Design, Fabrication, and Mechanical Characterization of 3D Hollow Ceramic Nano-Architectures

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Korea Advanced Institute of Science and Technology (KAIST)

Sept 19th 2018
Department of Mechanical Science and Engineering
Manufacturing Interest Group Seminar

Design and Fabrication of Deformable Ceramic

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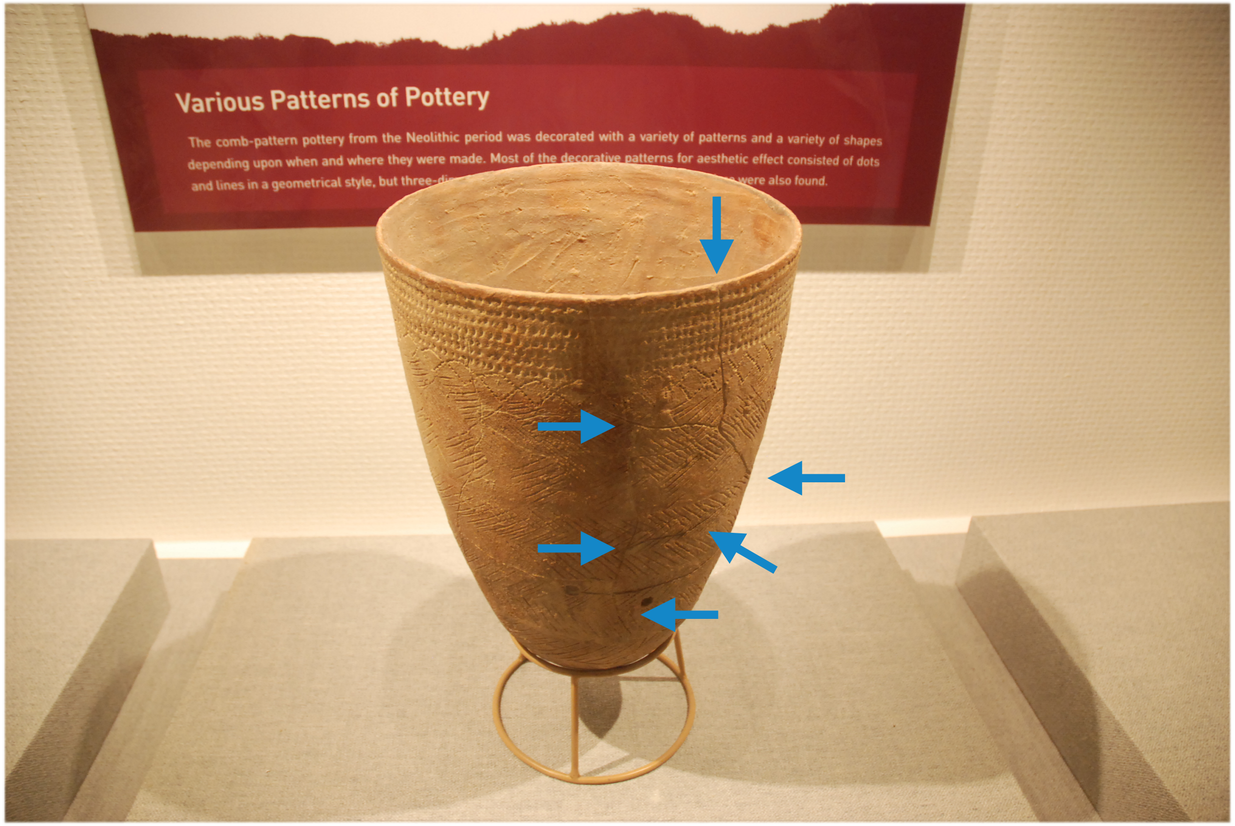
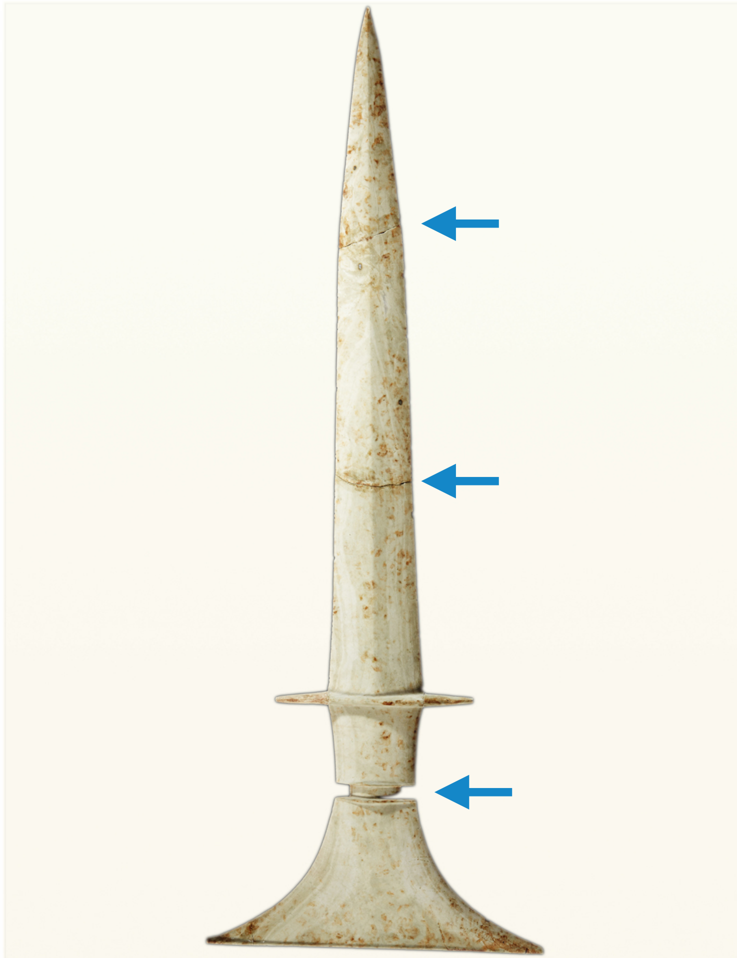
What is the Most Long-Lasting Engineering Issue of Human Beings Since Stone Age?

Overcoming Brittle Failure in Ceramics

The Most Long-lasting Engineering Issue of Human-being

Overcoming Brittle Failure in Ceramics





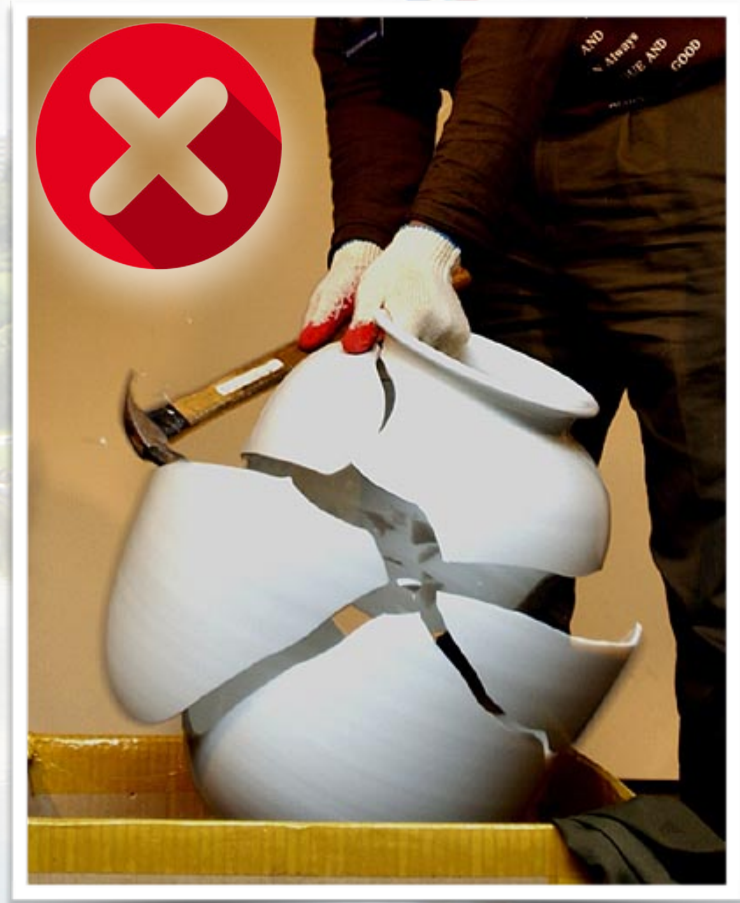
https://ko.wikipedia.org/wiki/빗살무늬토기_시대
https://www.museum.go.kr/site/main/relic/search/view?relicId=4337#

