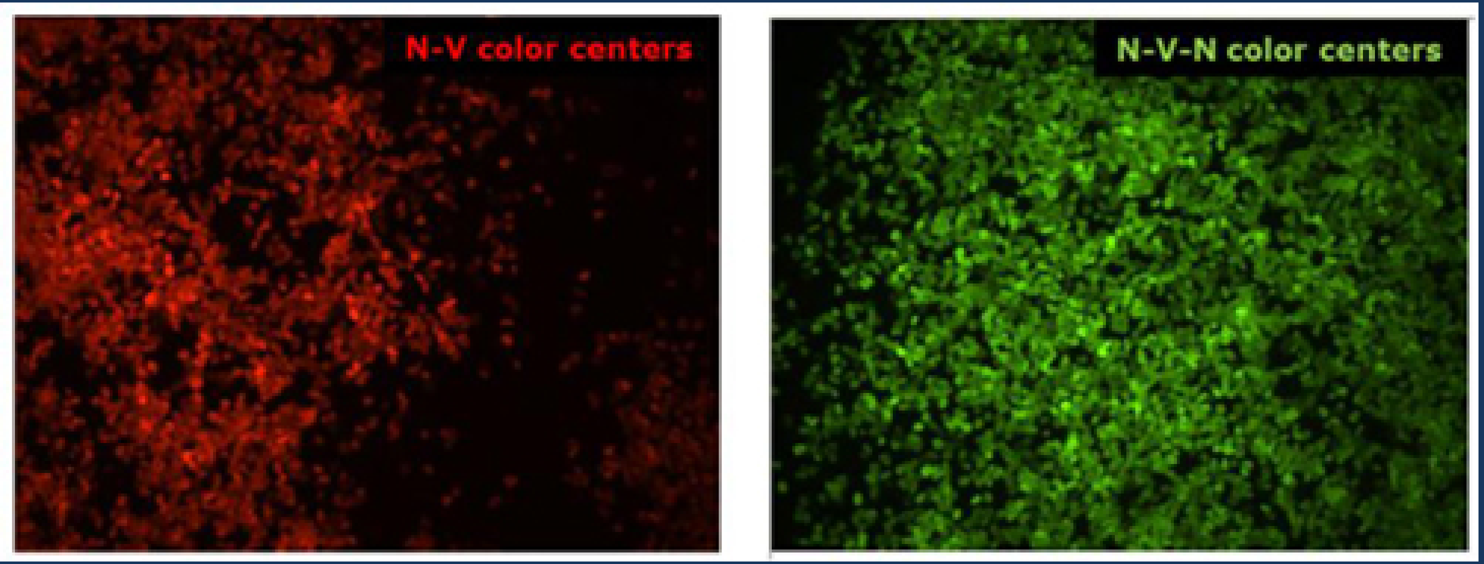




Nanodiamonds – from q-bit to stardust

- **Nanodiamonds are broadly scientifically important**
  - Common polishing compound, wear-resistant coatings
  - Biocompatible, functionalizable surface
  - Nitrogen and Si vacancy complexes for photoluminescence, spintronics / quantum computing

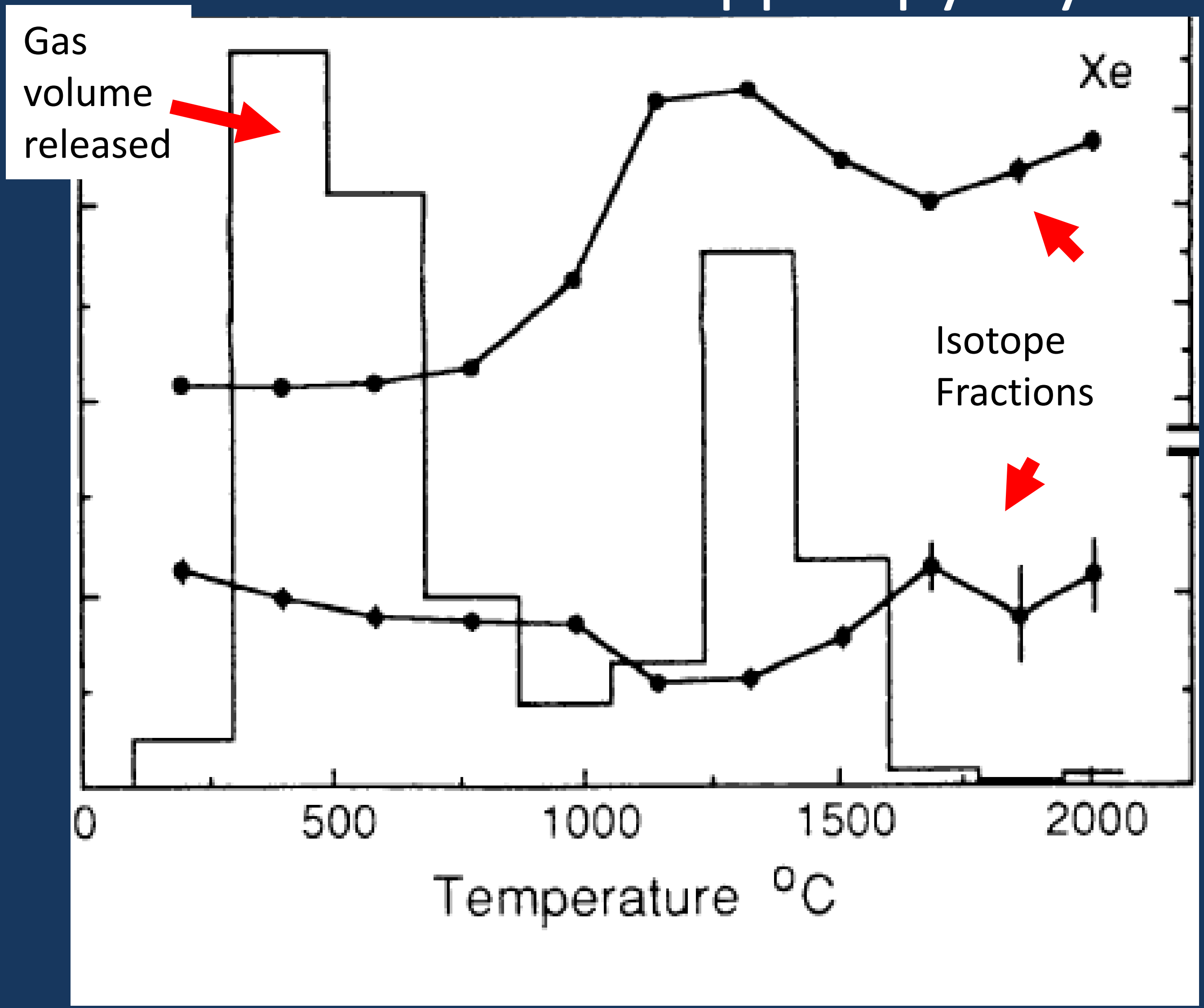
Commercial
irradiated nanodiamonds
Sigma-Aldrich



Cosmic Nanodiamonds

- Nanodiamond residue prepared by acid dissolution of meteorite
- First phase found in meteorites with an isotopic signature of extrasolar origin
  - Kr and Xe isotopes indicative of supernova origin, Lewis et al., *Nature* (1987)
- But only ~ 1 Xe per 10^5 nanodiamonds, and solar C and N isotopic composition
- Most nanodiamonds could have formed in Solar System

