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Direct Quantitative Observation of Plasticity and Fracture of Alumina Nanoparticles

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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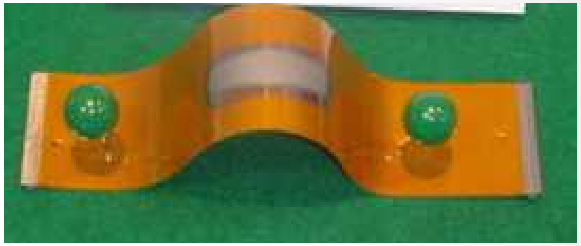
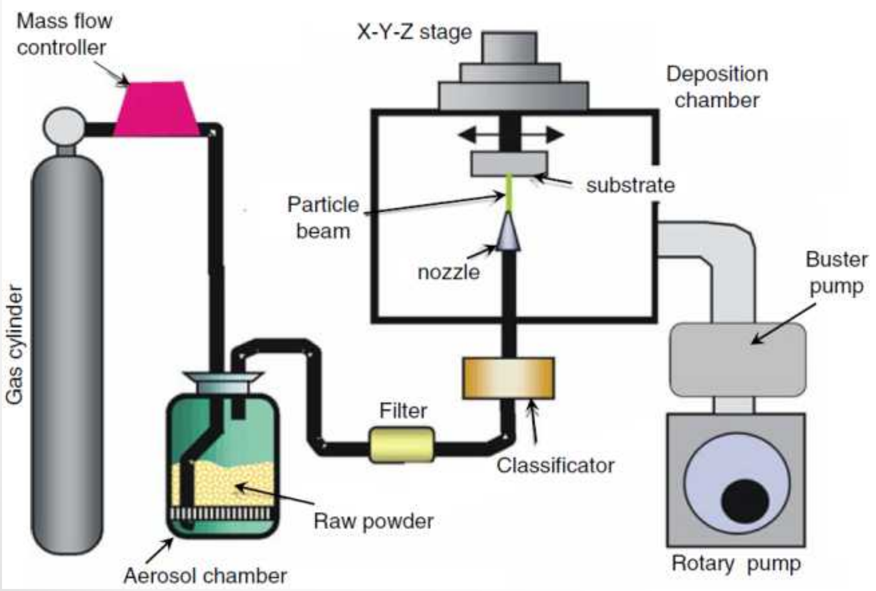
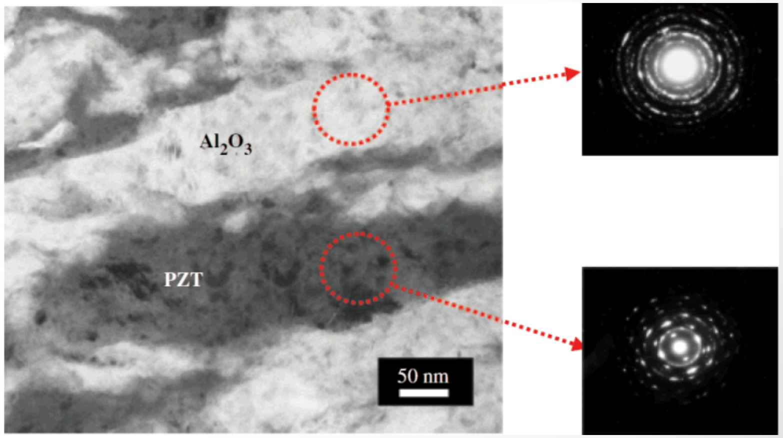
Background

- Ceramics are conventionally processed at high temperature.
- Aerosol Deposition (AD) process
  - Room temperature (RT) in vacuum
  - Sub-micron particles travel @ 200-600 m/s, impact, and consolidate on substrate to form a film.
- AD ceramic film microstructures
  - Small final grain sizes (20-75 nm)
  - Planar defects and amorphous regions.



AD Flexible electronics from J. Akedo. *JTTEE5.*, 2007:17:181



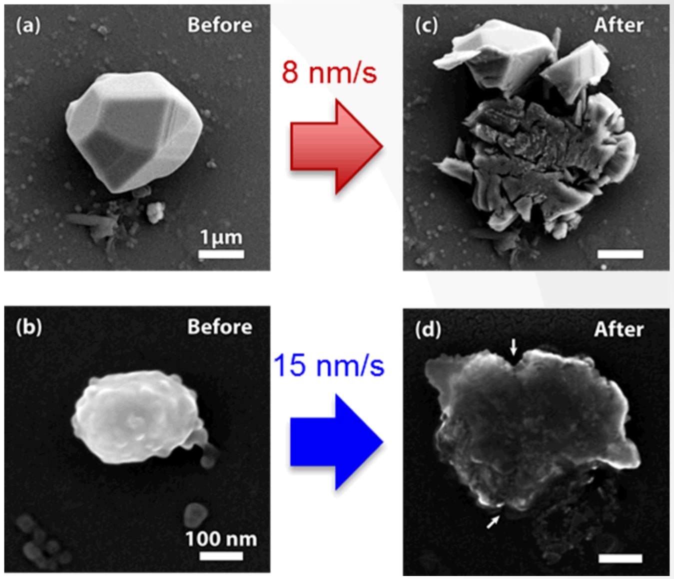


AD Al2O3 and PZT composite film from J. Akedo. *J. Am. Ceram. Soc.*, 2006:89:1834

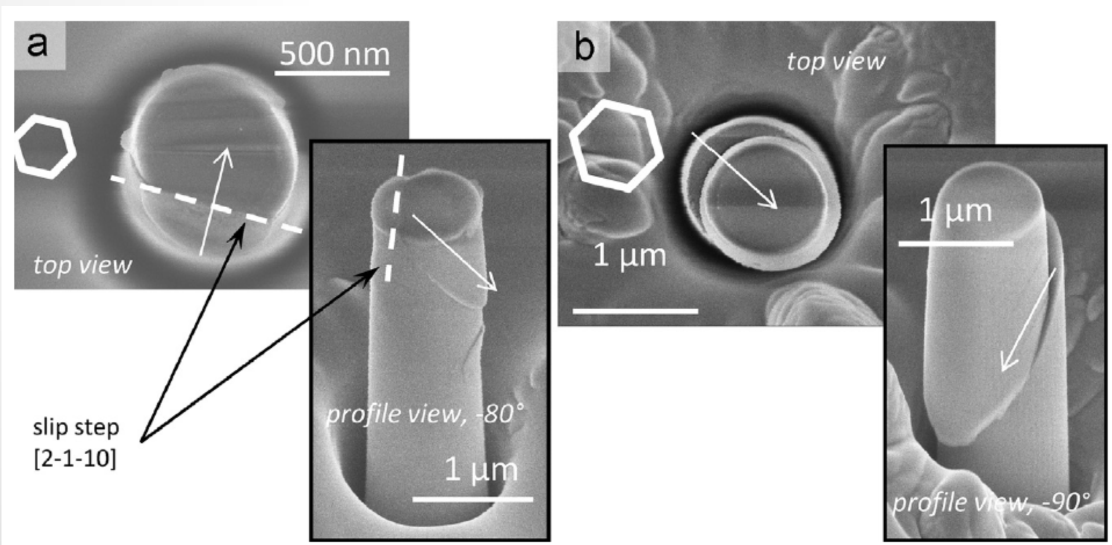
Consolidation process is not yet well understood.