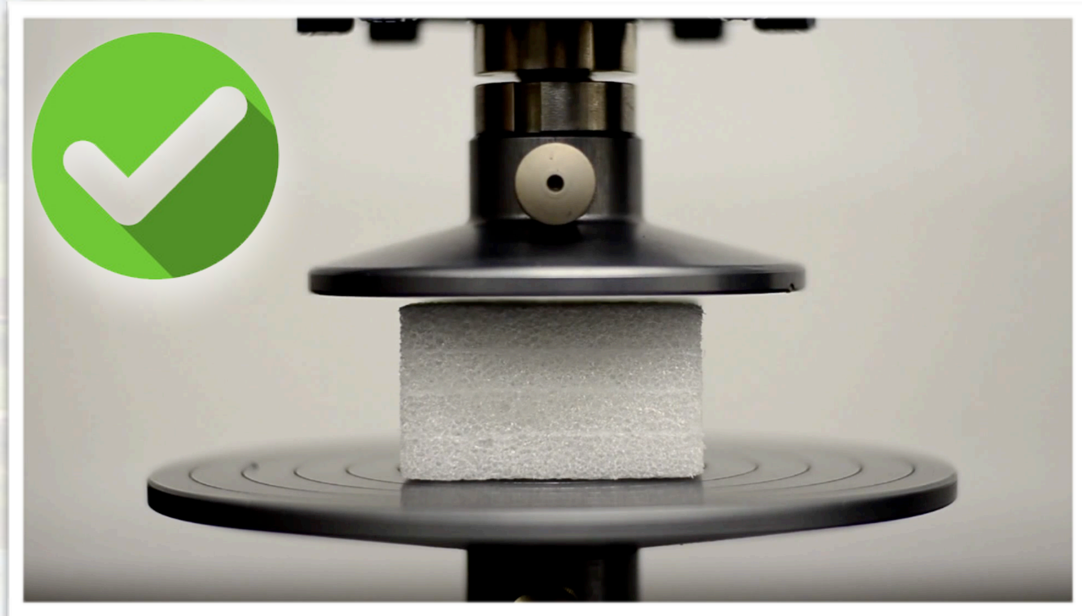
KAIST
College of Engineering
Department of Nuclear and Quantum Engineering

Strong and **deformable** lightweight pristine ceramics: Is it possible to make by any chance?

YES !!

Overcoming Brittle Failure in Ceramics





Scientific Backgrounds

Size Effects in Mechanical Properties

Size Effects

Coupling of Extrinsic Dimensions and Materials' Behavior Typically at Micron- and Nano-Scale

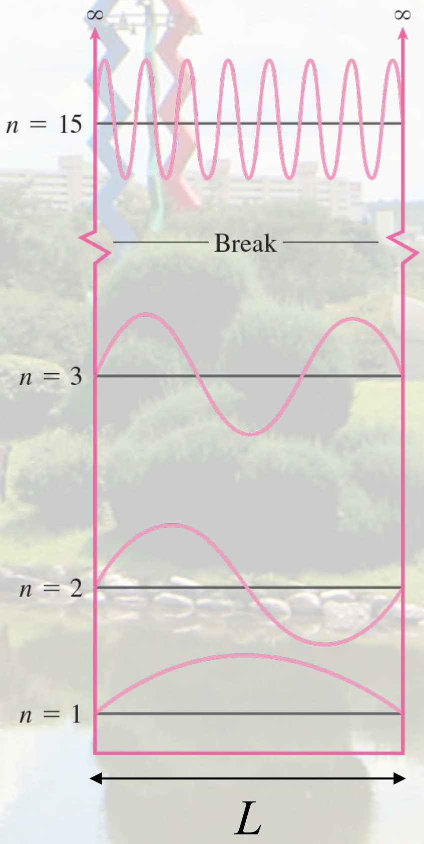
Origin of Size Effects

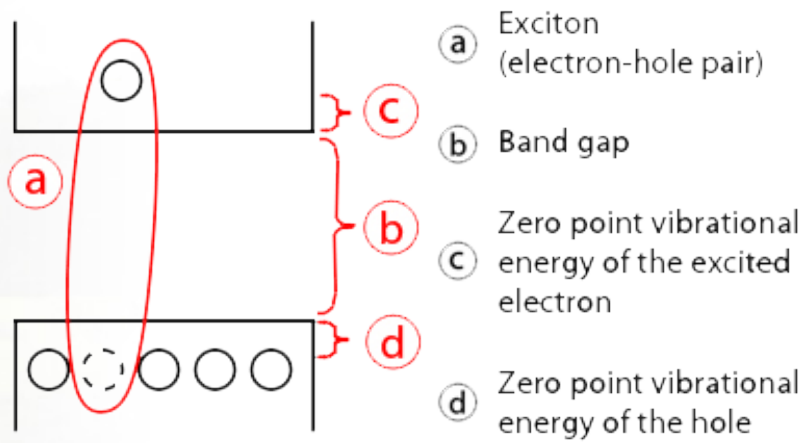
Confinement

Surface

Origin of Size Effects

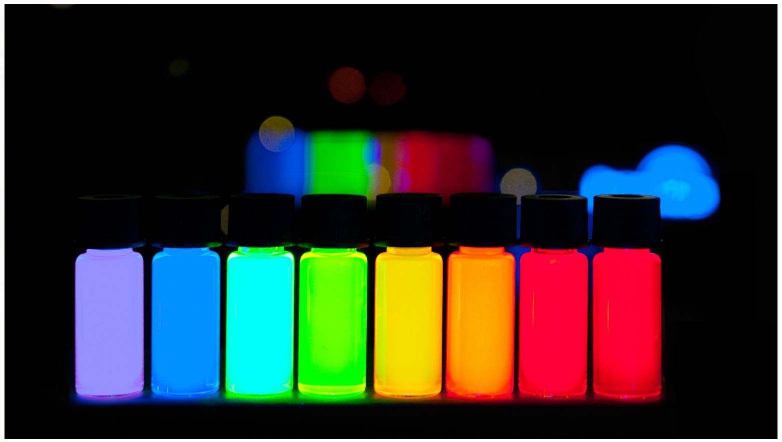
Confinement





$$\psi_{n_x,n_y,n_z} = \sqrt{\frac{8}{L_x L_y L_z}} \sin\left(\frac{n_x \pi x}{L_x}\right) \sin\left(\frac{n_y \pi y}{L_y}\right) \sin\left(\frac{n_z \pi z}{L_z}\right)$$

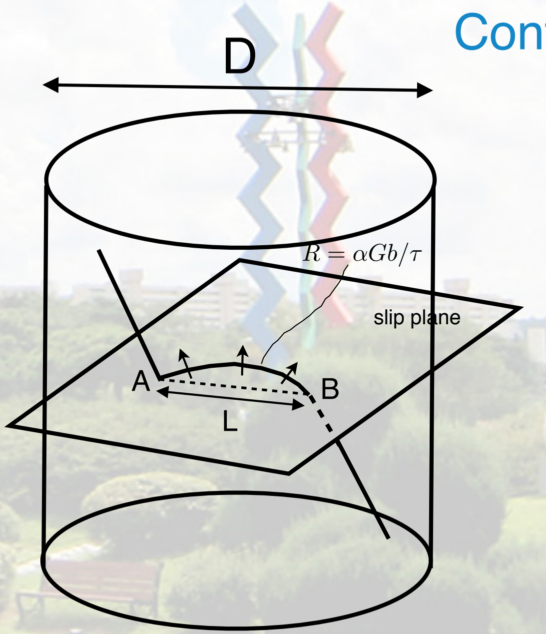
$$E_{n_x,n_y,n_z} = \frac{\hbar^2 \pi^2}{2m}\left[\left(\frac{n_x}{L_x}\right)^2 + \left(\frac{n_y}{L_y}\right)^2 + \left(\frac{n_z}{L_z}\right)^2\right]$$



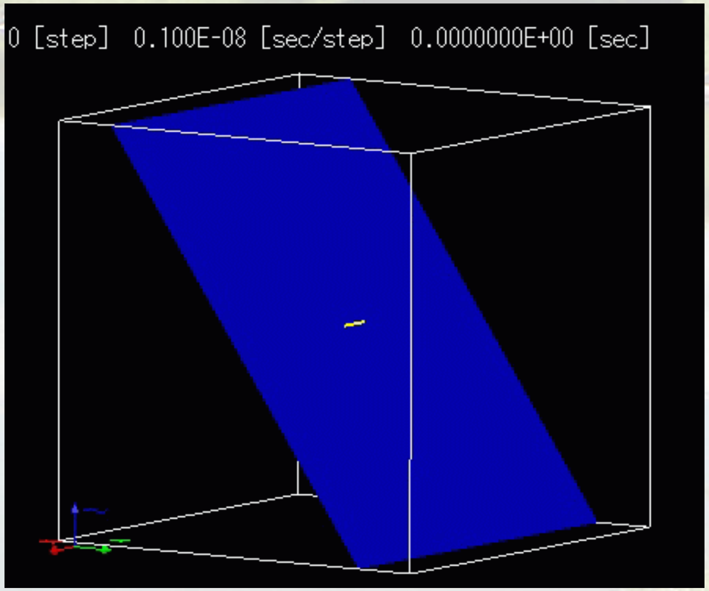
https://en.wikipedia.org/wiki/Quantum_dot

