

Nanomaterials Characterization For Industry

Supapan Seraphin

Department of Materials Science & Engineering
University of Arizona, Tucson, Arizona 85721 U.S.A.

Thailand Metal and Materials Technology Center (MTEC) and
Nanotechnology Center (NANOTEC)
Thailand National Science and Technology Development Agency (NSTDA)

ssupapan7@gmail.com



Supapan Seraphin

University of Arizona, Tucson, Arizona

FROM 1990 - 2016

PROFESSOR in three colleges:

- Dept. Materials Science & Engineering, **College of Engineering**
- Dept. Agriculture Biosystems Engineering, **College of Agriculture and Life Sciences &**
- **College of Optical Sciences**

DIRECTOR of University Spectroscopy and Imaging Facilities 1990 – 2014



USIF
Envision Tomorrow



Director of Electron Microscope Facilities

1990-2007

TEM at AML six miles east of campus





UNIVERSITY SPECTROSCOPY & IMAGING FACILITIES

2007 – moved to Marley building



High-resolution SEM S4500



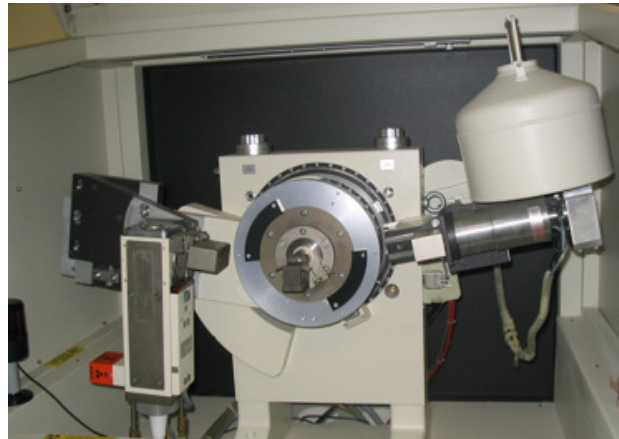
High-resolution SEM S4800



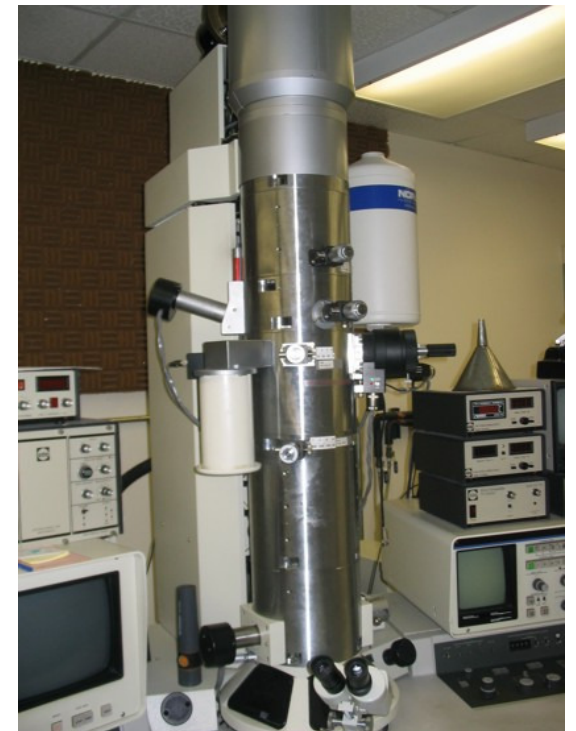
Variable pressure SEM S3400



Bio TEM J-100cx



X-ray diffraction



High-resolution TEM H-8100[®]

Why microscopy is exciting?

- “Use a picture. It’s worth a thousand words.” advice appeared in 1911 newspaper.

"Speakers Give Sound Advice".
Syracuse Post Standard (page 18). March 28, 1911

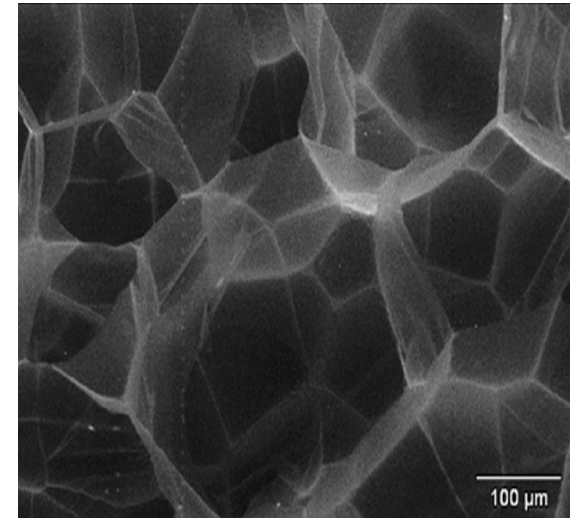
- Most of us are visual learners.
- We are drawn to an image because basically ***Seeing is believing.***
- ***Seeing may be believing but understanding is still science.***

