

New Methods of Sample Preparation for Atom Probe Specimens



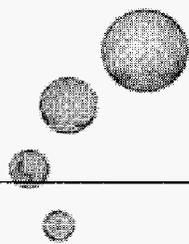
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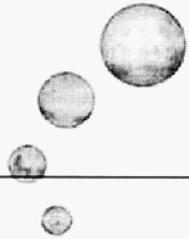




“Nano” isn’t just for Moore anymore

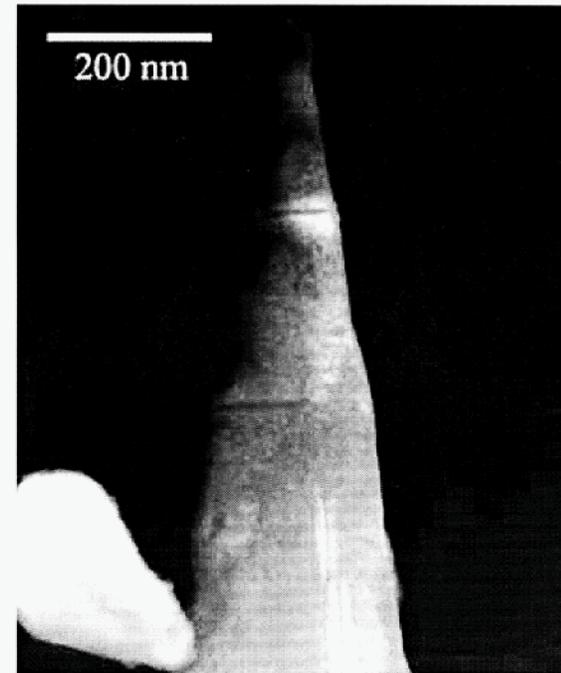
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- “There’s Plenty of Room at the Bottom” - Richard Feynman, 1959.
 - “There's plenty of room at the bottom: Nanoscience in geochemistry” - Michael F. Hochella (2002) *Geochimica et Cosmochimica Acta*.
 - Nanoparticles in the Environment - Jillian Banfield and Alexandra Navrotsky, eds. Reviews in Mineralogy and Geochemistry, Mineralogical Society of America Volume 44 volume





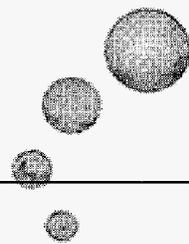
A Geological Application of APFIM

- Magnetite is a common mineral on Earth and Mars.
- Magnetite is one of the more conductive minerals.
 - Resistivity = 52×10^{-4} ohm-cm.
- Disk-shaped precipitates approx. 40 nm in diameter, 1-3 nm thick and about 10^4 platelets/ μm^3 .
- EDS shows Mn and Al concentrated in these precipitates.
- Quantitative analysis has been limited by the thickness of this second phase.



Atom Probe is the only technique that can potentially quantify the composition of these precipitates.

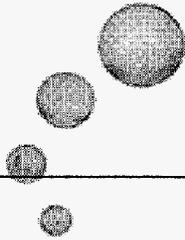




JPL Objectives for APFIM

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- Geological and planetary materials
 - Genesis
 - Stardust
 - Mars Sample Return
 - Lunar South Pole Aitkens Basis Return
 - Analyses of samples of interest for geomicrobiology
 - Rock varnish
 - Extremophilic microorganisms and spores
 - Metrology of nanoscale instrumentation
 - Quantum dot infrared detectors (QDIPs)
 - Delta-doped charge coupled devices (CCDs)