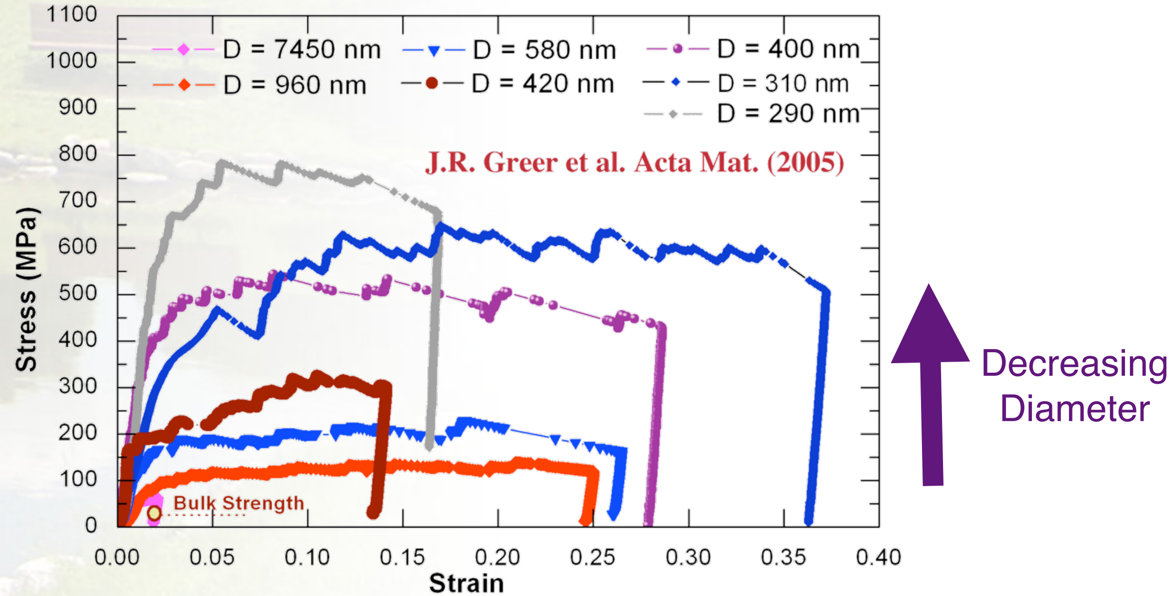
Origin of Size Effects

Confinement: Plasticity in Single Crystals



- Mechanism: dislocation multiplication by Frank-Read source
  - characteristic length: L
  - geometric size parameter: sample parameter, D

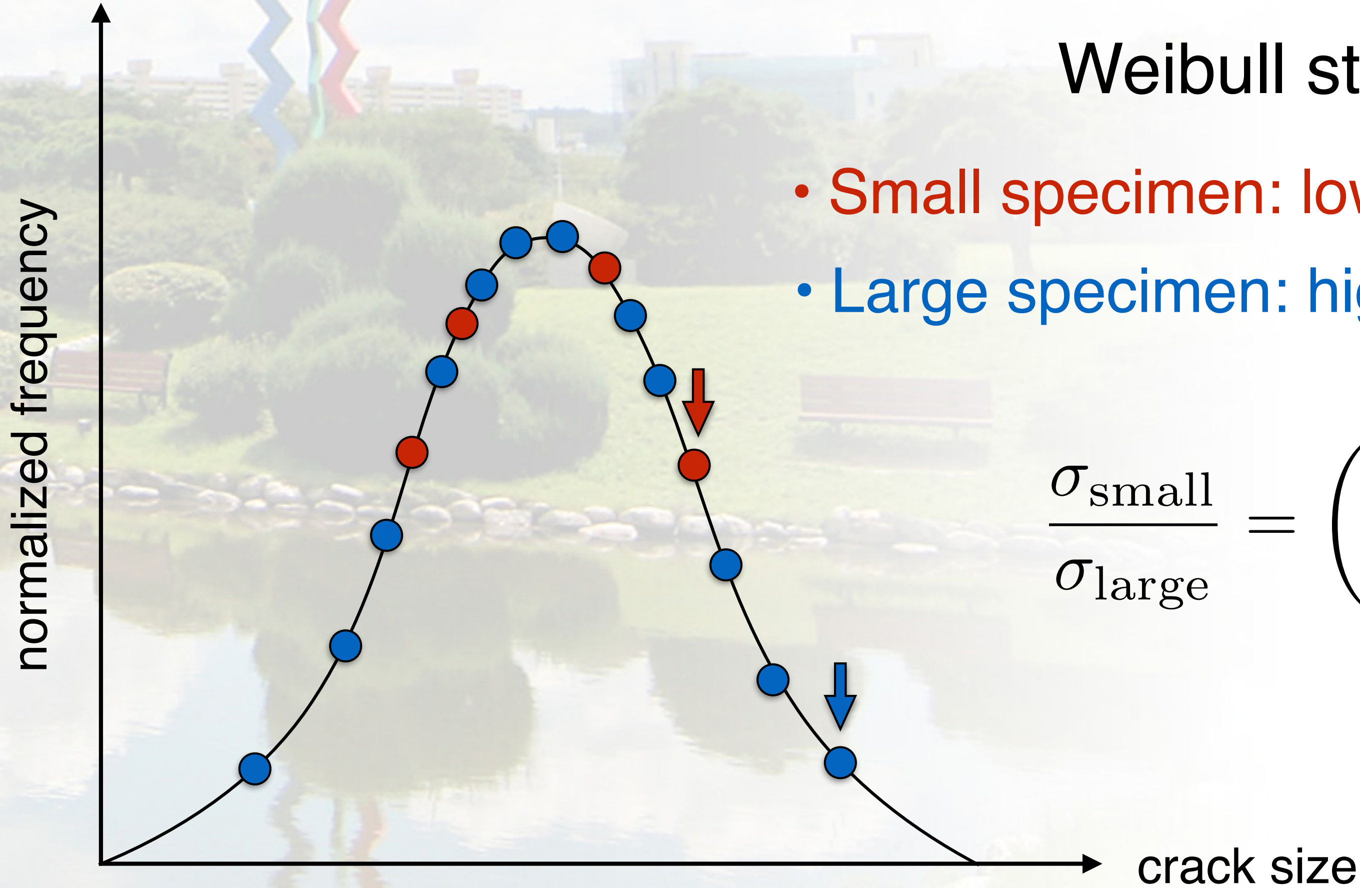




https://en.wikipedia.org/wiki/Frank-Read_source

Origin of Size Effects

Confinement: Weibull Statistics



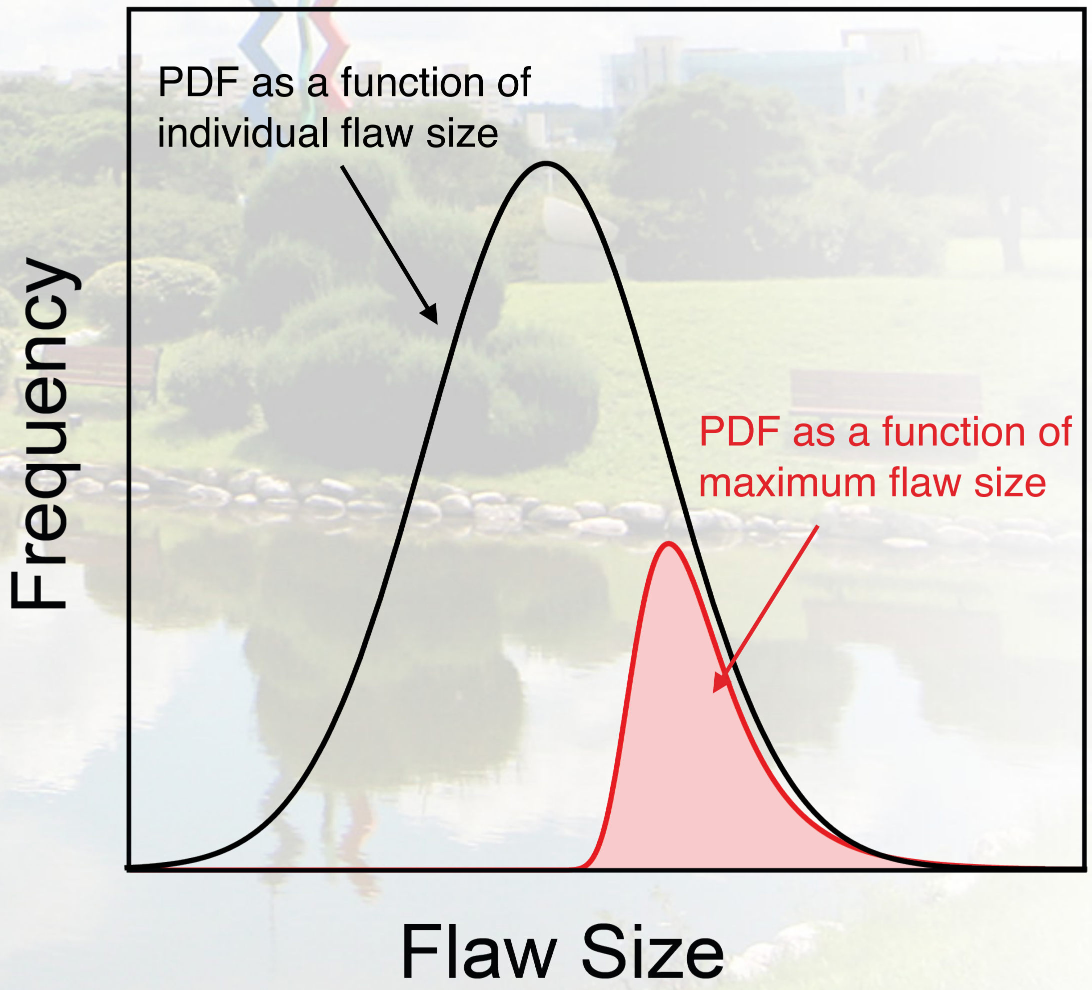
Weibull statistics

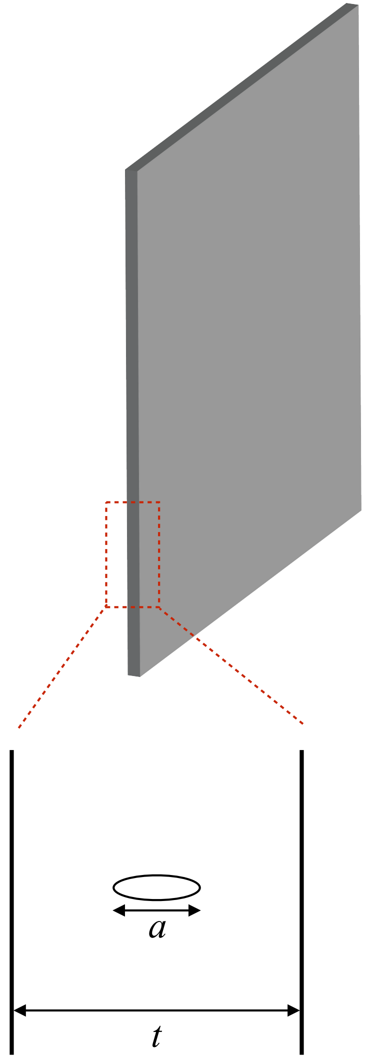
- Small specimen: low sampling number
- Large specimen: high sampling number

