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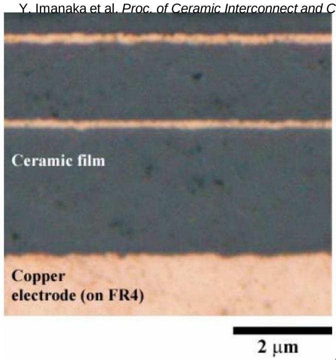
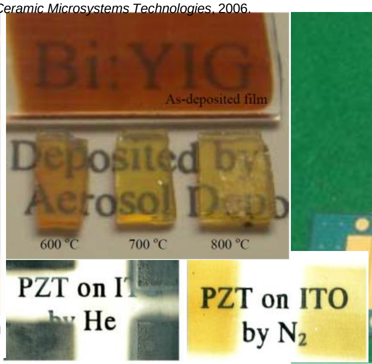
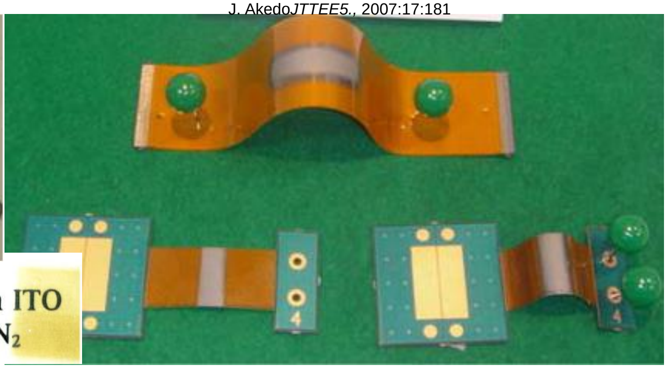
Y. Imanaka et al. *Proc. of Ceramic Interconnect and Ceramic Microsystems Technologies*, 2006.

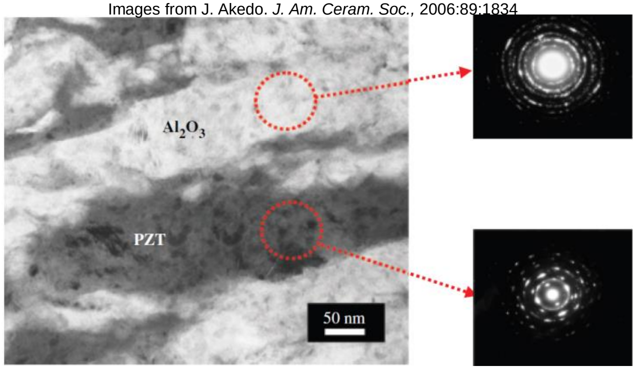




J. Akedo *JTTEE5.*, 2007:17:181



Images from J. Akedo. *J. Am. Ceram. Soc.*, 2006:89:1834



Deformation Behaviors of Sub-micron and Micron Sized Alumina Particles in Compression

Pylin Sarobol, Michael E. Chandross, Jay D. Carroll, William M. Mook, Brad L. Boyce, Paul G. Kotula, Bonnie B. McKenzie, Daniel C. Bufford, and Aaron C. Hall

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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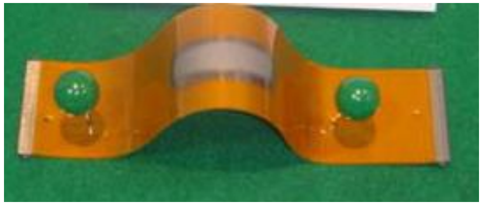
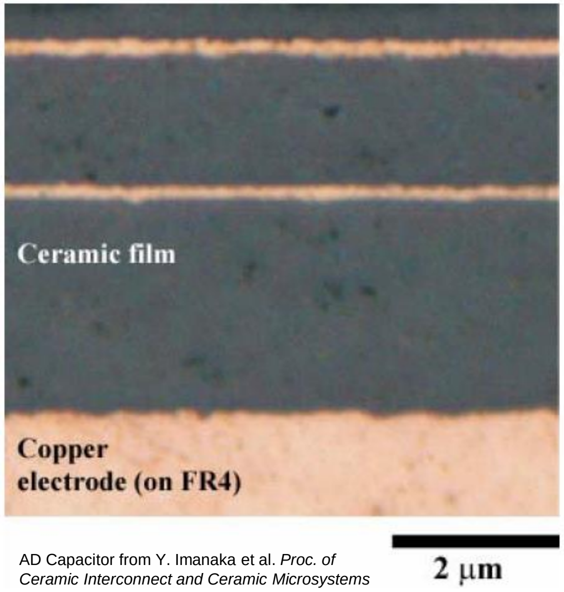
Fabrication of **Ceramic** Films at **Room Temperature**?

Motivation: Conventional ceramic fabrication involves high temperatures, limiting integration.

Current State-of-the-art: Researchers demonstrated a room temperature (RT) deposition process for engineering and electronic ceramics on plastic and glass substrates. *Mechanisms **not** well understood.*



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



AD Capacitor from Y. Imanaka et al. *Proc. of Ceramic Interconnect and Ceramic Microsystems Technologies*, 2006.

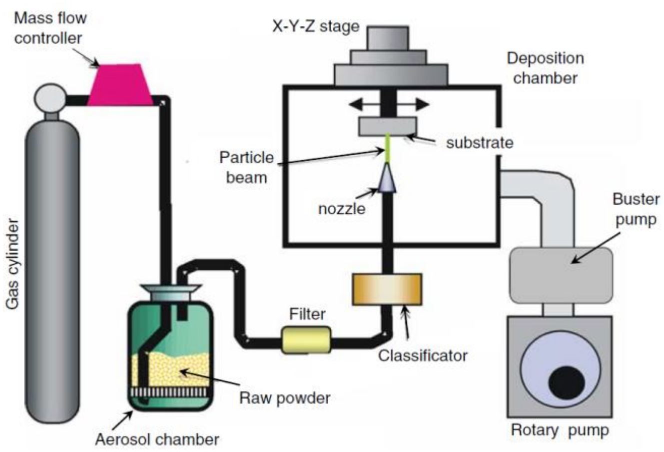
Fabrication of **Ceramic** Films at **Room Temperature**?

Motivation: Conventional ceramic fabrication involves high temperatures, limiting integration.

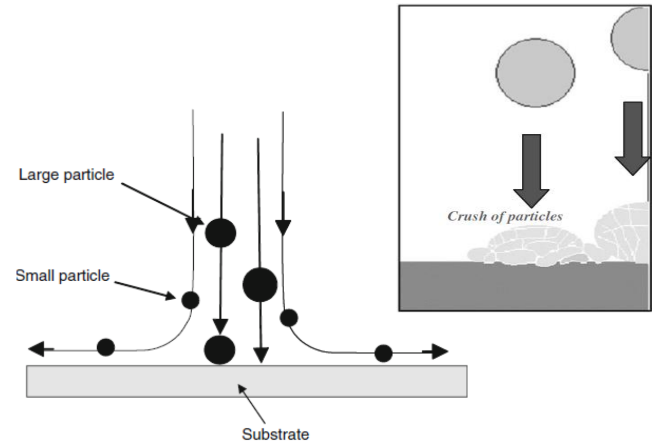
Current State-of-the-art: Researchers demonstrated a room temperature (RT) deposition process for engineering and electronic ceramics on plastic and glass substrates. *Mechanisms **not** well understood.*

Background: Sub-micron sized ceramic particles are accelerated to high velocity by pressurized gas, impacted, *plastically deformed*, and consolidated to form a dense film on the substrates at room temperature in vacuum, at high rates (10-30μm/min).

Goal: Pursue underlying fundamental mechanisms for sub-micron sized ceramic particle deformation and consolidation in a RT deposition process.



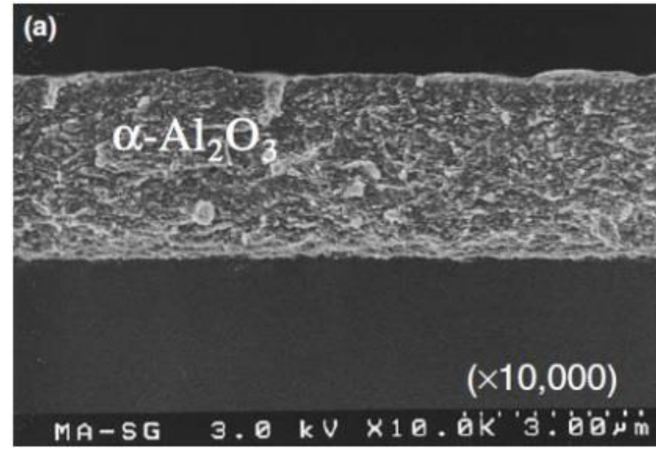
Particle velocity and gas consumption for a nozzle of 10mm x 0.4mm from . Akedo *J. Am. Ceram. Soc.*, 2006;89:1834.