C. Kisielowski, National Center for Electron

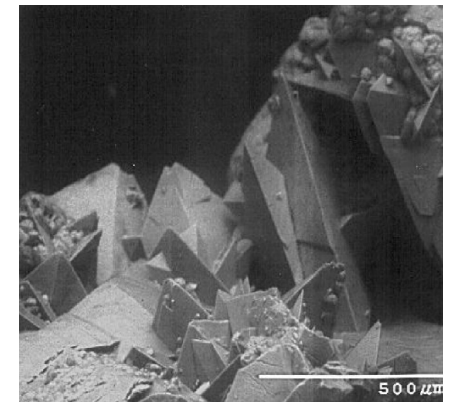
Microscopy

Lawrence Berkeley National Laboratory

azengineering



SEM image of Styrofoam

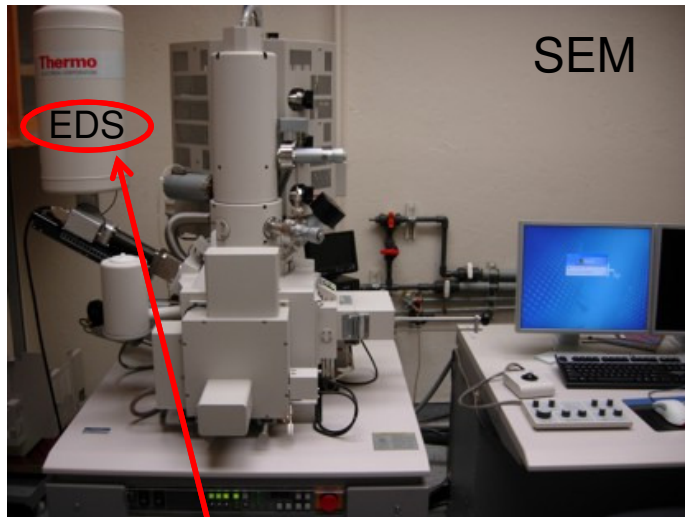


SEM image of kidney stone

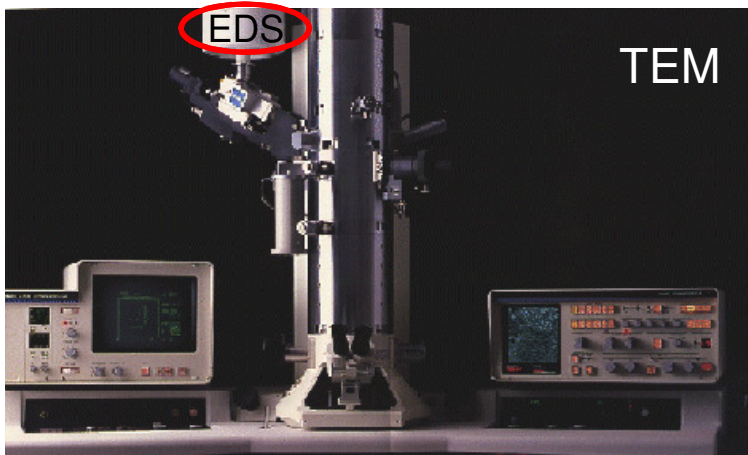


Why do we need both SEM & TEM?

SEM vs TEM



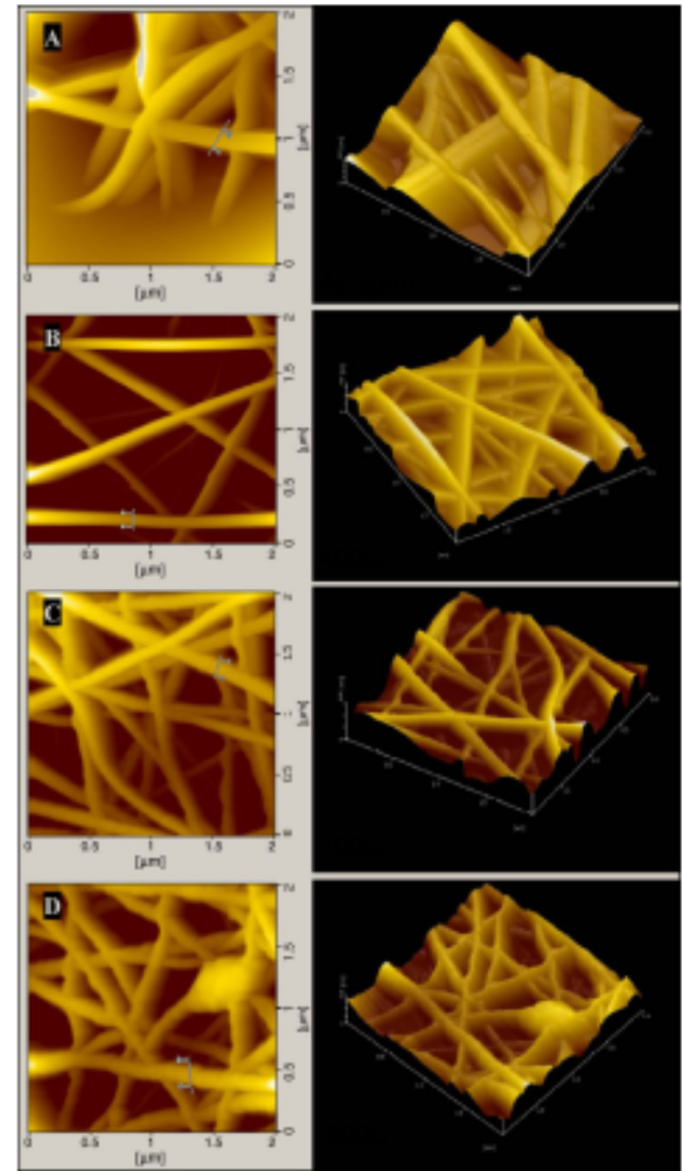
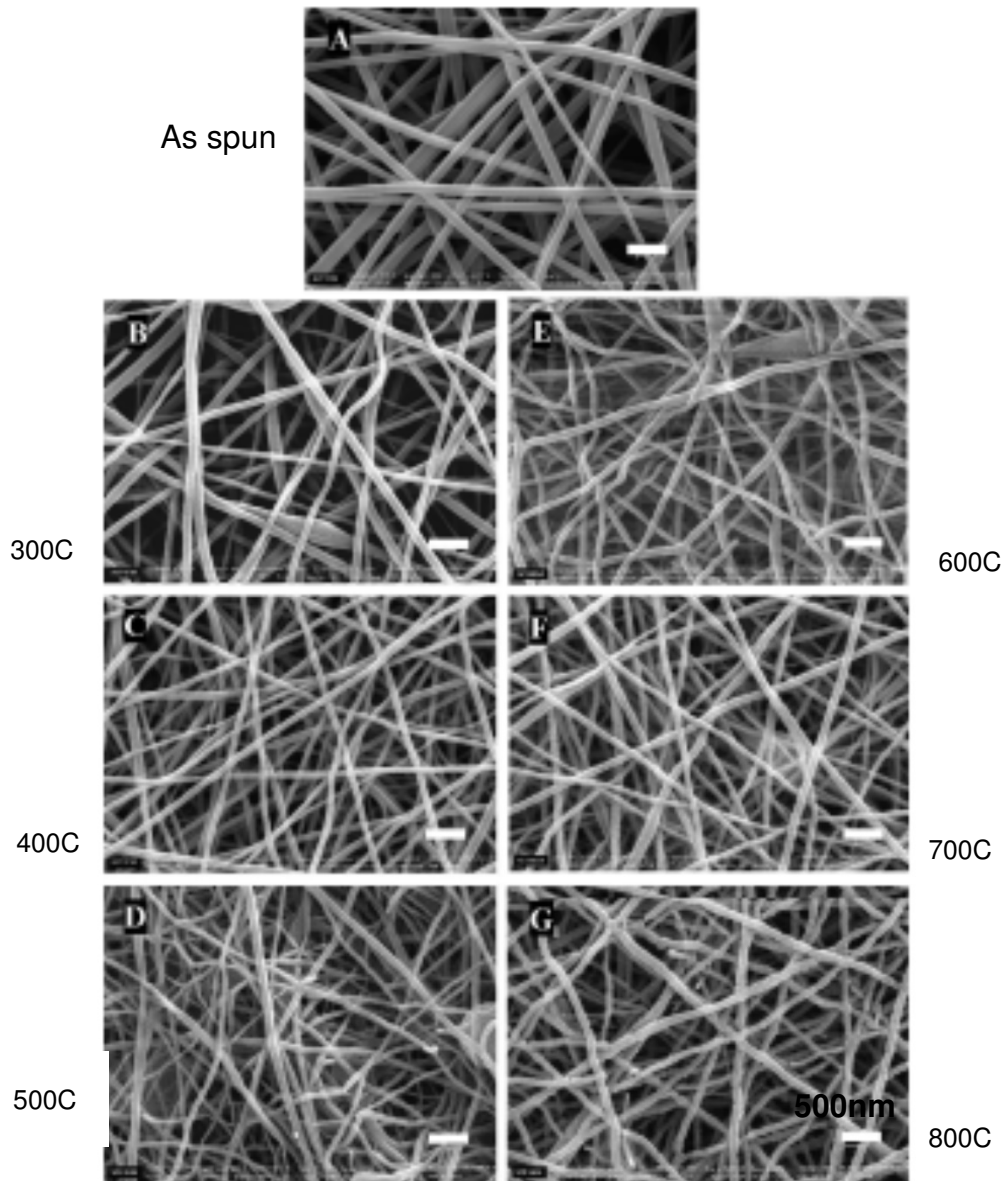
Energy Dispersive X-ray
Spectrometer – elemental analysis