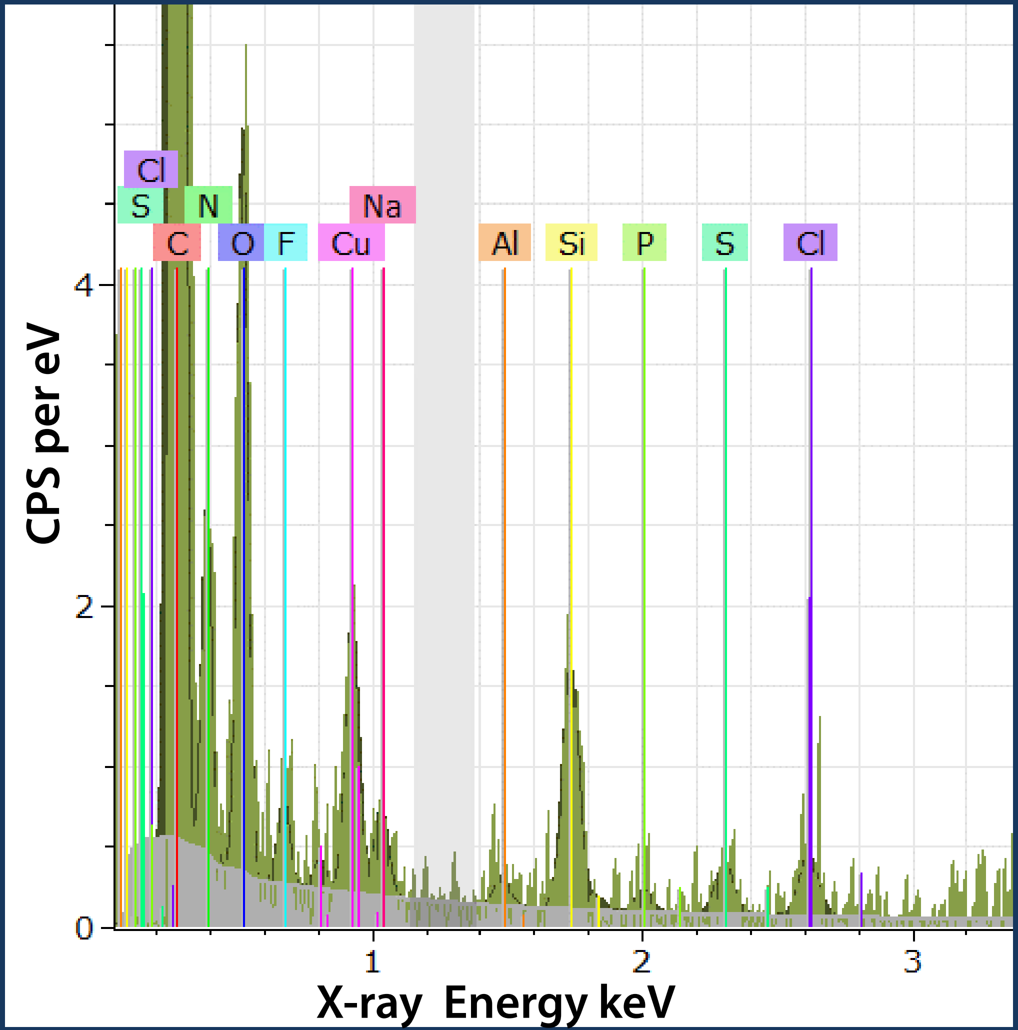
stepped pyrolysis

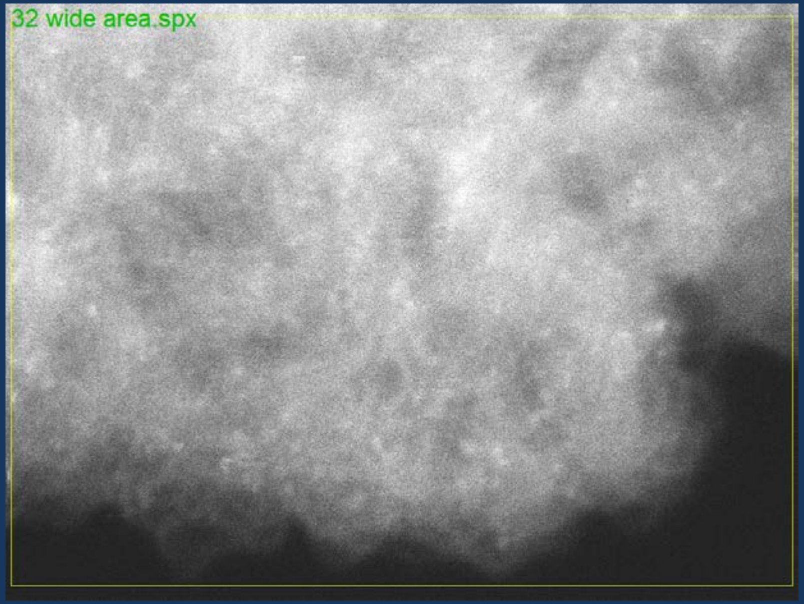


Huss and Lewis,MAPS (1994).

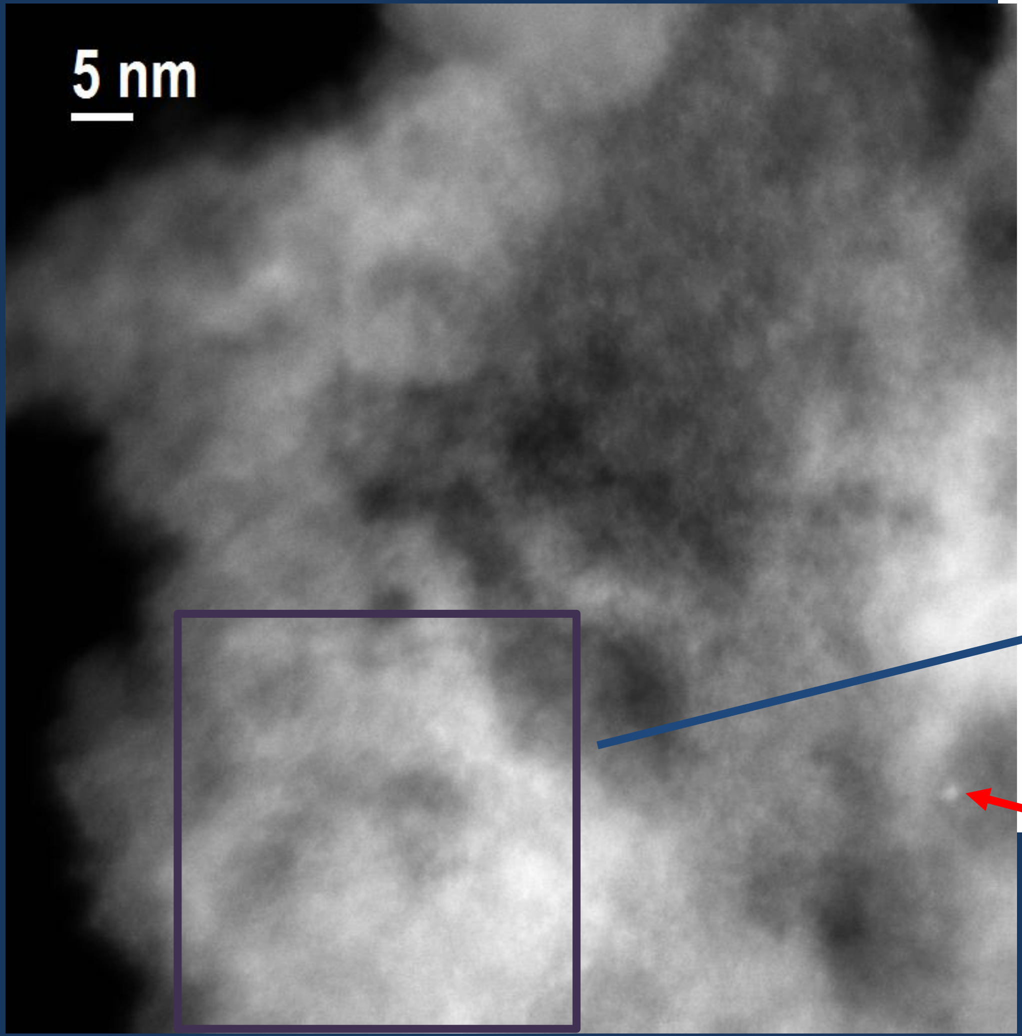
EDS "Average" Nanodiamond Composition (~ 9x12 nm^2)

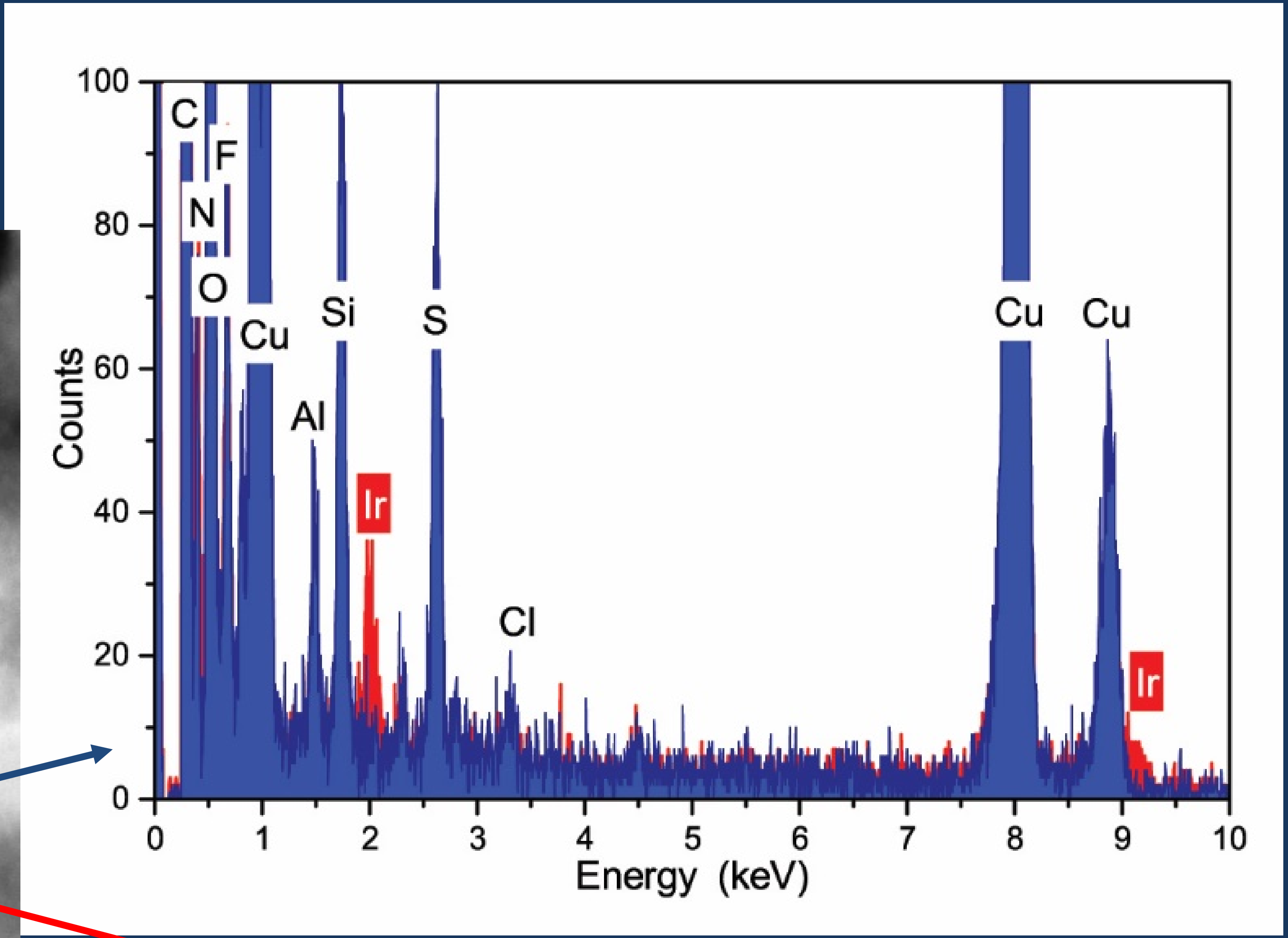
C (at.%)	N	O	F	Cu	Na	Al	Si	P	S	Cl
96.75	0.84	1.45	0.16	Sys.	0.11	0.05	0.4	0.04	0.08	0.11





