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Strength and Fracture Toughness of Ultrafine Grained Alumina Ceramic

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Outline

- Introduction
- Experimental Procedures
- Results and Discussion
 - Bending Process
 - Fracture Morphology
 - Statistical Analysis
 - Fracture Toughness
- Conclusions
- Acknowledgements



Introduction

■ Griffith's Theory

$$\sigma = \frac{K_{Ic}}{Y\sqrt{a}}$$

σ -- Flexure strength

K_{Ic} -- Fracture toughness

Y -- Stress intensity factor

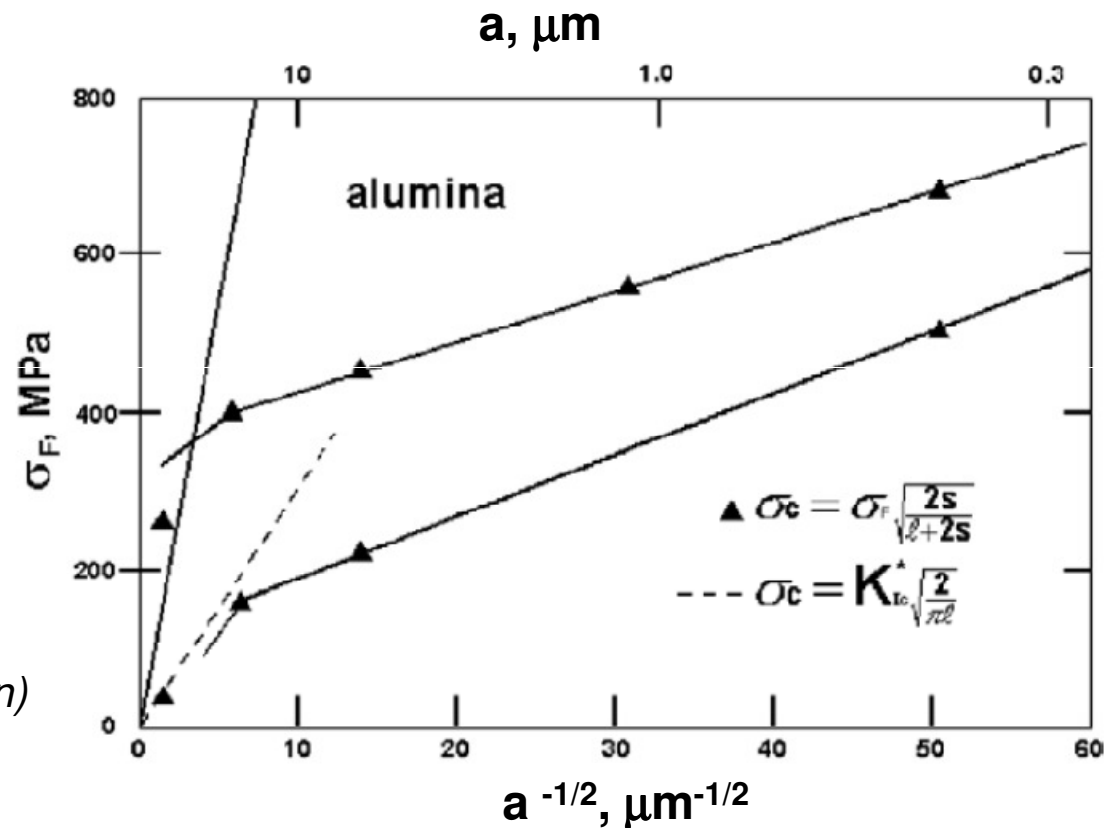
a -- Critical flaw size

$$\sigma = \sqrt{\frac{2E\gamma}{\pi a}} \quad (\text{Modified version})$$

E -- Elastic modulus

γ -- Surface energy

a -- Critical flaw size

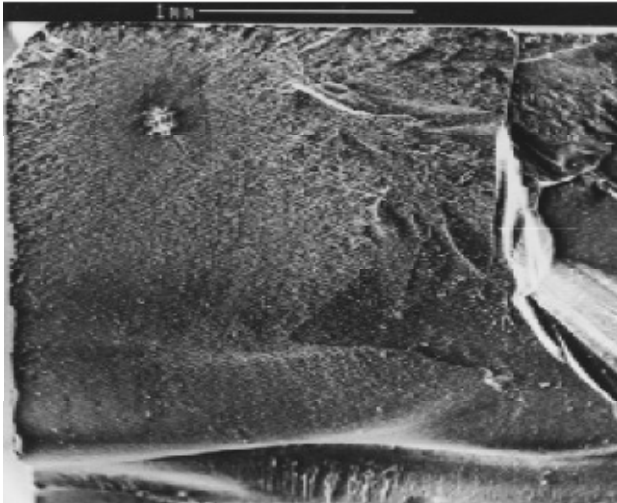


Bending strength dependence on grain size of alumina

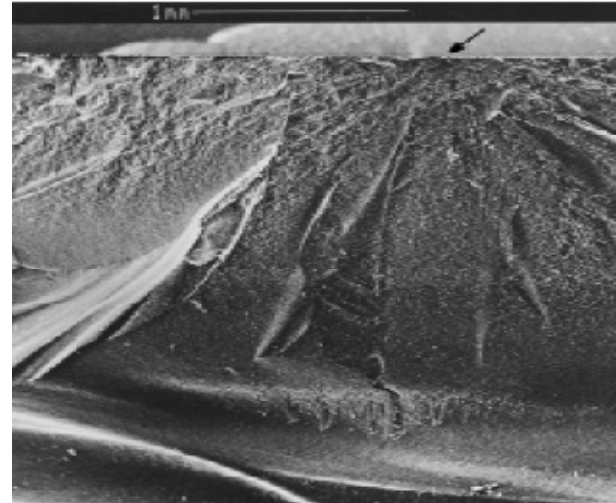


Introduction

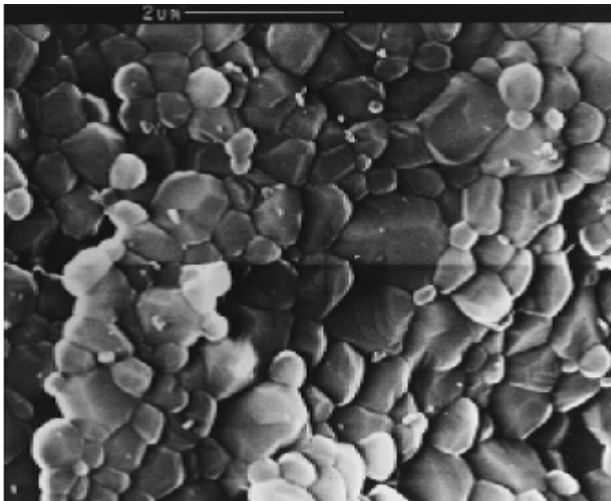
**Volume
Flaw**



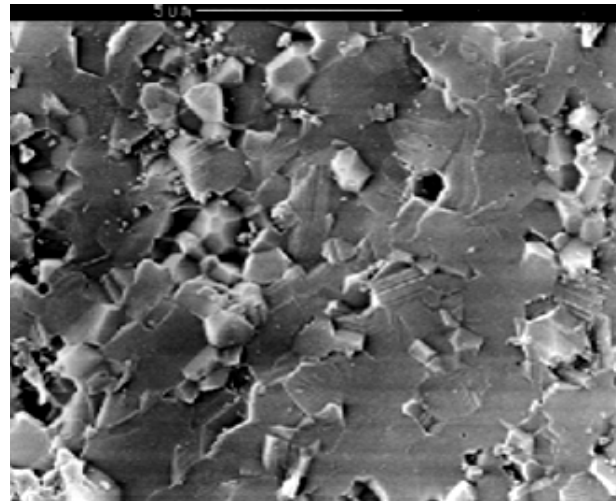
**Surface
Flaw**



**Intergranular
Fracture**



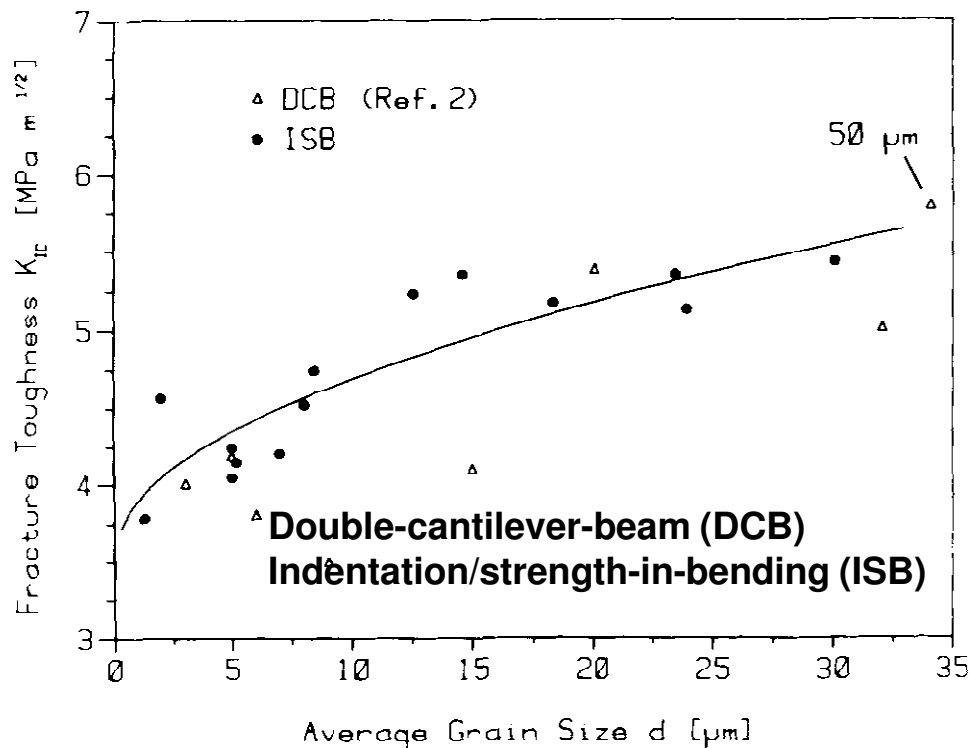
**Transgranular
Fracture
as well as
Intergranular
Fracture**