- SEM shows **external** information such as surface feature, particle shape and size, topography in micrometer and nanometer scale.
- TEM gives information about **internal** structures and defects of a sample at atomic level.
- The two instruments complement each other.
- **EDS** provides elemental composition.

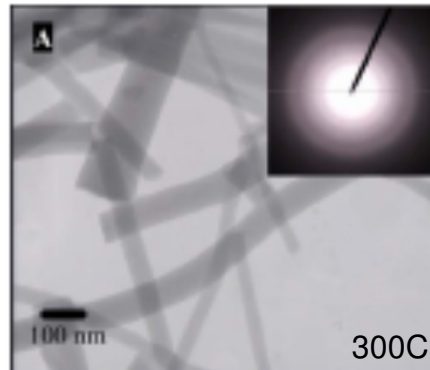


SEM and AFM of TiO₂

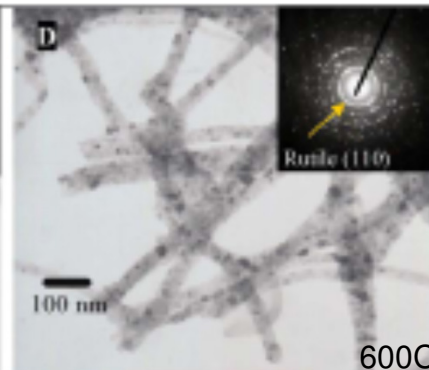


TEM & Diffraction patterns

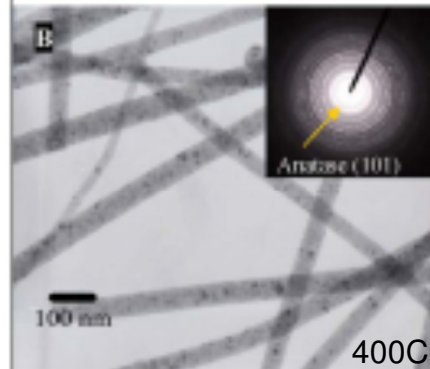
amorphous



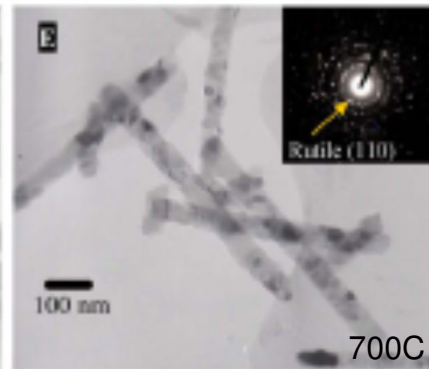
More rutile



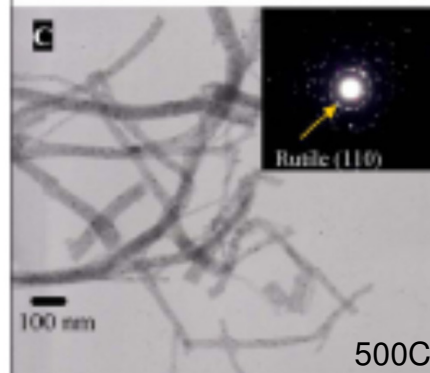
anatase



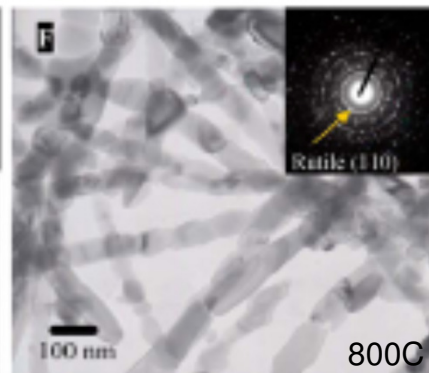
Larger grains



Anatase+rutile



Grains as large
As the fiber diameter



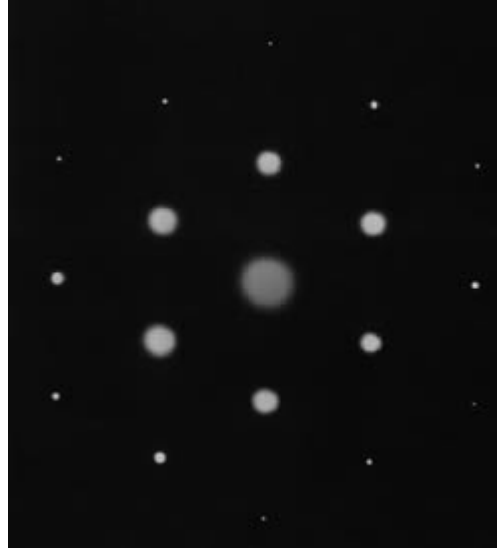
Diffraction

- Diffraction techniques depend on interference of transmitted or reflected beam from the sample.
- Those diffraction patterns reveal information about the sample:
 - Phase
 - Crystal structure
 - Orientation
- Relevant terms
 - FWHM of Diffraction pattern (fat or skinny peaks in XRD)
 - Spot pattern (single crystal)
 - Ring pattern (polycrystalline)
 - Spotty ring (mixed size powders)