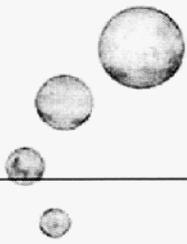
Prior Art of APFIM

Sample Preparation

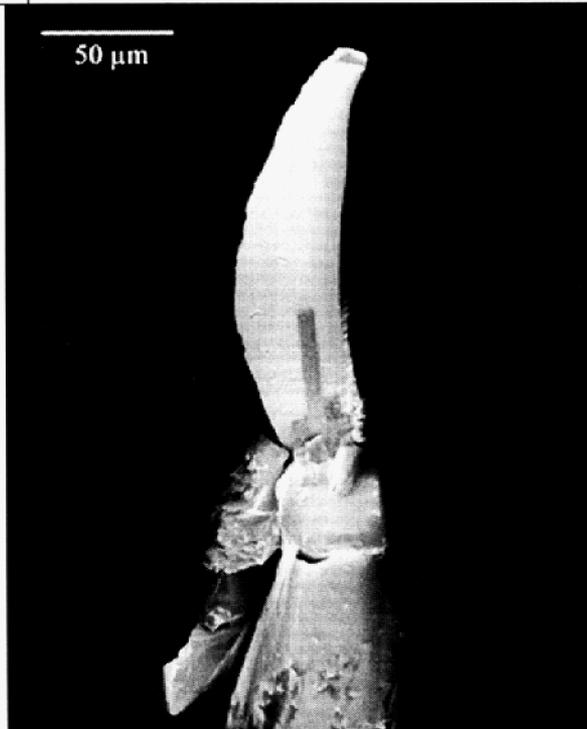


- Electropolishing
 - Suitable for most metallic samples
 - Used for decades
- Method of Sharp Shards
 - Camus, Melmed and Banfield (1991)
 - Good for samples that can not be electropolished,
 - Requires Focused Ion Beam (FIB) milling to generate useable samples.
- Bosch Process (Deep Reactive Ion Etching)
 - Larsen, et al. (1998, 1999)
 - Very useful for multilayer structures on Si
 - Limited to Si substrates, requires extensive development for other materials
 - Requires FIB milling as final step

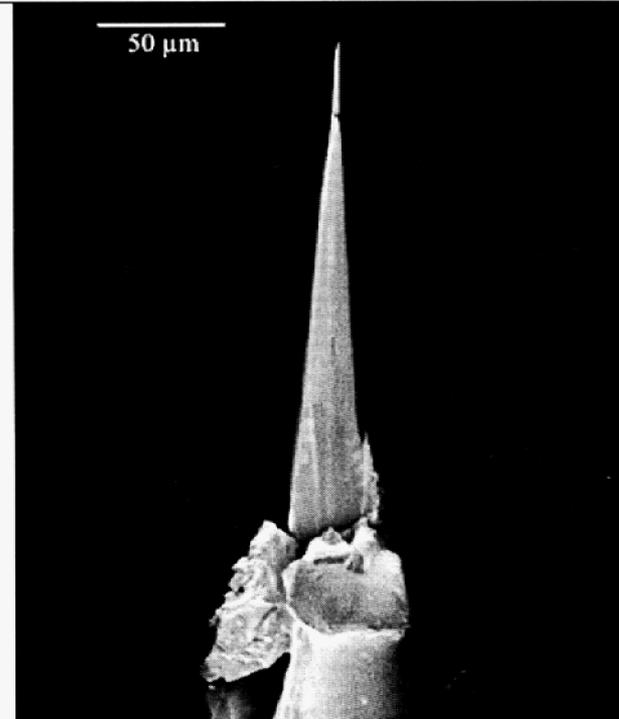




Method of Sharp Shards with FIB



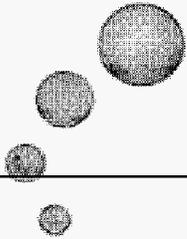
Sample as mounted with Pt deposition at the base to improve electrical connection.



Sample after extensive ion milling using various beam currents and orientations.