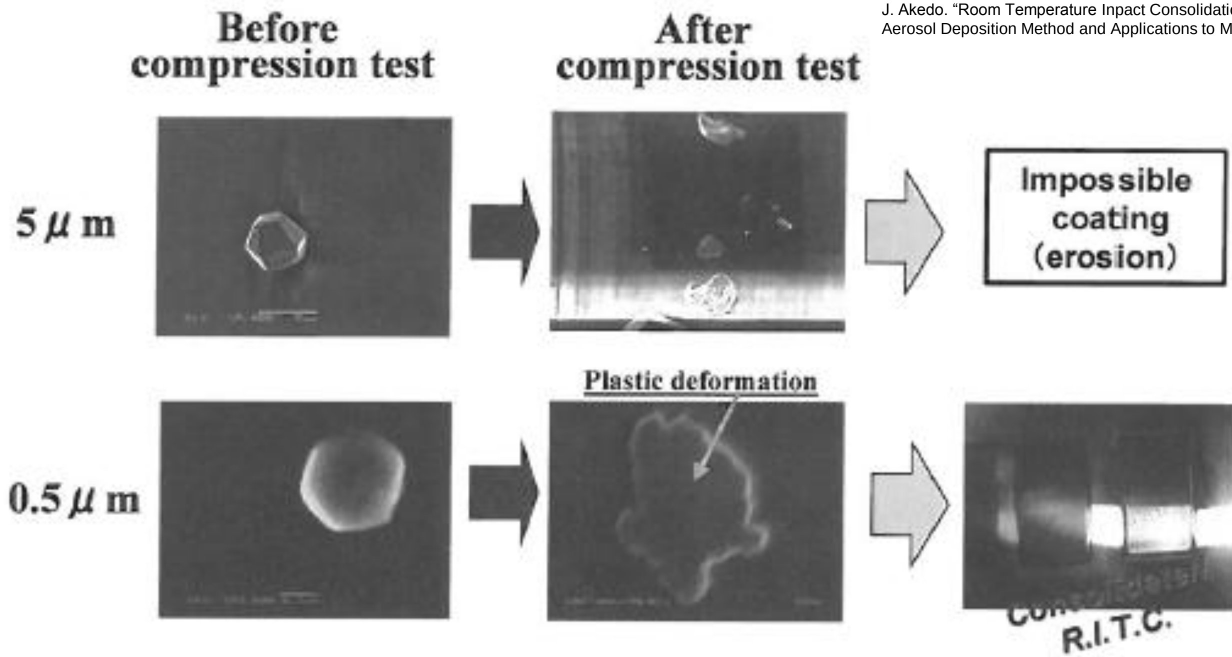
J. Akedo. "Room Temperature Impact Consolidation (RTIC) of Fine Ceramic Powder by Aerosol Deposition Method and Applications to Microdevices." *JTTEE5.*, 2007:17:181

Plastic Deformation of Small Scale Ceramics

- Common mechanisms for plastic deformation in small scale ceramics.
- Small particles deform/fracture
- Size → ductile-to-brittle transition



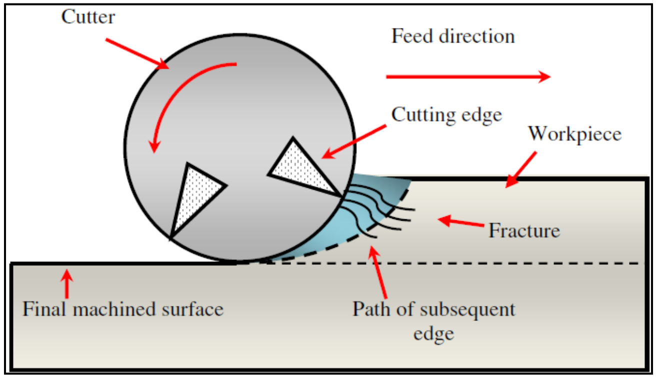
J. Akedo. "Room Temperature Inpact Consolidation (RTIC) of Fine Ceramic Powder by Aerosol Deposition Method and Applications to Microdevices." *JTTEE5.*, 2007:17:181

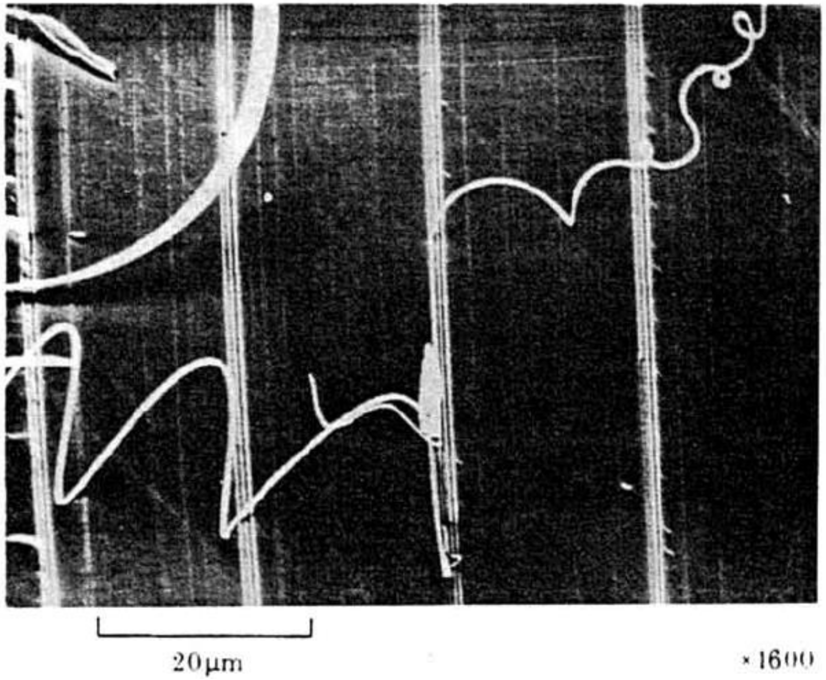


J. Akedo and H. Ogiso, "Room Temperature Impact Consolidation (RTIC) of Ceramic Fine Powder on Aerosol Deposition," Journal of Thermal Spray Technology, Volume 17, 2008, pp. 181-198.

Plastic Deformation of Small Scale Ceramics

- Common mechanisms for plastic deformation in small scale ceramics.
- Ductile-regime machining of ceramics
- Griffith's theory*:
  - a crack in a remote uniform tensile stress filed.
  - Balance of decrease in stored elastic energy and the increase in surface energy from crack.
  - Extension of crack only if surface energy is supplied externally



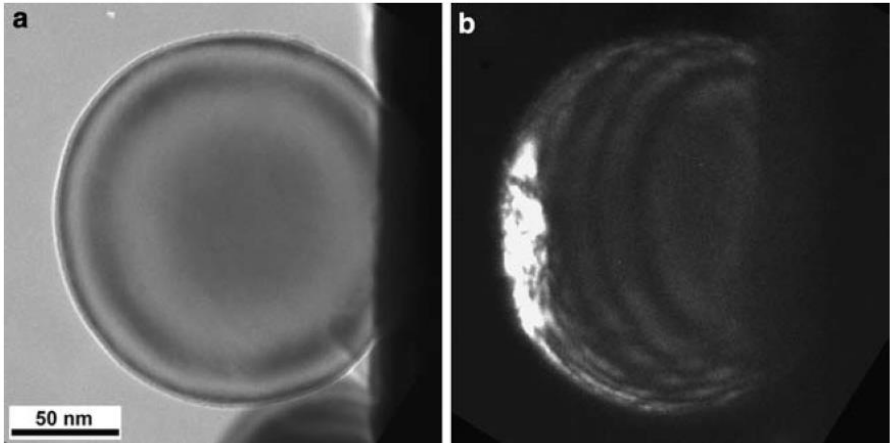


R. Komanduri, D.A. Lucca, and Y. Tani, Annals of the CIRP Vol. 46/2/1997

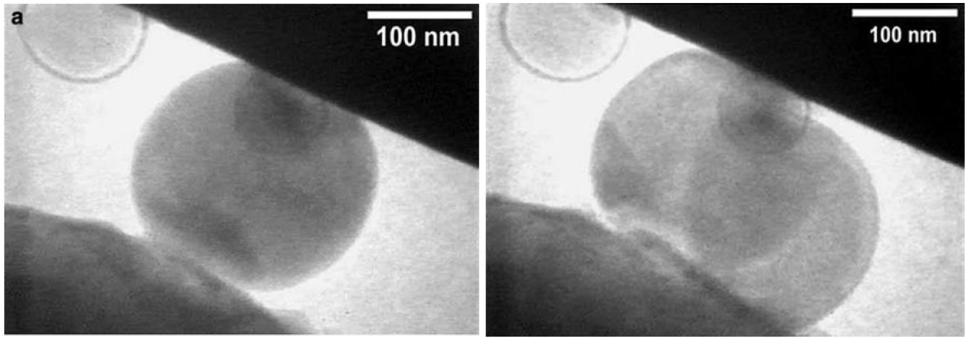
*Watchman JB, Cannon WR, Matthewson MJ (2008) Mechanical properties of ceramics. Willey, New York and Griffith AA (1924) The phenomena of rupture and flow in solids. In: Proceedings of the first international congress on applied mechanics. ICTAM, Delft, p 55

Plastic Deformation of Small Scale Ceramics

- Common mechanisms for plastic deformation in small scale ceramics.
- Ductile-regime machining of ceramics
- Compressed single crystal Si particles
  - Dislocation slip and fracture



Bright field image of a particle prior to deformation and Dark field image of after compression showing plastic deformation

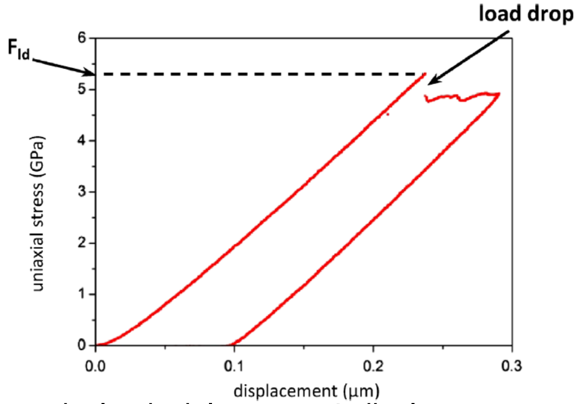


Just before the moment of fracture large strain fields are present in the particle which partially disappeared upon release. With further compression, particle fractured.