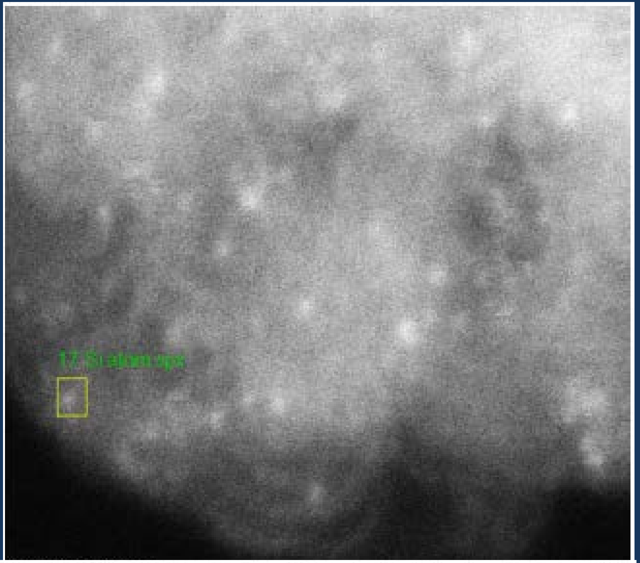
EDS of individual impurity particles

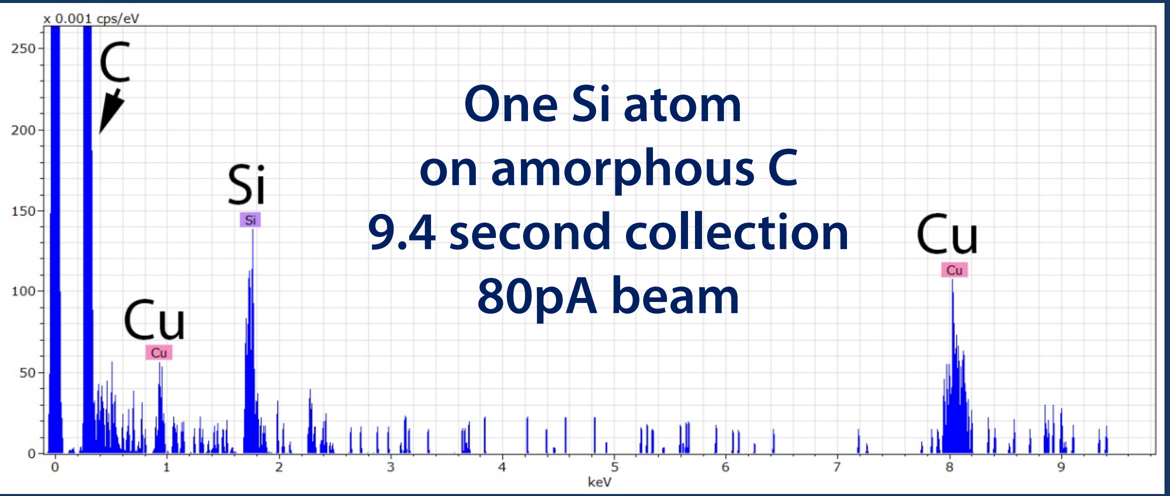


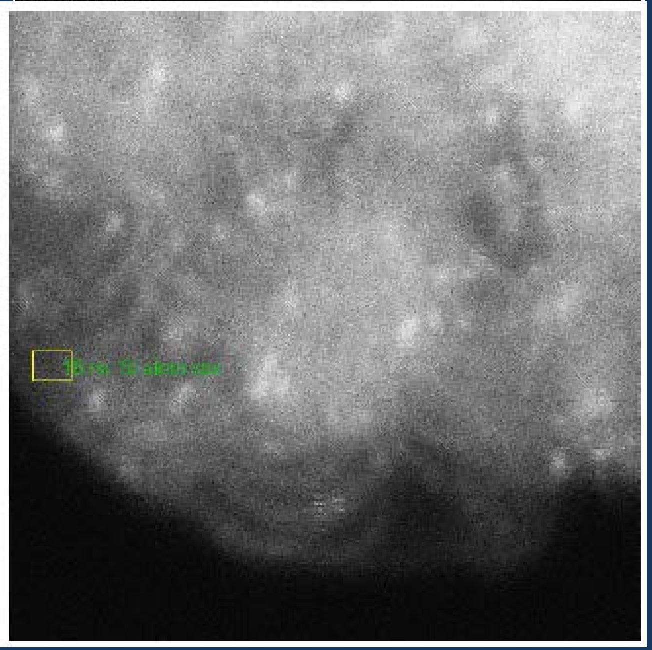


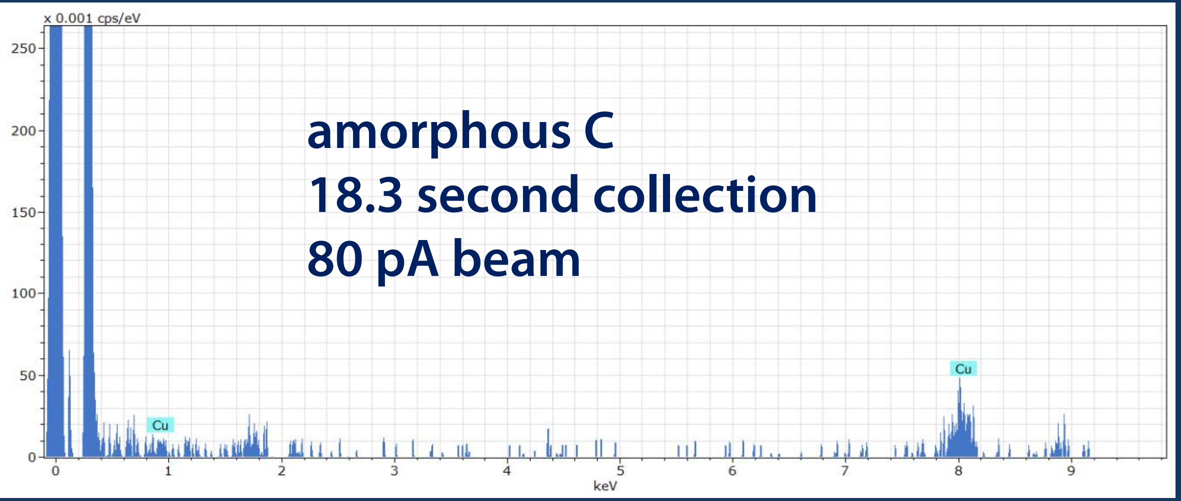
0.8 nm Ir particle

EDS on and off a Si atom





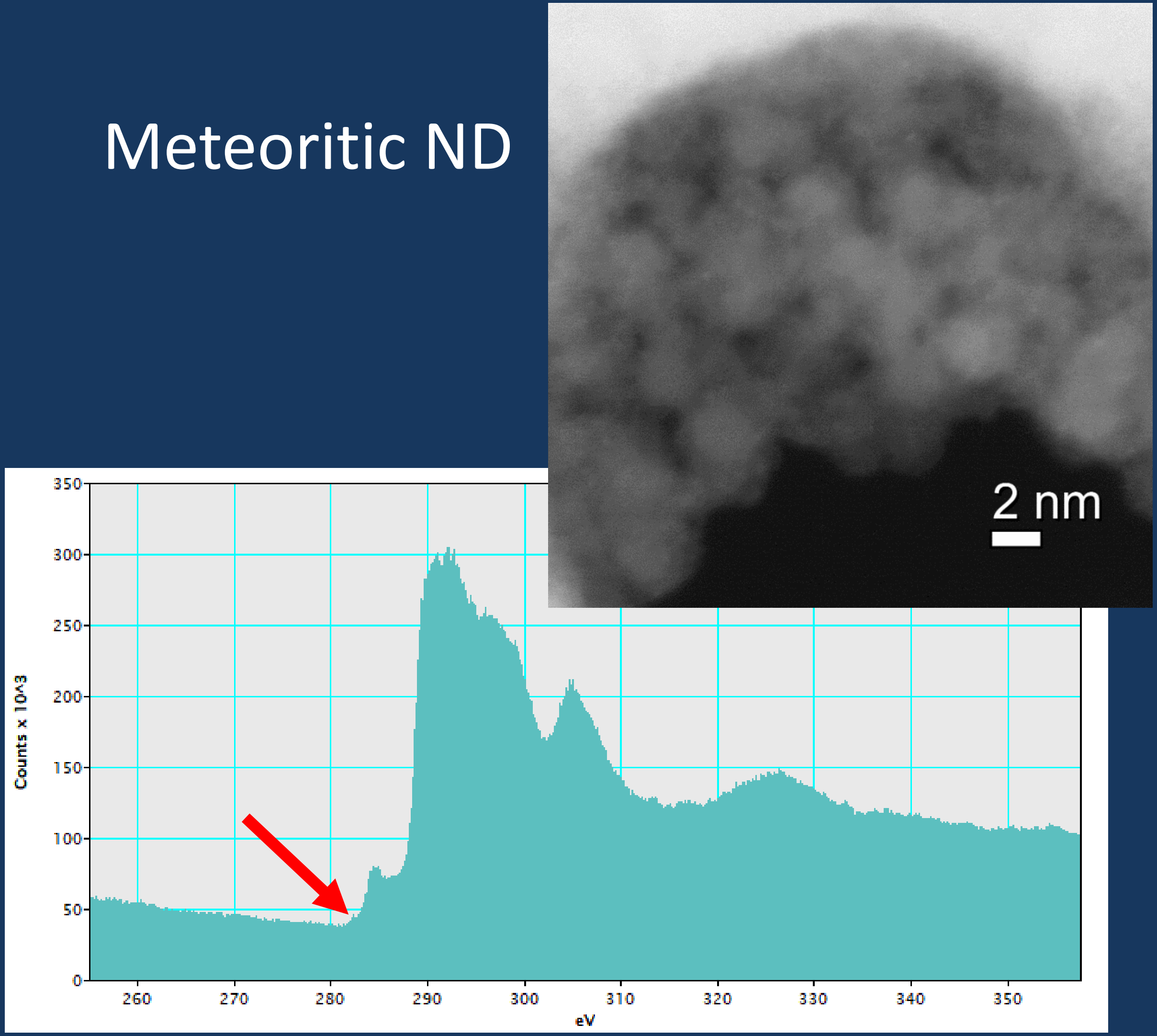




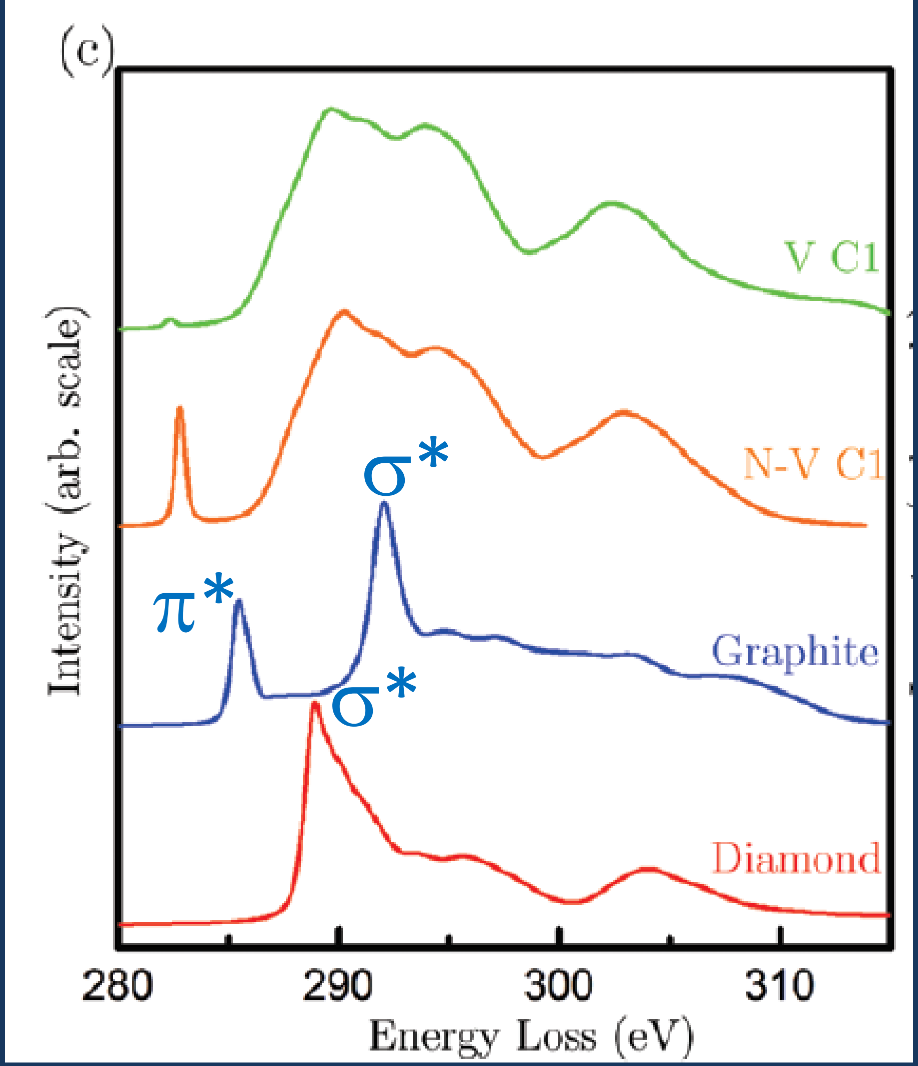
Stroud et al., Applied Phys. Lett. (2016)