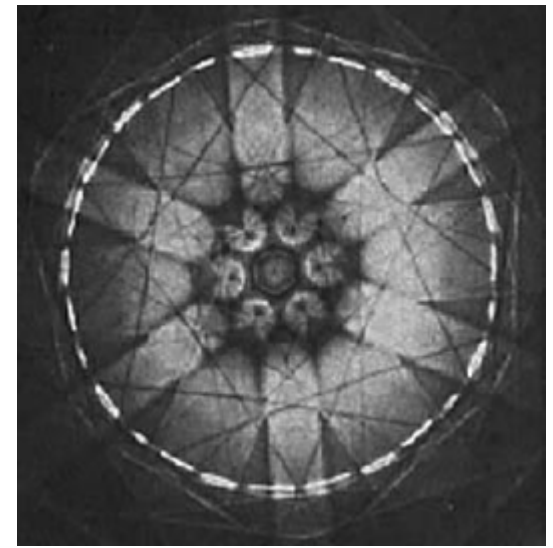
Diffraction Patterns



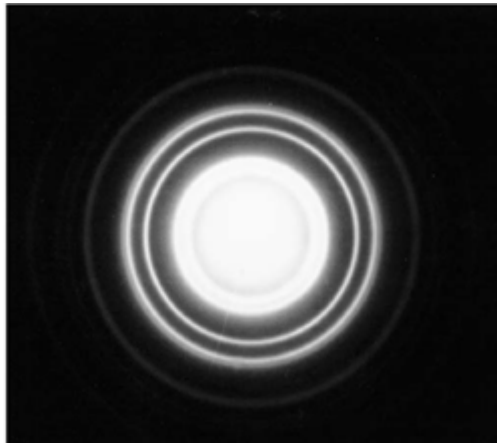
Polycrystalline



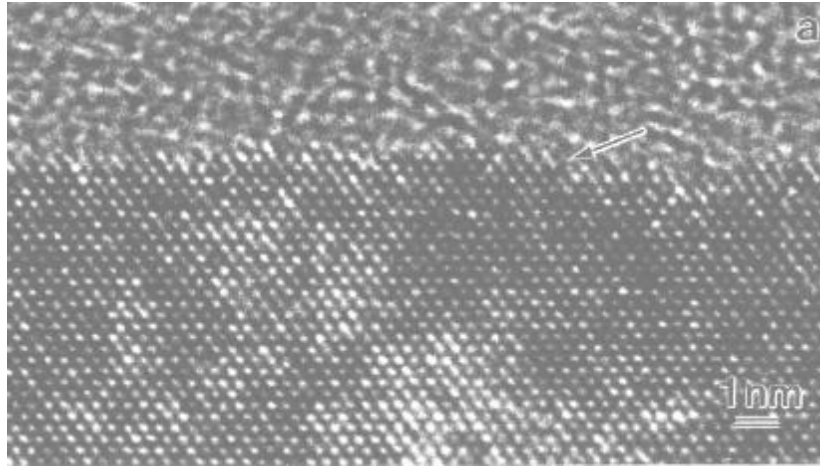
Single crystal



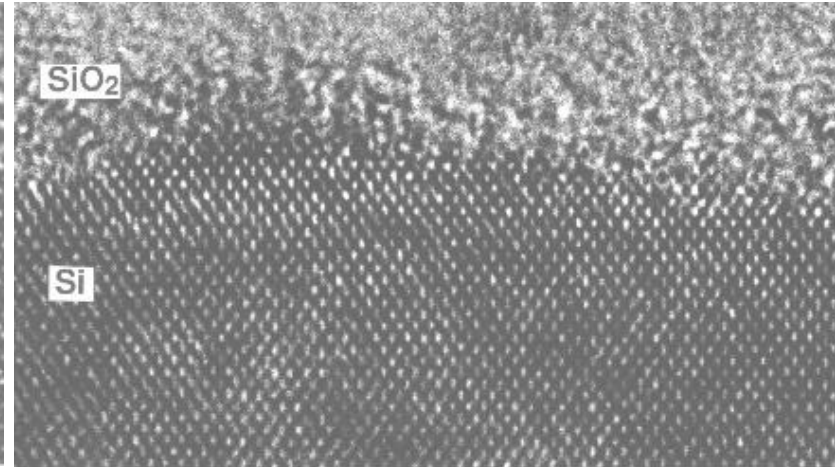
CBED single crystal



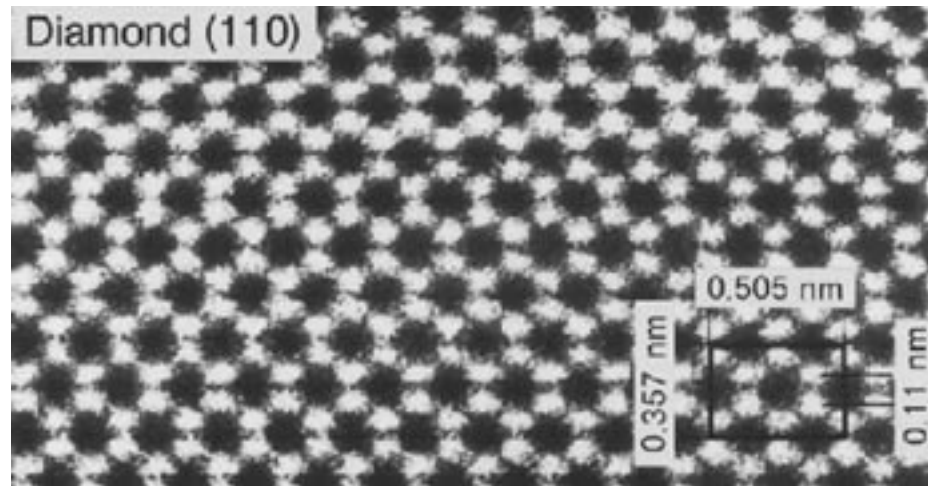
TEM – atomic structure



Smooth interface

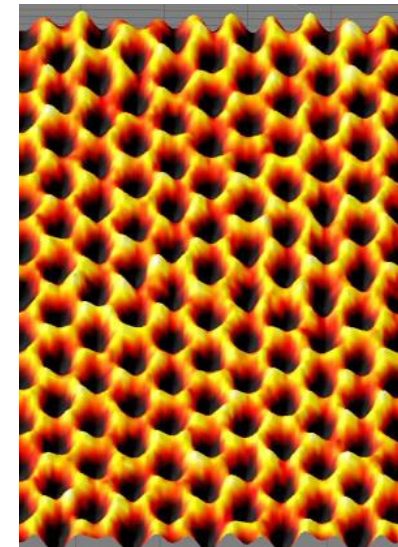
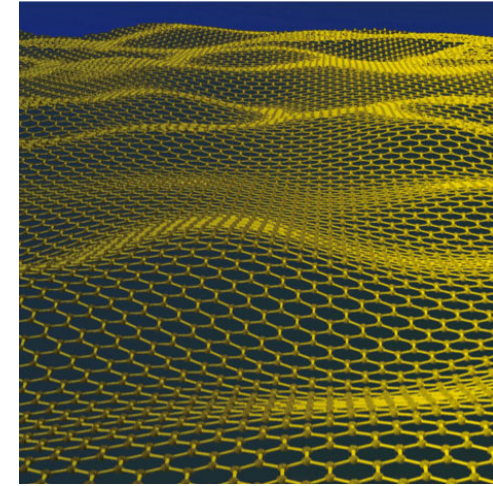
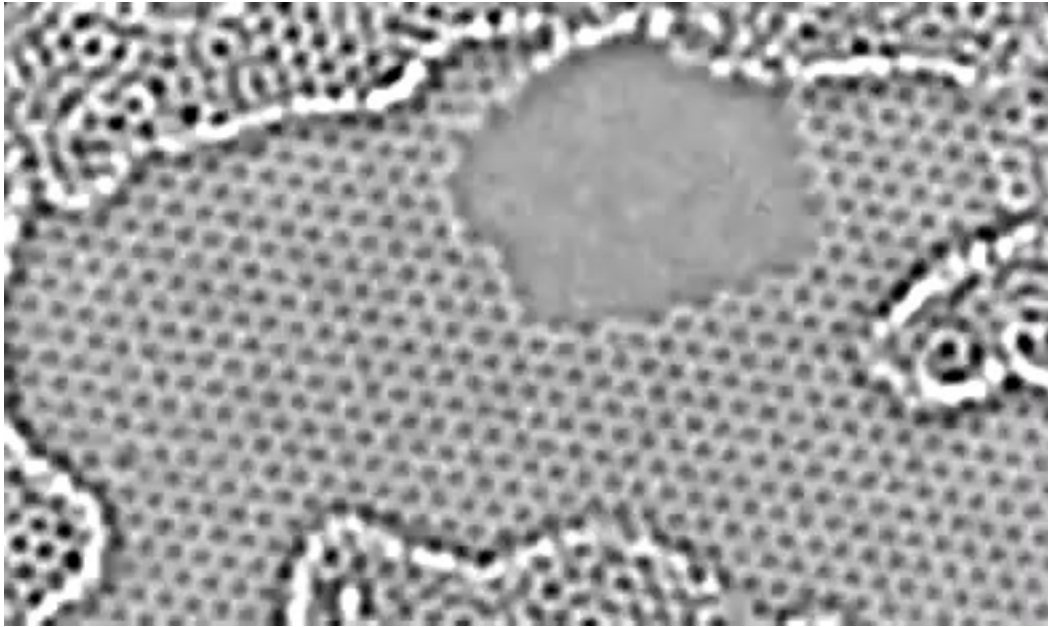


Rough interface



TEM – atomic structure

CARBON atoms moving at the edge of a hole in graphene



Science 27 March 2009: Vol. 323. no. 5922, pp. 1705 - 1708 DOI: 10.1126/science.1166999, "Graphene at the Edge: Stability and Dynamics," Çağlar Ö. Girit, Jannik C. Meyer, Rolf Erni, Marta D. Rossell, C. Kisielowski, Li Yang, Cheol-Hwan Park, M. F. Crommie, Marvin L. Cohen, Steven G. Louie, A. Zettl

How we use these to serve industry

Think win-win-win

- NSTDA's mission – providing services
- NSTDA has state-of-the-art instrument
- Industry - increasing profit and revenue
- For the country's healthy economy and increasing competitiveness

Ex. 1: Ibis Technology

- In early 1990s, Ibis was a company making silicon-on-insulator wafers by oxygen implantation into silicon wafer, called SIMOX
- They provided funding and sending samples for my Ph.D. study because they did not have a TEM.