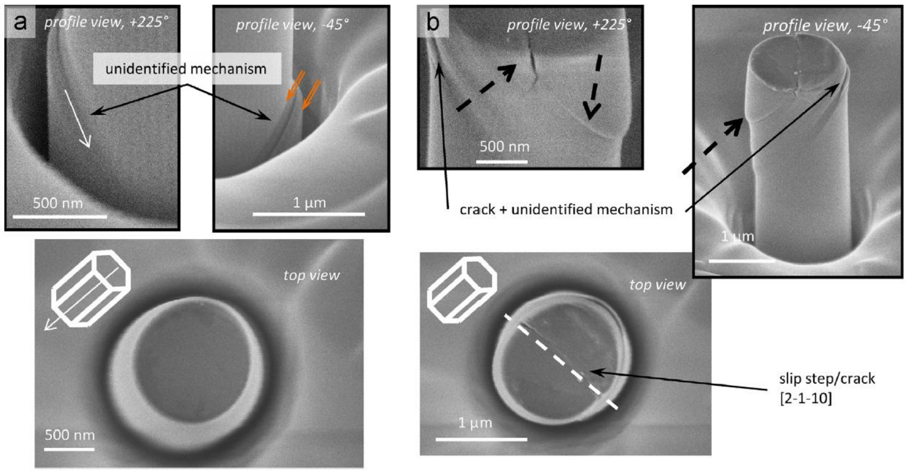
J. Deneen, W.M. Mook, A. Minor, W.W. Gerberich, C.B. Carter. J Mater Sci 41 (2006) 4477-4483

Plastic Deformation of Small Scale Ceramics

- Common mechanisms for plastic deformation in small scale ceramics.
- Ductile-regime machining of ceramics
- Compressed single crystal Si particles
- Compressed single crystal sapphire pillars:
  - Dislocation slip (8 planes leading to 17 slip systems).
  - Twinning (basal & rhombohedral systems).
  - Cracking



Typical uniaxial stress VS displacement curve on a sapphire pillar with a load drop.

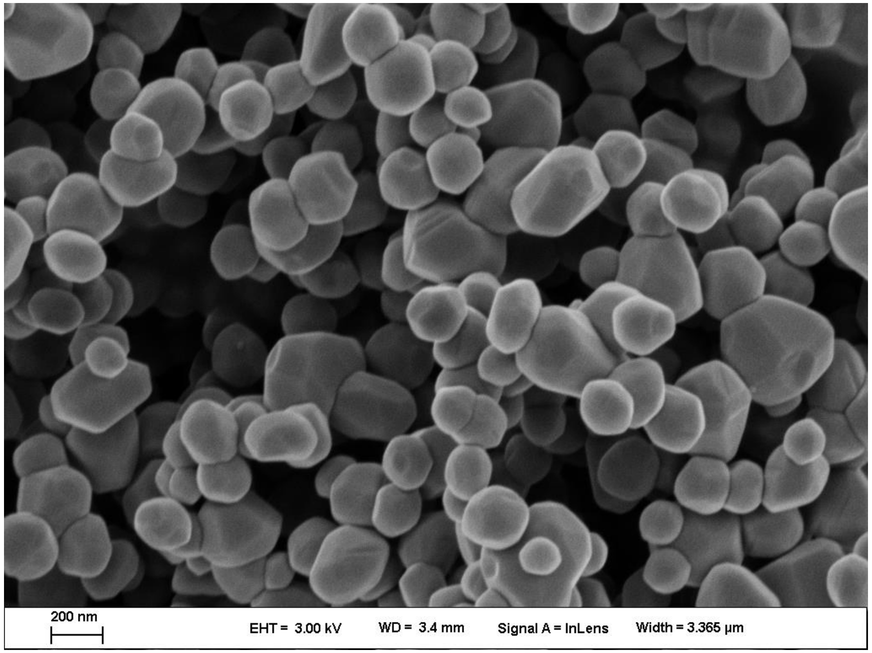


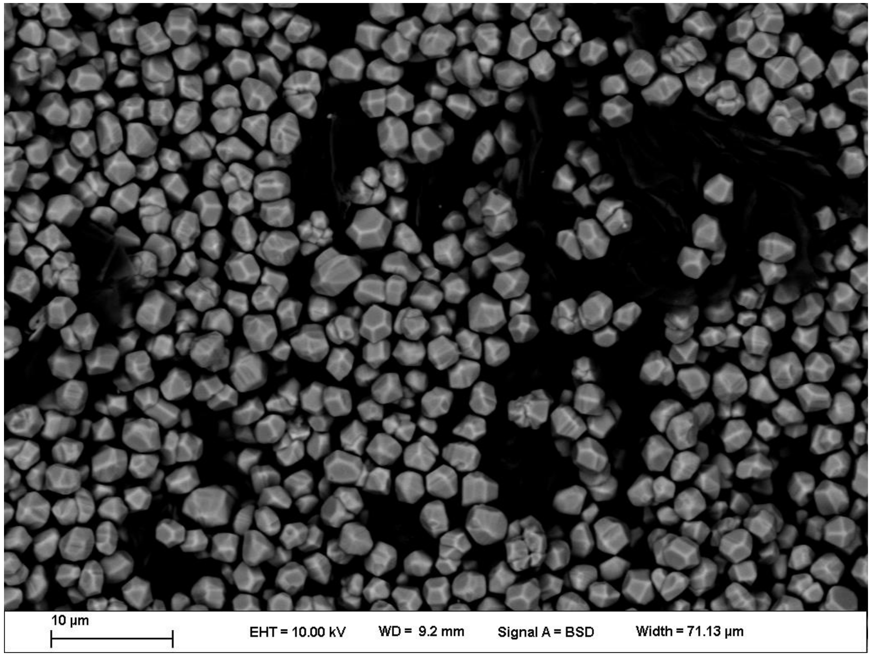
SEM images of sapphire pillars after compression on the R plane showing slip step and cracks.

A. Montagne, S. Pathak, X. Maeder, and J. Michler, *Ceram. Int*. 40 (2014) 2083-2090.

Alumina particles

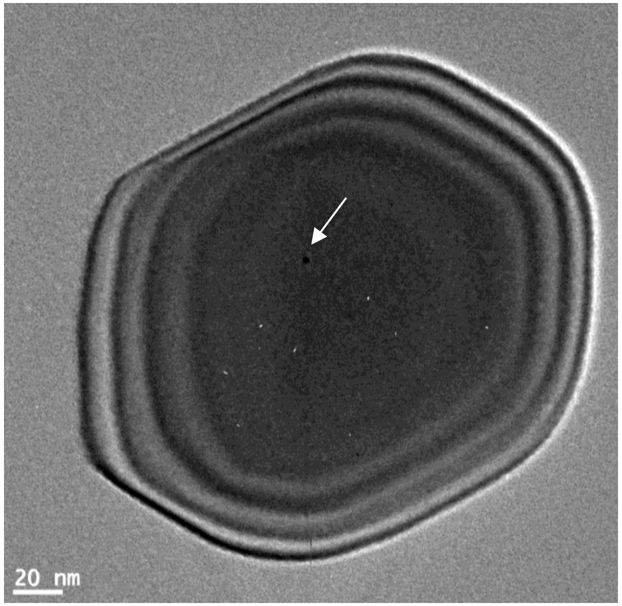
- Single crystal, ultra pure 0.3µm & 3µm, α-Al_2O_3 particles