A few clues…

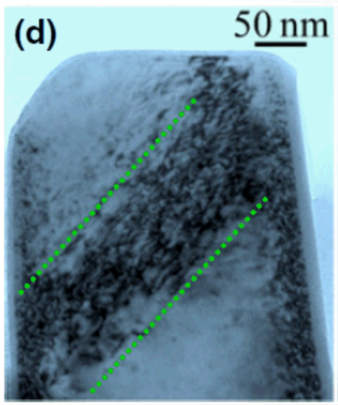
- Empirical observation that micron-sized particles do not consolidate.
- Length-scale dependent plasticity in Al_2O_3



Compressed sapphire particles. P. Sarobol, *et al.*, *JTST.*, 2016:25



Compressed Sapphire pillars S. Montagne, et al, *Ceram Int*. **40**, 2083 (2014).



ZrC pillar S. Kiani, *et al. J. Am. Ceram. Soc.*, 2015:98:2313

Clear evidence of a strong size effects on deformation.

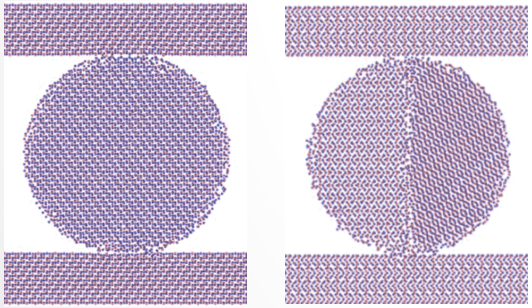
A multi-scale problem…

Tens of nm

Hundreds of nm

Thousands of nm

Atomistic Simulations



P. Sarobol, *et al.*, *JTST.*, 2016:25

Experimental Characterization

What microstructural features and processes enable this size-dependent behavior?

Initial Particle Structures

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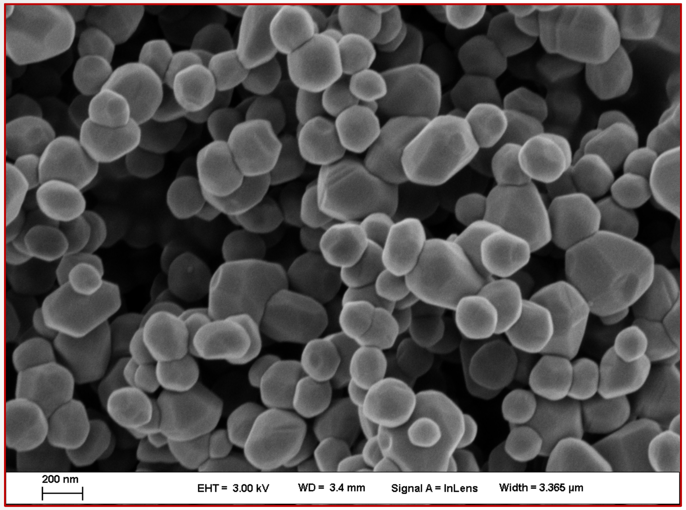
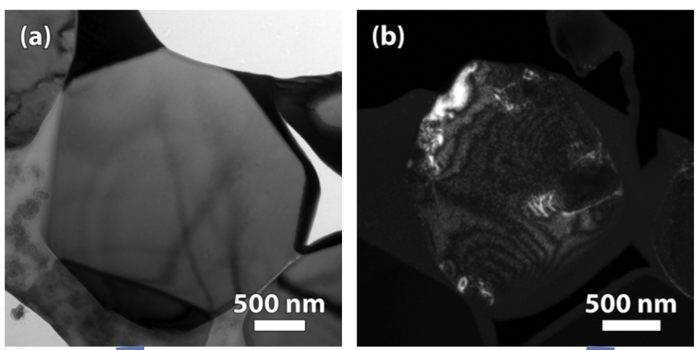
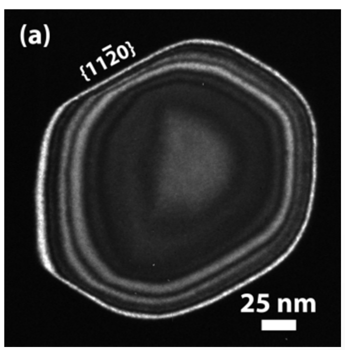


- Particles received in 300 nm and 3 μm diameters
- Faceted surfaces
- Varying internal defect densities



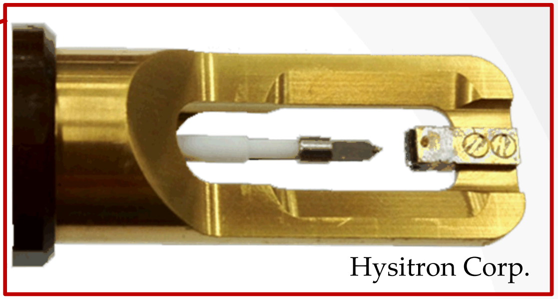
3.0μm Highly Defective



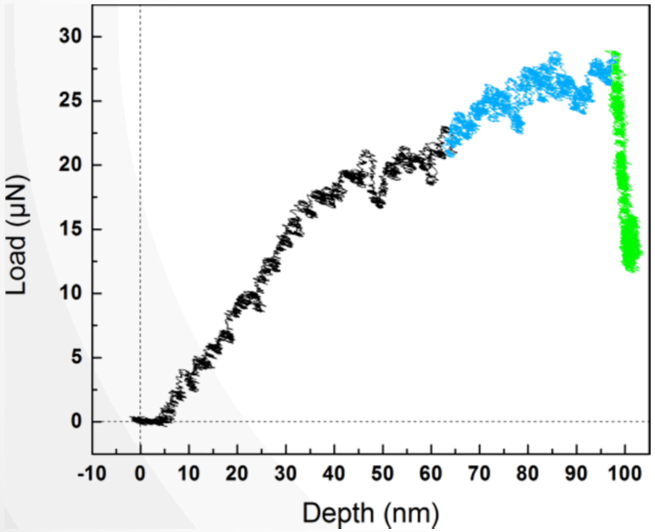
0.3μm Nearly Defect Free

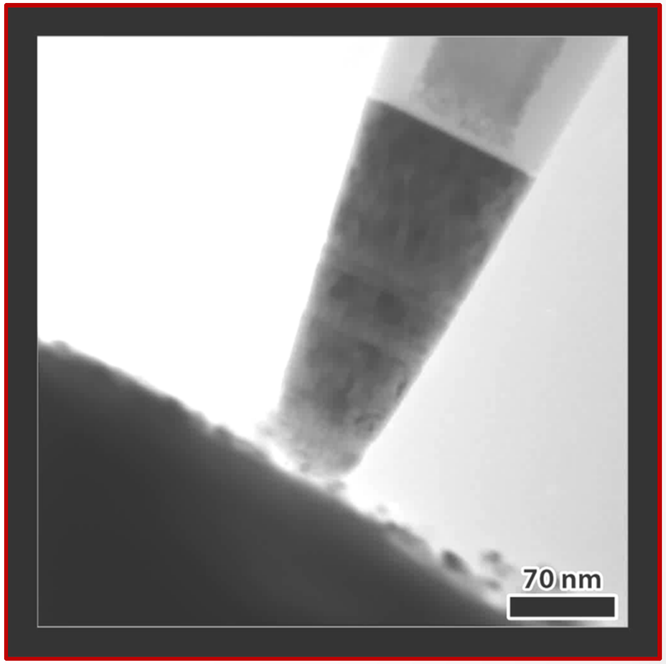
Smaller particles have lower initial internal defect densities.