Oxygen dose at 65 keV

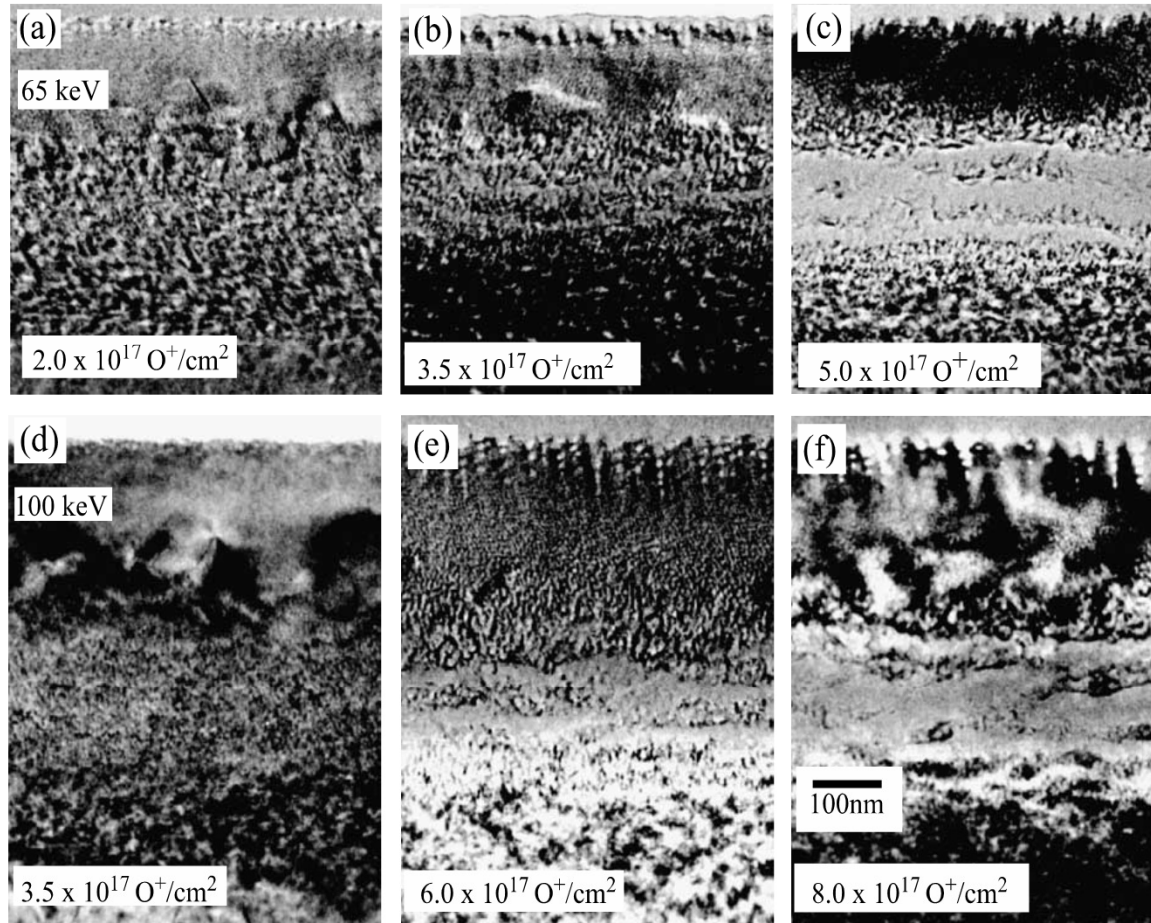


Fig. 3. TEM micrographs of as-implanted SIMOX wafers implanted at 65 keV with doses of (a) $2.0 \times 10^{17} \text{ O}^+/\text{cm}^2$, (b) $3.5 \times 10^{17} \text{ O}^+/\text{cm}^2$, and (c) $5.0 \times 10^{17} \text{ O}^+/\text{cm}^2$ and