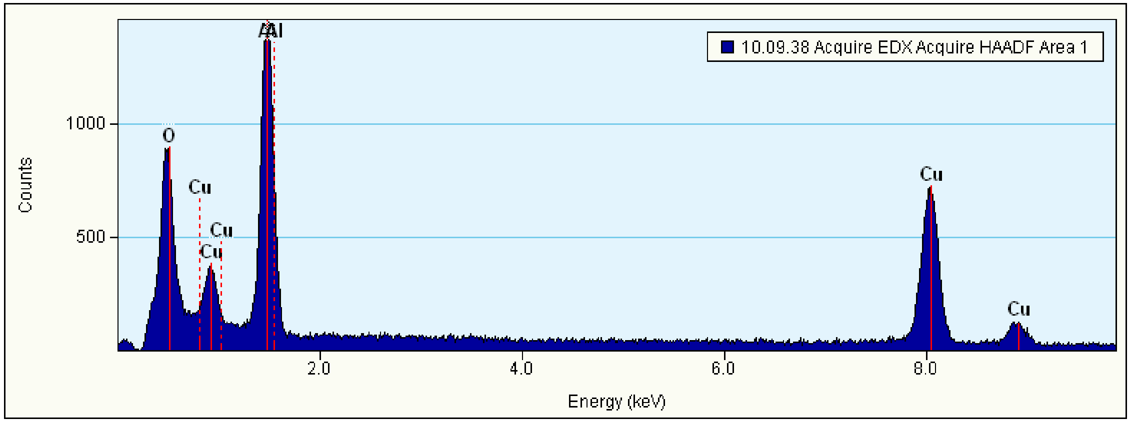


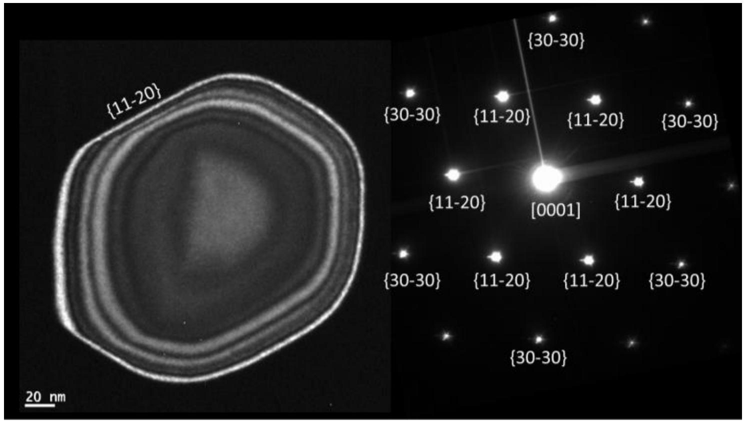


Alumina particles

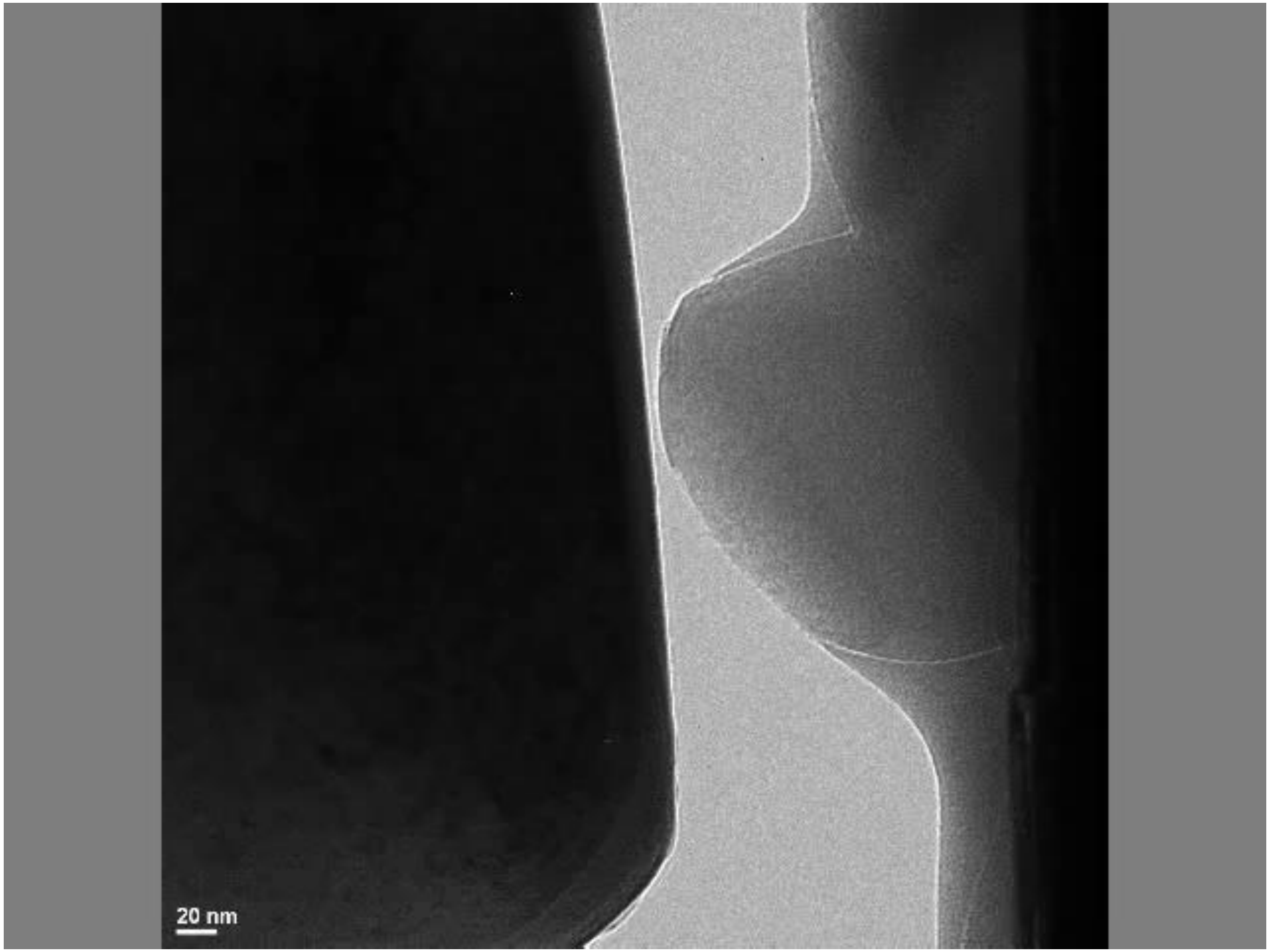
- Single crystal, ultra pure 0.3µm & 3µm, α-Al_2O_3 particles
- XRD confirmed corrundum
- TEM confirmed purity, relatively defect-free single crystals





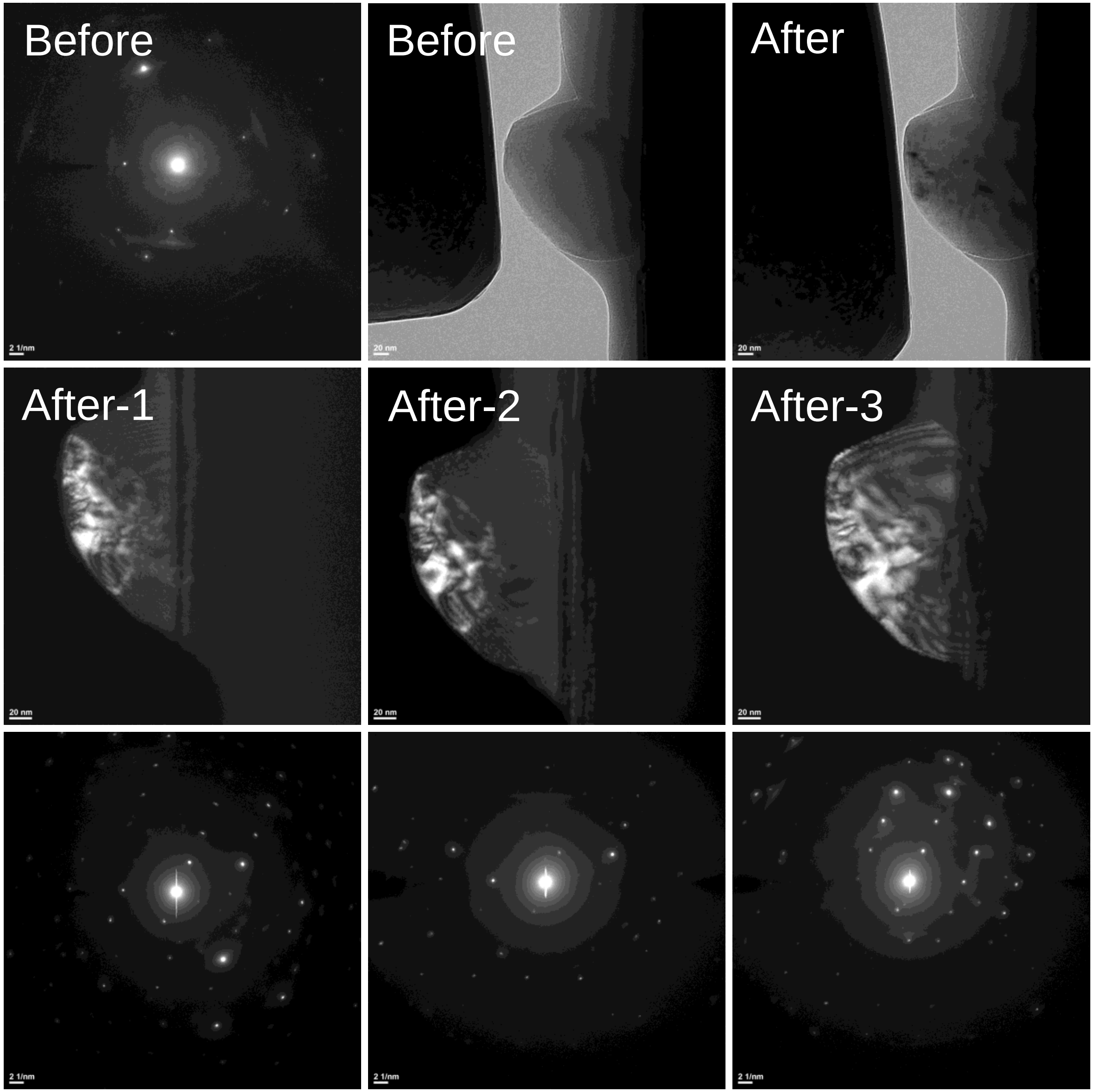


In situ compression in the TEM, 0.3μm



Sub-micron sized Alumina particles exhibited ductile behavior in compression.