Experimental Tools



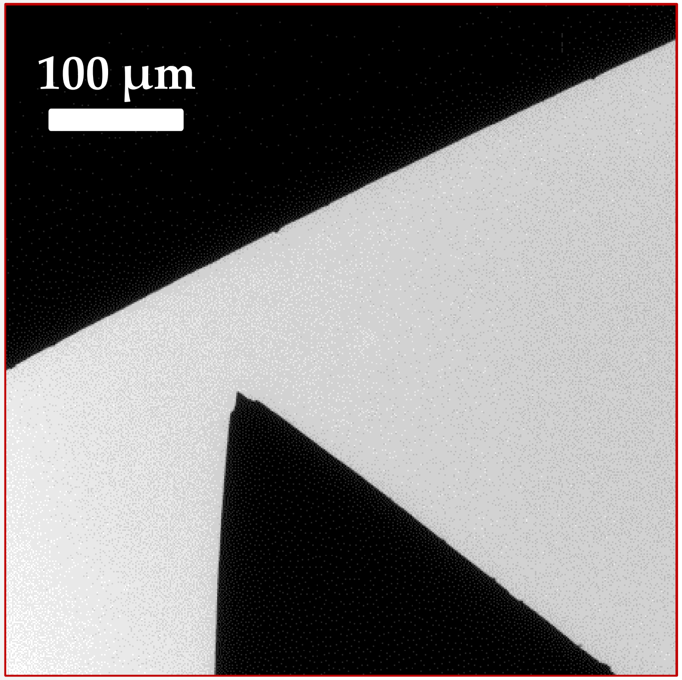
Hysitron PI95 *In Situ* Nanoindentation TEM Holder
- Sub nanometer displacement resolution
- Quantitative force information with μN resolution
- Concurrent real-time imaging by TEM

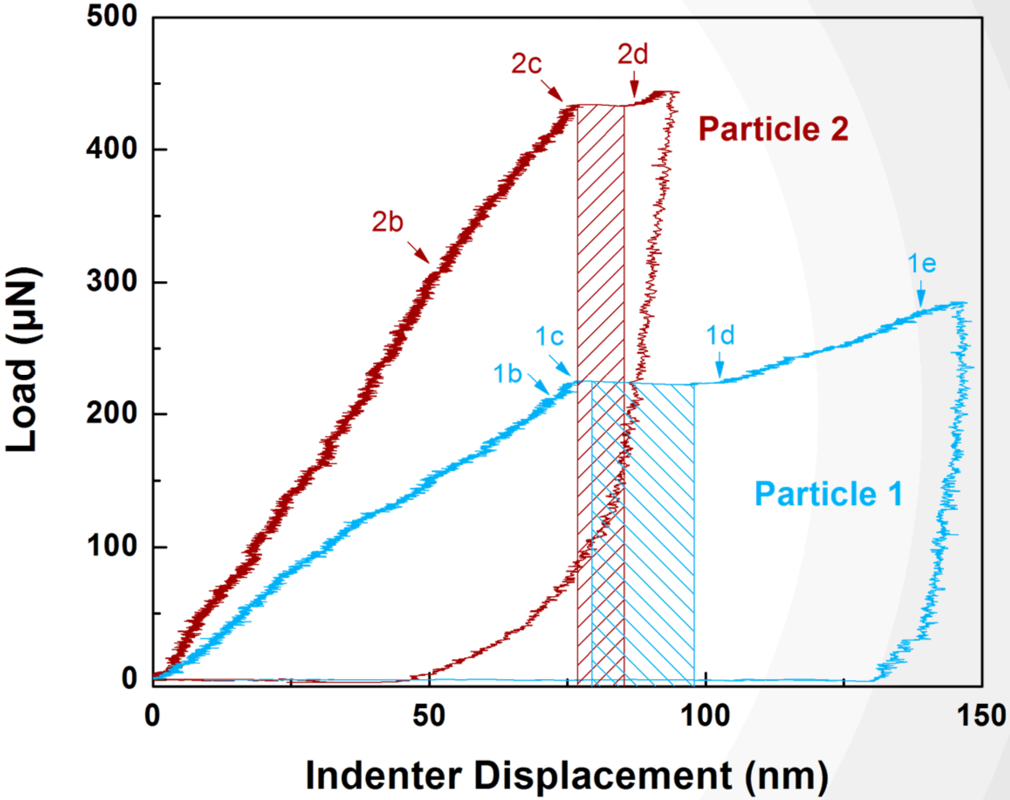




Correlates microstructural processes with quantitative mechanical loading.