Identification of Vacancy Centers in Diamond with EELS

Meteoritic ND

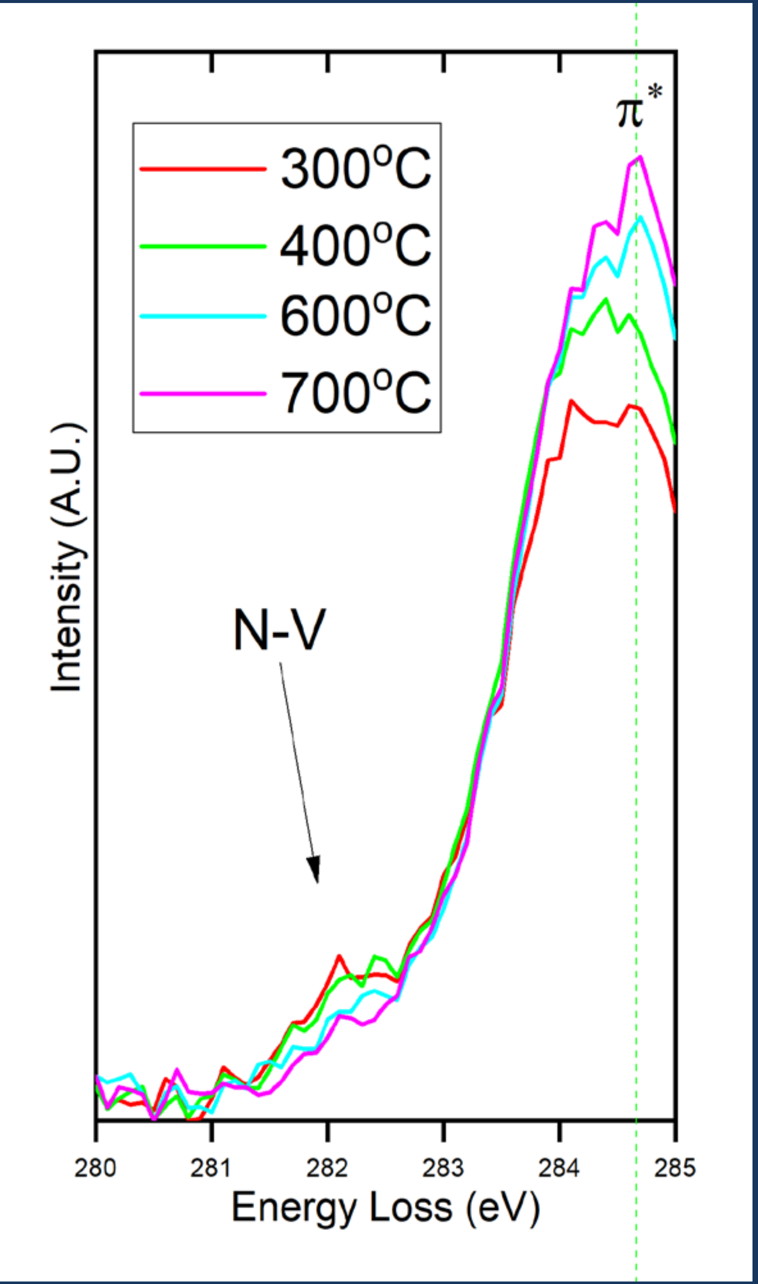


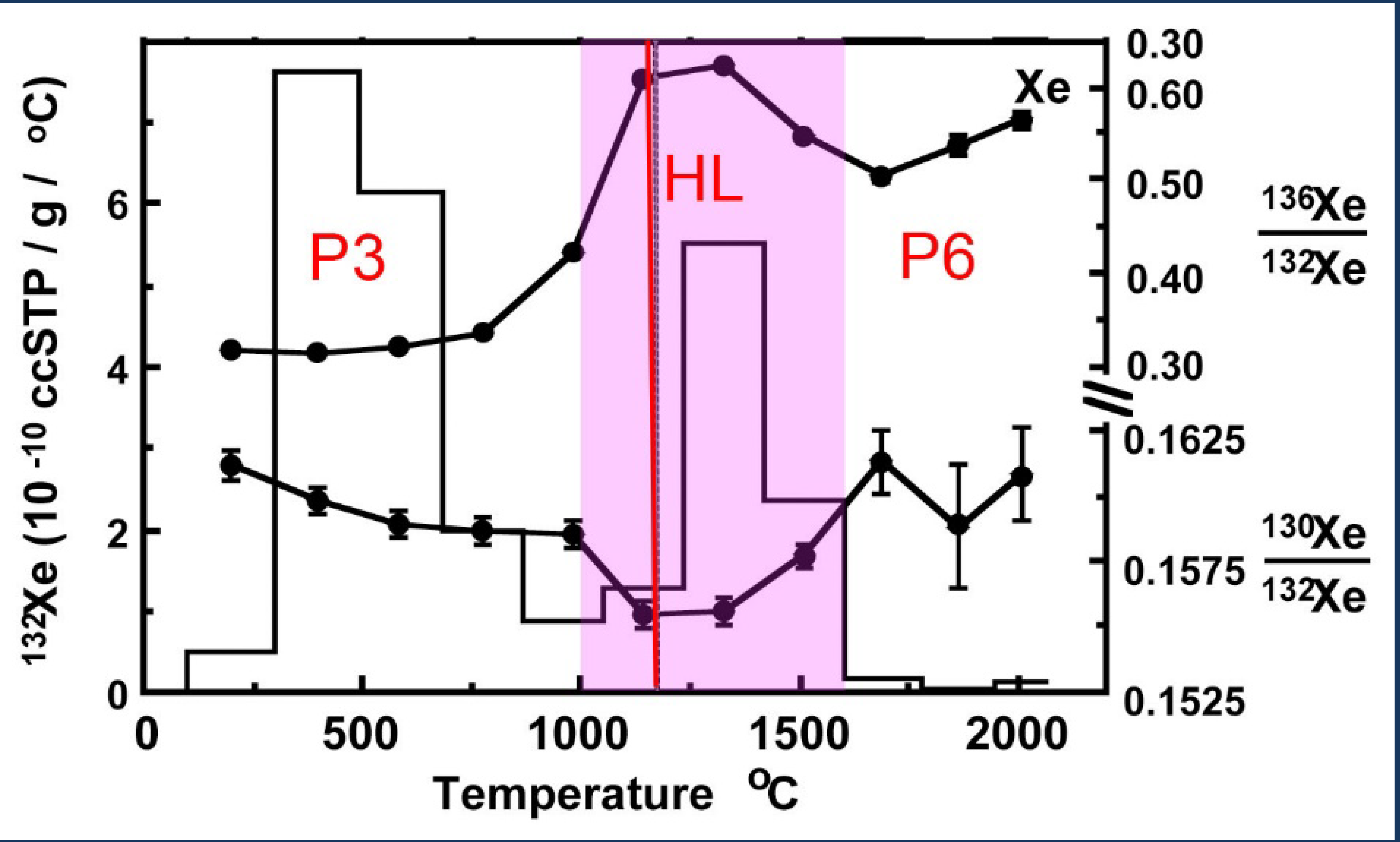
Calculated C-K edge spectra



Chang et al., Nanoscale 2016

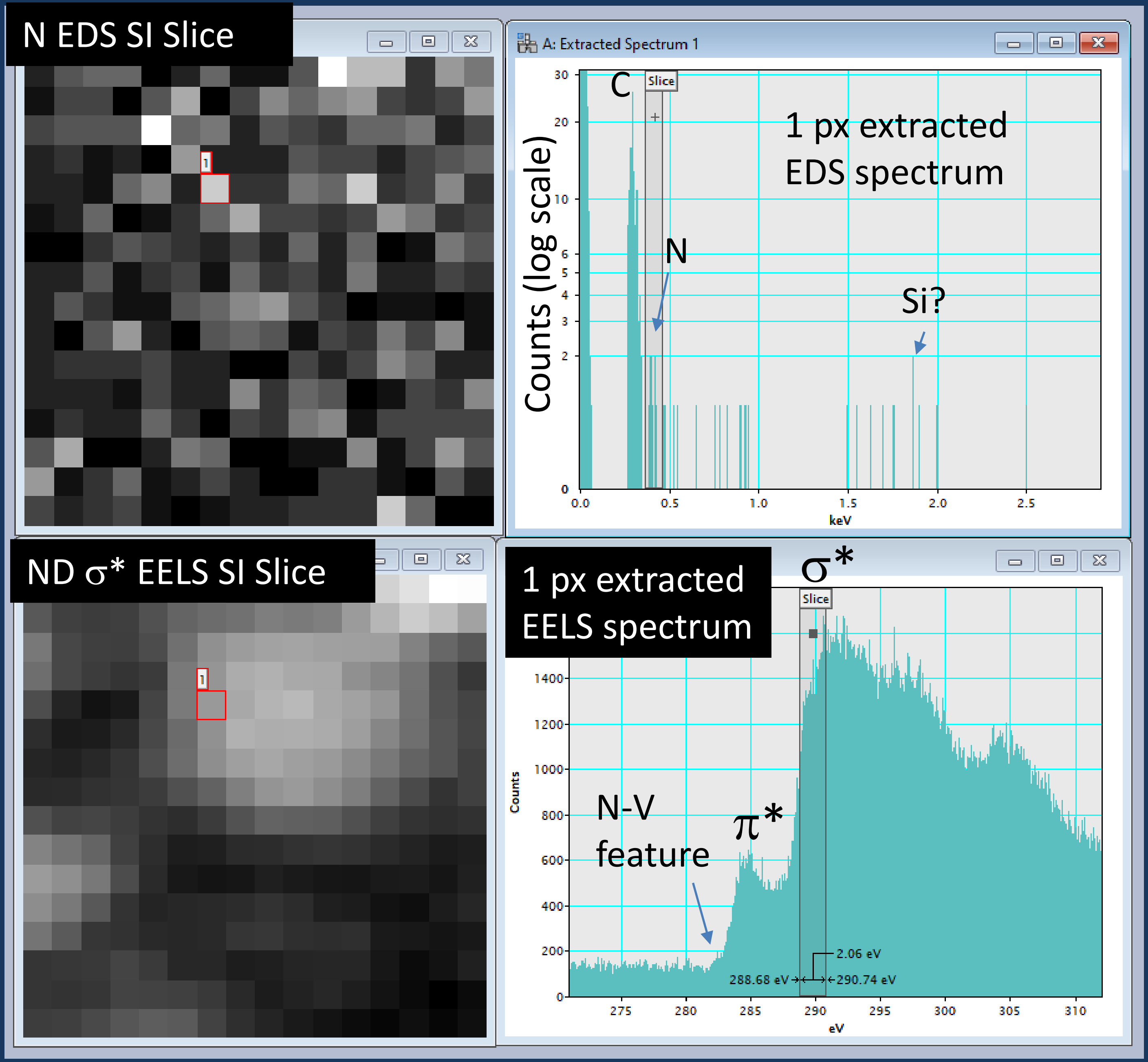
Hot stage STEM-EELS Comparison with Stepped Pyrolysis

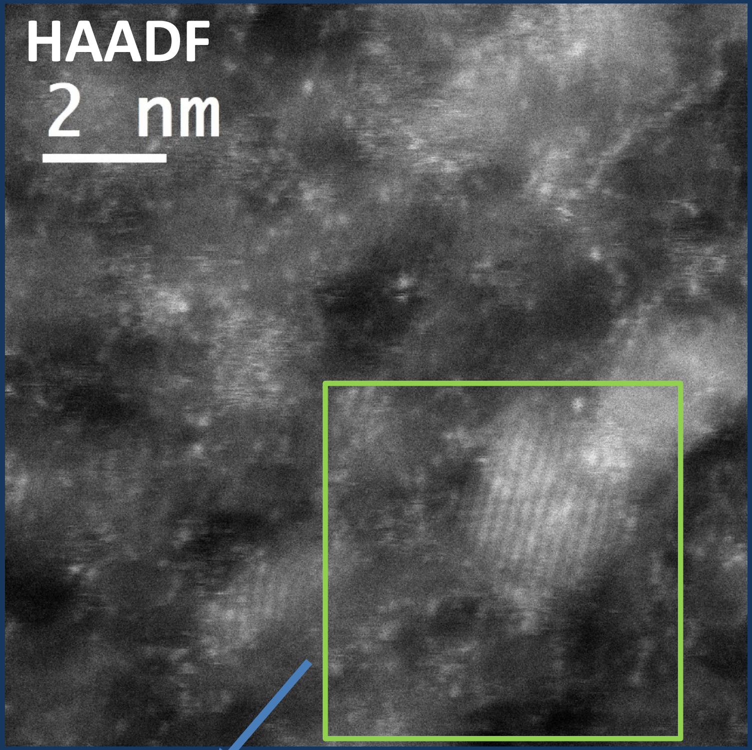


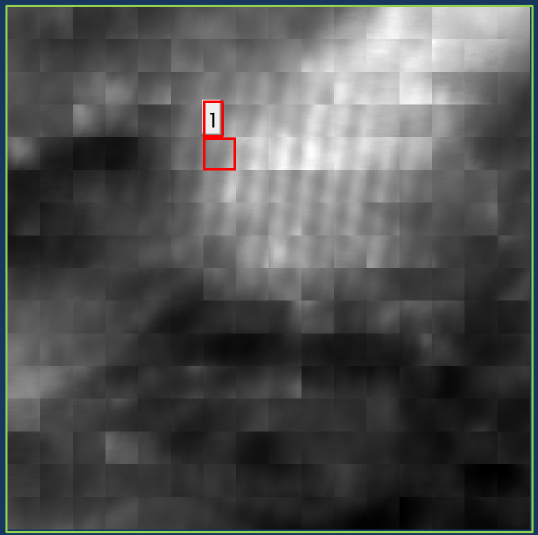


Nitrogen released from diamonds in "P3" temperature range, i.e., most likely solar system formed