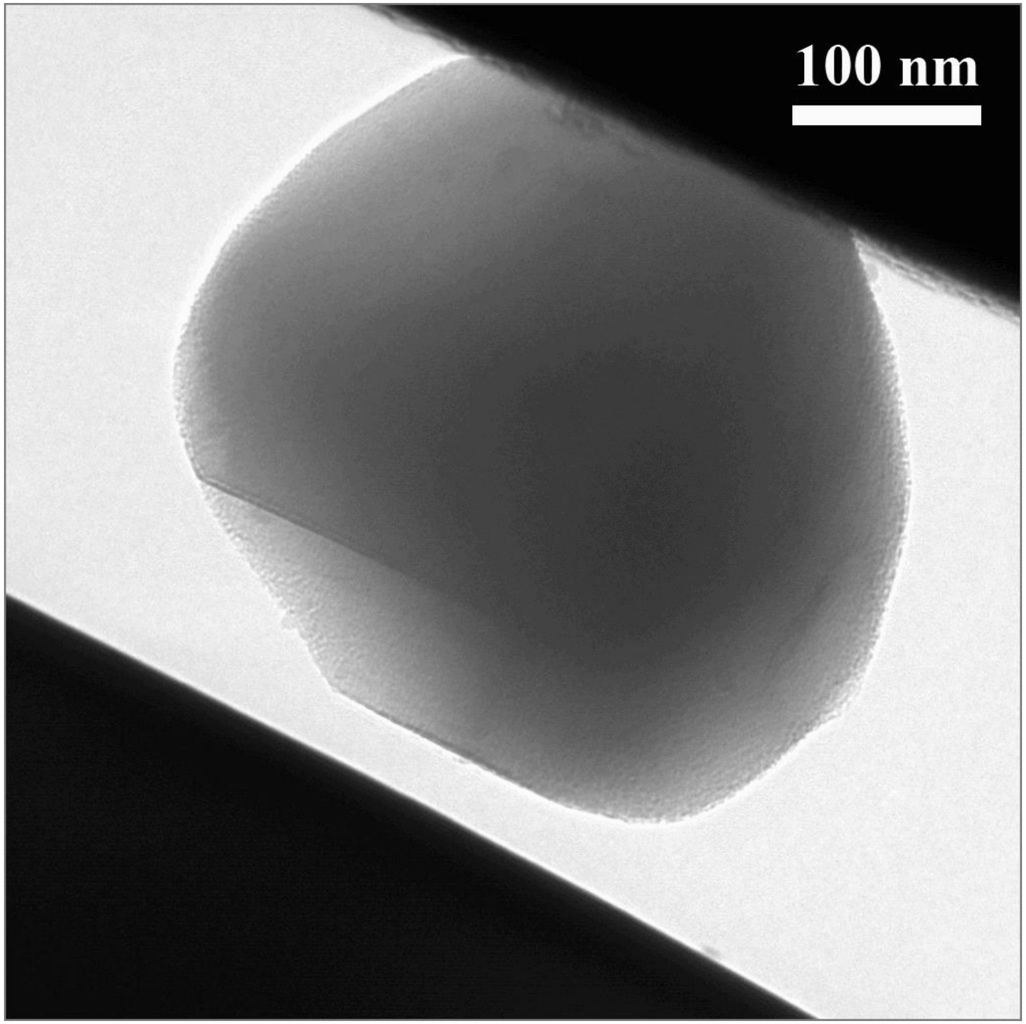
In situ compression in the TEM, 0.3µm



- 0.3µm particle
- Plastic deformation
- Pockets of high dislocation density
- Fracture?
- Became polycrystalline
- Intact

Sub-micron sized Alumina particles exhibited ductile behavior in compression.

In situ compression in the TEM, 0.3µm



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In situ compression in the TEM, 0.3μm

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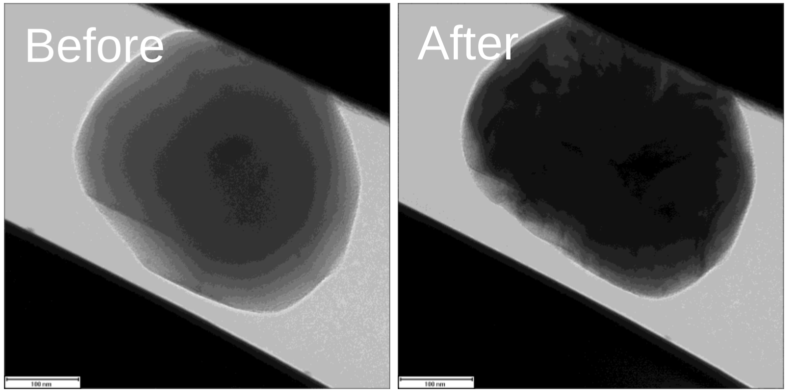
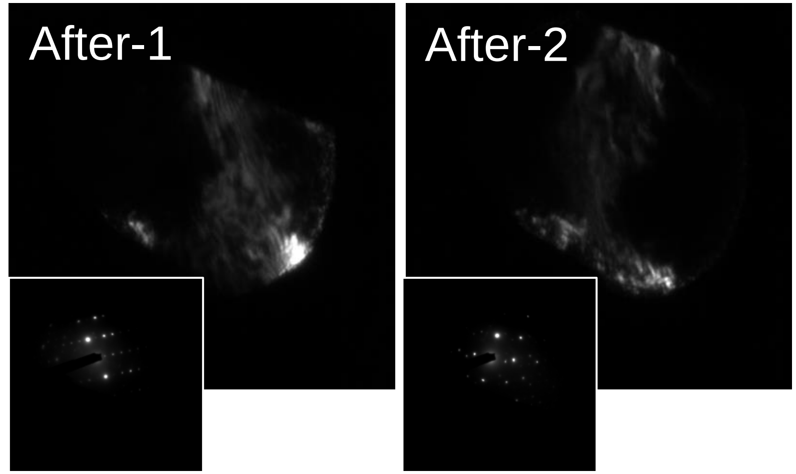
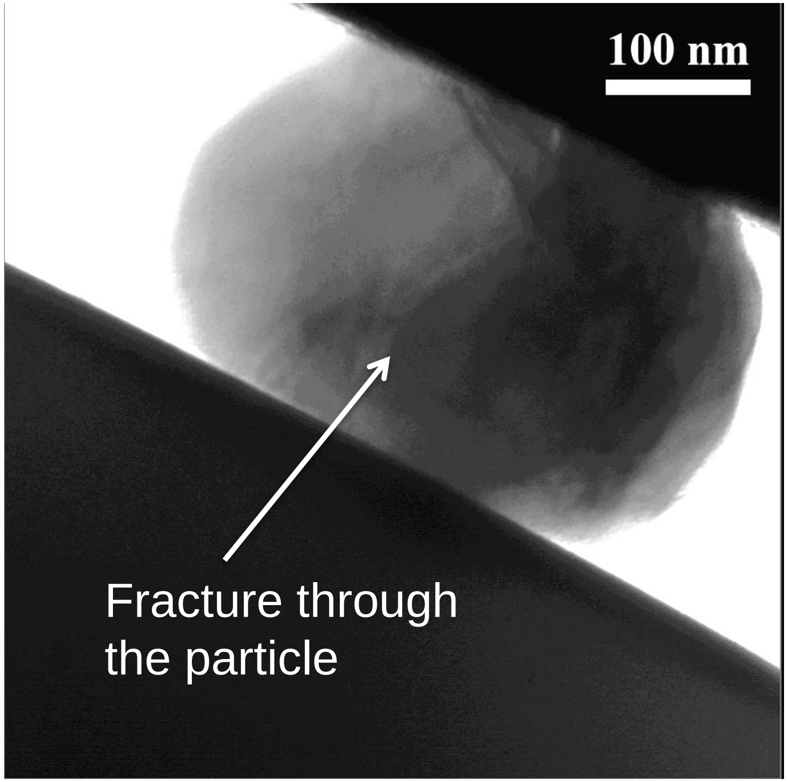