In Situ TEM Compression

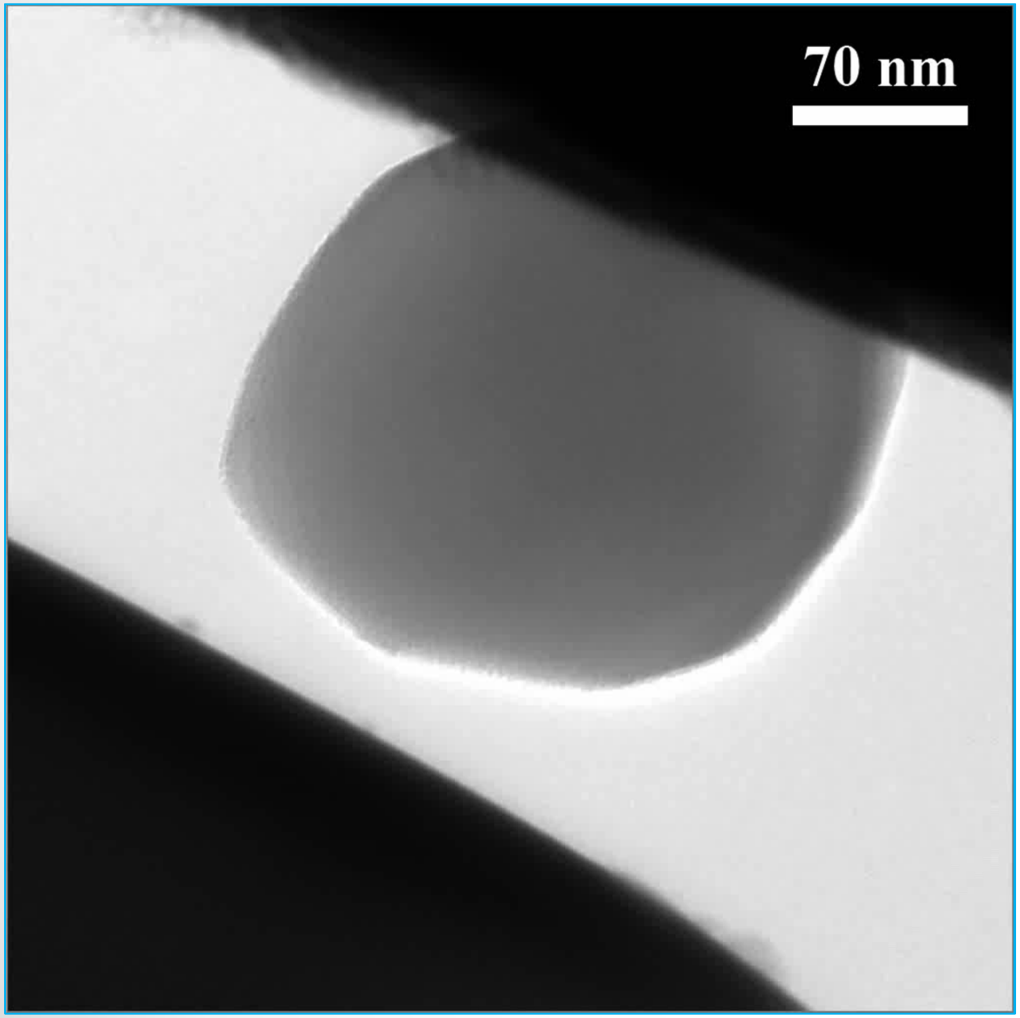




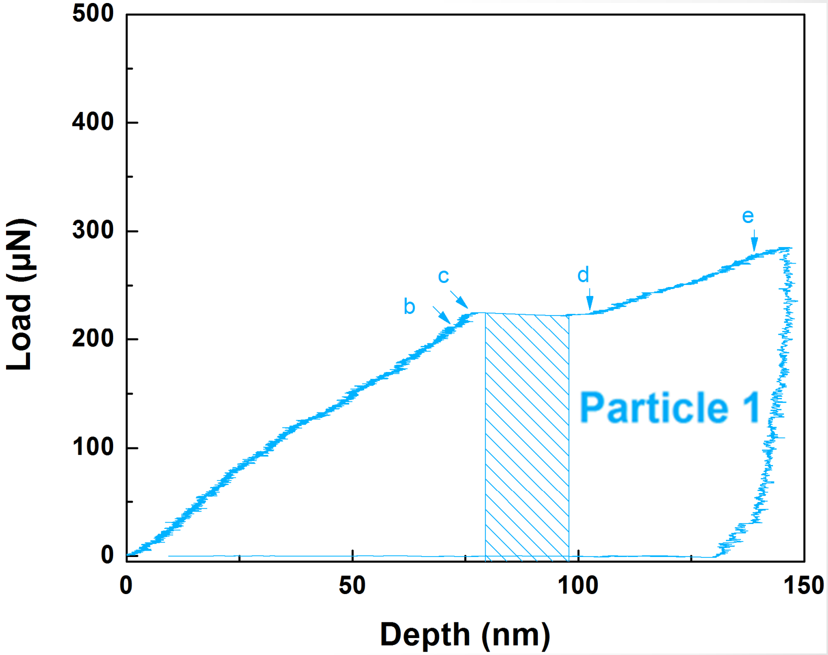
- Elastic to Plastic transitions are unclear
- Differences in strain burst behavior

In Situ TEM Compression

Diameter ~ 0.24 μm, Compression rate ~ 0.009 s^{-1}

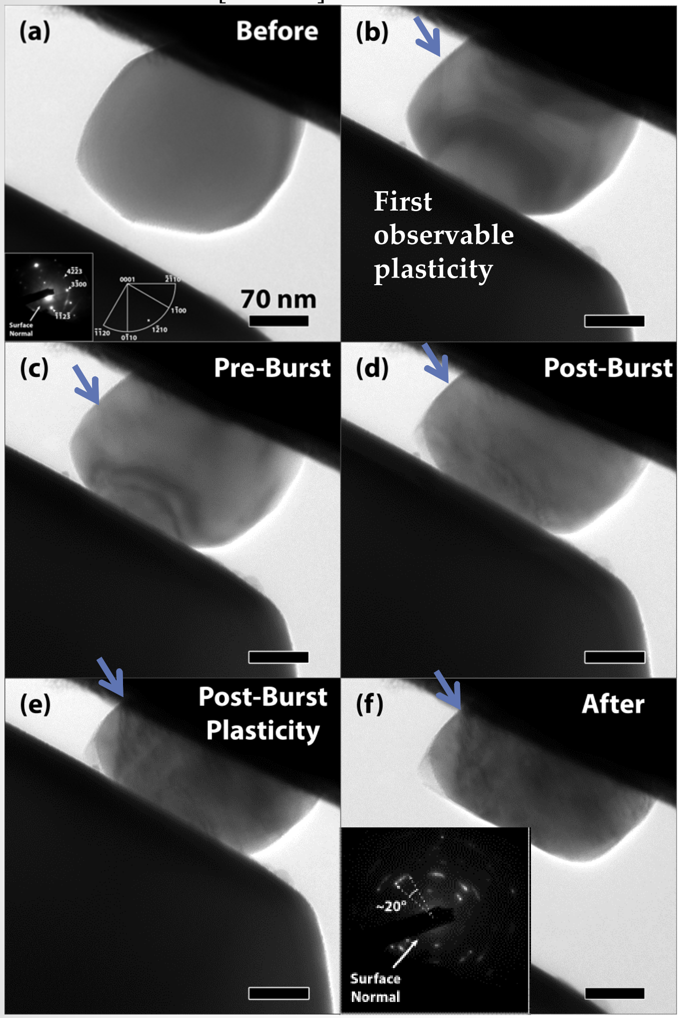


Large displacement burst at a constant load corresponds to particle fracture.