Introduction

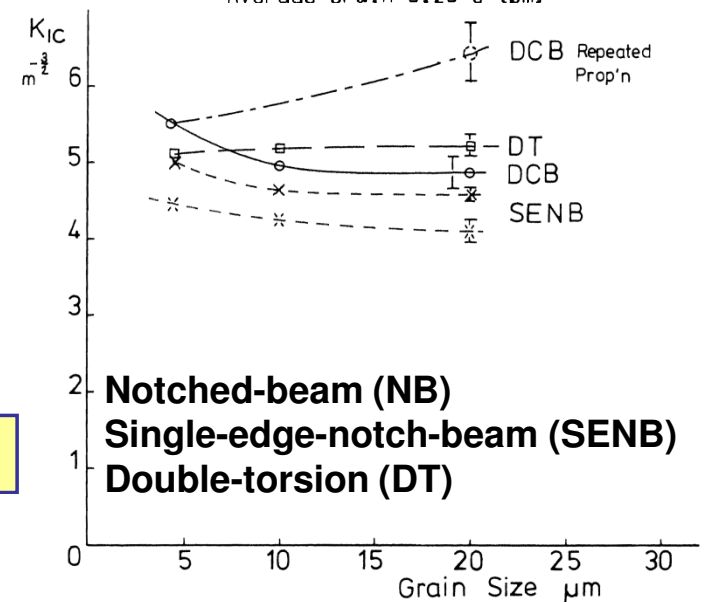
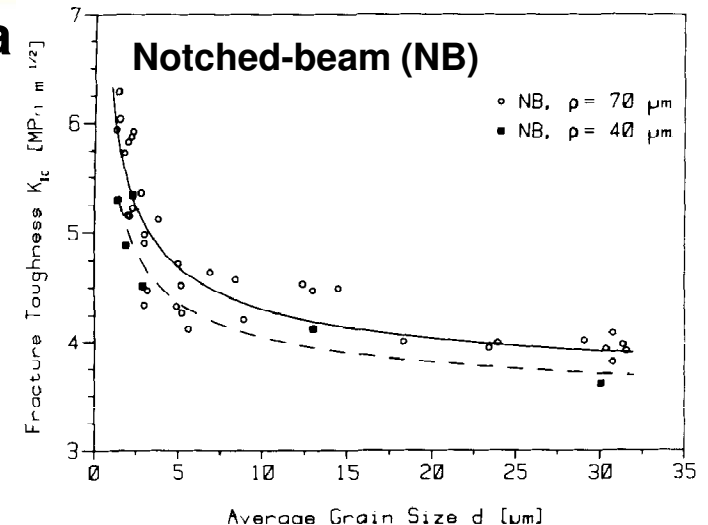
- Dependence of fracture toughness of alumina on grain size



Previous work on ultrafine grained alumina is limited.

B. Mussler et al., *J. Am. Ceram. Soc.*, 1982, 65(11), 566-572

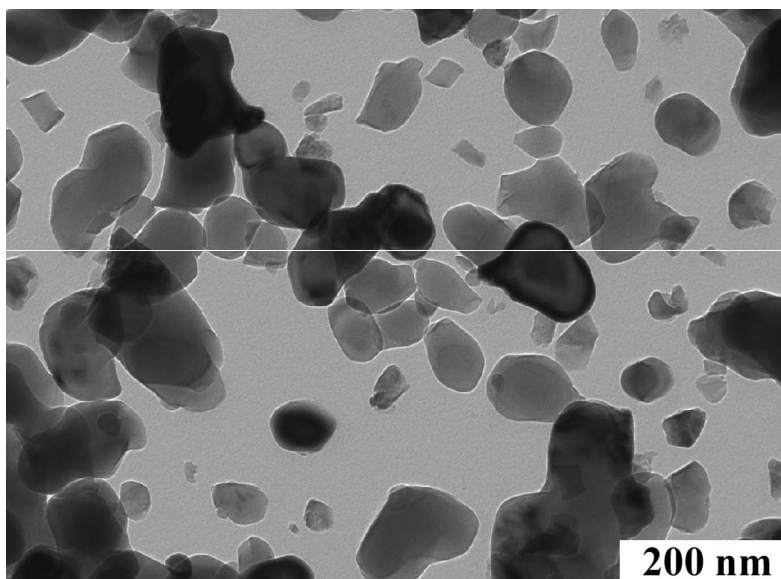
P. L. Pratt, *Fracture*, 1977, 3, 909-912





Experimental Procedures

α -Al₂O₃ (TM DAR 6914)
~100nm, 99.99 %



Composition of feedstock powder

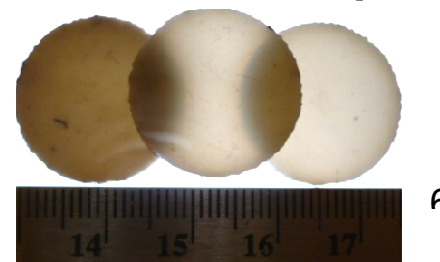
Powder Composition	α - Al ₂ O ₃ (wt. %)	Impurity (ppm)					
	99.99	Si	Fe	Na	K	Ca	Mg
		1	8	3	2	3	1



Spark Plasma Sintering
(SPS-825S, DR. Sinter)



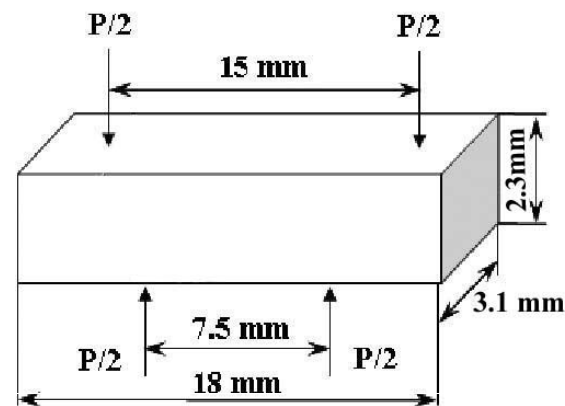
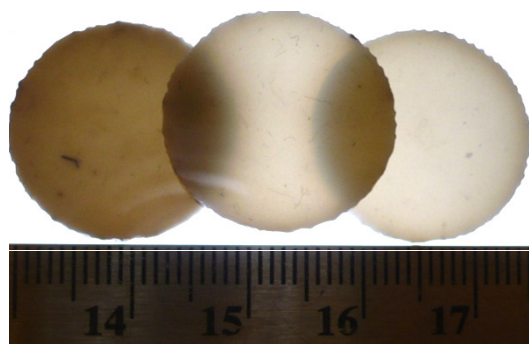
As-Sintered Samples





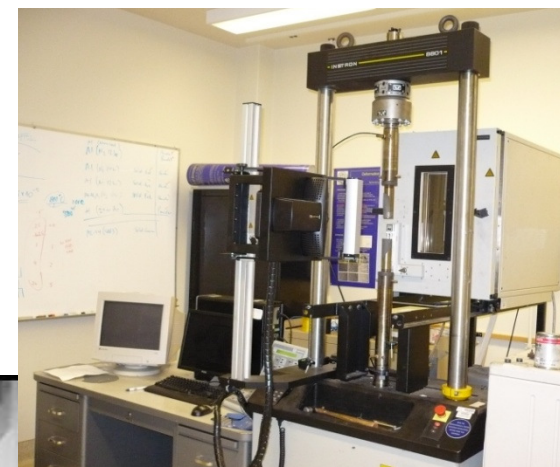
Experimental Procedures

As-Sintered Samples

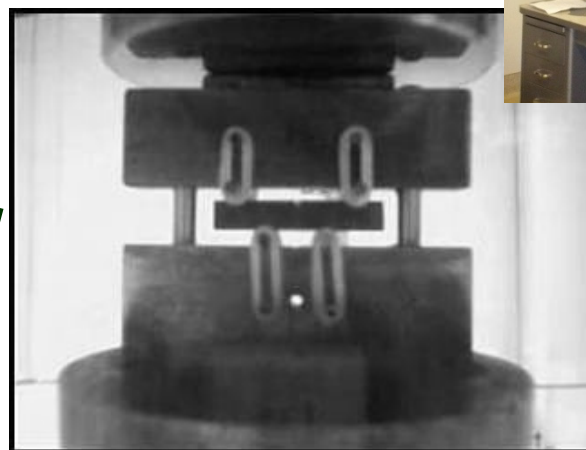
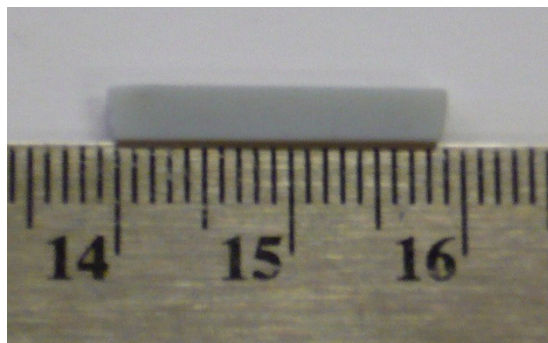