California Institute of Technology



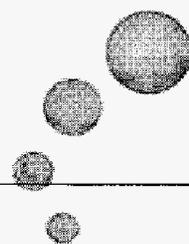
New Methods Required for Difficult Samples



- Many materials are not easily fabricated using electropolishing, MSS or the Bosch process.
 - Materials science
 - Biology and Bioengineering
 - Geology
 - Environmental Science
 - Nanoscale device fabrication
- FIB milling is slow and expensive.
- Wet chemistry and the reactive ion etching are typically limited to Si and other semiconductors.

⇒ **Bulk mechanical micromachining.**



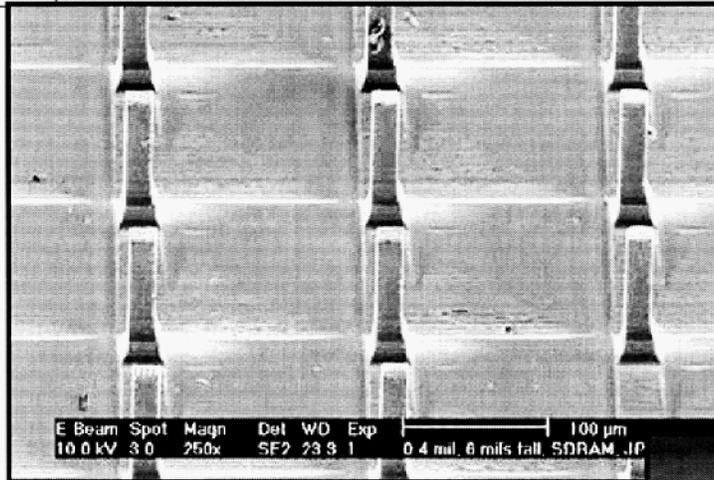


APFIM Sample Prep using the Dicing Saw

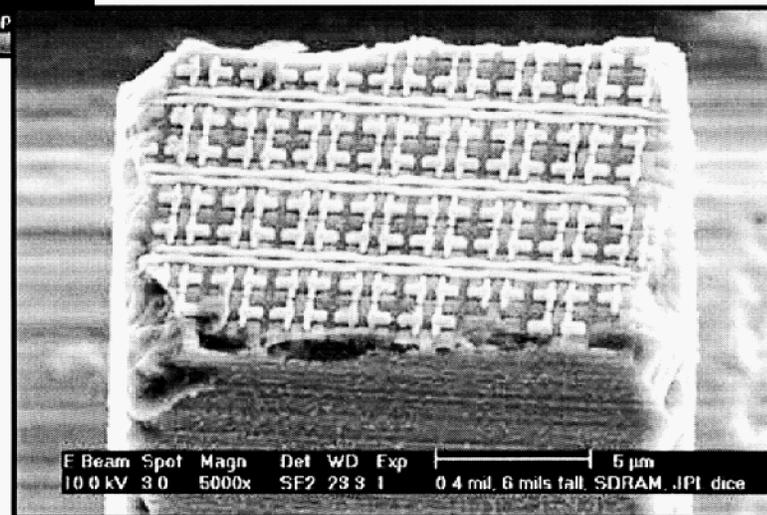
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- Commonly used to section semiconductor wafers into individual devices following manufacture
 - Blades are optimized for Si, GaAs and ceramics
 - Specimen preparation is relatively independent of sample size and material, providing a thin slab can be made (150 to 500 μm thick)
 - Large arrays of high aspect ratio posts can be fabricated very quickly, typically about 10-20 μm by 150-300 μm long.
 - Posts can then be broken off and mounted on pins as for the MSS or the Bosch formed posts.
 - Multi-tip samples can be fabricated and separated at the same time.