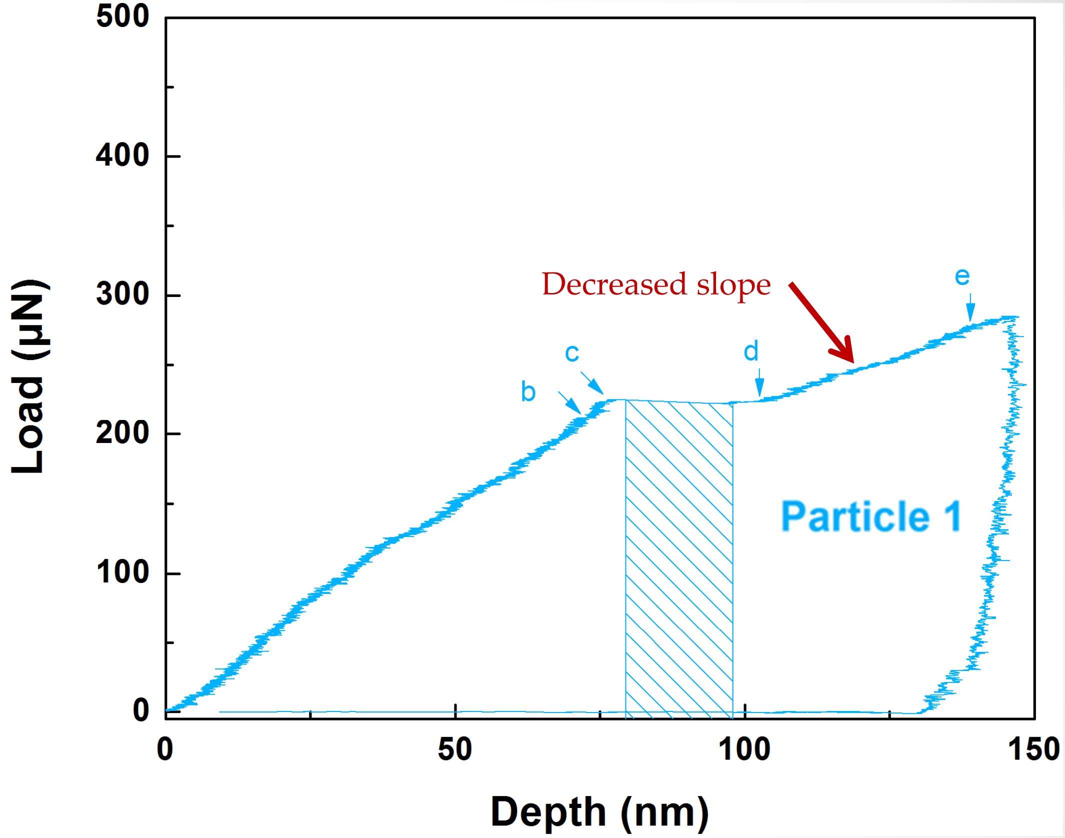


In Situ TEM Compression

Zone axis near [$\bar{9}\,\bar{9}$ 18 6]



Multiple orientations within 20 degree rotation of original orientation.



- Pre-burst plasticity: little dislocation activity.
- Crack nucleation and propagation
- Post-burst plasticity: high dislocation activity, change in deformation mechanism as indicated by lower slope.
- Mosaicity with a 20 degree orientation spread.
- Strain energy release rate = 17 J/m^2
- Contact stress estimated at 14 GPa

In Situ TEM Compression

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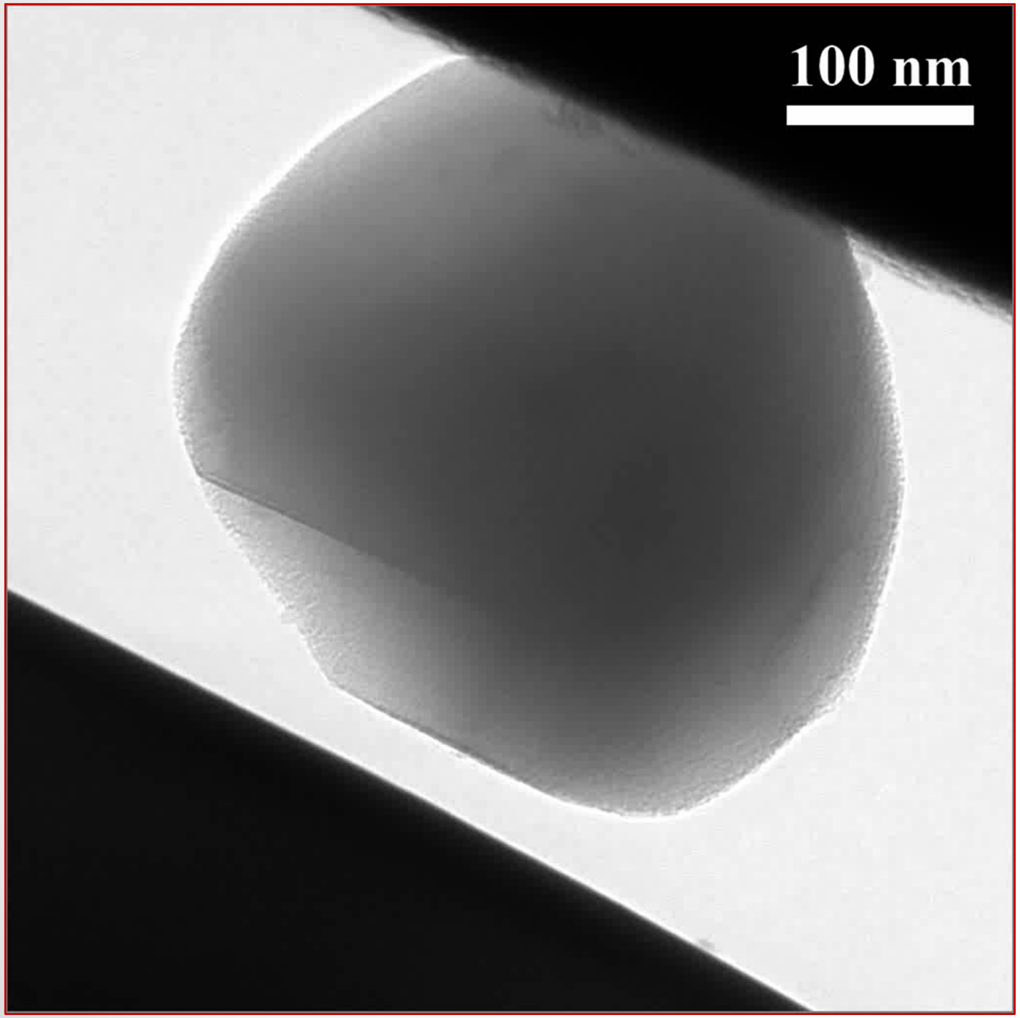
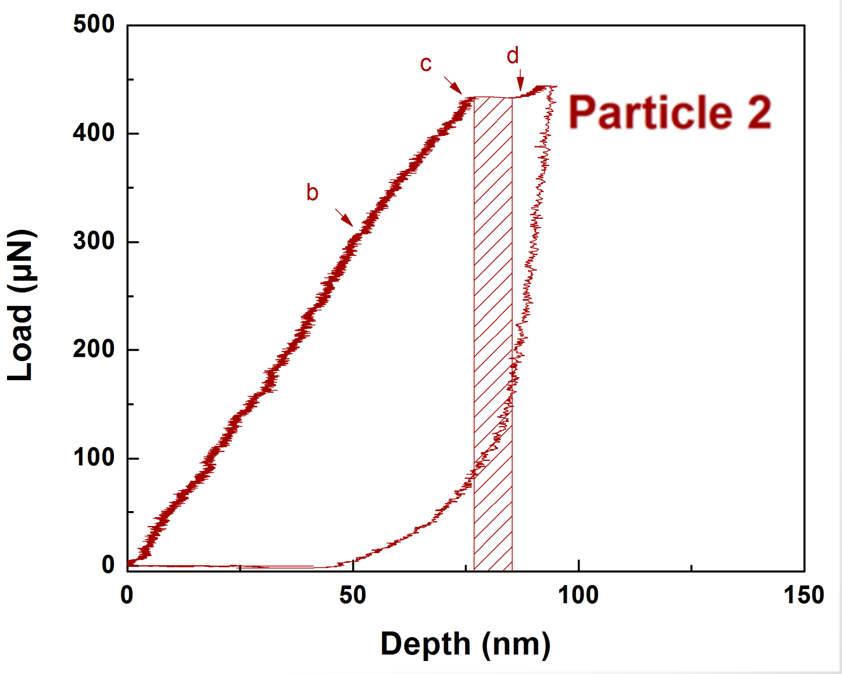
Diameter ~ 0.38 μm, Compression rate ~0.005 s^{-1}