Dimensions of bending beam

Instron universal testing machine



Strain rate: 0.0015 s^{-1}

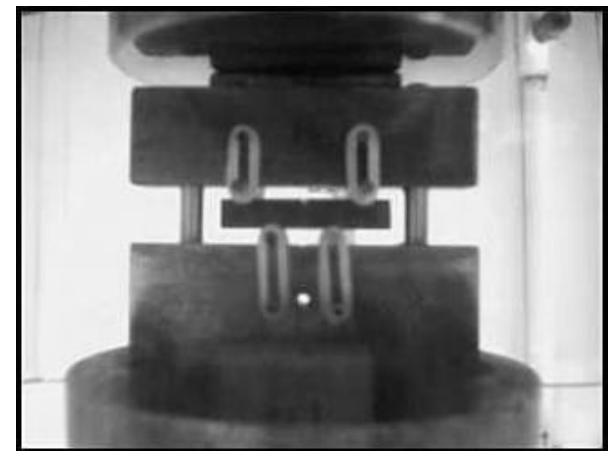


miniature 4-point bend test fixture

Experimental Procedures



- Determination of Fracture Toughness of Ultrafine Grained Alumina
 - Vickers indentation
 - Surface Crack in Flexure
 - Single-edge-V-notched beam (SEVNB)



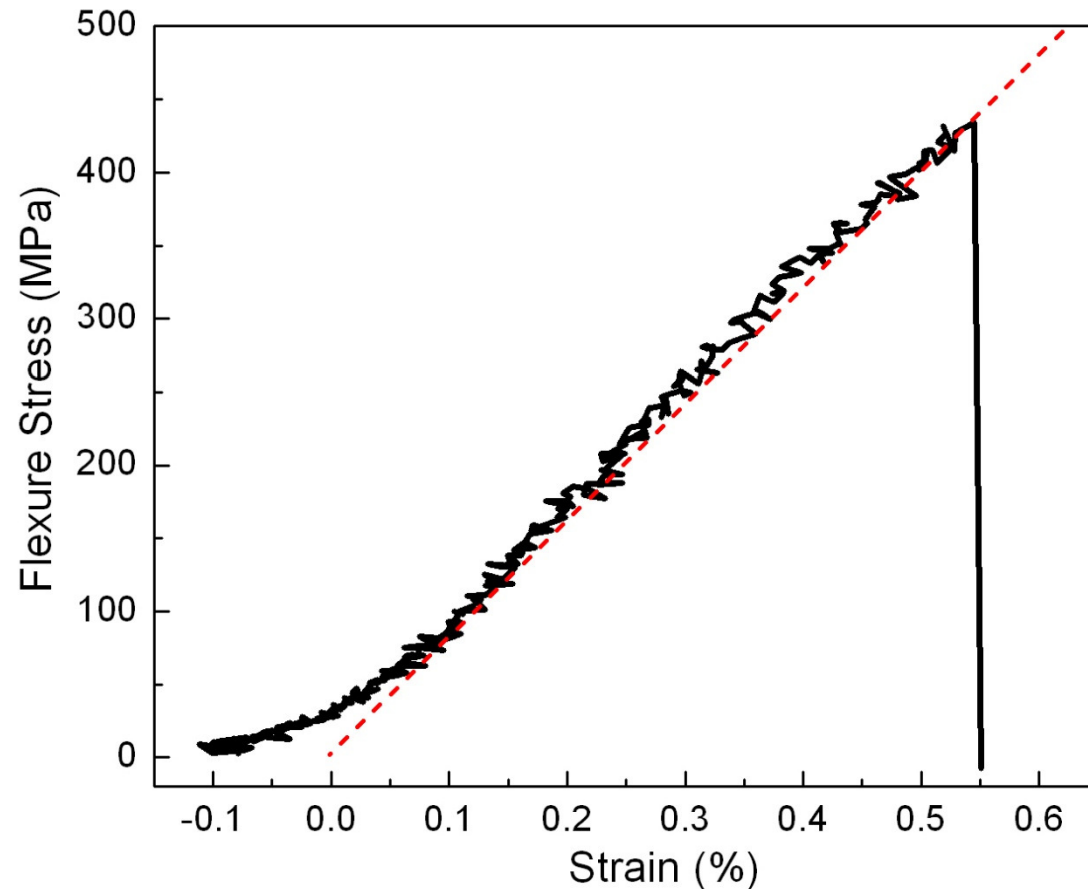
Results and Discussion



- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness



Results and Discussion



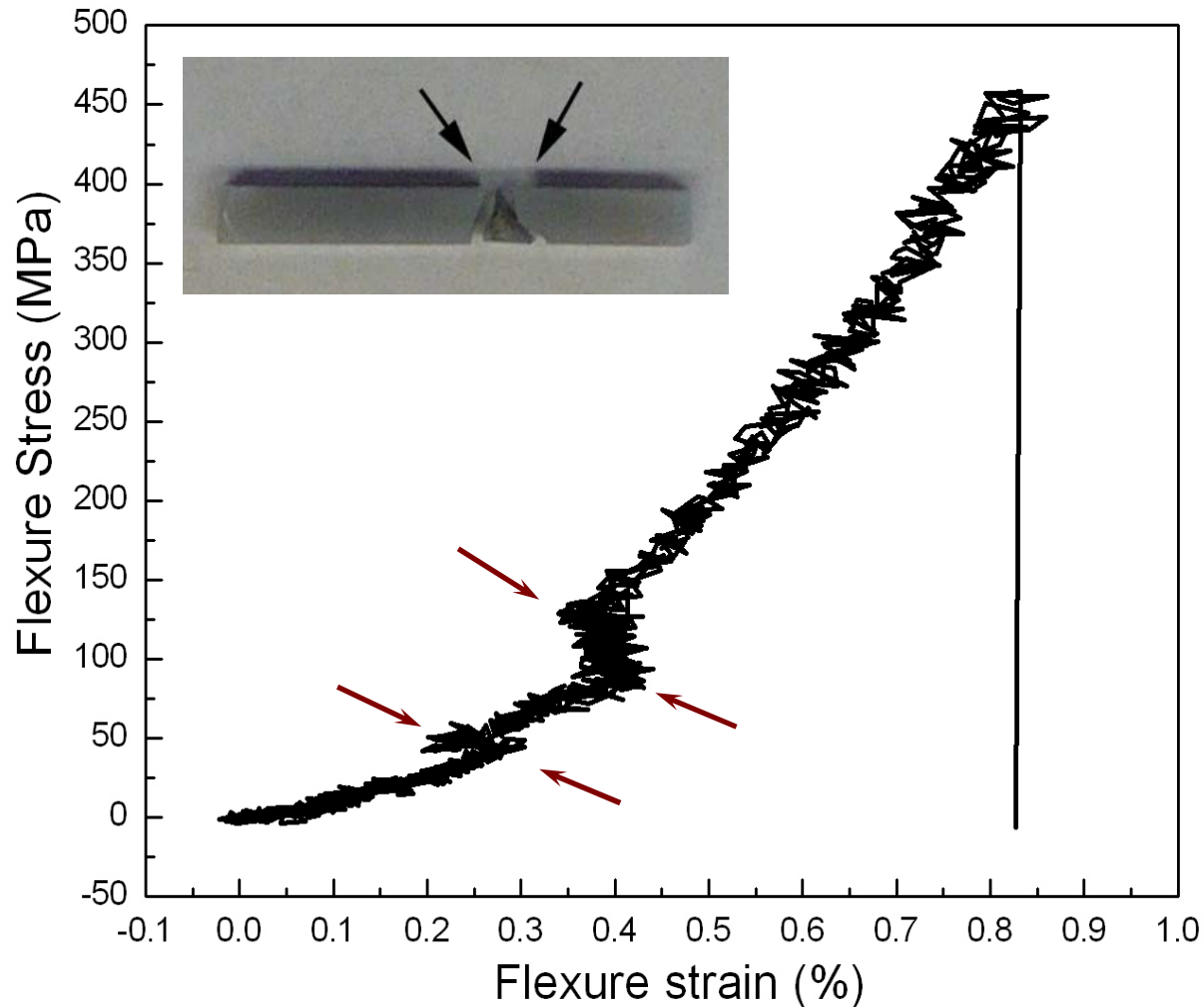
Typical Bending Stress-Strain Curve

- **Average Grain Size -- 363 40 nm**
- **Relative Density -- 99.1%**
- **Strength -- 434MPa**

- **Sample preparation methods (chamfering the edges; annealing samples for a short time before bending)**
- **Preloading conditions and strain rate**



Results and Discussion



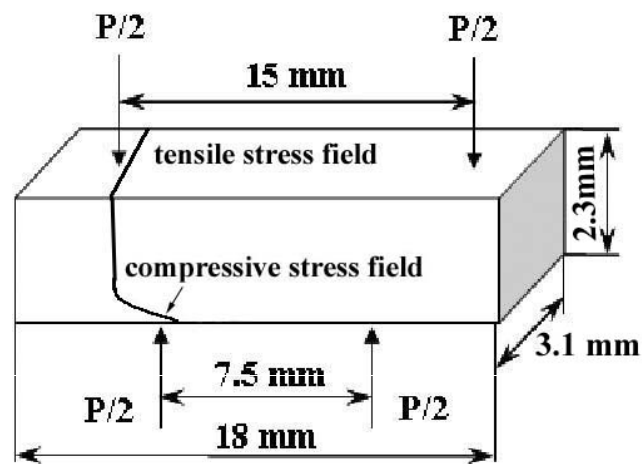
- **Average Grain Size -- 320 20 nm**
- **Relative Density -- 99.5%**
- **Strength -- 458MPa**