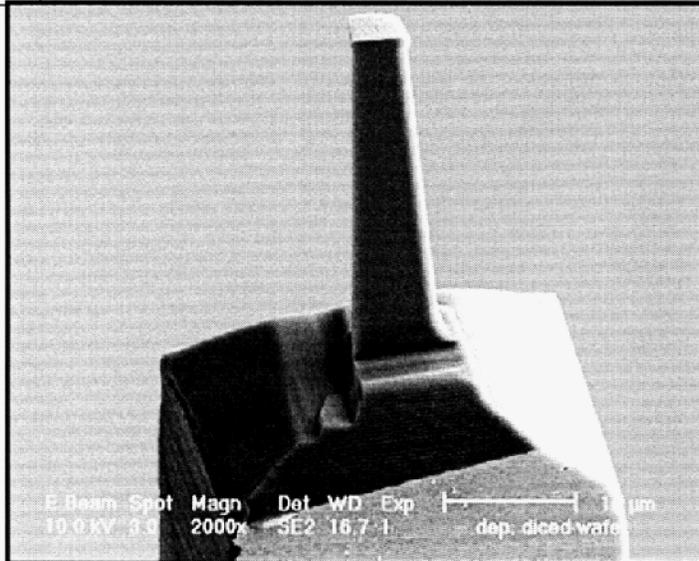
Fabrication of DRAM Posts



- Posts fabricated from a completed wafer of DRAMs
- Posts approx. 15 μm square and 150 μm long. Pitch is approx. 150 μm.

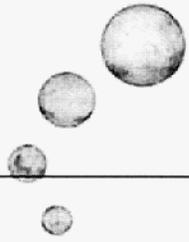


Fabrication of Coated Si Wafer

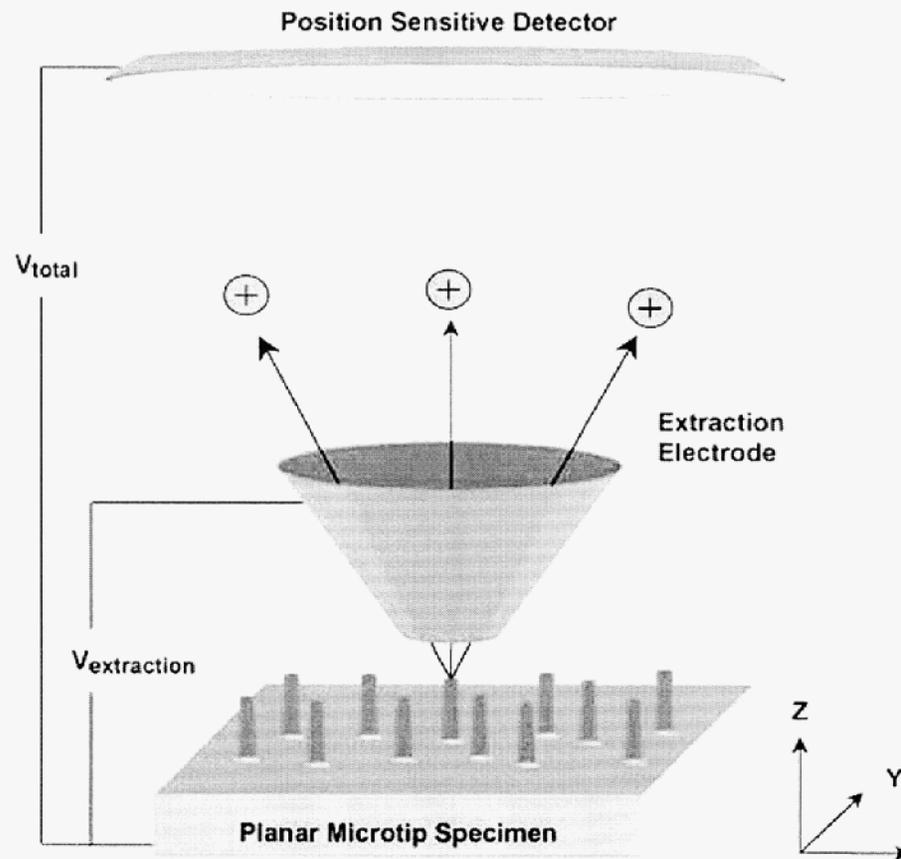


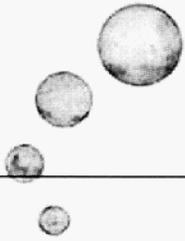
- Metal coated Si after dicing saw and rough FIB milling.