$$\frac{\sigma_{\text{small}}}{\sigma_{\text{large}}} = \left(\frac{V_{\text{large}}}{V_{\text{small}}}\right)^{\frac{1}{m}}$$

Origin of Size Effects

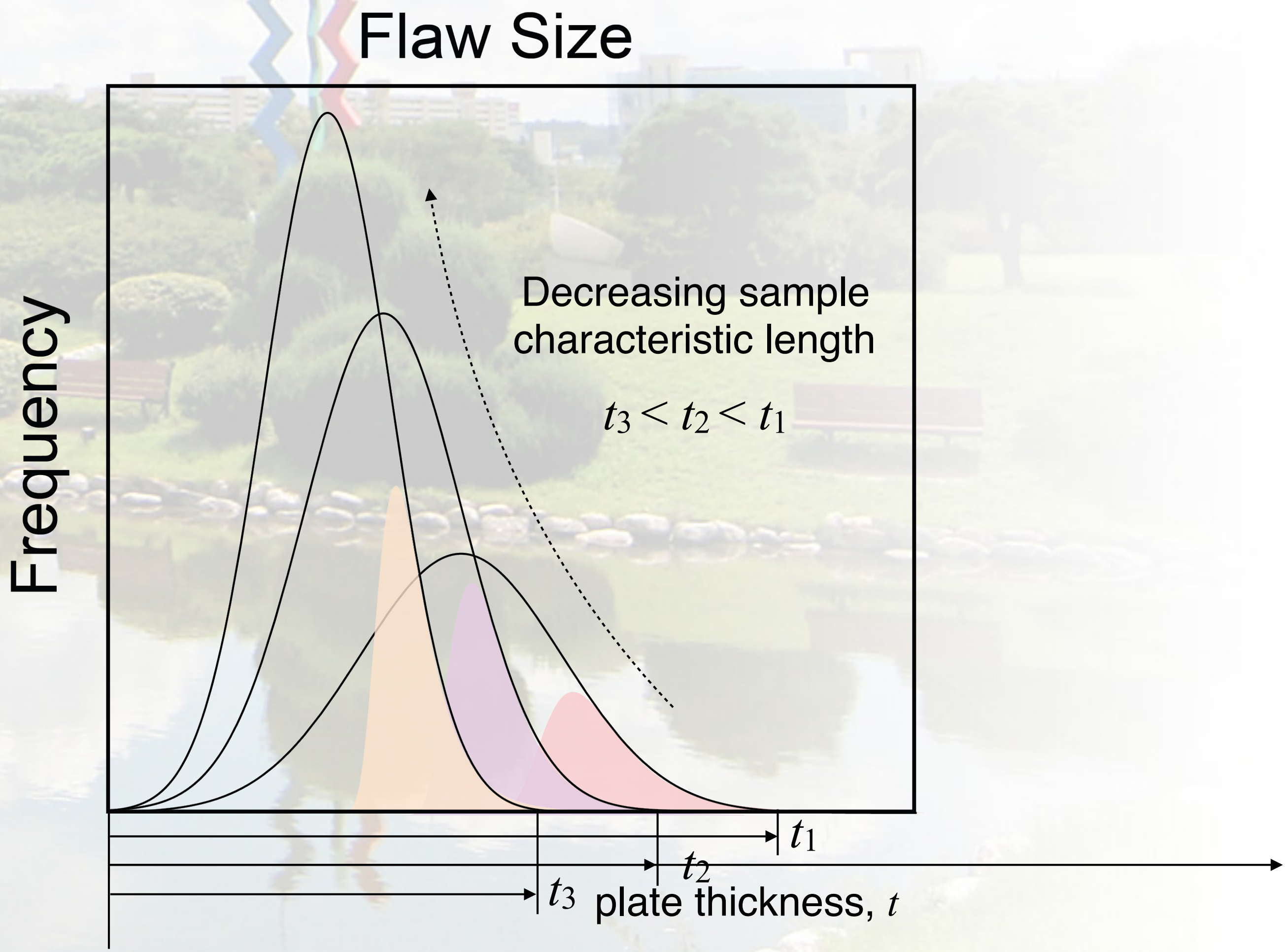
Confinement: Weibull Statistics

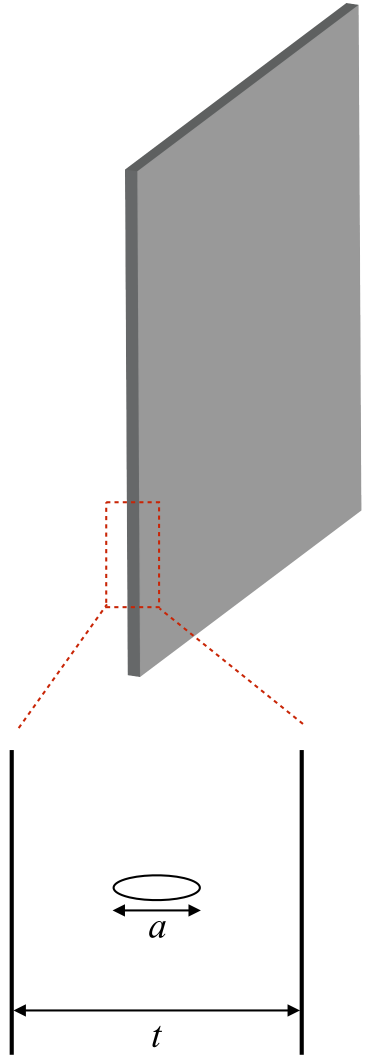




Origin of Size Effects

Confinement: Weibull Statistics





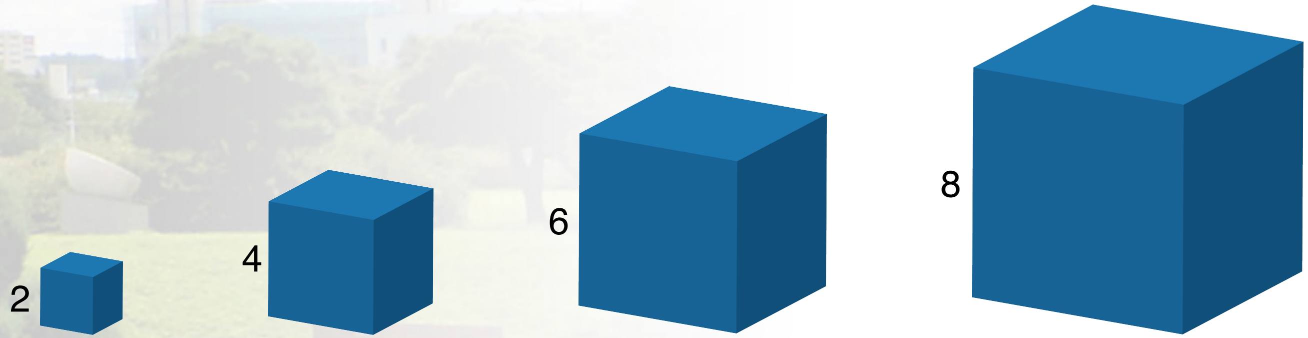
Origin of Size Effects

Confinement

Surface

Origin of Size Effects

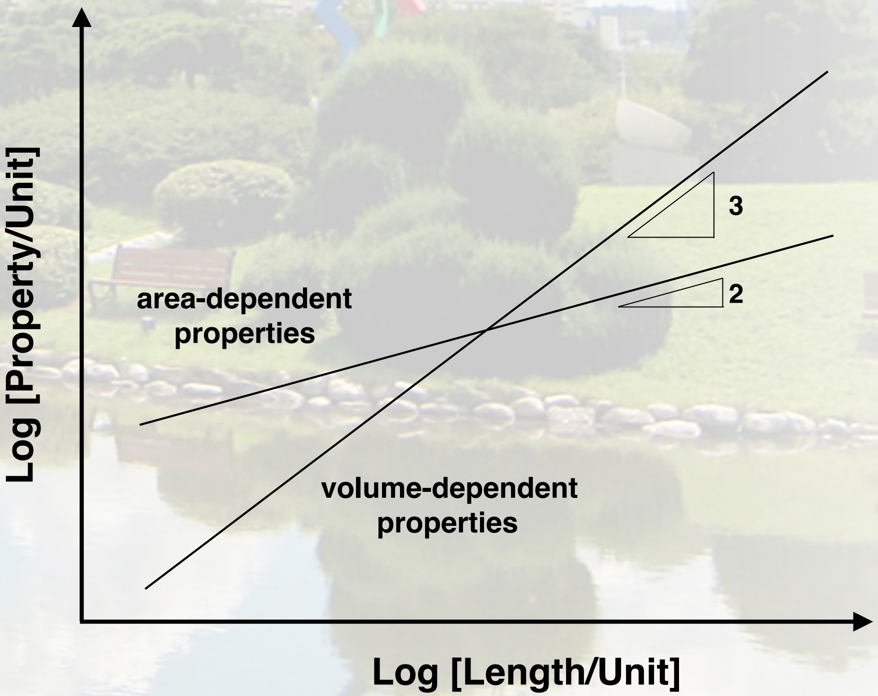
Surface



Surface area	24	96	216	384
Volume	8	64	216	512
Surface/Volume	**3.0**	**1.5**	**1.0**	**0.75**