Results and Discussion

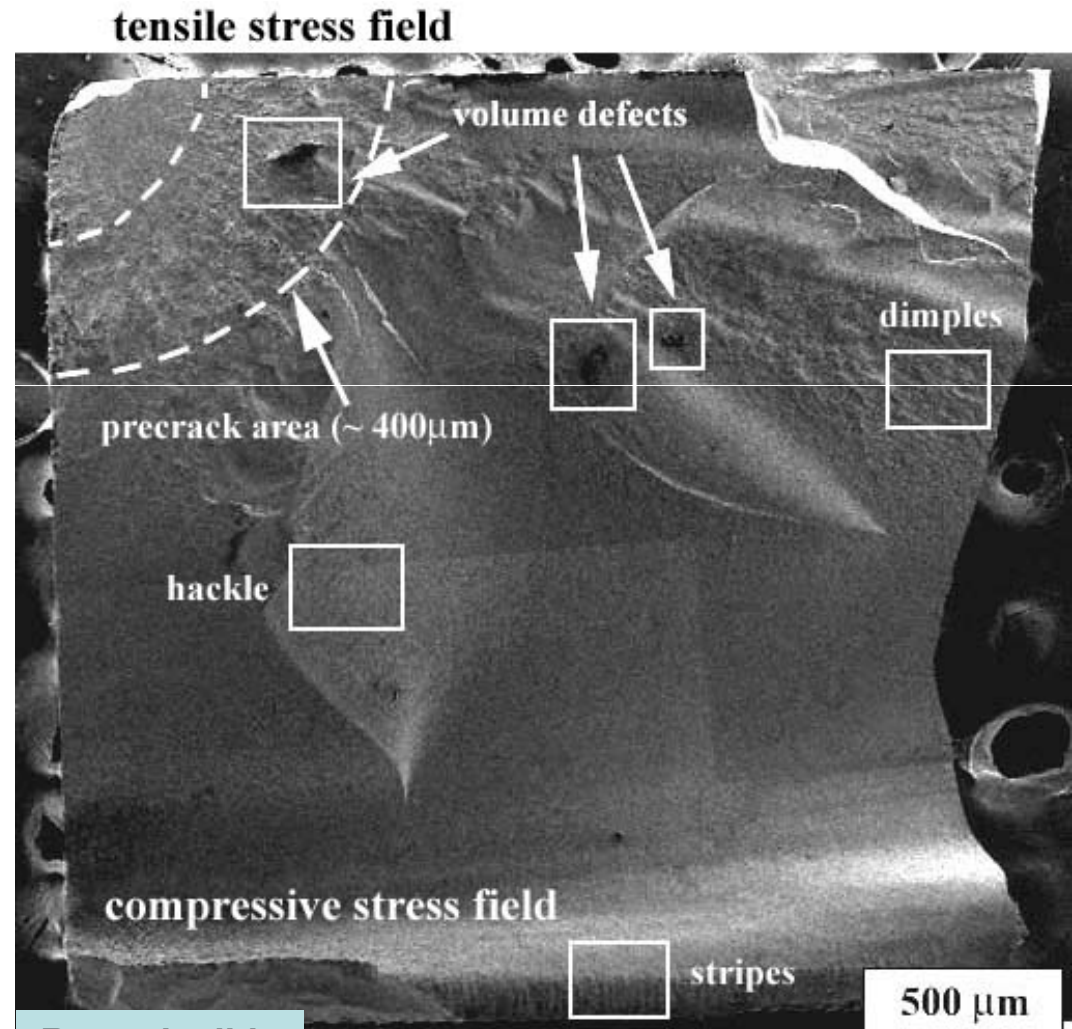


- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness

Results and Discussion

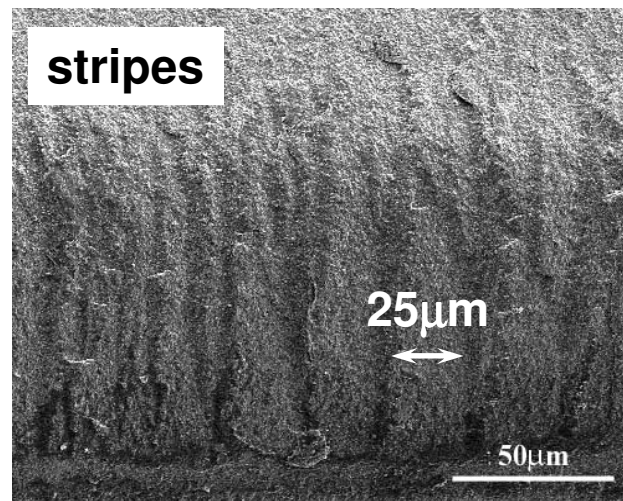
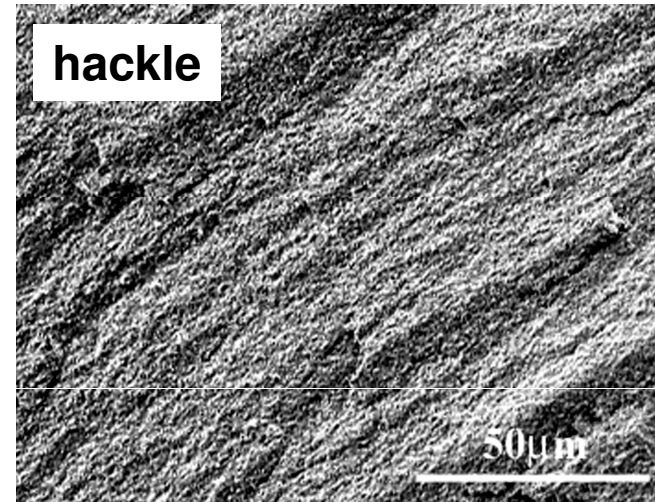
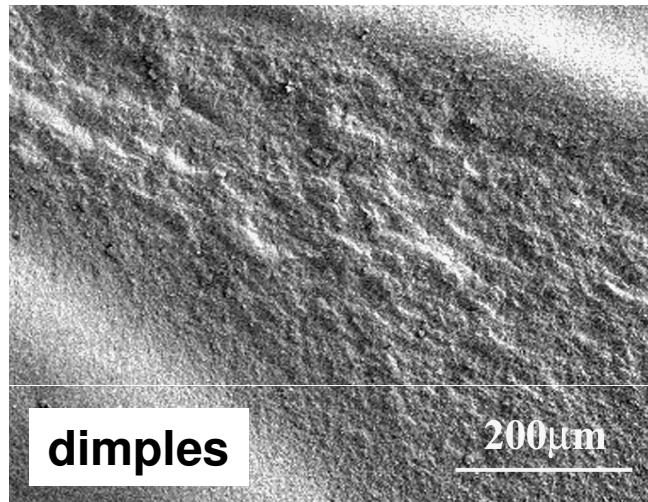


- Average Grain Size -- 363 40 nm
- Relative Density -- 99.1%
- Strength -- 434MPa