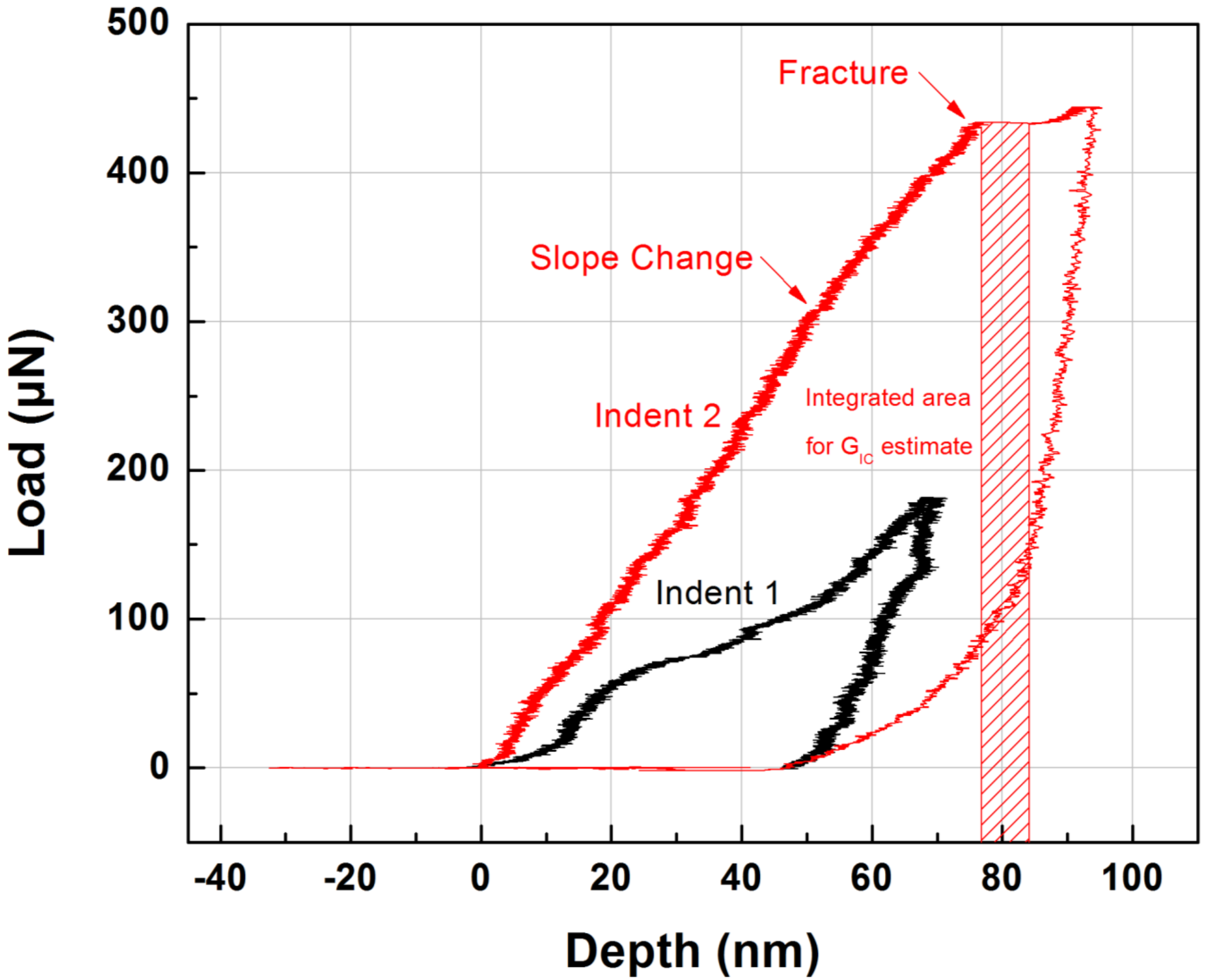




- 0.3μm particle
- Plastic deformation
- Pockets of high dislocation density
- Macro-fracture in half
- Micro-fracture?
- Became polycrystalline
- Intact

Sub-micron sized Alumina particles exhibited ductile behavior in compression.

In situ compression in the TEM, 0.3µm



Energy – area under displacement burst for G_{IC} estimation:

7nm*435uN=305pJ,
diameter ~ 370 nm,
area $\pi * r^2 = 1.1e\text{-}13\ m^2$,
$G_{IC} = (305pJ)/(2*1.1e\text{-}13 m^2)$
$= 1386\ J/m^2$.

Sub-micron sized Alumina particles exhibited ductile behavior in compression.

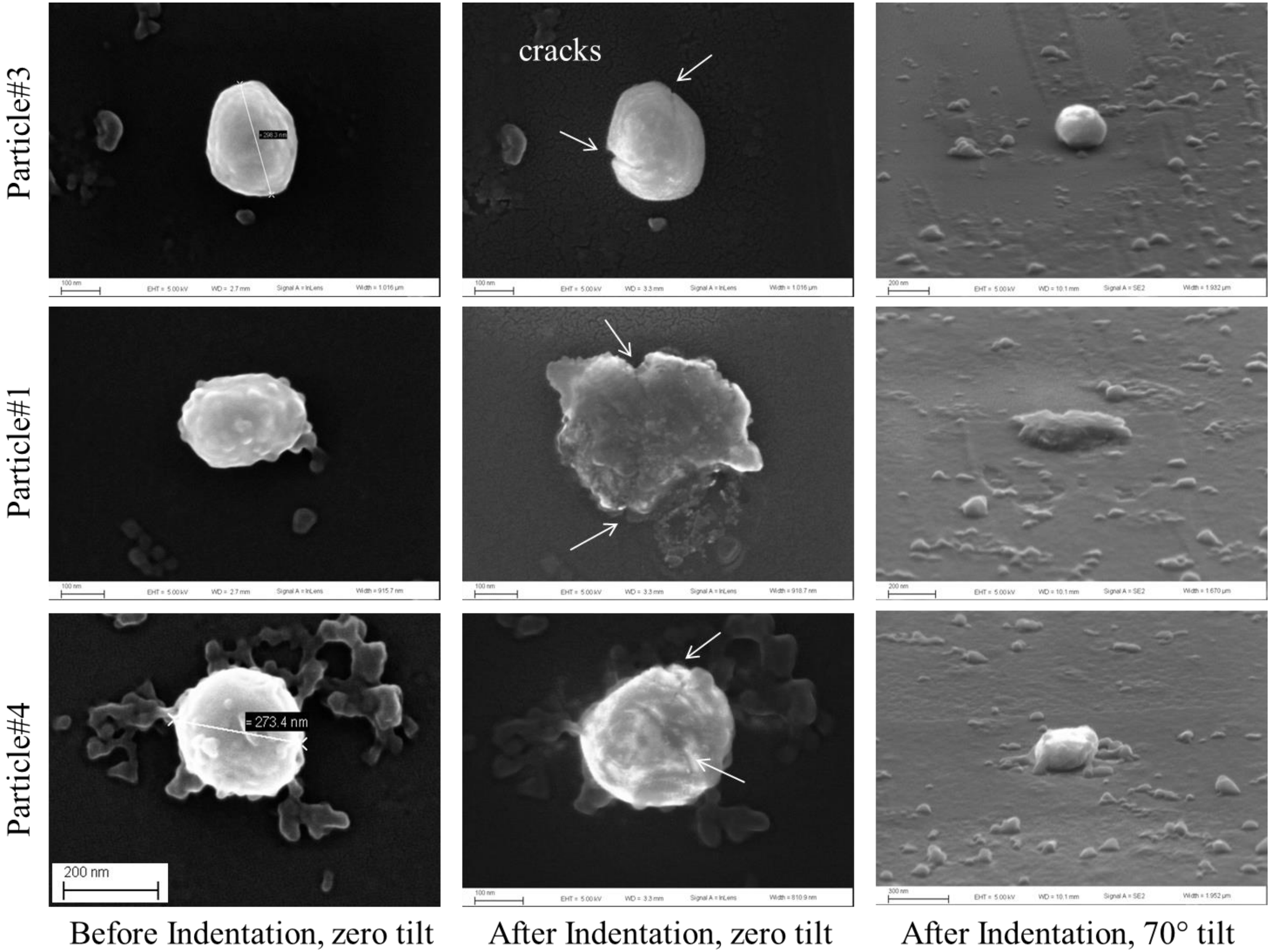
Strain Energy Release Rate

- G_{IC} = (305pJ)/(2*1.1e-13m^2)
  = 1.4 kJ/m^2.
- Bulk alumina G_{IC}= 0.04 kJ/m^2.
- This fracture energy is much higher than bulk ceramics.