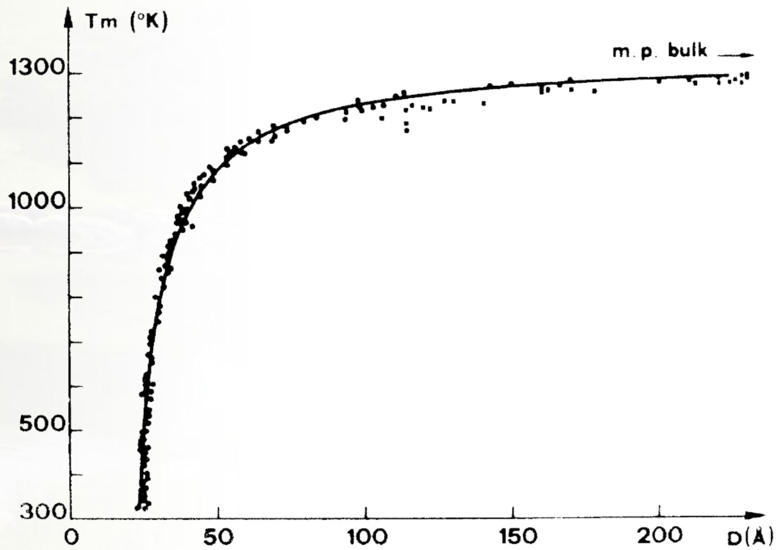
Origin of Size Effects

Volume- vs Area-Dependent Properties



$$\Delta T_m(r) = -T_m(\infty)\frac{4\gamma_{SL}}{H_f(\infty)\,\rho_s r}$$

Gibbs-Thomson Equation



Buffat, P. & Borel, J.-P. Size effect on the melting temperature of gold particles. Phys. Rev. A 13, 2287–2298 (1976).

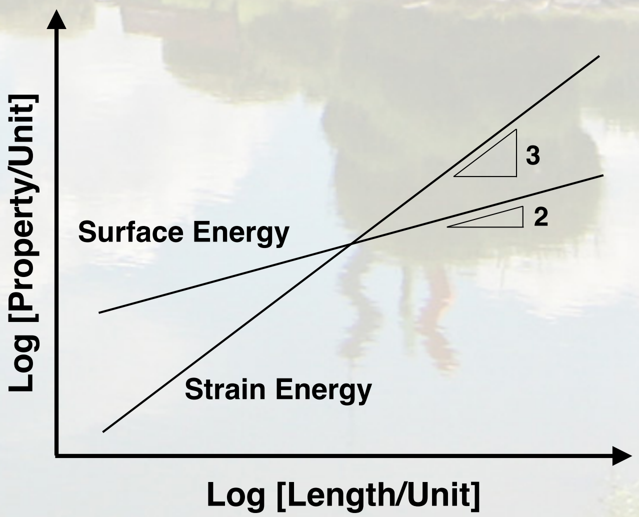
Origin of Size Effects

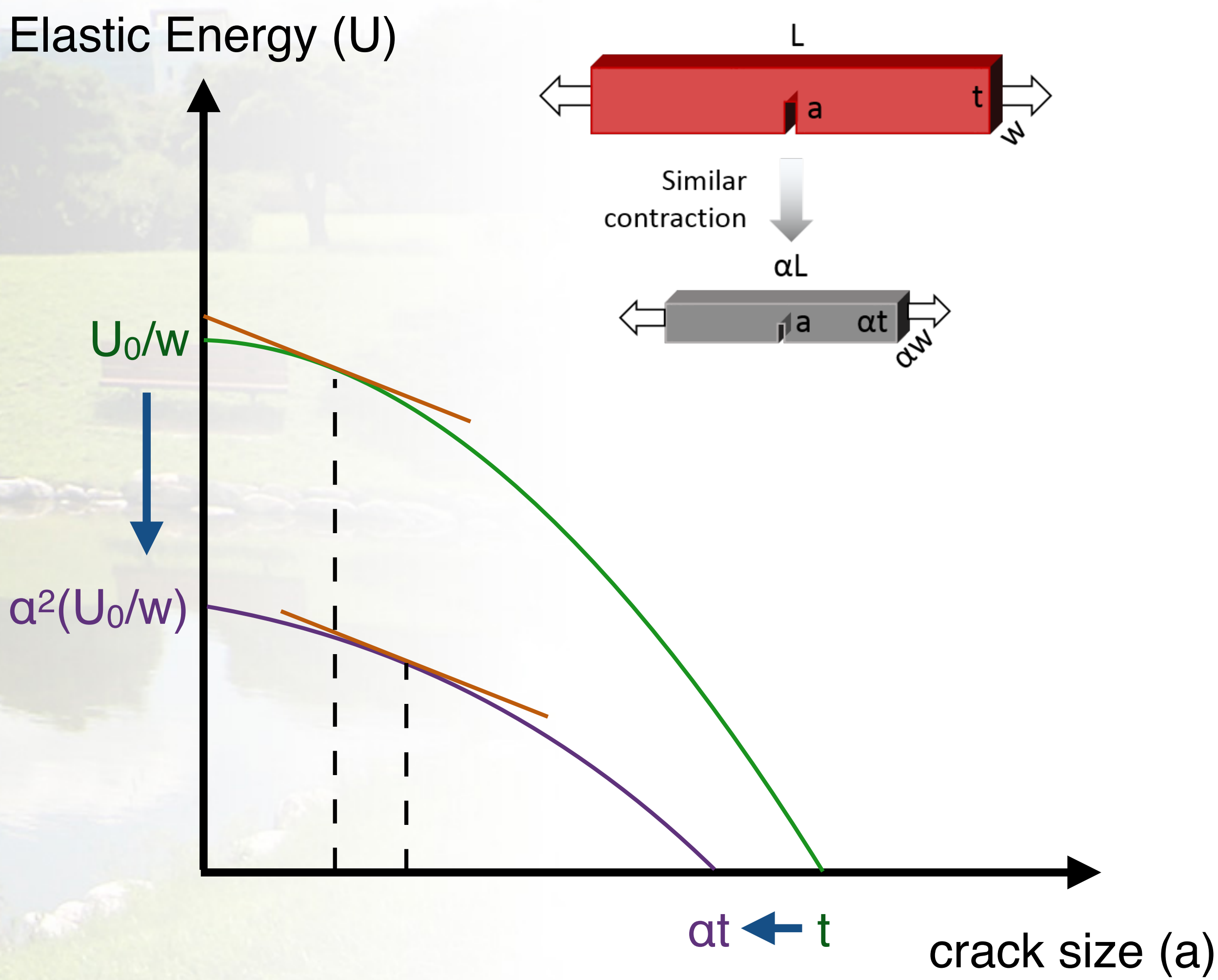
Volume- vs Area-Dependent Properties: Griffith Criterion

Criterion for crack propagation at constant displacement condition

$$-\frac{d\Pi}{dA} = -\frac{dU}{dA} \geq 2\gamma_s$$

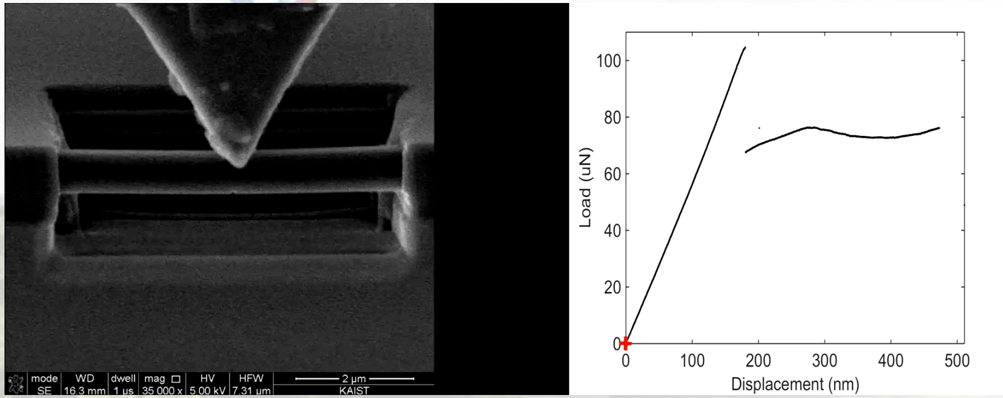
$$-\frac{dU}{d(wa)} = -\frac{d(U/w)}{da} \geq 2\gamma_s$$