Reproducible

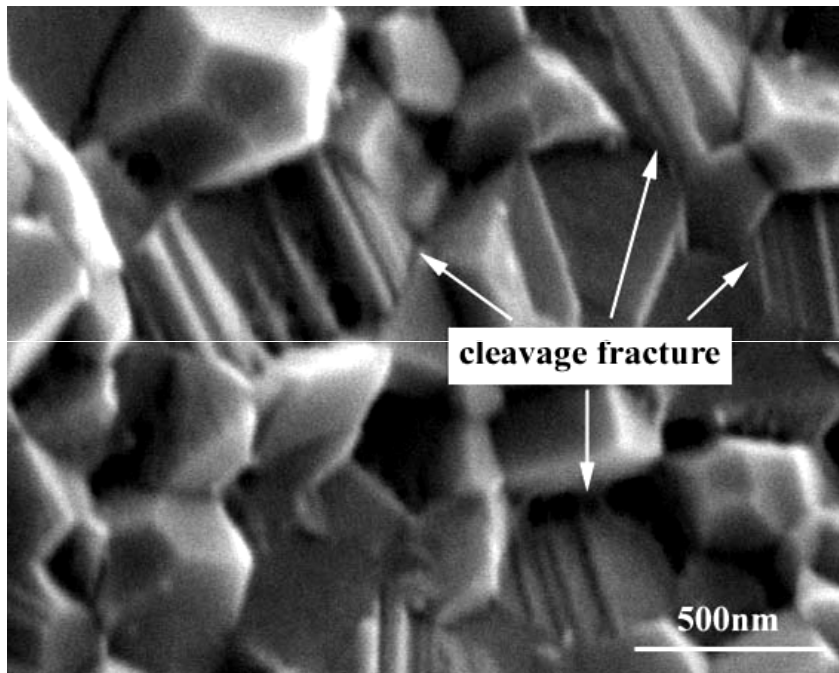
Results and Discussion



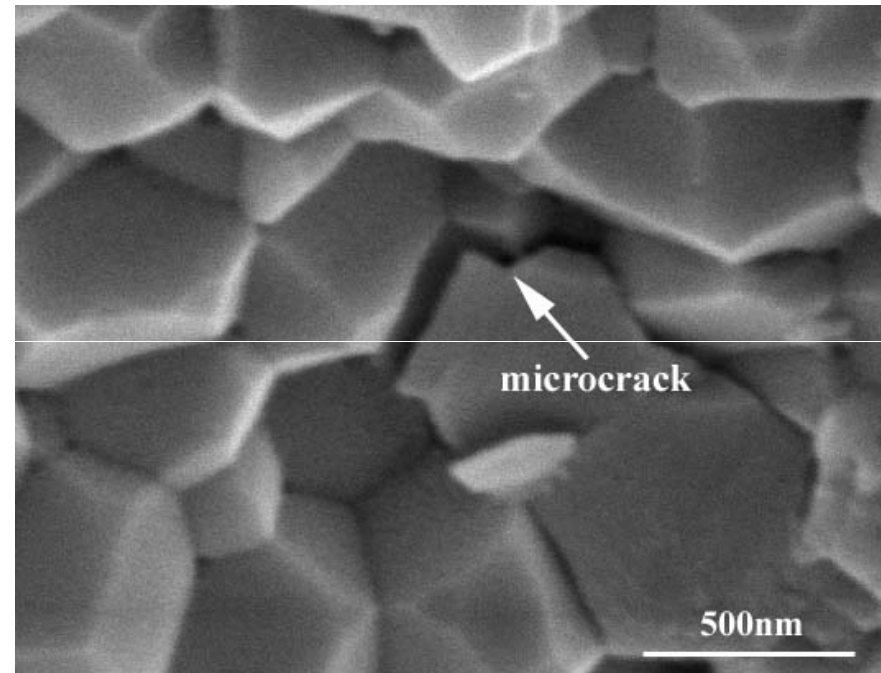
- Average Grain Size -- 363 40 nm
- Relative Density -- 99.1%
- Strength -- 434MPa

Local magnifications

Results and Discussion



Mixed Fracture (transgranular cleavage fracture + intergranular fracture)

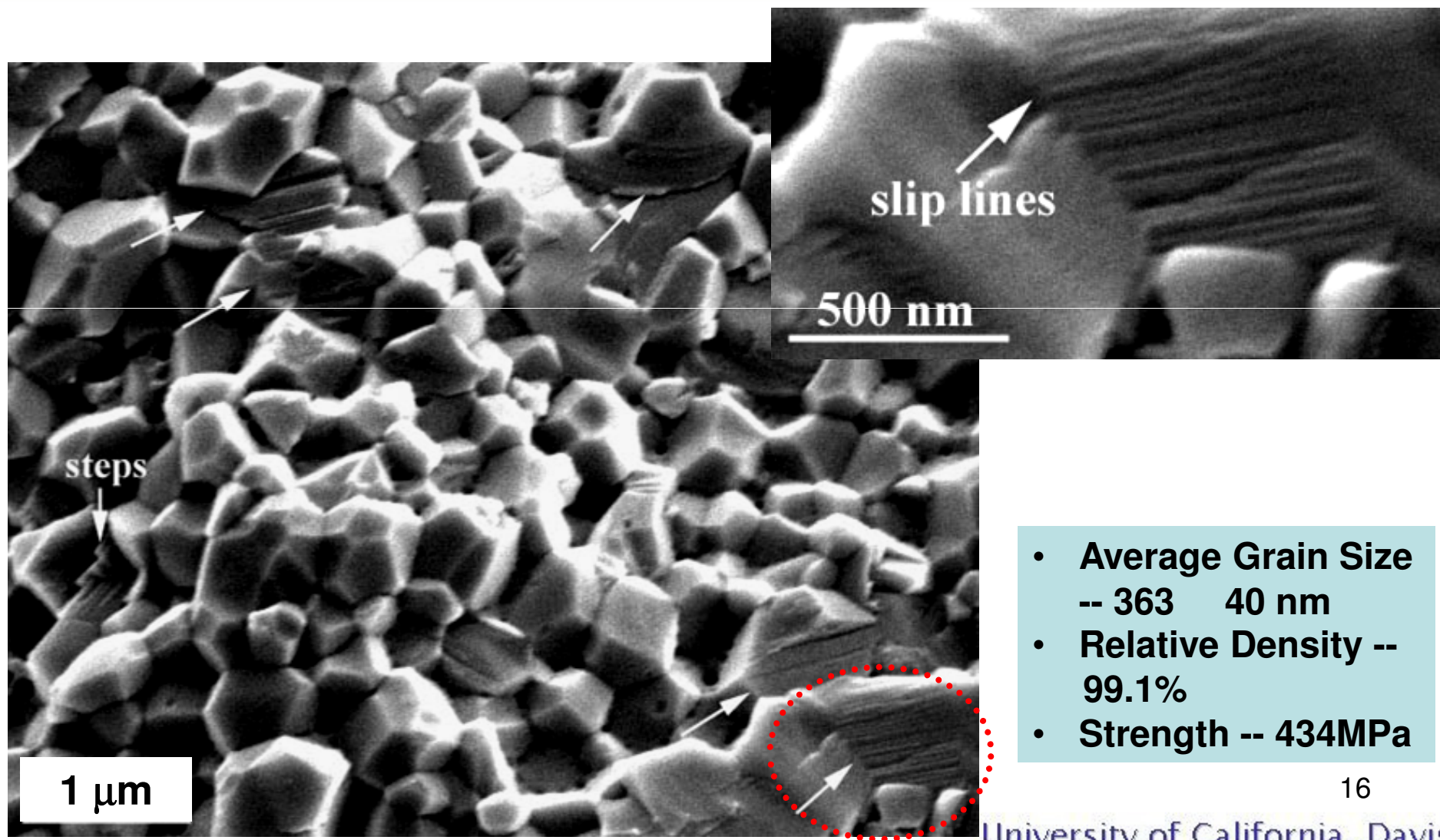


Intergranular Fracture

- **Average Grain Size -- 363 40 nm**
- **Strength -- 434MPa**



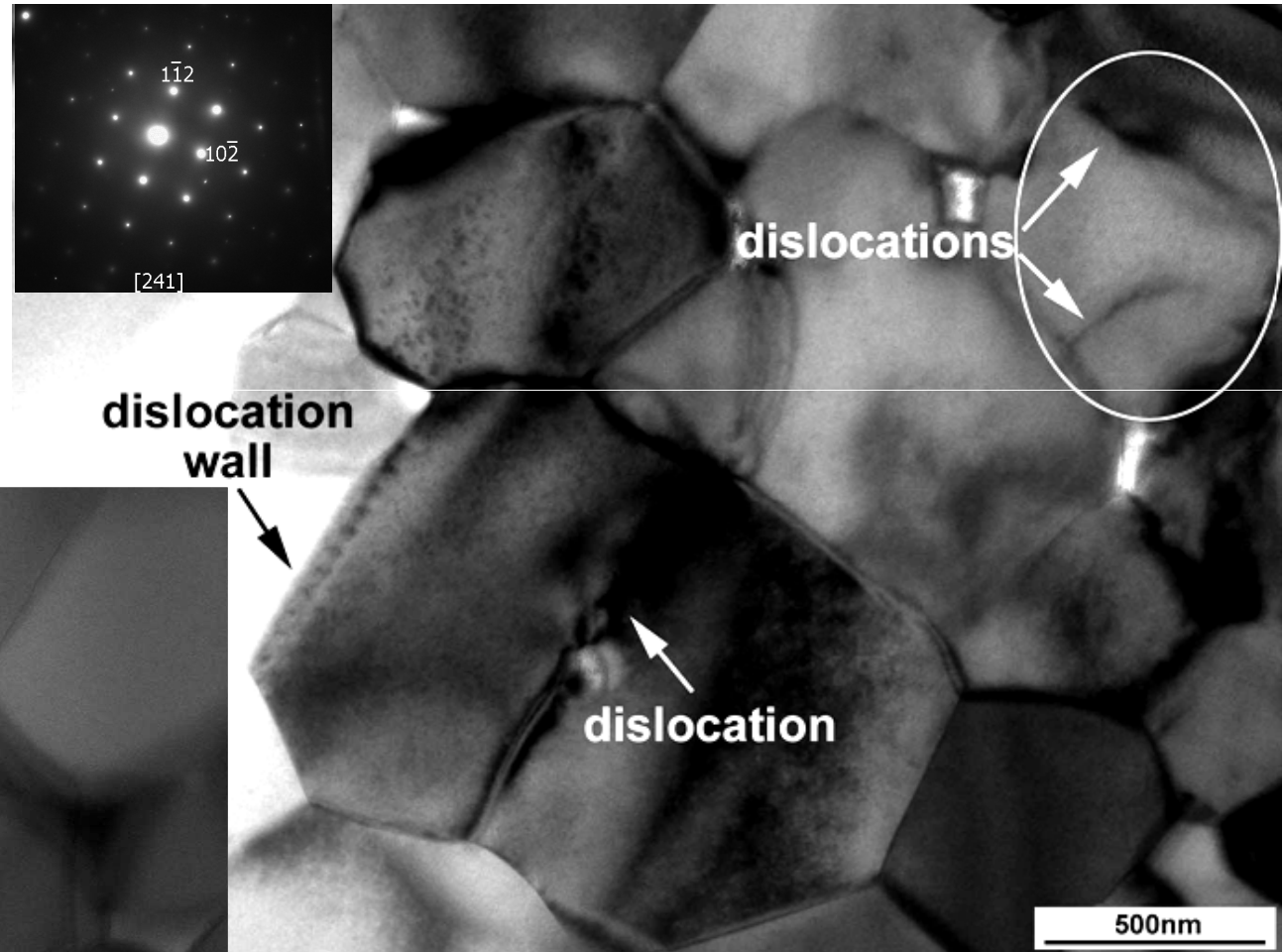
Results and Discussion



- Average Grain Size -- 363 40 nm
- Relative Density -- 99.1%
- Strength -- 434MPa