wafers implanted at 100 keV with doses of (d) $3.5 \times 10^{17} \text{ O}^+/\text{cm}^2$, (e) $6.0 \times 10^{17} \text{ O}^+/\text{cm}^2$, and (f) $8.0 \times 10^{17} \text{ O}^+/\text{cm}^2$, respectively.

Energy, Dose, Anneal

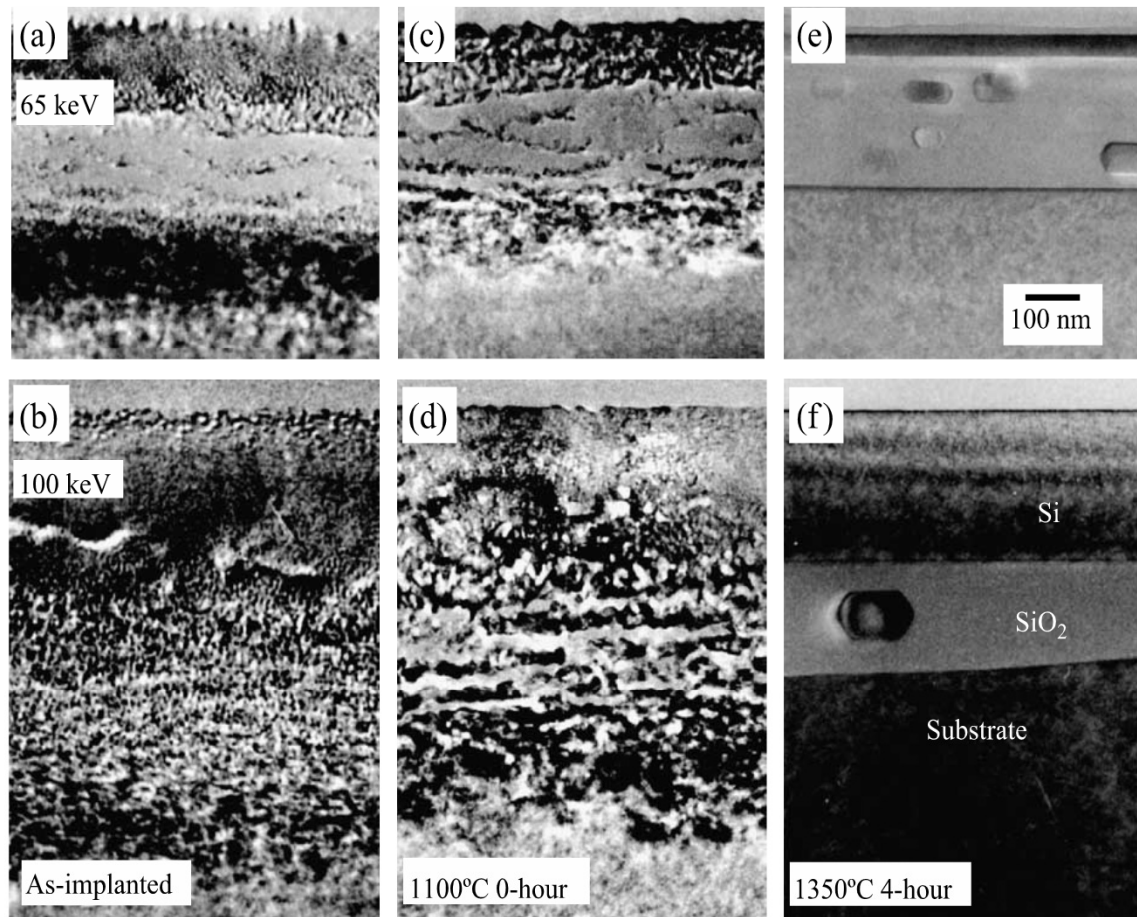
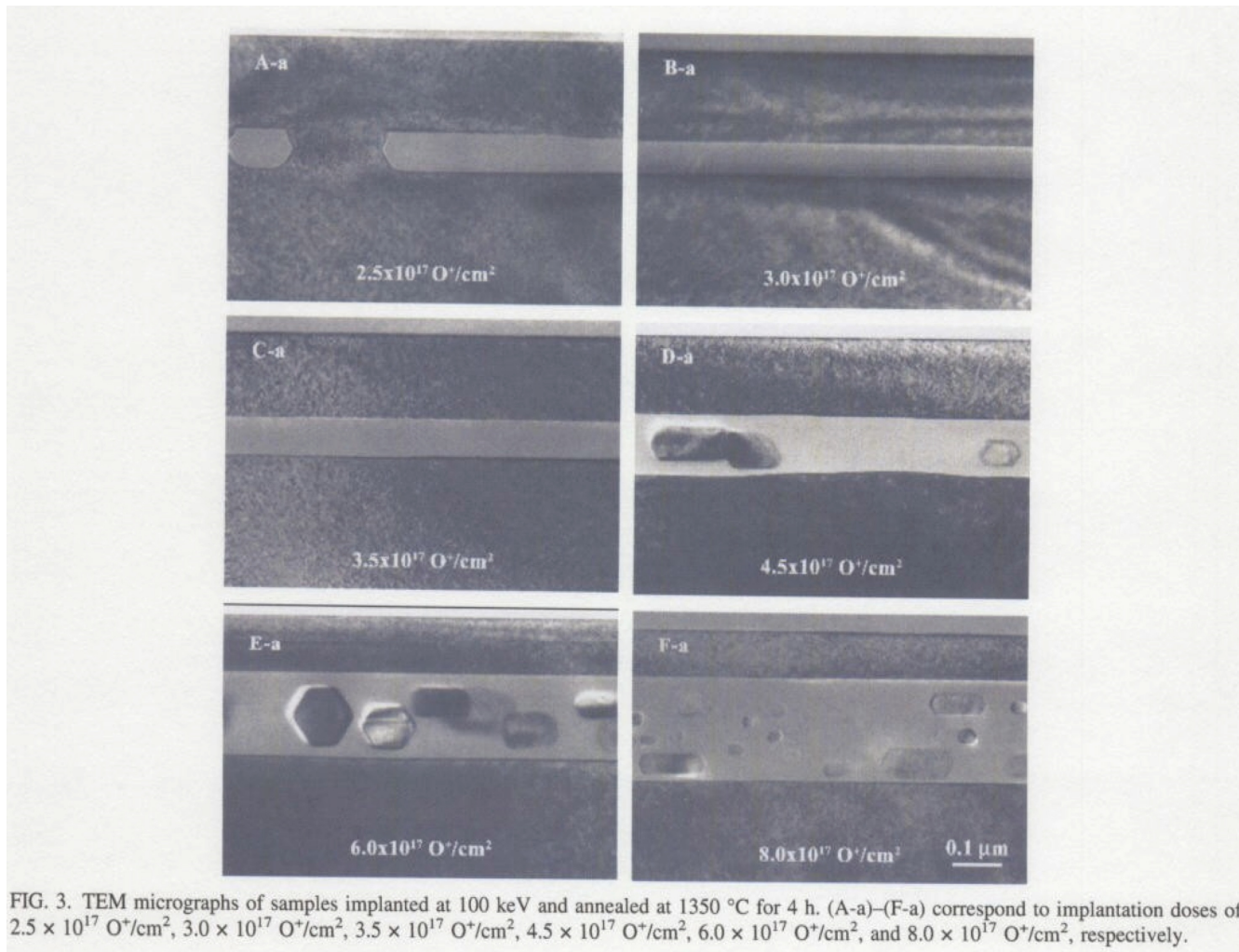