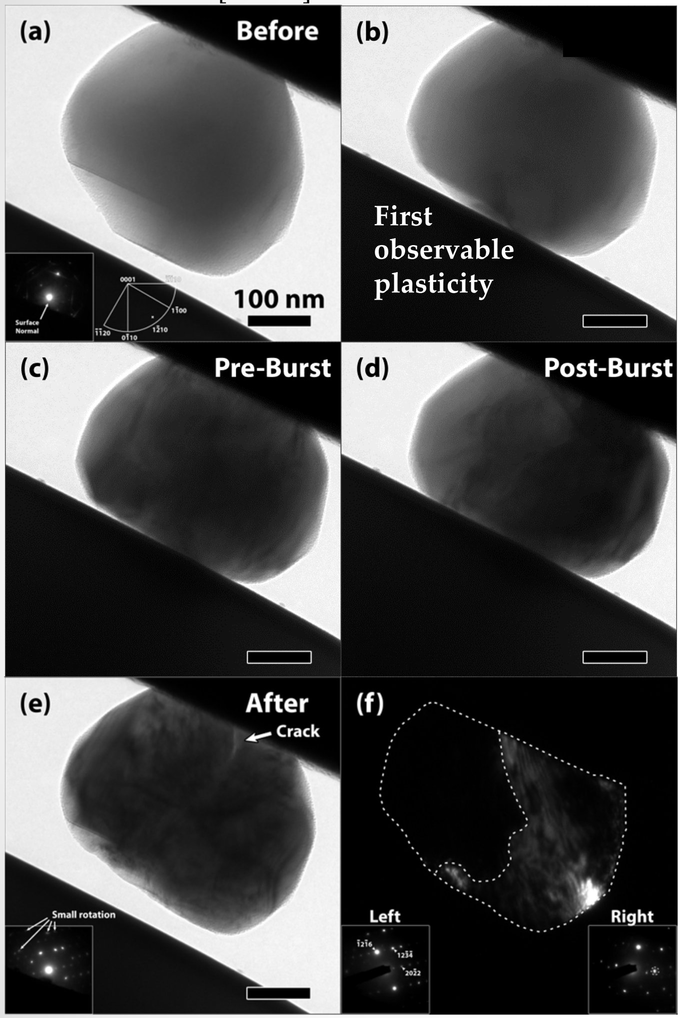


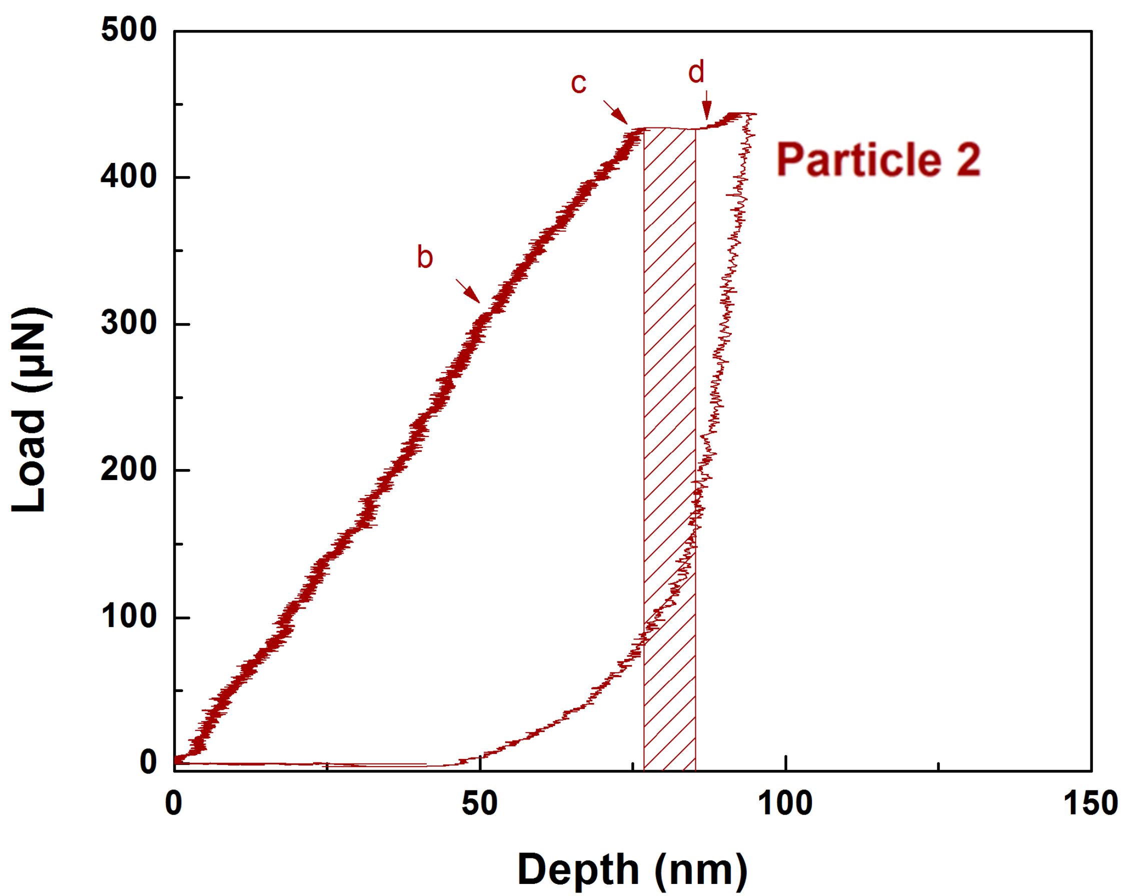
Large displacement gain at a constant load ("burst") corresponds to particle fracture.