Results and Discussion

- Average Grain Size -- 363 40 nm
- Relative Density -- 99.1%
- Strength -- 434MPa



Results and Discussion

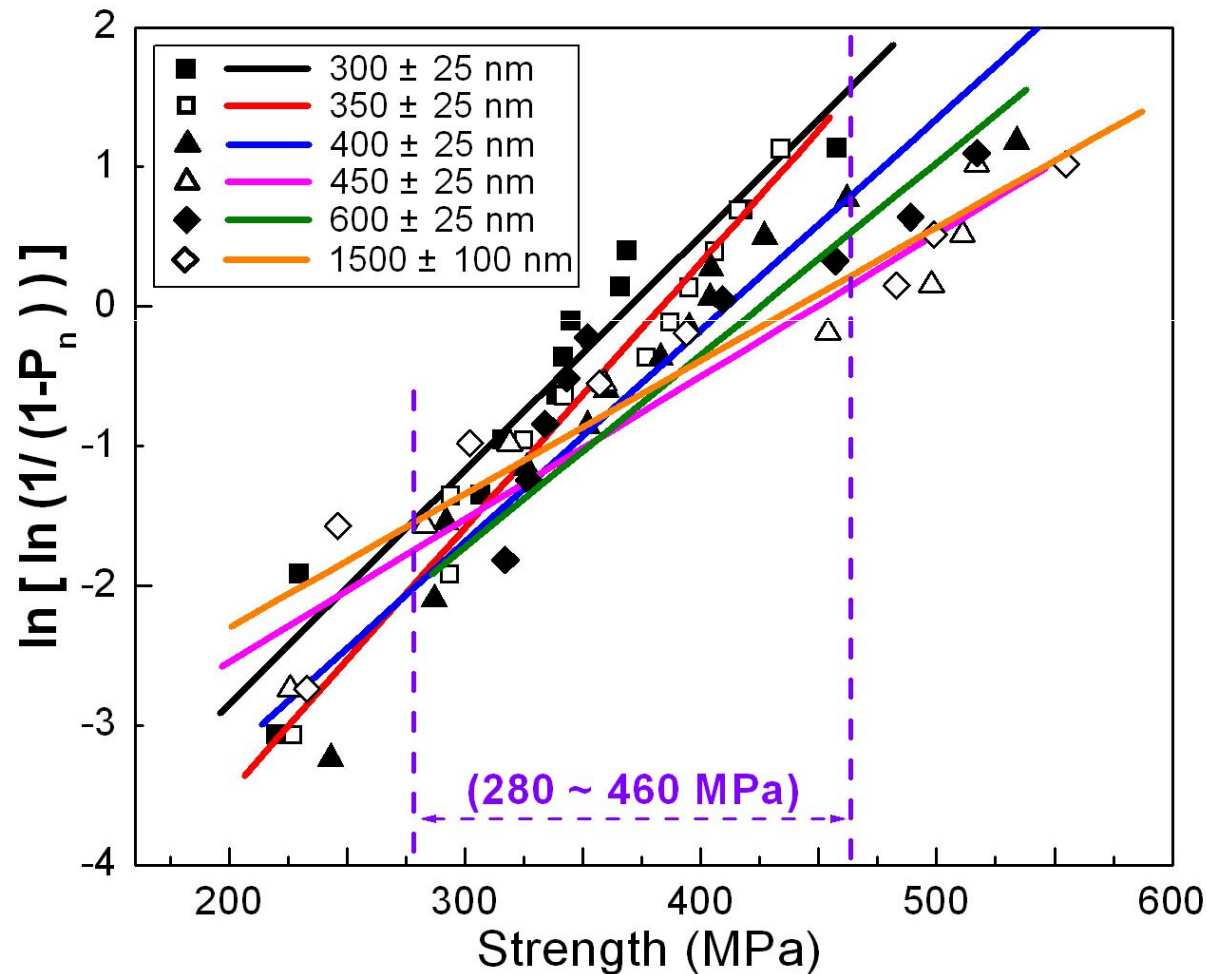


- Bending Process
- Fracture Surface Morphology
- **Statistical Analysis**
- Fracture Toughness



Results and Discussion

Strength Distribution (Weibull Statistics)



Samples

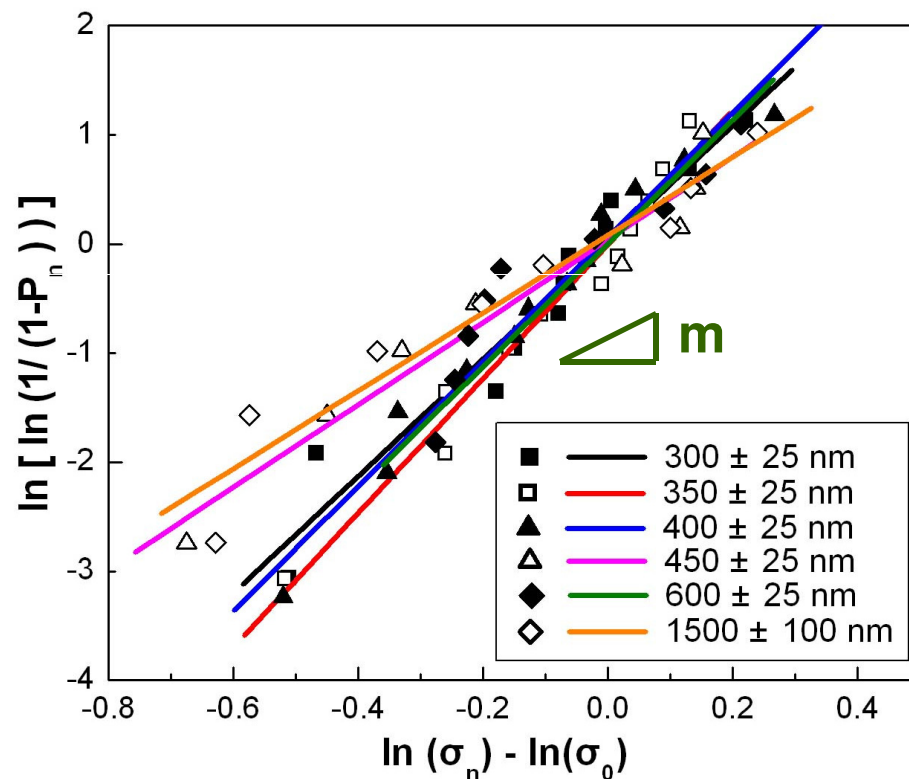
Average Grain Size (nm)		Number of Samples
300	25	11
350	25	11
400	25	13
450	25	8
600	25	10
1500	100	8

$$\ln \left[\ln \left(\frac{1}{1-P_n} \right) \right] = m \ln \sigma_n - m \ln \sigma_0$$