Fig. 4. TEM micrographs of (a), (b) as-implanted SIMOX wafers at 65 and 100 keV with the dose of $4.5 \times 10^{17} \text{ O}^+/\text{cm}^2$; (c), (d) annealed at 1100 °C without holding; and (e), (f) annealed at 1350 °C for 4 h, respectively.

Dose & Annealing



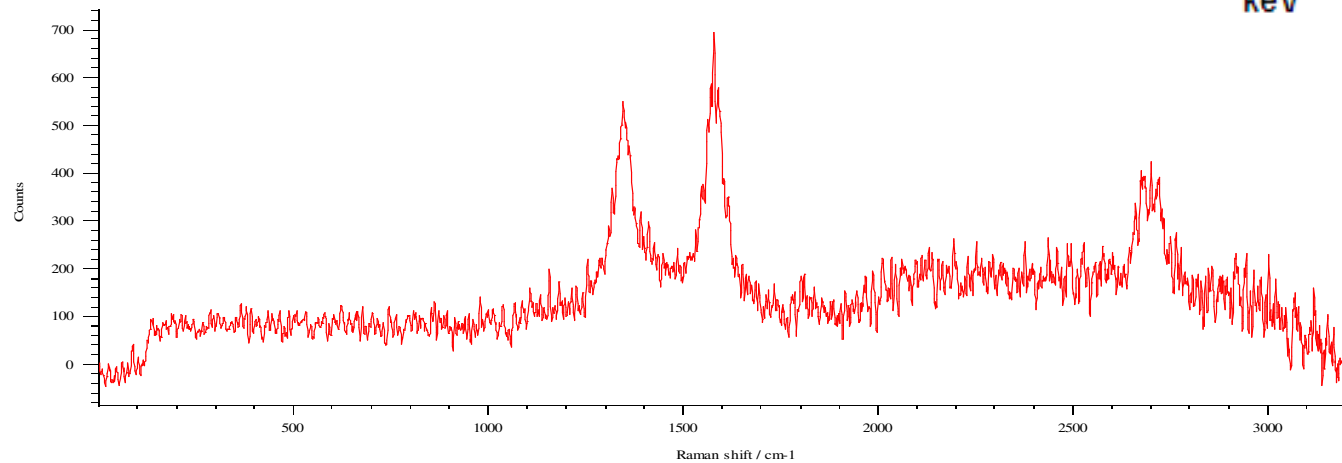
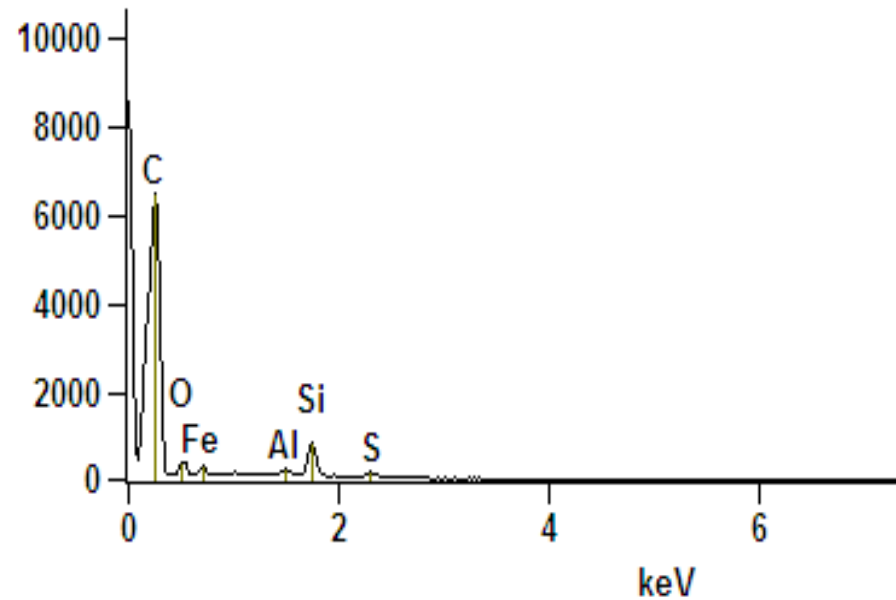
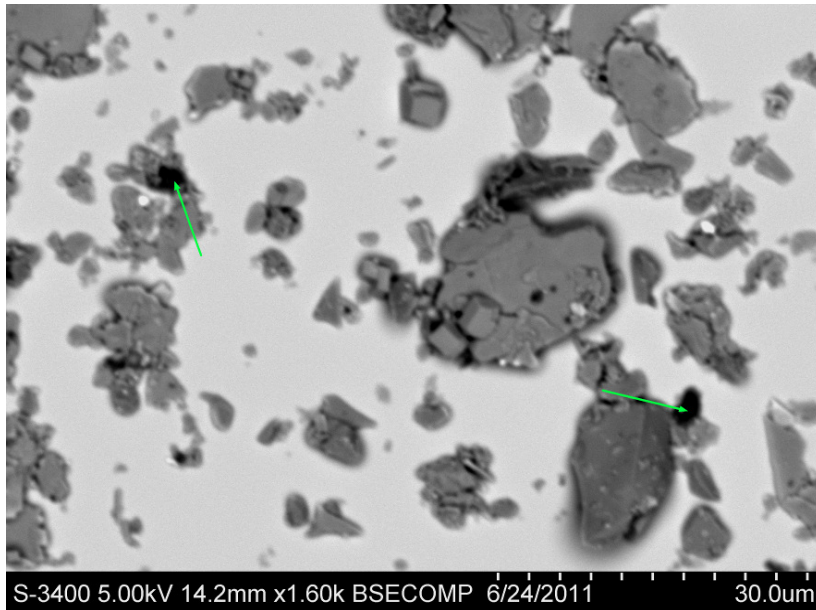
Ex 2: Burr Brown