Simultaneous EDS and EELS Spectrum Image of N-V Center in Nanodiamond

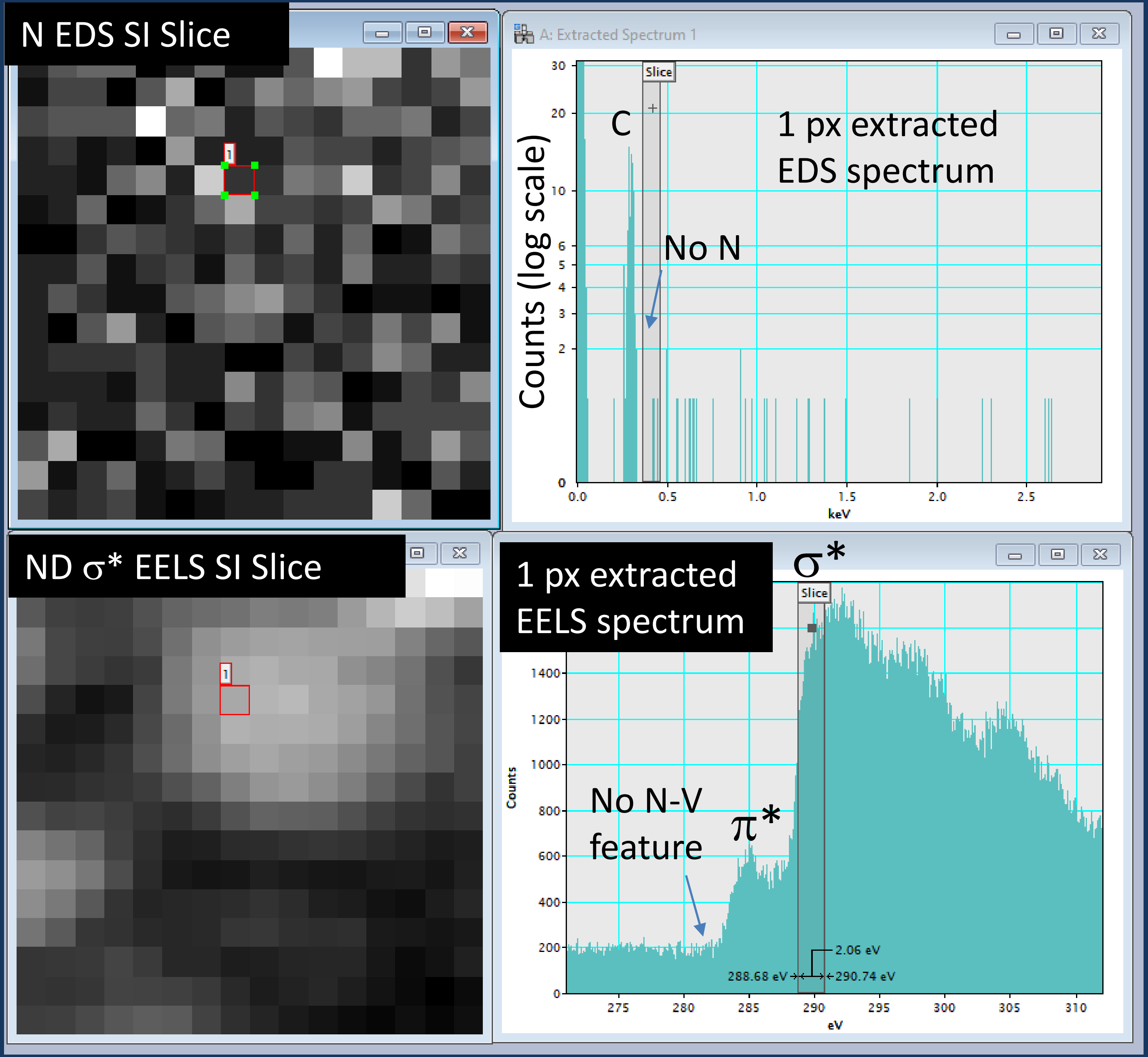


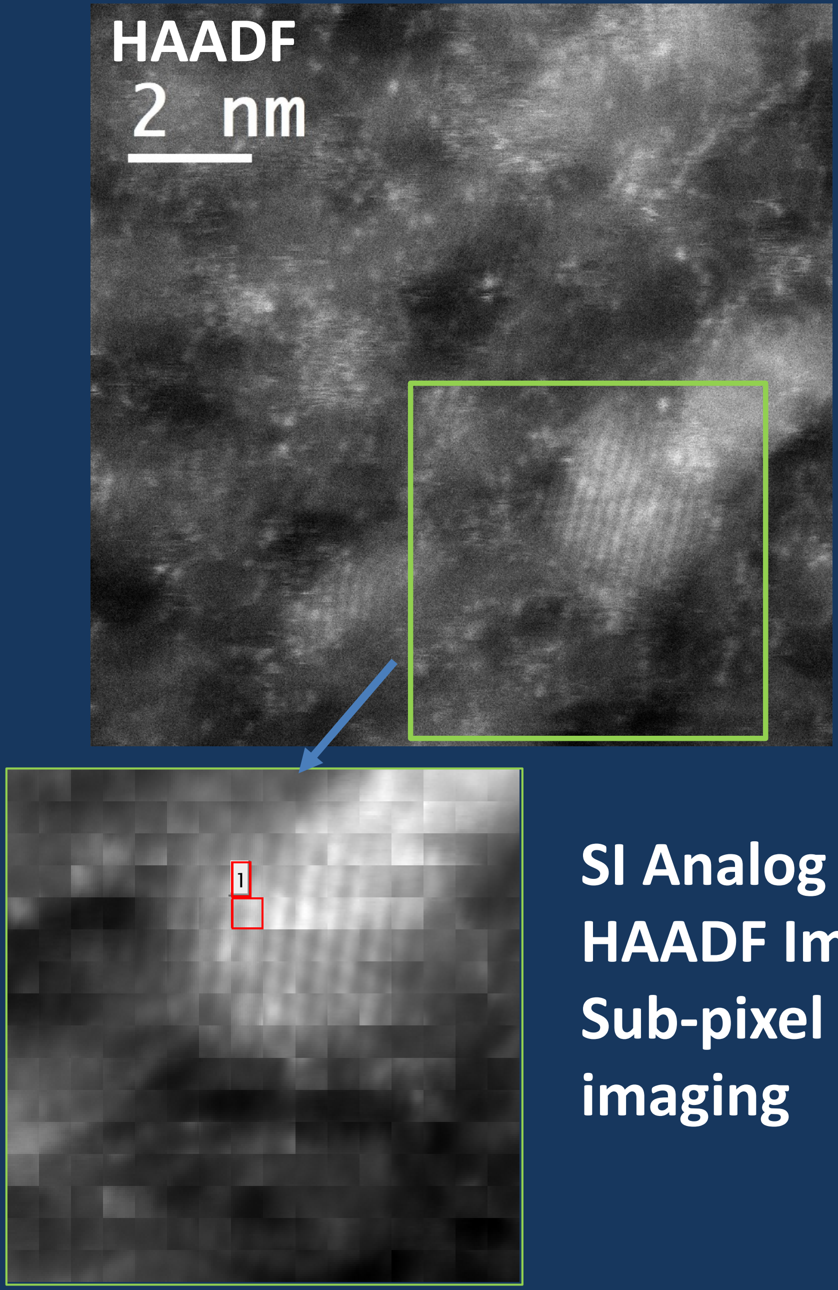




SI Analog HAADF Image Sub-pixel imaging

Simultaneous EDS and EELS Spectrum Image of ~~N-V Center in~~ Nanodiamond