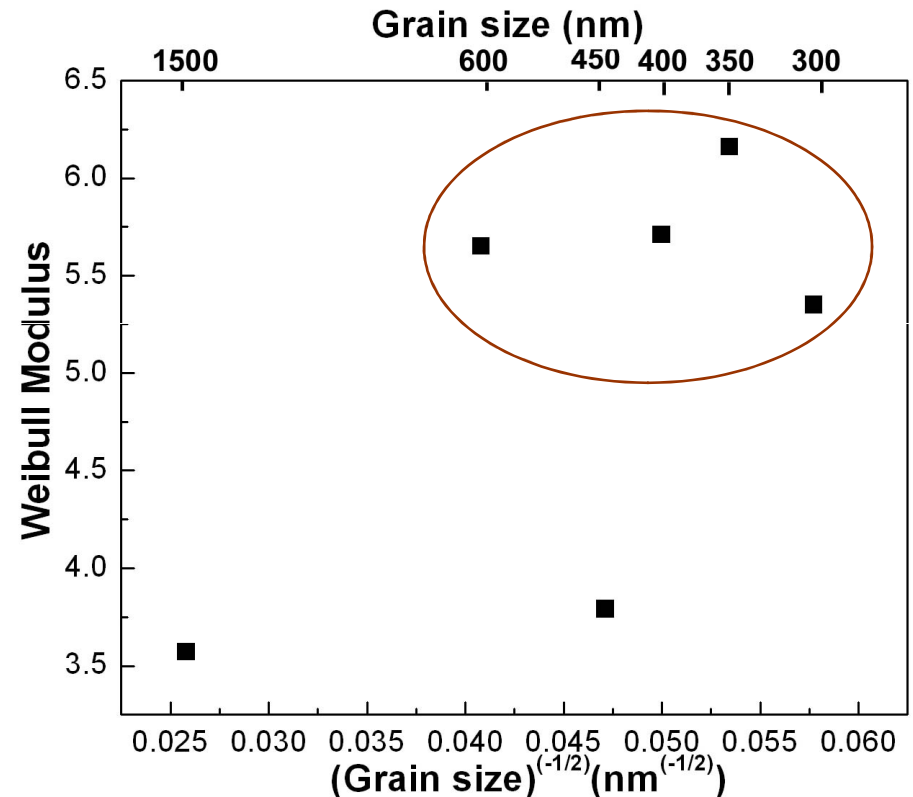


Results and Discussion

m – Weibull modulus



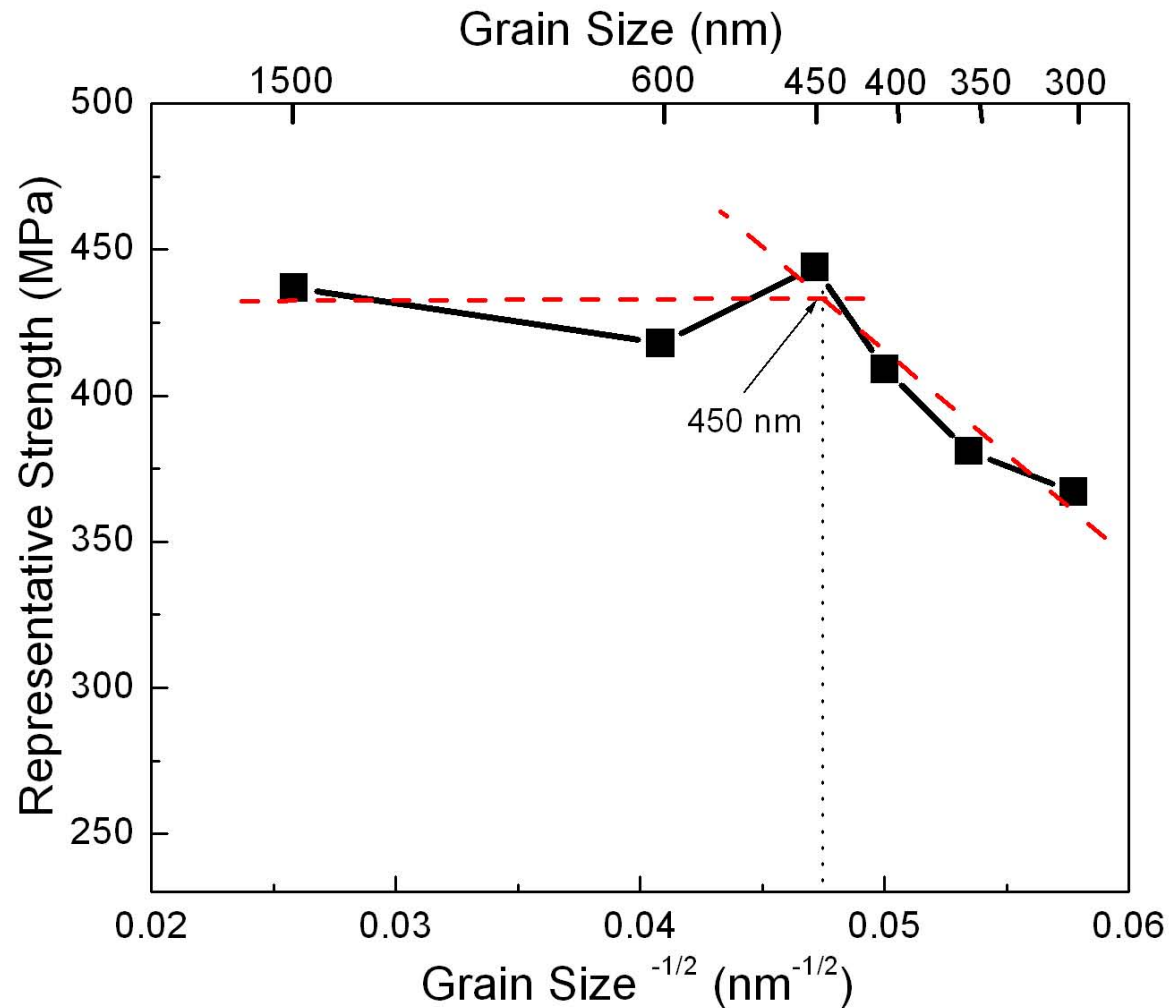
$$\ln\left[\ln\left(\frac{1}{1-P_n}\right)\right] = m \ln \sigma_n - m \ln \sigma_0$$



Typical Value: 5 - 20 for technical ceramics



Results and Discussion



Results and Discussion



- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness

Determination of Fracture Toughness of Ultrafine Alumina

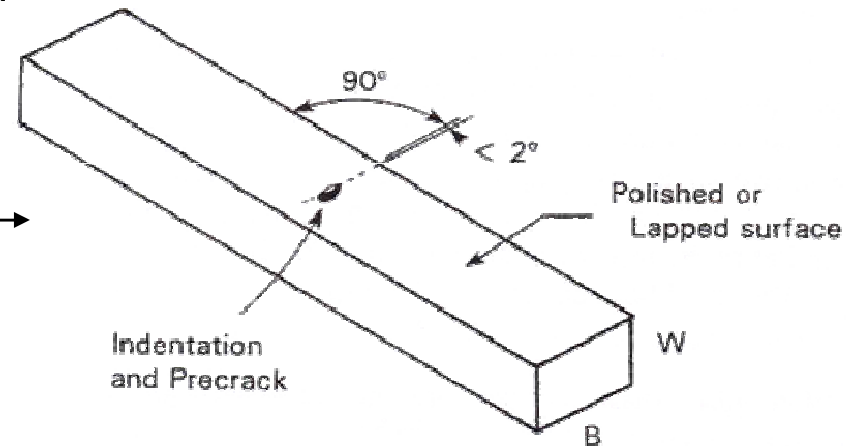


- Vickers indentation

Simple. SEM is used to measure the length

- Surface Crack in Flexure →

ASTM C1421-01b, 2007



- Single-edge-V-notched beam (SEVNB)

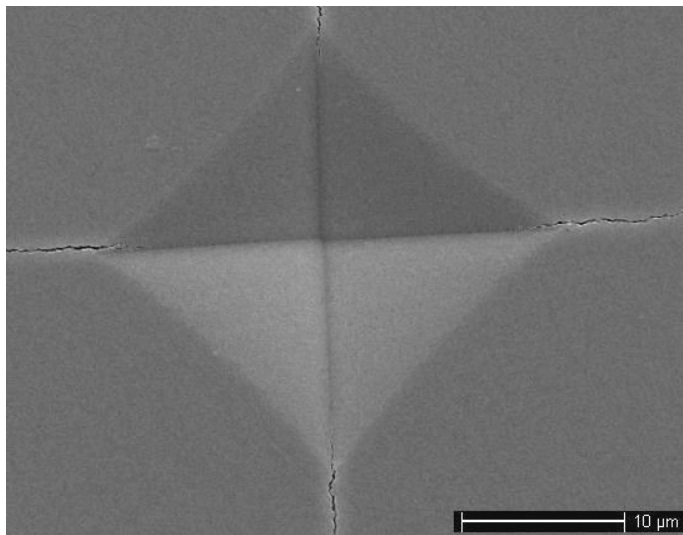
ASTM STP 1409, 2002

* It didn't give consistent results and was abandoned in our lab.

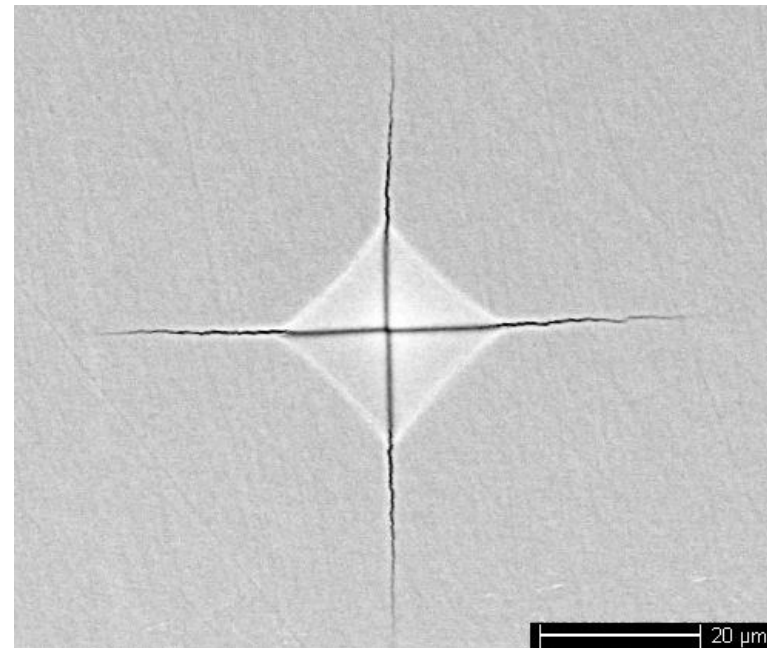
Fracture Toughness determined by Vickers Indentation



- Surface of alumina was polished to 1 μm finish before 10 indentations being made