- A microelectronic company in Tucson
- They paid for an energy dispersive X-ray spectrometer (EDS) that was attached to a SEM in my facilities.
- In turn, they got access to the SEM one day a week.
- They later-on got their own SEM and allowed us to use it as our back-up.
- They hired our graduates.

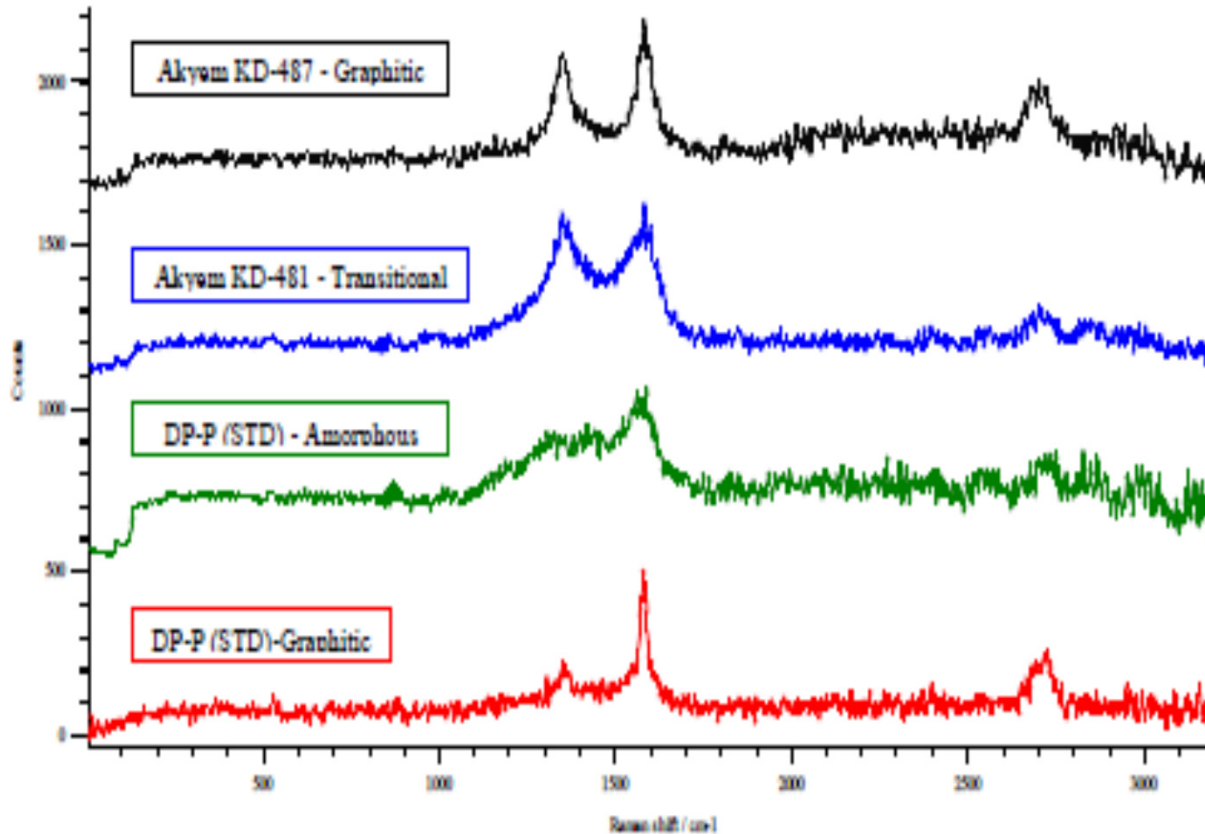
Ex 3: Mining

- Provide funding to carry out research
- They wanted to figure out carbon phases in gold ore before further invest in extracting gold.

Carbon in Gold Ore



Carbon robbing gold



Amorphous carbon robs gold

Graphitic carbon will NOT

Raman can distinguish them

Summary

In the Era of Thailand 4.0

- It is time to maximize available tools for innovation in materials research and development.
- NSTDA is playing a key role in partnering with industry and works together to increase the economical competitiveness.