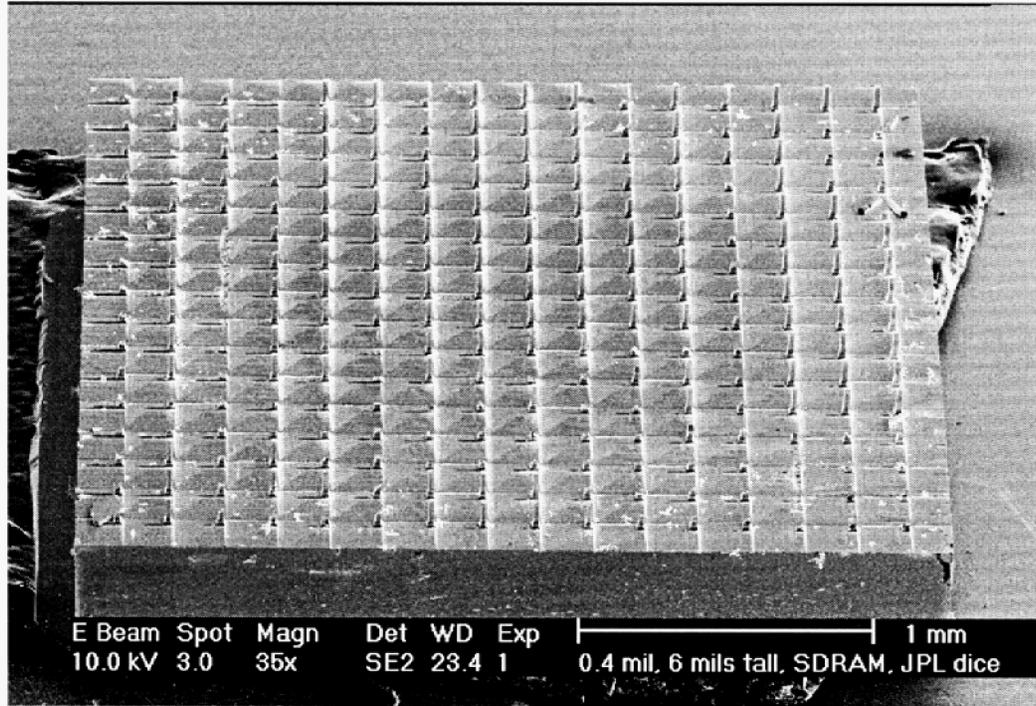


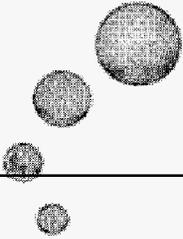
Schematic Illustration of Multi-tip sample in the LEAP





Multi-tip Sample Fabrication



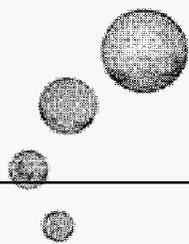


Conclusions



- The dicing saw is a time-effective method for preparing high aspect ratio posts of poorly conducting materials
 - Pros: Speed, relative material independence, COTS
 - Cons: Water cooled
- Femtosecond laser micromachining is also suitable for preparation of posts
 - Pros: Operates in air or vacuum, no water requirement
 - Cons: Proprietary setup, additional development required, parameters must be developed for each new material, “slag” produced.
- Reduction of FIB time required by about a factor of 10
- Multi-tip specimens can easily be fabricated using the dicing saw





Acknowledgements

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 - Patent Pending:
 - U.S. Provisional Patent Application serial no. 60/401,428 filed August 5, 2002. Conversion PCT utility patent filed July 29, 2003.

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