Secondary electron image



Back scattering electron image

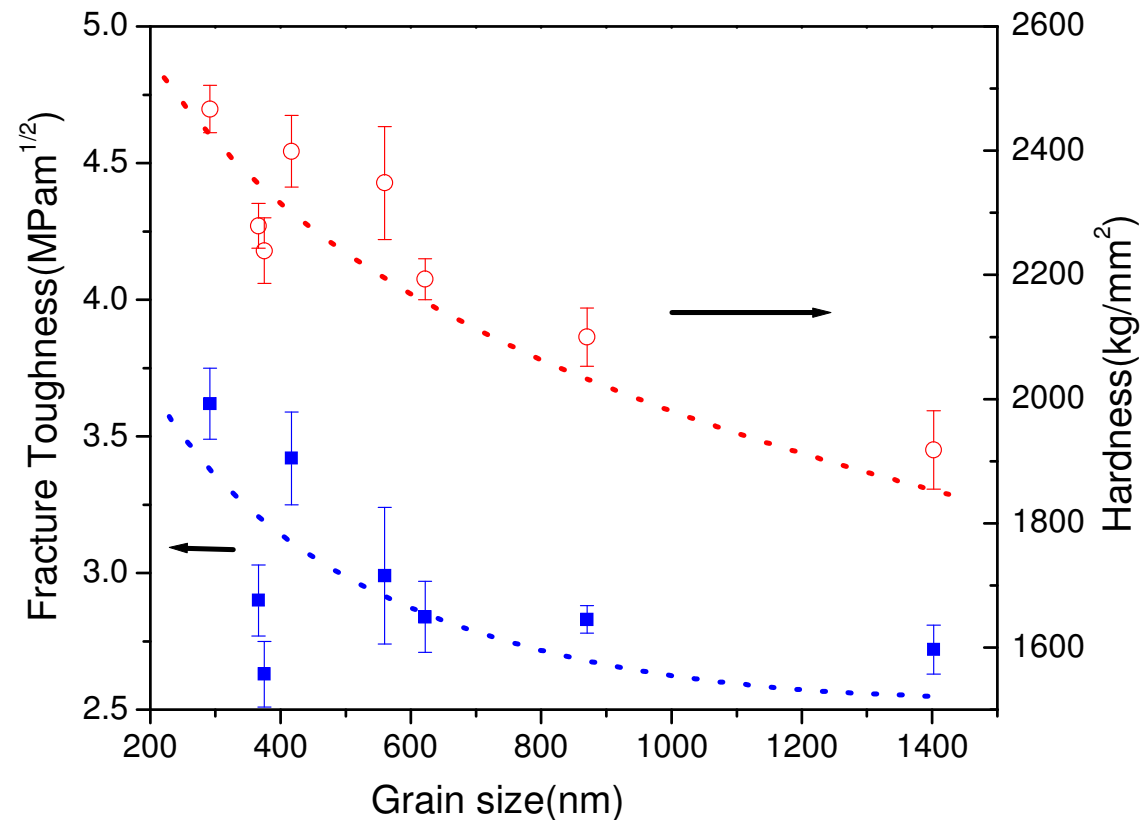
$$\text{Anstis equation } K_{IC} = 0.016(E/H)^{1/2} P/c^{3/2} \quad H = 1.8544(P/d^2)$$

H: Hardness E: Young's modulus P: Load d: length of diagonal of indentation

2c: length of diagonal of indentation and radial crack

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Fracture Toughness and Hardness from Vickers Indentation

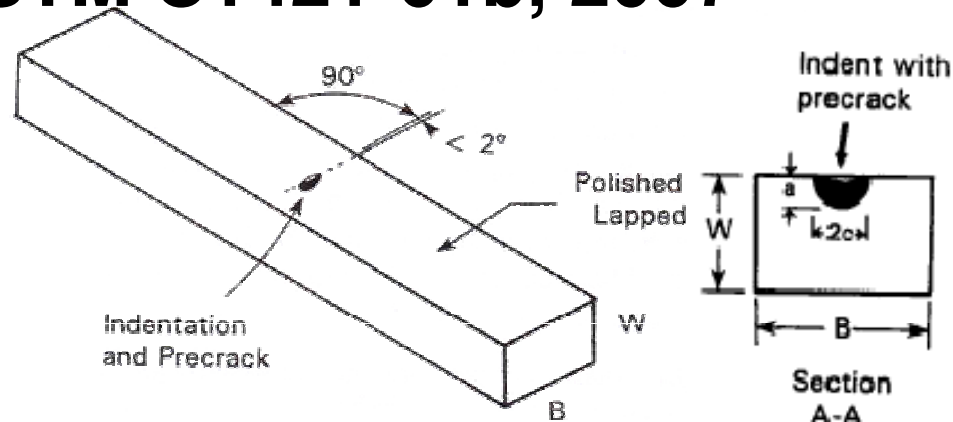


Dependence of fracture toughness and hardness of alumina on grain size

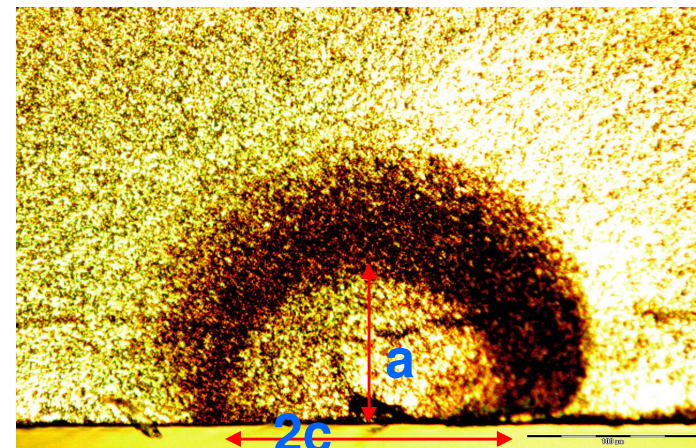
Surface Crack in Flexure

(Determination of Fracture Toughness of Advanced Ceramics)

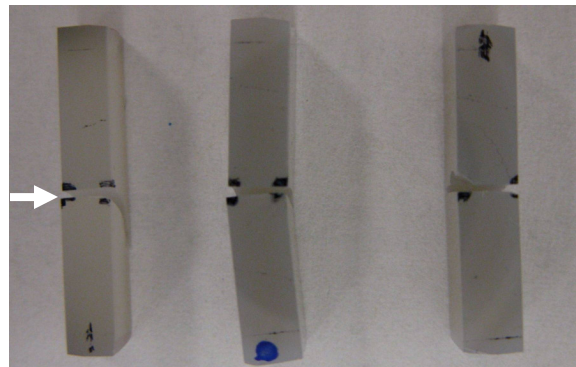
ASTM C1421-01b, 2007



Surface-Crack in Flexure Test Specimen



Knoop indenter precrack in a sintered alumina as photographed in optical microscope. The detect and measurement of precrack shape of width $2c$ and depth a can be done in SEM or optical microscope.





Surface Crack in Flexure

$$K_{IC} = Y\sigma\sqrt{a}$$

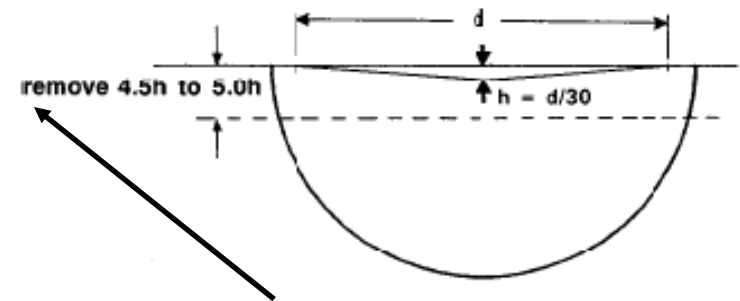
Y : stress intensity factor, $f(a, c, W)$

σ : flexure strength at fracture

a : precrack depth

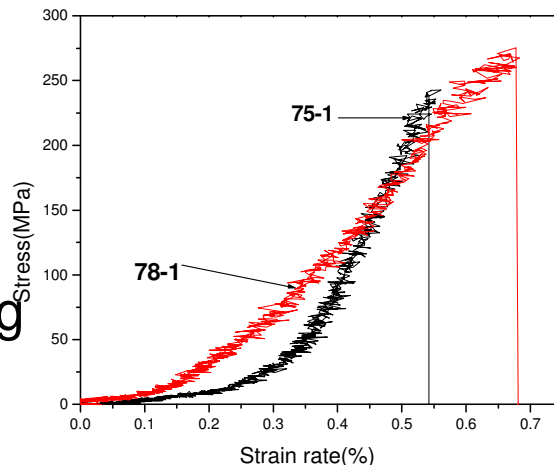
c : half width of precrack shape

W : thickness of test specimen



Flexure strength is most important to determine K_{IC}

Typical four point bending curves



Sample ID	Grain size (nm)	Flexure Stress (MPa)	Remove indenter and Damage zone?	Fracture Toughness ($\text{MPa}\cdot\text{m}^{1/2}$) (Surface crack in flexure)
75-1	366 28	243	Yes	2.61
78-1	622 58	275	Yes	3.11
78-2	622 58	238	No	2.78
81-1	870±77	224	No	2.68
92-2	1402 249	257	No	3.02

Comparison of Fracture Toughness of Alumina determined by Surface Crack in Flexure and Vickers Indentation



Sample ID	Grain size (nm)	Fracture toughness ($MPa \cdot m^{1/2}$) (Surface crack in flexure)	Fracture toughness ($MPa \cdot m^{1/2}$) (Vickers indentation)
75-1	366 28	2.61*	2.90 0.13
78-1	622 58	3.11*	2.84 0.13
78-2	622 58	2.78	2.84 0.13
81-1	870±77	2.68	2.83 0.05
92-2	1402 249	3.02	2.72 0.09

Single-Edge-V-Notched Beam (SEVNB):
 K_{IC} varies from 3 to 5 $MPa \cdot m^{1/2}$

Surface Crack in Flexure and Vickers indentation gave more consistent fracture toughness value in our lab.

* Removing indenter and damage zone



Conclusions

- For dense ultra-fine grained alumina, most strength values fall within the interval of 280-460 MPa. The proper selection of appropriate sample preparation methods and preloading conditions is important to achieve accurate measurements.
- No dependence of Weibull modulus on grain size can be observed.
- Cleavage, transgranular and intergranular fracture as well as microcracks, slip lines and dislocations can be observed at or near the fracture surface.
- Surface crack in flexure and Vickers indentation gives more consistent fracture toughness values than does single-edge-V-notched beam (SEVNB).



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- ***Collaborators/Contributors***

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Thank You

for

Your Attention!