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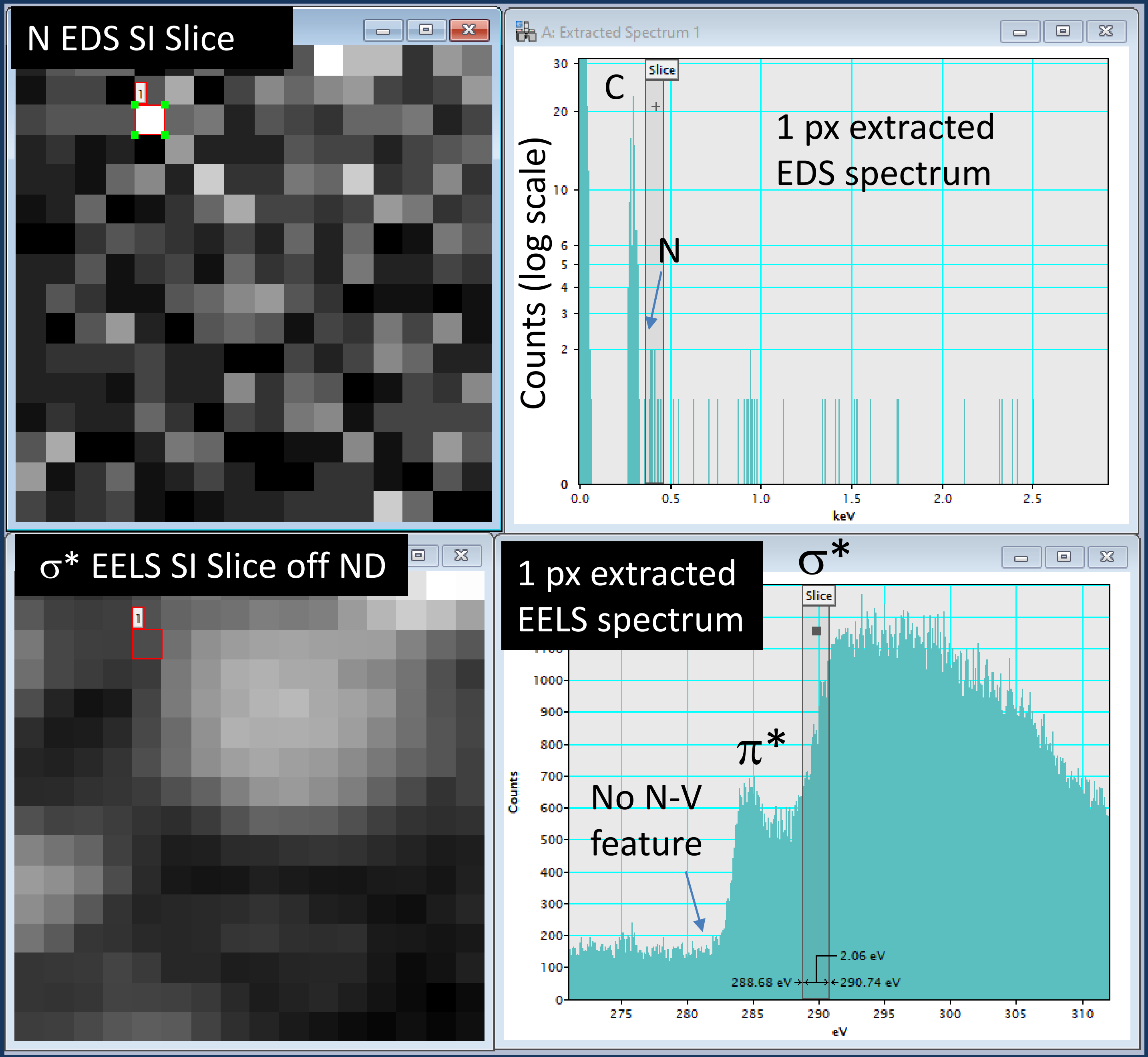
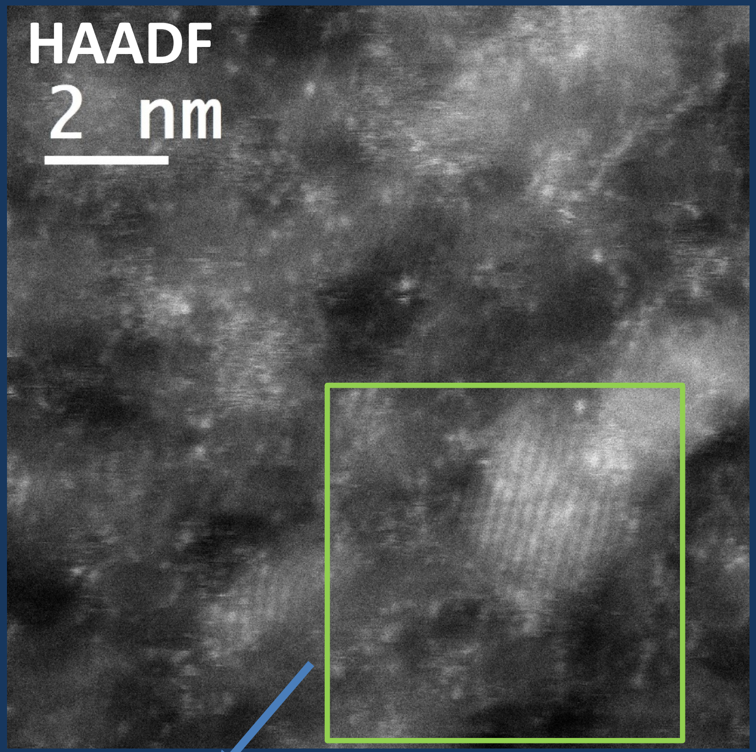
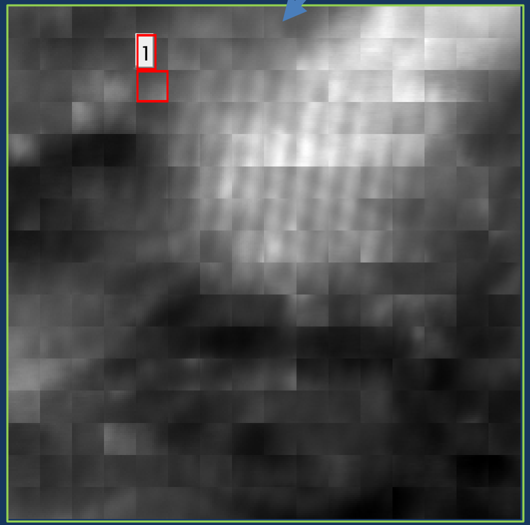
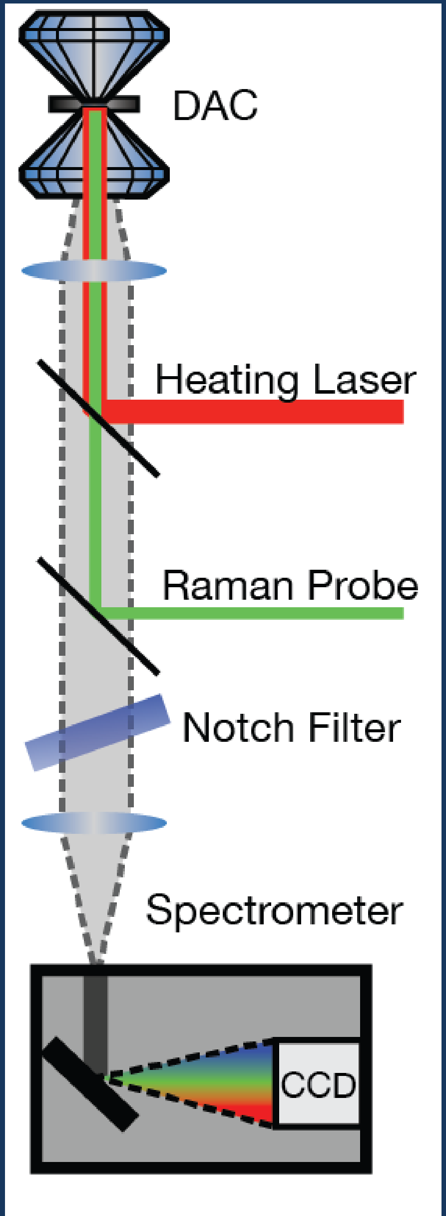