In Situ TEM Compression

Zone axis near $[\bar{2}\ 5\ \bar{3}\ 2]$

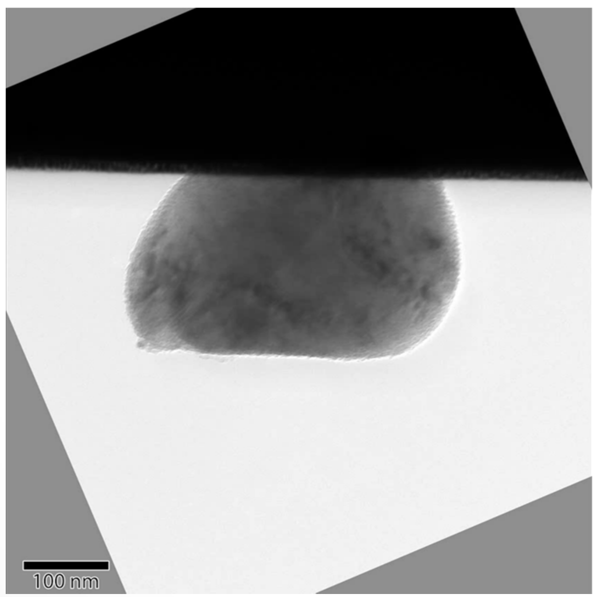


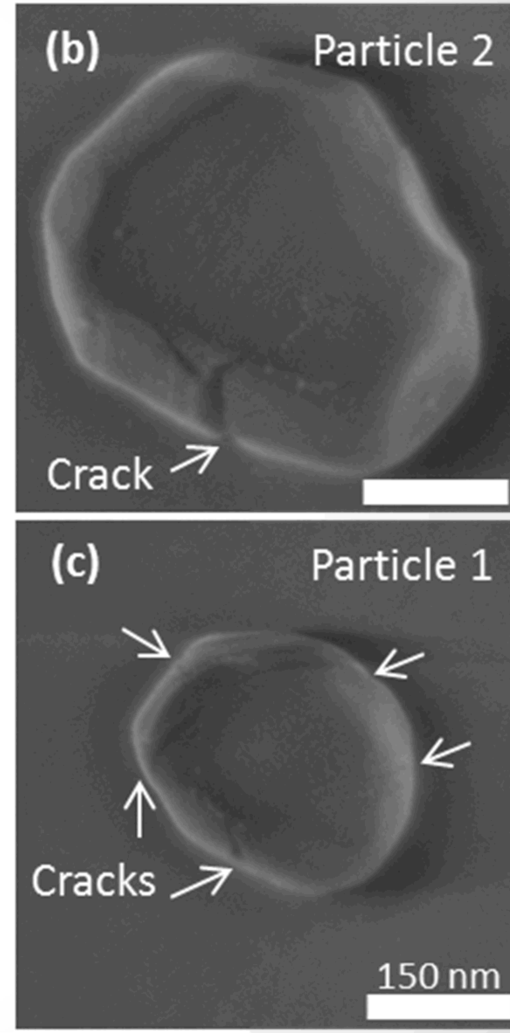


- Pre-burst plasticity: moredislocation activity.
- Crack nucleation and propagation
- Strain energy release rate = 17 J/m^2
- Contact stress estimated at 14 GPa

Post-Deformation

- Cracks more clearly evident
- Possible evidence of coordinated shear process
- Qualitatively similar final structures

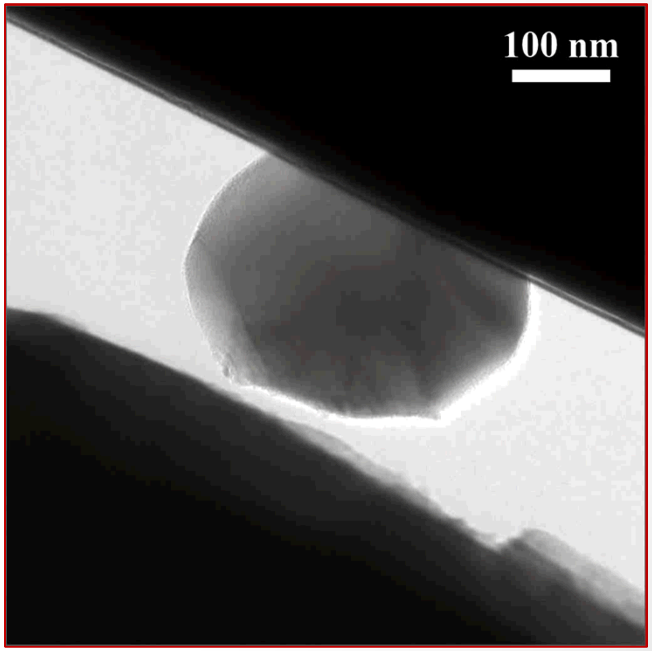




Flattened shapes and arrested cracks suggest plasticity.

Summary & Conclusions

- Experiments enable estimates of contact stress, strain, and fracture information, and observations of internal defect behavior
- Substantial dislocation activity and shape change before fracture, and crack arrest suggest plasticity.



- Open questions
  - Strain rate?
  - Compression axis?
  - Kinetic energy?