Material	$G_{Ic}(\mathrm{kJm}^{-2})$	$K_{Ic}(\mathrm{MNm}^2)$	$E(\mathrm{GPa})$
Steel alloy	107	150	210
Aluminum alloy	20	37	69
Polyethylene	20 (J_{Ic})	—	0.15
High-impact polystyrene	15.8 (J_{Ic})	—	2.1
Steel — mild	12	50	210
Rubber	13	—	0.001
Glass-reinforced thermoset	7	7	7
Rubber-toughened epoxy	2	2.2	2.4
PMMA	0.5	1.1	2.5
Polystyrene	0.4	1.1	3
Wood	0.12	0.5	2.1
Glass	0.007	0.7	70

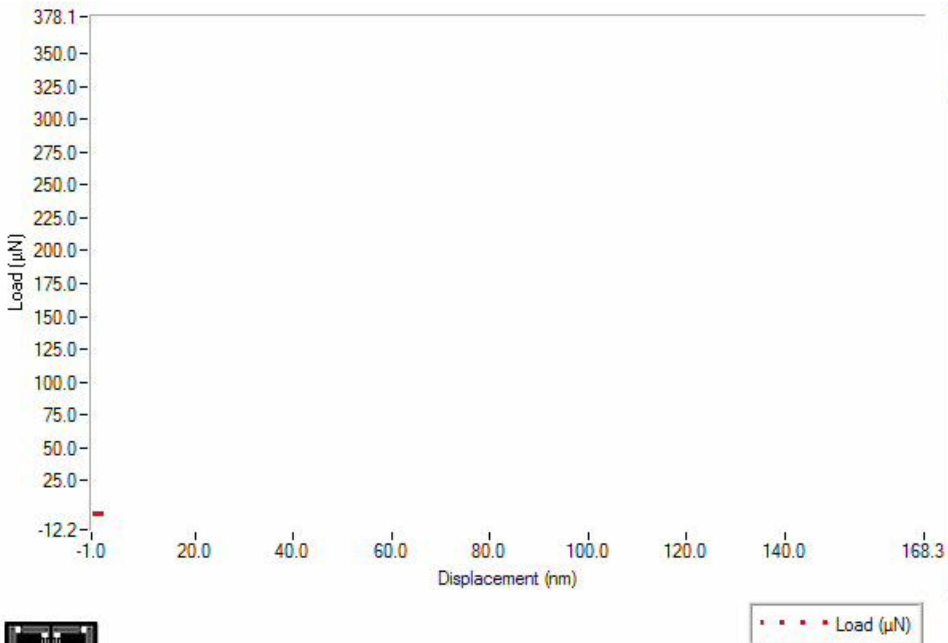
Roylance, D. Introduction to Fracture Mechanics, Massachusetts Institute of Technology: Cambridge, 2001.
http://ocw.mit.edu/courses/materials-science-and-engineering/3-11-mechanics-of-materials-fall-1999/modules/frac.pdf

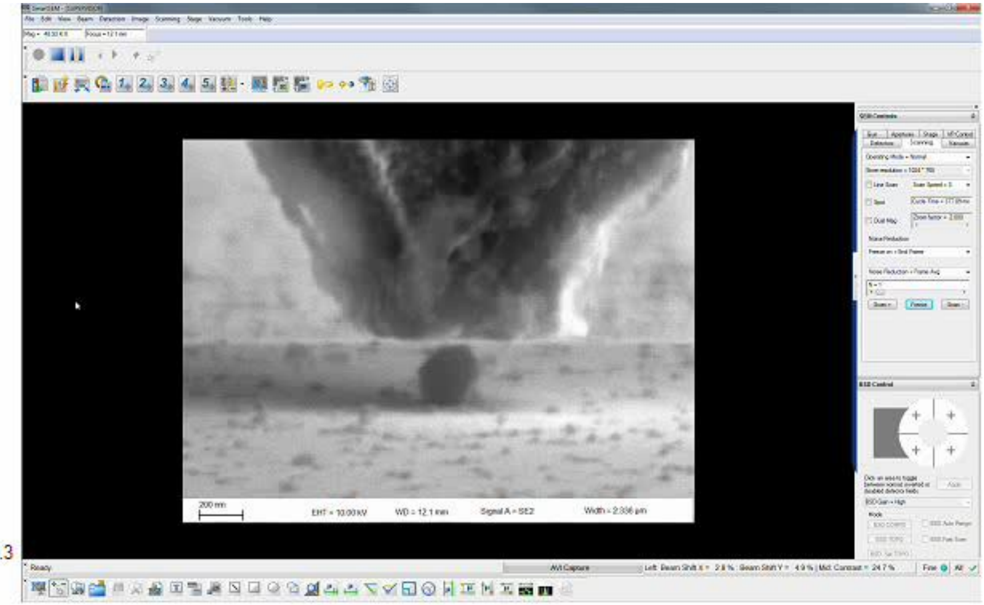
In situ compression in the SEM, 0.3μm



Sub-micron sized Alumina particles exhibited ductile behavior in compression.

In situ compression in the SEM, 0.3µm





HYSITRON™

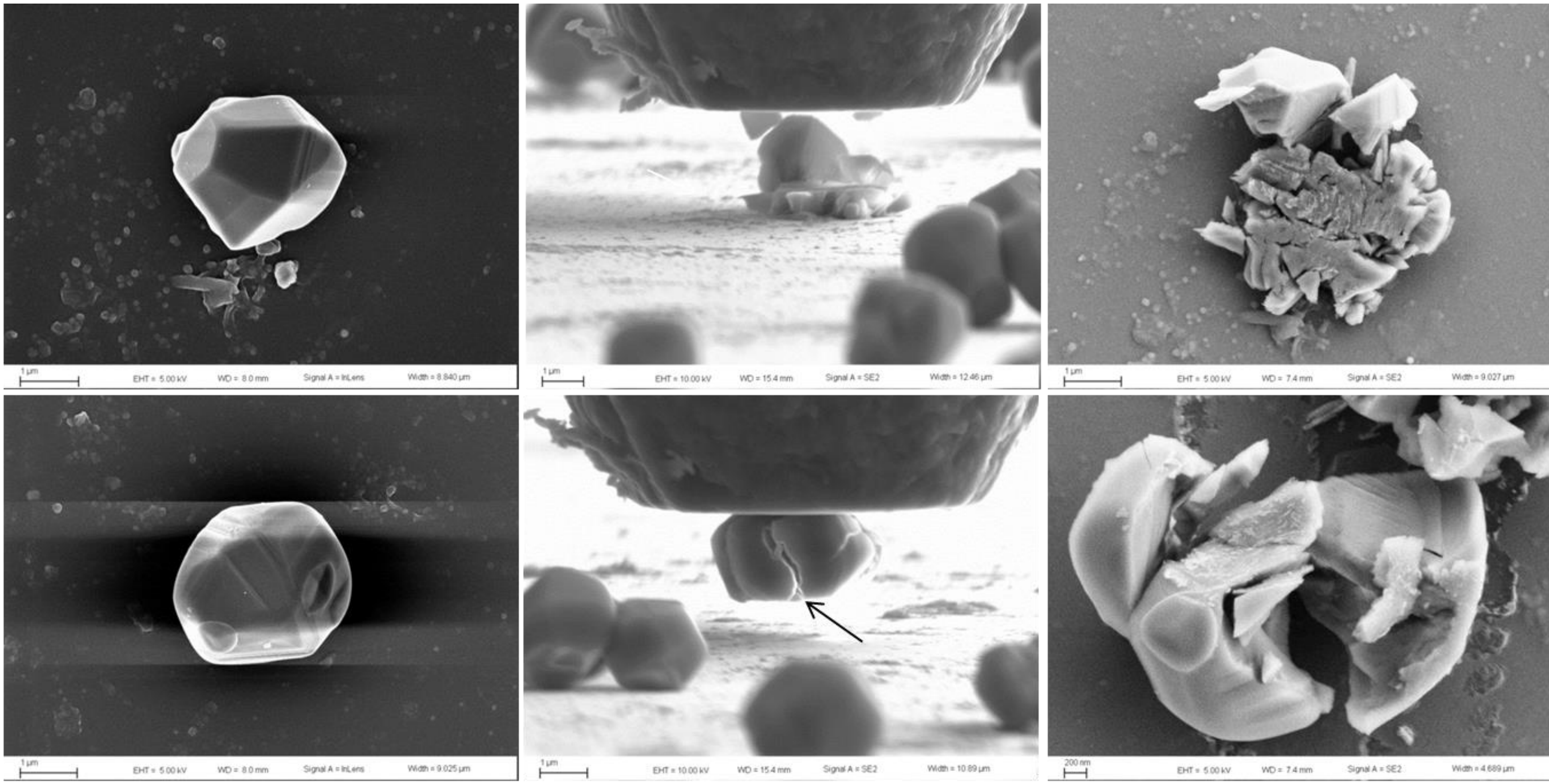
- Significant plastic deformation, change in shape, *fracture*
    - Pockets of high dislocation density
    - Became polycrystalline but stayed intact

Sub-micron sized Alumina particles exhibited ductile behavior in compression.

In situ compression in the SEM, 3μm

Particle#2

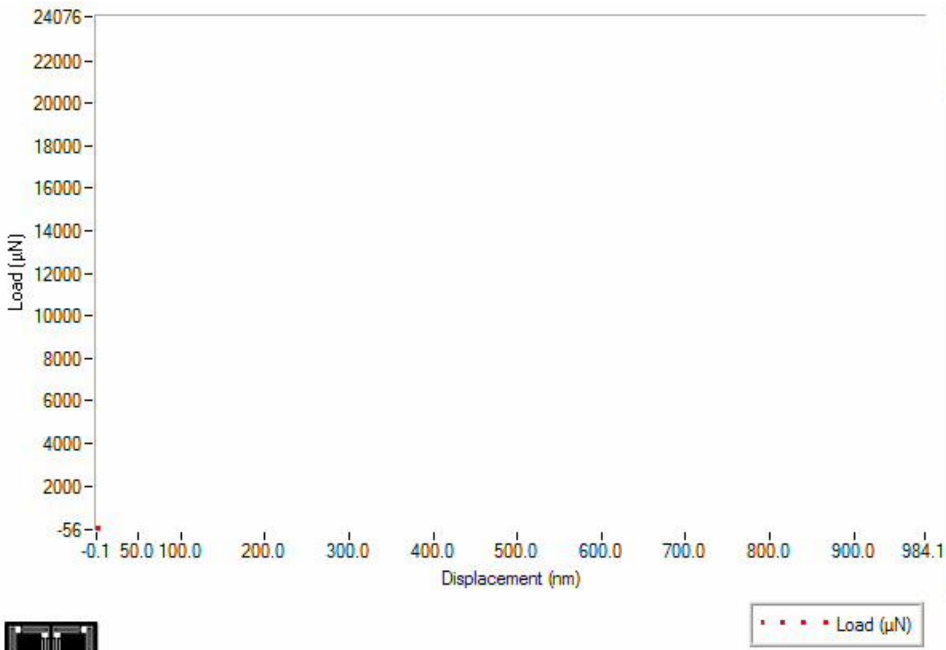
Particle#5



Before Indentation, zero tilt | After Indentation, 86° tilt | After Indentation, zero tilt

Micron sized Alumina particles exhibited brittle behavior in compression.