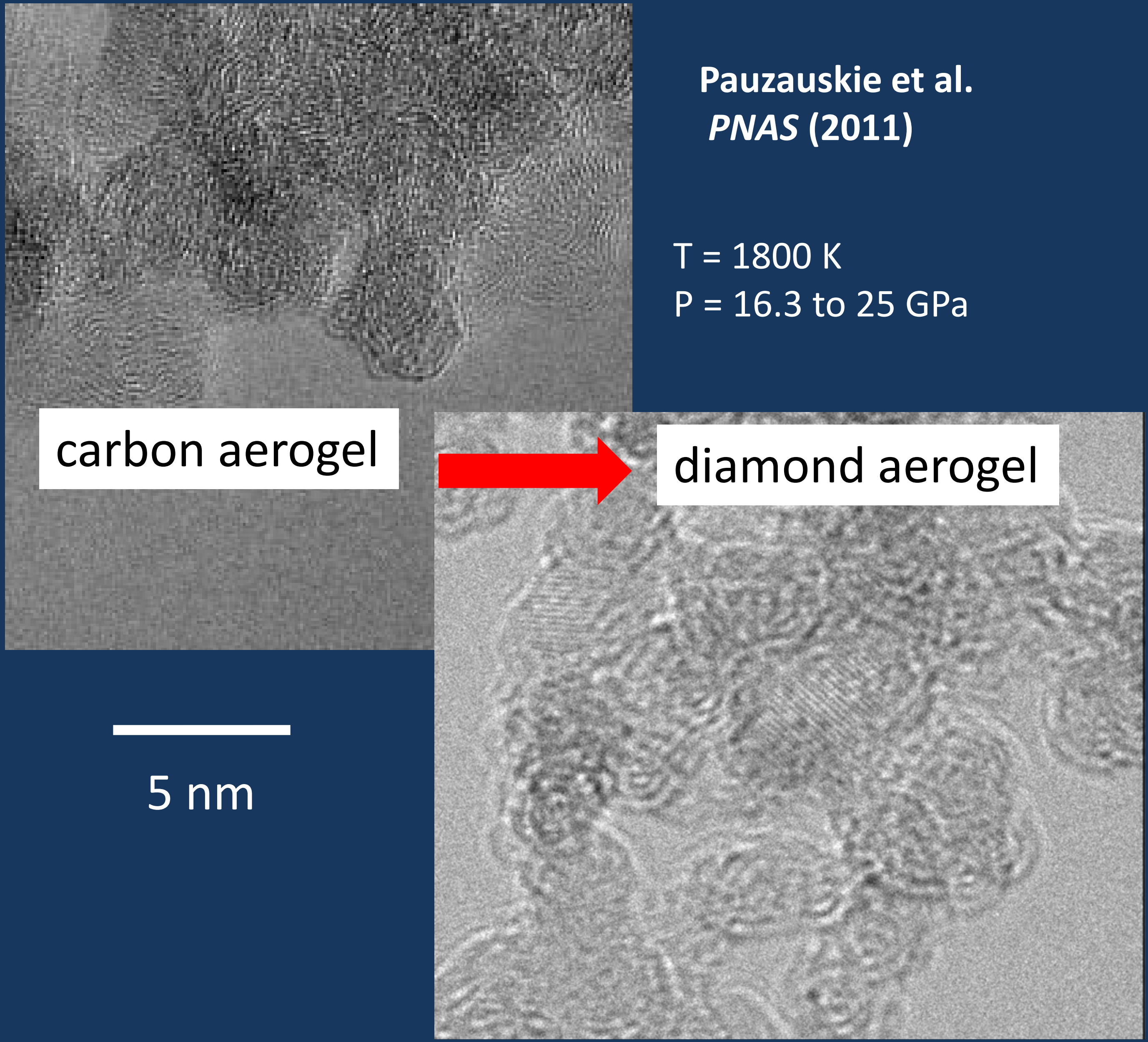




SI Analog HAADF Image Sub-pixel imaging

Diamond anvil cell synthesis of nanodiamond aerogel



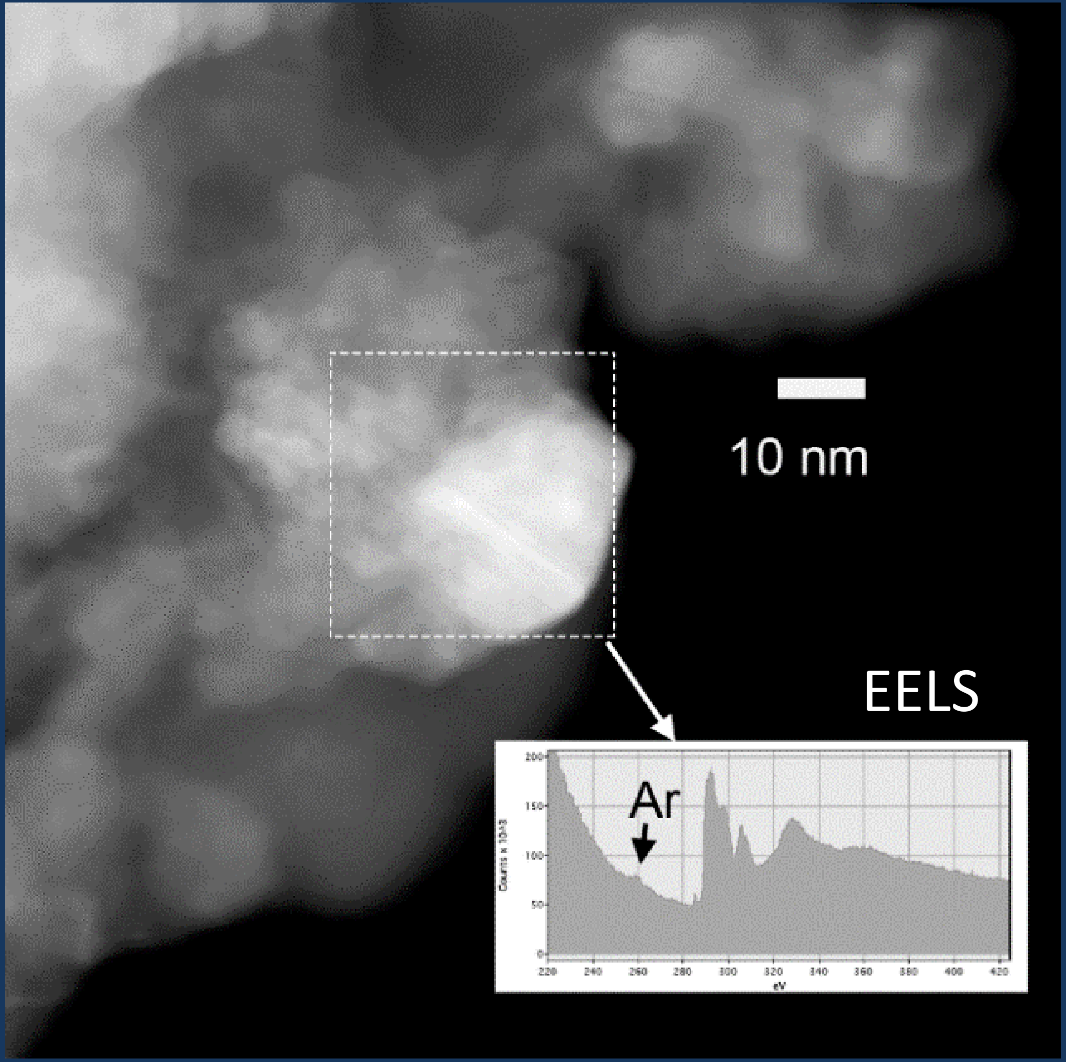


Pauzauskie et al.
PNAS (2011)