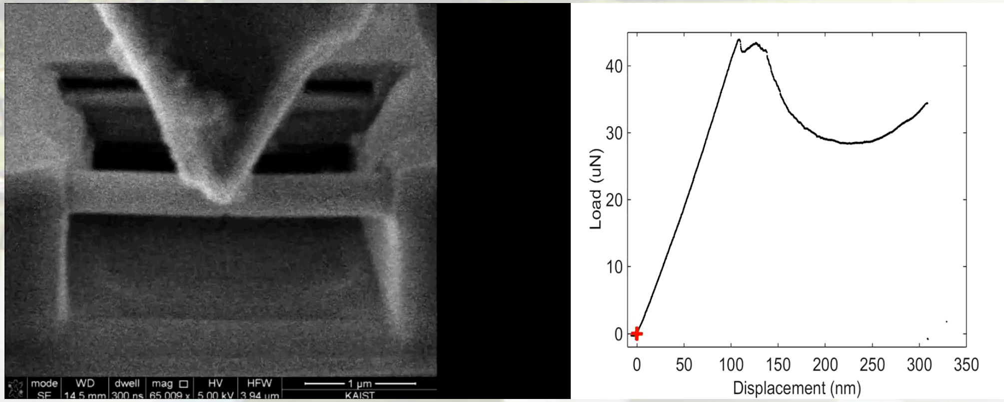


Origin of Size Effects

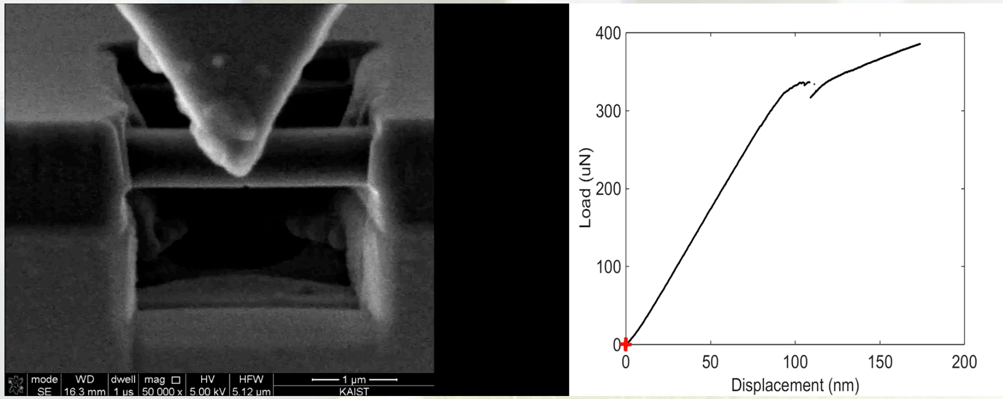
Volume- vs Area-Dependent Properties: Griffith Criterion



- Length: 6 micron
- Thickness: 500 nm



- Length: 3 micron
- Thickness: 250 nm



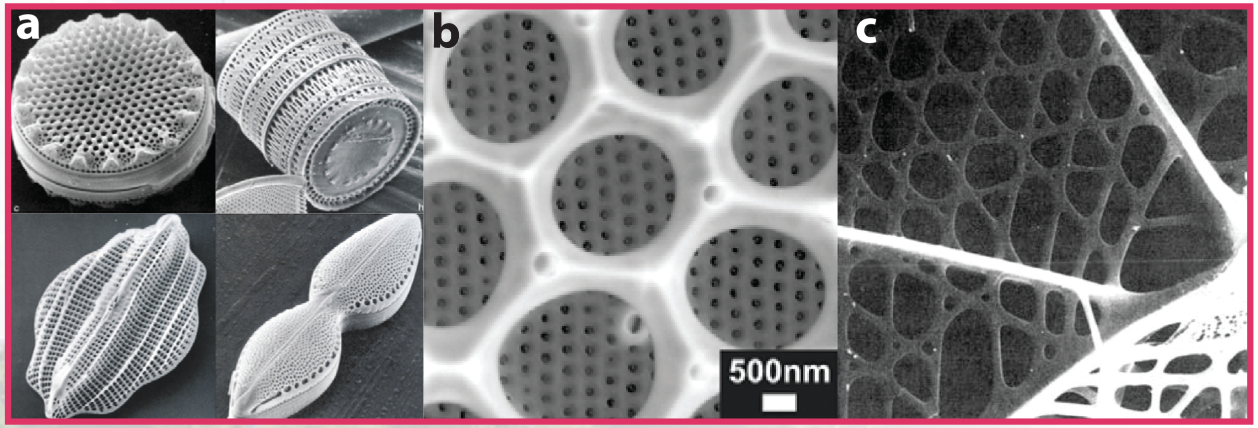
- Length: 3 micron
- Thickness: 500 nm

Seemingly Contradicted

How can we render small-only properties to be attainable at larger scales?