In situ compression in the SEM, 3μm



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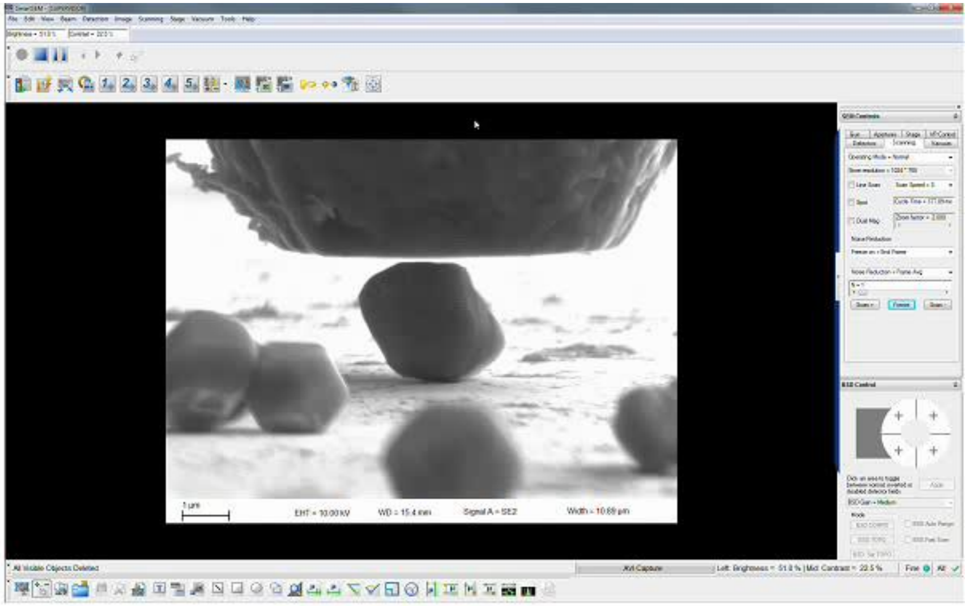
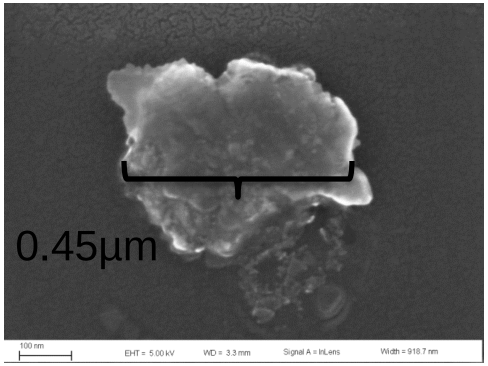


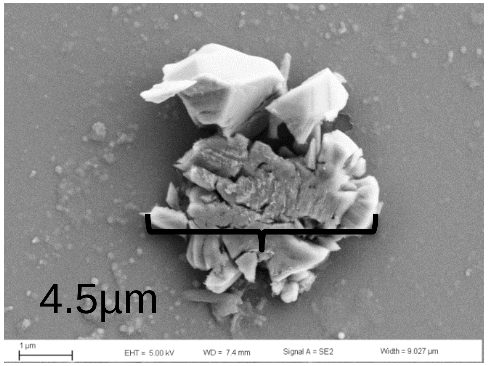
- Very little plastic deformation
- Fracture into pieces
- Brittle

Micron sized Alumina particles exhibited brittle behavior in compression.

Summary on deformation Behaviors - Experimentation

- Appearance
  - Ductile – change shape, fracture but intact
  - Brittle – fracture into pieces
- Strain to failure
  - 0.3 µm particles, ~13%
  - 3 µm particles, ~5%
- Strain energy release rate
  - 0.3µm particles in TEM
  - 1368 J/m^2 >> ceramics



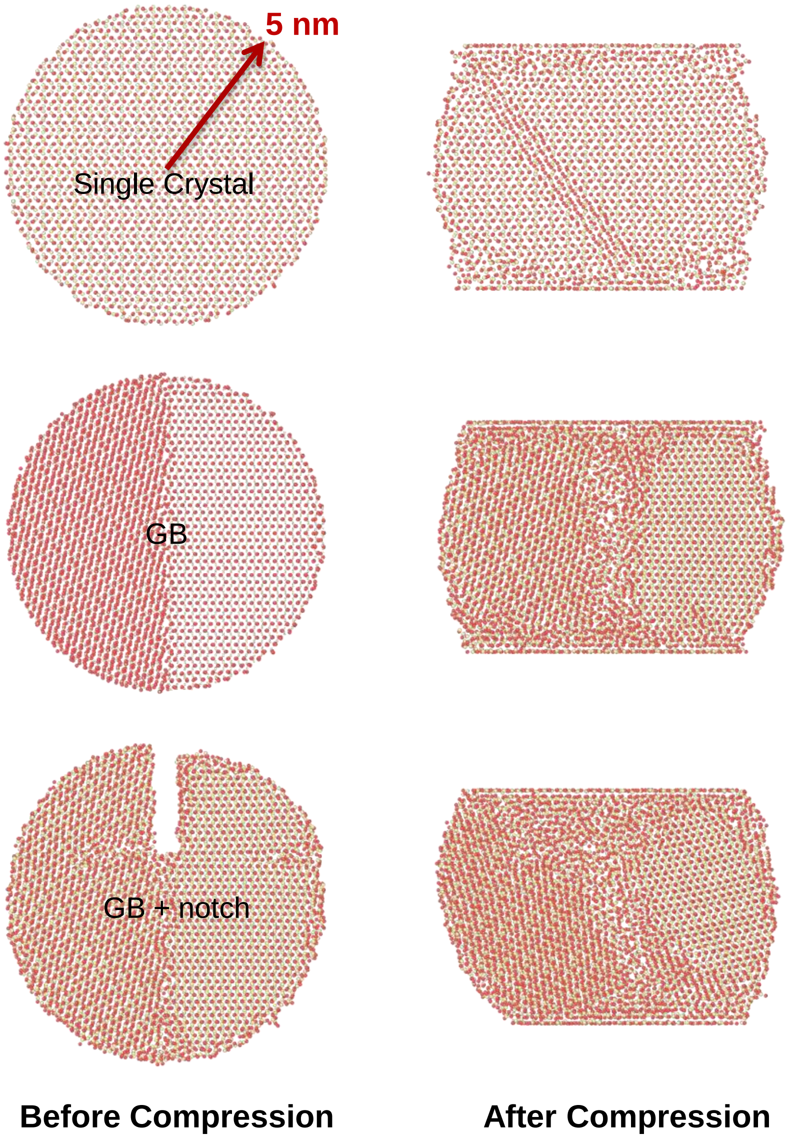


Particle Identifier	Diameter (µm)	Strain Rate (s^{-1})	Strain at First Fracture (%)
Large Particles			
D6P1	2.9	0.03	5
D6P2	2.6	0.006	5
D6P4	2.9	0.005	5
D6P5	2.9	0.003	7
Small Particles			
D5P2	0.17	0.09	11
D5P3	0.29	0.05	12
D5P4	0.28	0.05	13
D5P5	0.29	0.05	16

G_{IC} = (305pJ)/(2*1.1e-13m^2)
= 1386 J/m^2.
indicative of ***ductile fracture***.
Brittle ceramics are usually G_{IC}<10 J/m^2.

Summary on deformation Behaviors – Atomistic Simulations

- Molecular Dynamics
  - Assume large particles contain flaws → brittle. Small particles are defect-free → ductile
  - 3 types 10nm diameter particles- single crystal, GB, GB + notch
  - repulsive Lennard-Jones walls moved together at a constant velocity of 200 m/s. Compressed for an overall distance of 34 Å on particles then release.
- Ductile Behavior
  - Dislocation slip
  - Plastic deformation



Conclusions

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- The findings from the *in situ* compression experiments supported those from atomistic simulation: sub-micron sized Al_2O_3 particles exhibit ductile behavior in compression.
  - Dislocation slip, significant plastic deformation, fracture, polycrystalline.
- The micron sized Al_2O_3 particles exhibited brittle fracture in compression.
  - *In situ* compression experiments showed 3µm Al_2O_3 particles fractured into pieces.
- **Deformation mechanisms?**
- Use info to inform coating deposition parameters and particle-particle bonding in the consolidated coatings.