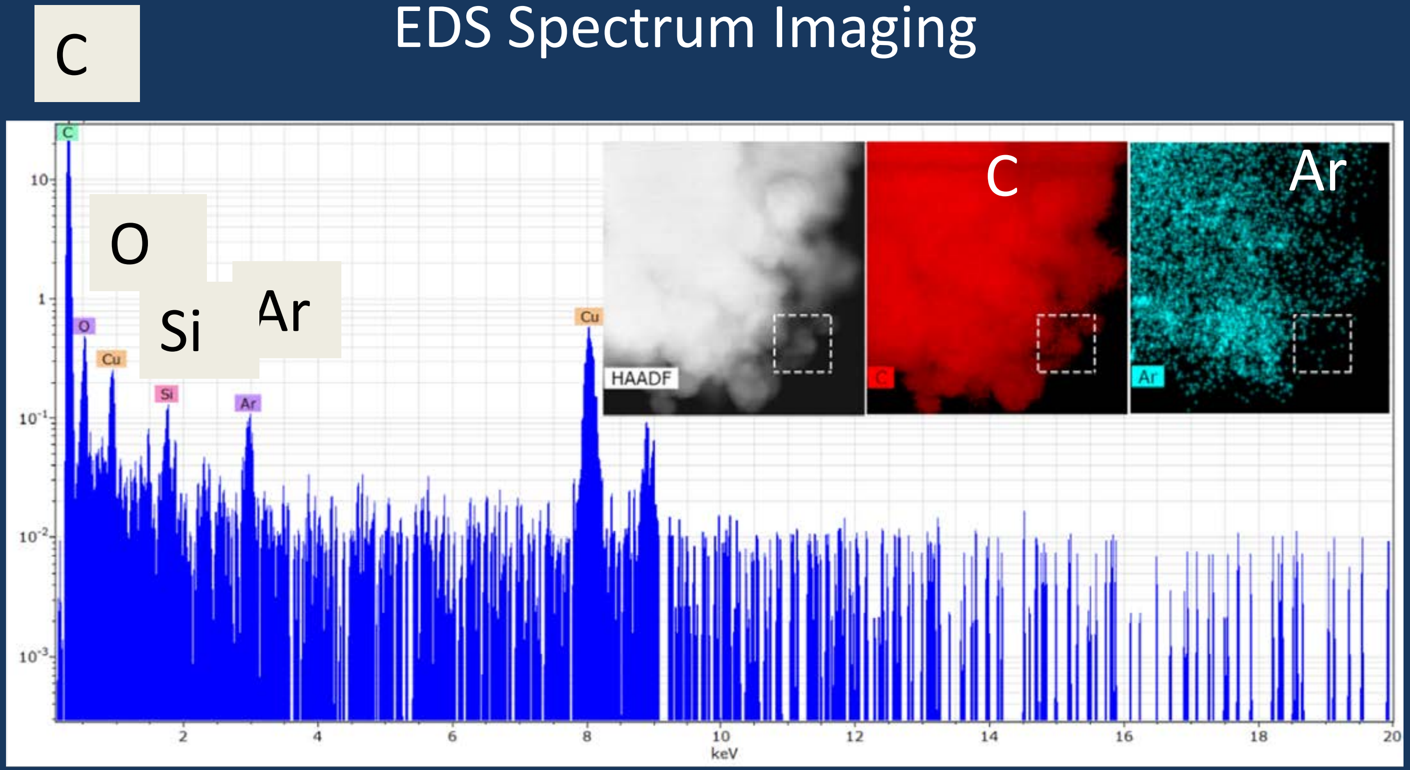
T = 1800 K
P = 16.3 to 25 GPa

STEM-EELS-EDXS of High Pressure / High Temperature Nanodiamond with incorporated Ar

HAADF

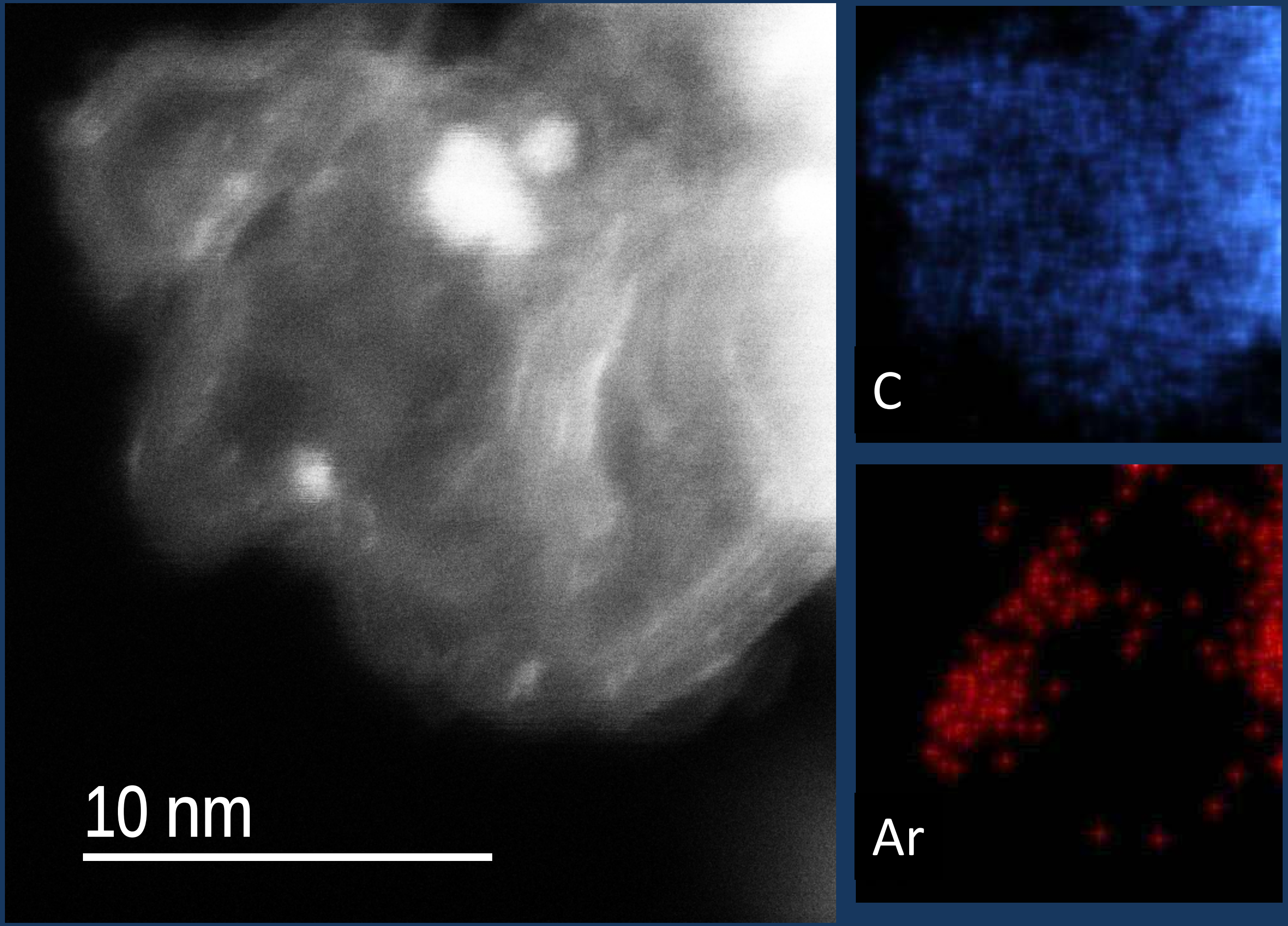


EDS Spectrum Imaging



M. Crane et al., *Science Advances (2019)*,
US Patent App. 16/297,338.

EDS Mapping of HPHT Graphitic Onion C with Incorporated Ar



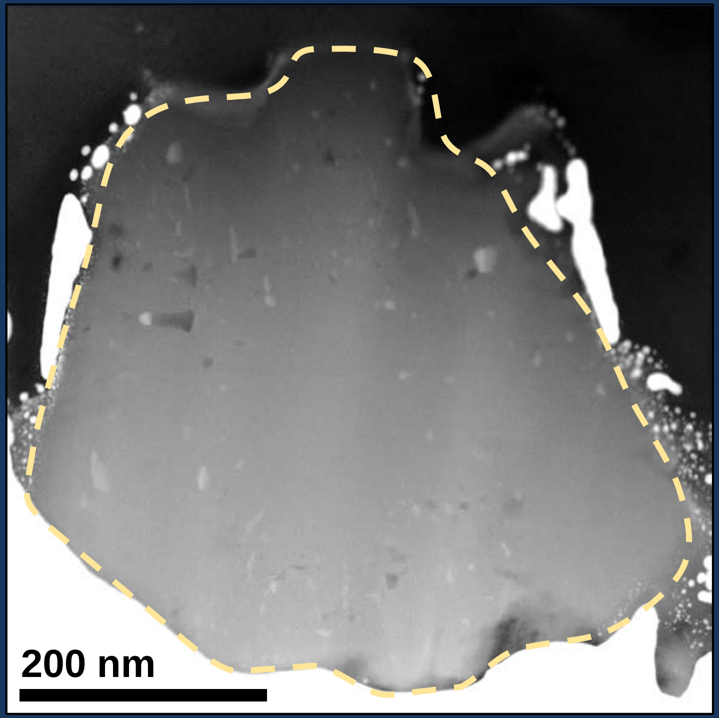
Some areas with up to 40% Ar observed

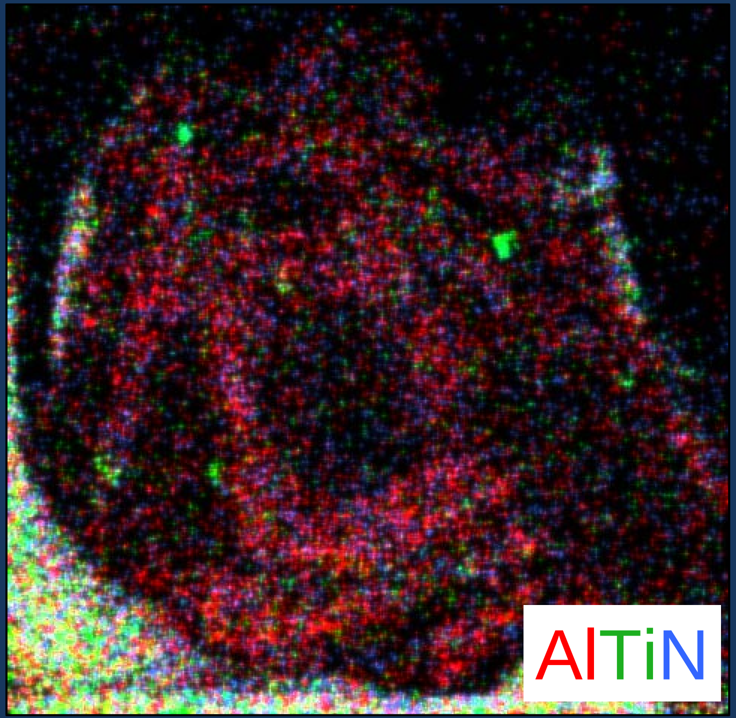
Potential mechanism for archival gas storage over billions of years

Summary

- Physics and chemistry of materials are the same in space as on Earth
- Nanomaterials are as old as the stars
- Atomic-scale structure key to materials growth, history, & properties

> 4.5 billion year old SiC





Q&A

Are there any questions?

Please type in the questions you might have

in the Q&A box and press *Send*.

Innovation with Integrity