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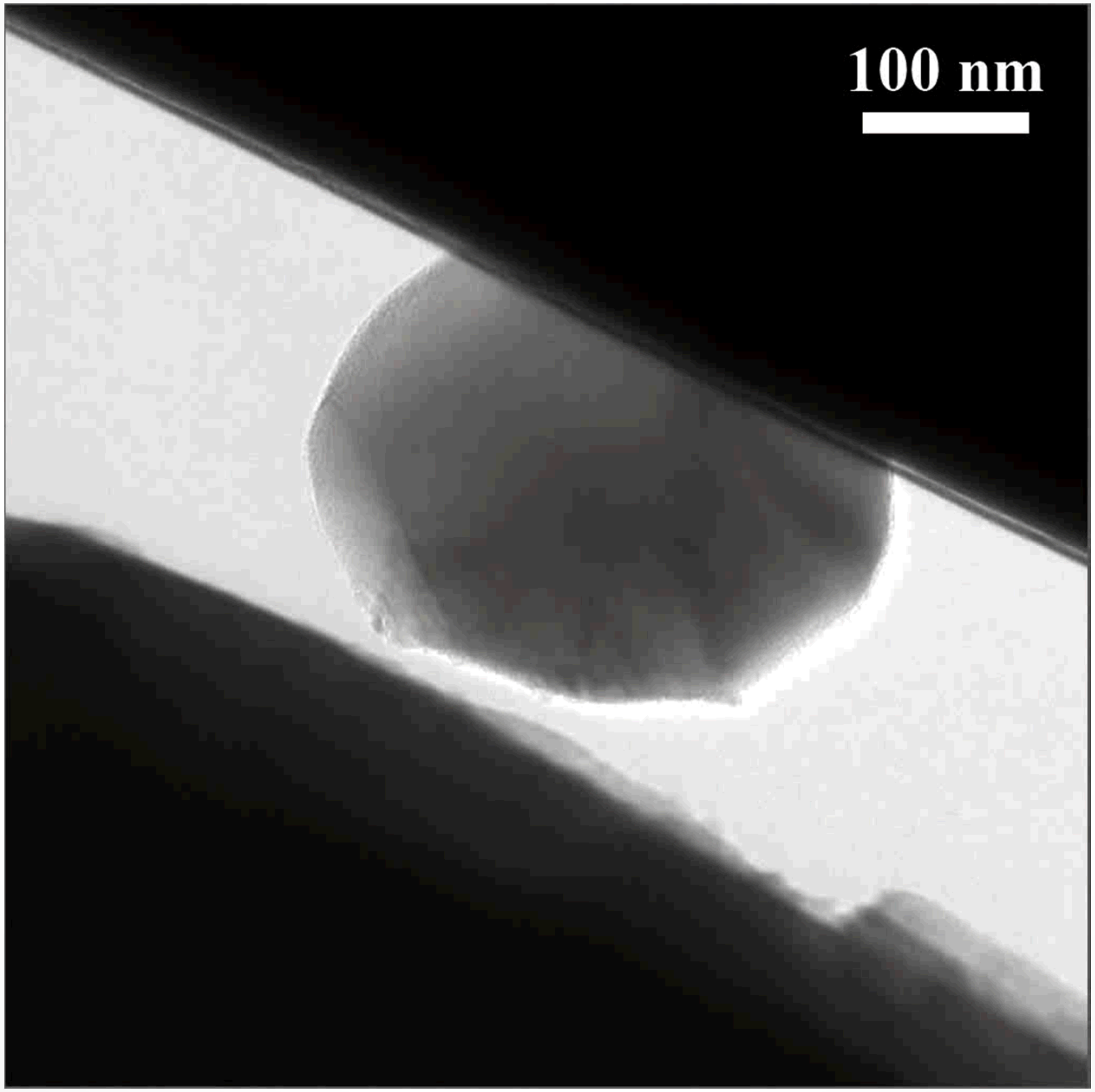
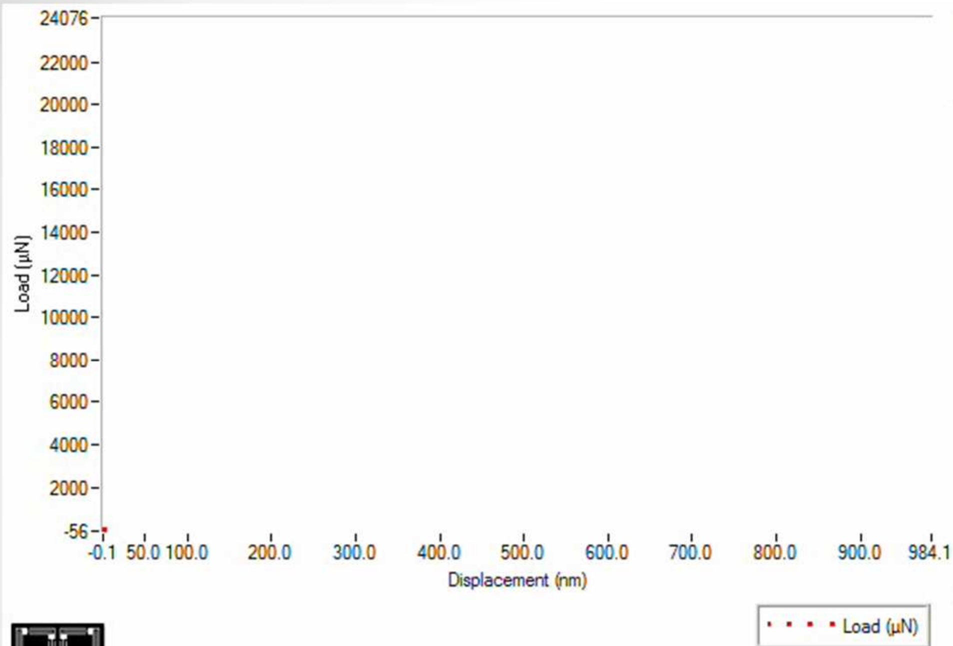
Particle Properties		Particle 1	Particle 2
Initial Particle Diameter (nm)		235	380
Indenter displacement during plasticity	(nm)	20.6	5.9
	Compression %	7.0	3.5
Indenter displacement during strain burst	(nm)	14.8	29.5
	Compression %	5.4	18.3
Load at first fracture (µN)		240	433
Maximum contact stress (GPa)	Before burst	12.6	14.4
	After burst	7.2	11.7
Stored Energy prior to first fracture (pJ)		8.9	16.9
Stored Strain Energy prior to first fracture per unit volume (GJ/m^3)		1.3	0.6
Estimated Energy Released during Displacement Burst (pJ)		3.9	3.9
Estimated Strain Energy Release Rate (J/m^2)		45	17


100 nm

In Situ SEM micro-compression – 3.0 µm

Displacement control, Strain rate ~ 0.003 s^{-1}



HYSITRON™

- Compressed 4 particles
- No observable shape change prior to fracture and fragmentation
- Displacement excursion corresponded to a fast fracture event
- Strain Energy Density before Fracture ~203 MJ/m^3
- Strain at fracture ~7%

Tip could not keep up with large displacement gained during fracture.

In Situ SEM micro-compression – 0.3 μm

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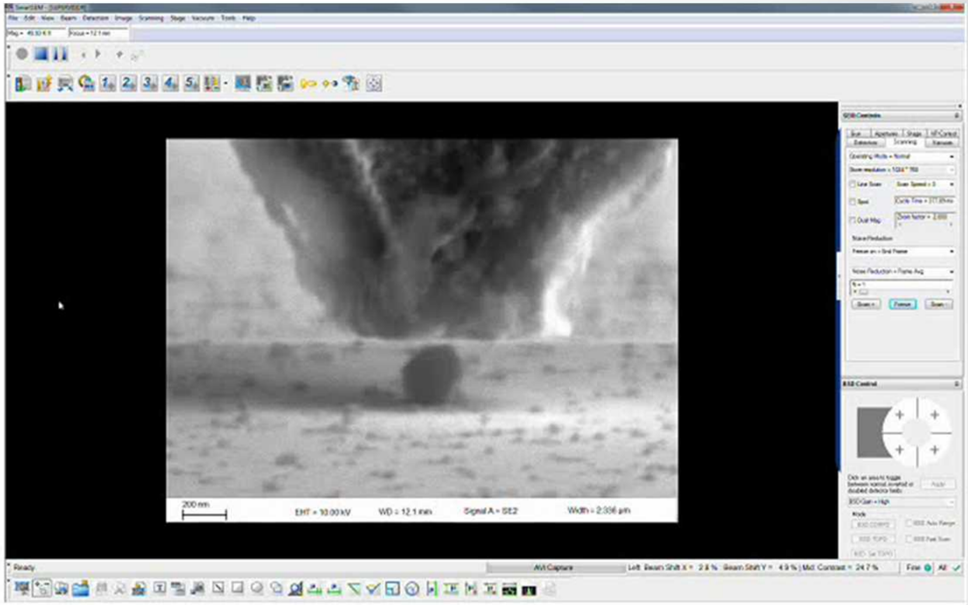
Displacement control, Strain rate ~ 0.05 s^{-1}



HYSITRON™

- Compressed 4 particles
- Significant plastic deformation/ shape change and stayed intact
- Displacement excursion corresponded to??? *Ex situ observation*
- Strain Energy Density before displacement excursion ~675 MJ/m^3
- Strain at displacement excursion ~16%

Tip could not keep up with large displacement gained during fracture.