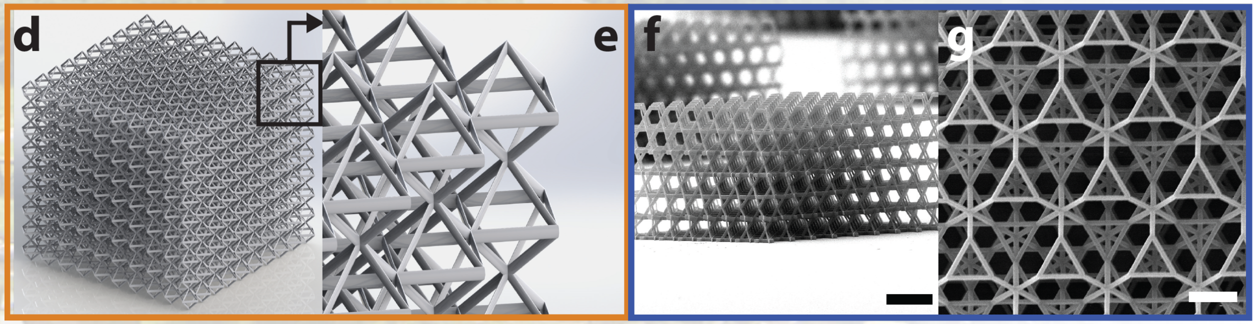
Ceramics Nano-architectures

Natural Materials

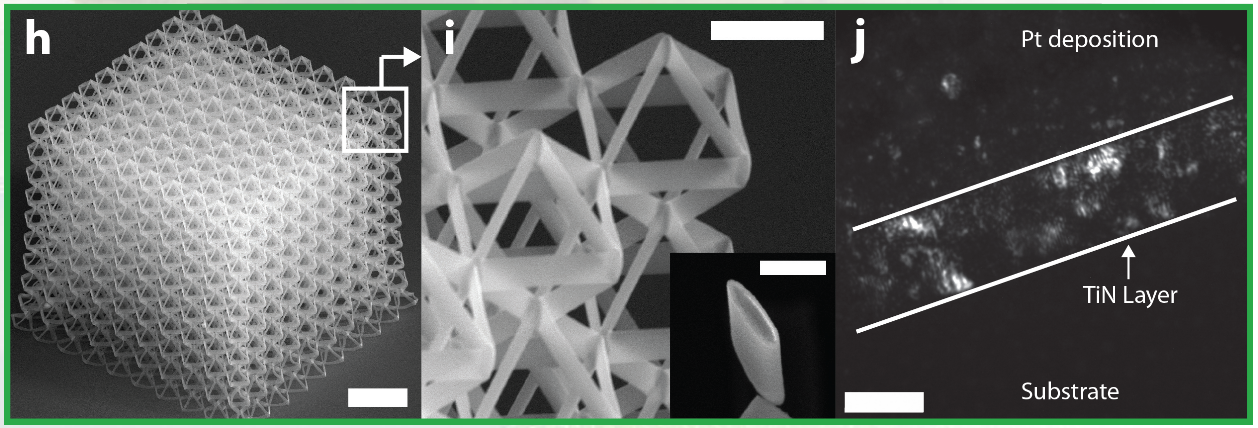


Computer-aided Design

Fabrication - 3D Kagome Unit Cell



Fabrication - Octahedral Unit Cell



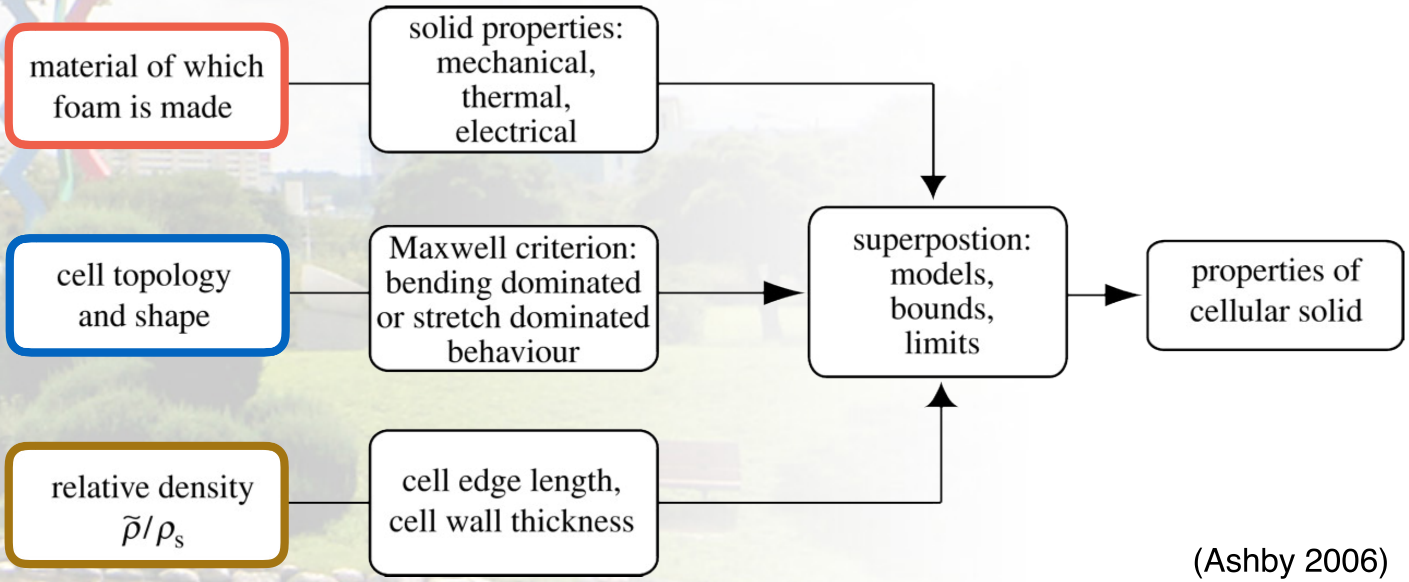
Scales of Nanotruss

Jang, D., Meza, L. R., Greer, F., & Greer, J. R. (2013). Fabrication and deformation of three-dimensional hollow ceramic nanostructures. Nature materials.

Design Factors to Consider:

1. Integration of Scaling Laws

Scaling Law for Porous Materials



(Ashby 2006)

$$\sigma_y = \sigma_o (\tilde{\rho})^m$$

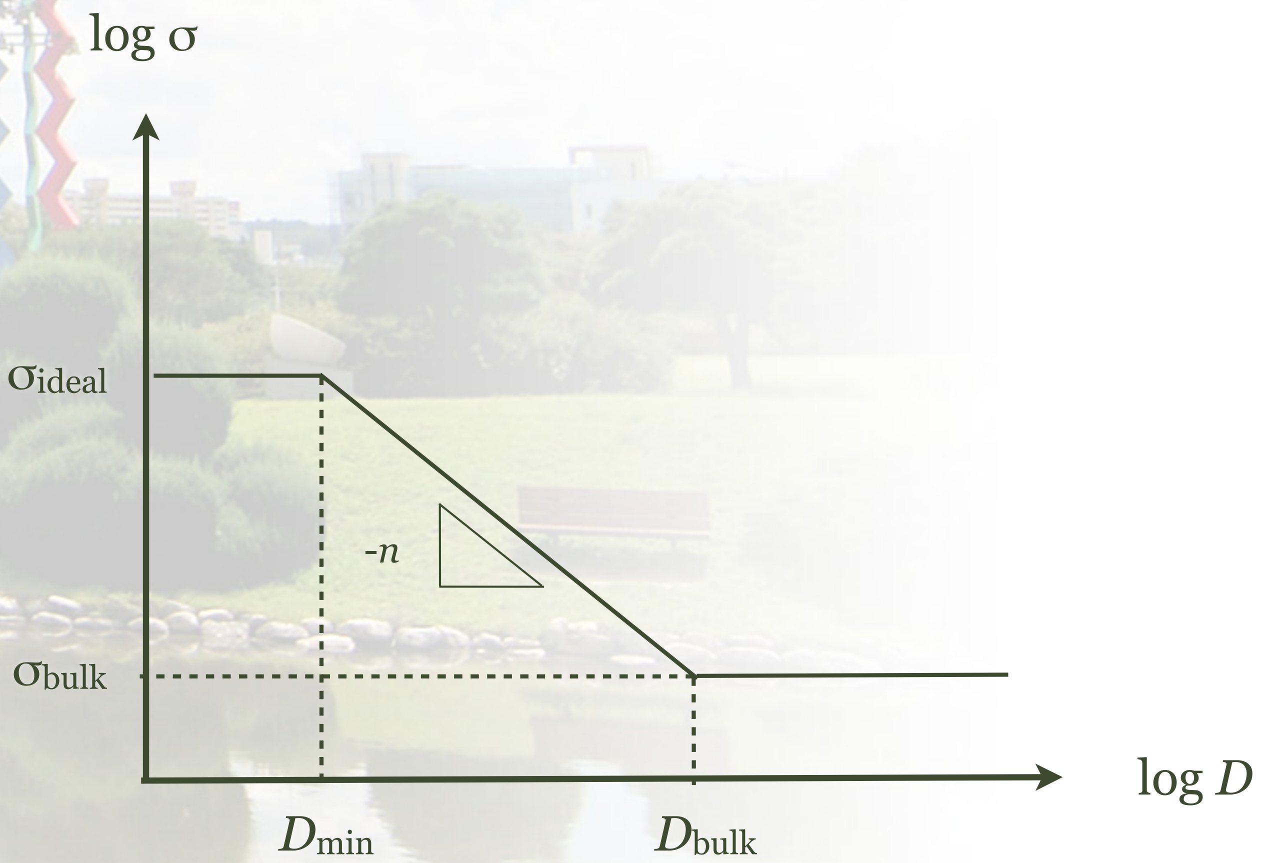
σ_y :Strength of structure

σ_o :Strength of base material

m :Geometric factor

$\tilde{\rho}$:Relative density

Scaling Law for Base Materials



$$\sigma_{\mathrm{small}} = \sigma_{\mathrm{bulk}} \left(\frac{D}{D_{\mathrm{bulk}}} \right)^{-n} , D_{\min} < D < D_{\mathrm{bulk}}$$

Combination of Two Scaling Laws

$$\sigma_{\text{porous}} = \sigma_{\text{solid}} (\tilde{\rho})^m$$

$$\sigma_{\text{solid}} = \sigma_{\text{bulk}} \left(\frac{D}{D_{\text{bulk}}} \right)^{-n}$$

$$\sigma_{\text{porous}} = \sigma_{\text{bulk}} \left(\tilde{\rho} \left(\frac{D_{\text{bulk}}}{D} \right)^{\frac{n}{m}} \right)^m$$

$$\sigma_{\text{porous}} = \sigma_{\text{bulk}} \left(\tilde{\rho}_e \right)^m$$

$$\tilde{\rho}_e = \tilde{\rho} \left(\frac{D_{\text{bulk}}}{D} \right)^{\frac{n}{m}}$$

The nano-architectures behave as if they have higher relative densities.

Design Factors to Consider:
2. Selection of Base Material

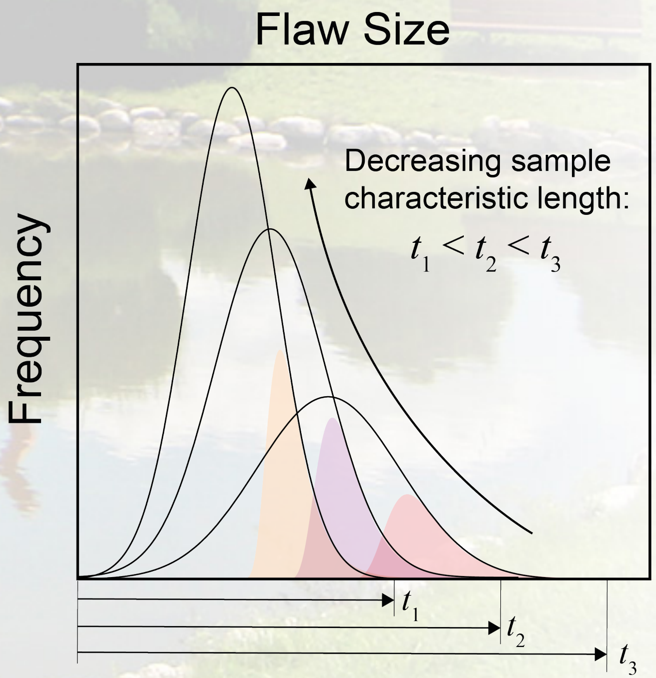
Size-dependent Fracture Strengths of Ceramics:

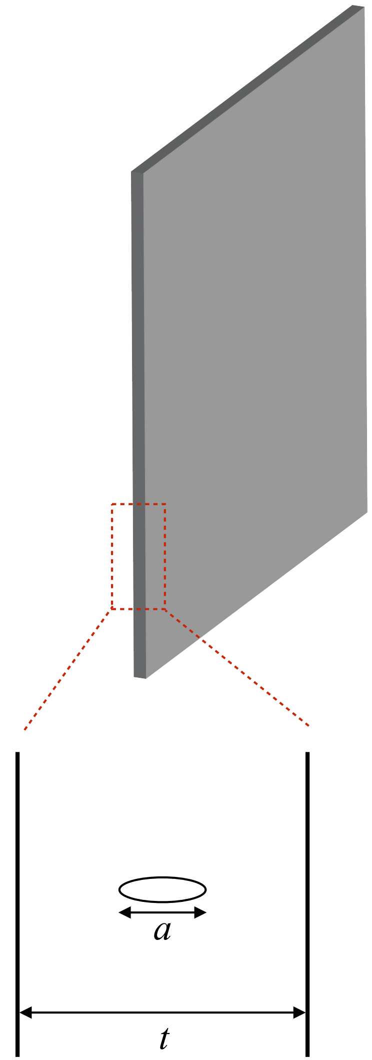
Linear Elastic Fracture Mechanics

$$f_v(a,t;m) = \frac{m}{2\alpha t}\left(\frac{A}{A_0}\right)^{-\left(\frac{m}{2}+1\right)} \exp\left[-\frac{A}{A_0}\left(\frac{a}{\alpha t}\right)^{-\frac{m}{2}}\right]$$

$$\bar{a} = \alpha t \left(\frac{A}{A_0}\right)^{\frac{2}{m}} \Gamma\left(1-\frac{2}{m}\right)$$

$$\bar{\sigma} = \frac{K_{\mathrm{IC}}}{\sqrt{\pi \bar{a}}} F\left(\bar{\phi}\right) = \frac{G(\beta,\nu)F(\bar{\phi})}{\sqrt{\alpha\Gamma\left(1-\frac{2}{m}\right)}}\frac{K_{\mathrm{IC}}}{\sqrt{\pi t}}\left(\frac{A_0}{A}\right)^{\frac{1}{m}}$$



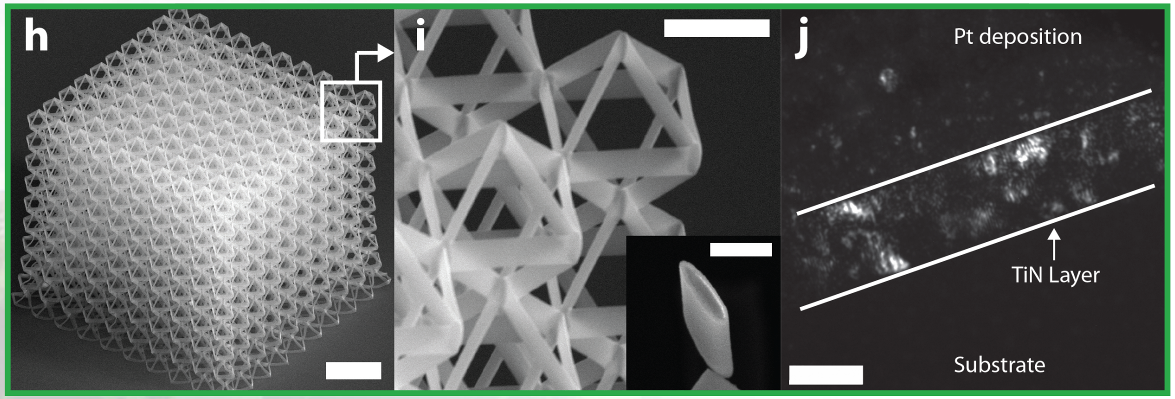


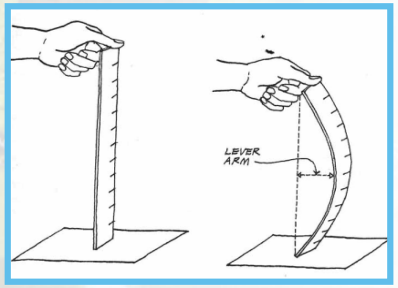
Design Factors to Consider:

3. Determination of Architectures & Dimensions

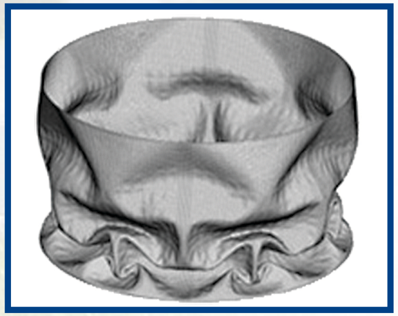
Geometric Conditions for Buckling Instabilities

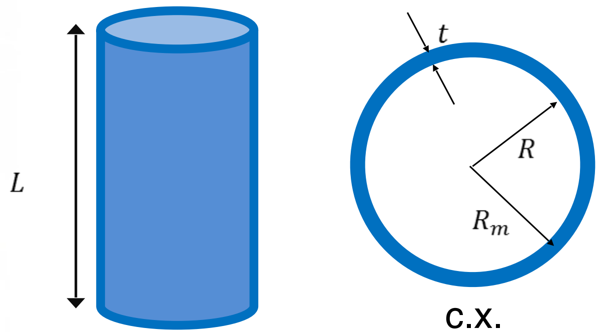
Fabrication - Octahedral Unit Cell





- Euler buckling: $\sigma_{EB} = \frac{\pi^2 E}{2k^2}\left(\frac{R_m}{L}\right)^2$

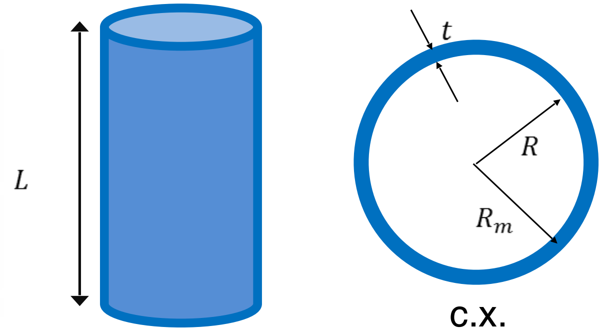




- Shell buckling: $\sigma_{SB} = \frac{E}{\sqrt{3(1-\nu^2)}}\frac{t}{R}$

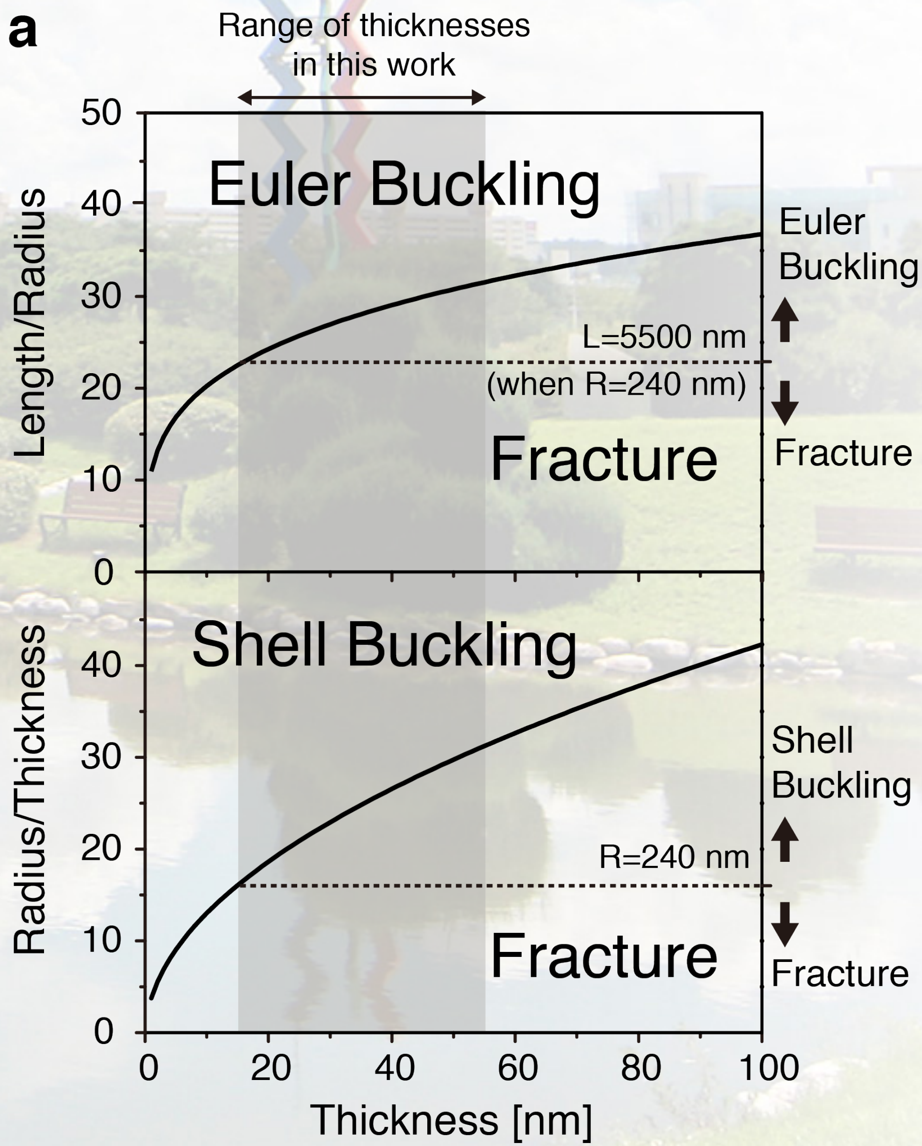
Geometric Conditions for Buckling Suppression

$$\sigma_{SB} = \frac{E}{\sqrt{3\left(1-\nu^2\right)}}\frac{t}{R} \qquad \sigma_{EB} = \frac{\pi^2 E}{2k^2}\left(\frac{R_m}{L}\right)^2$$

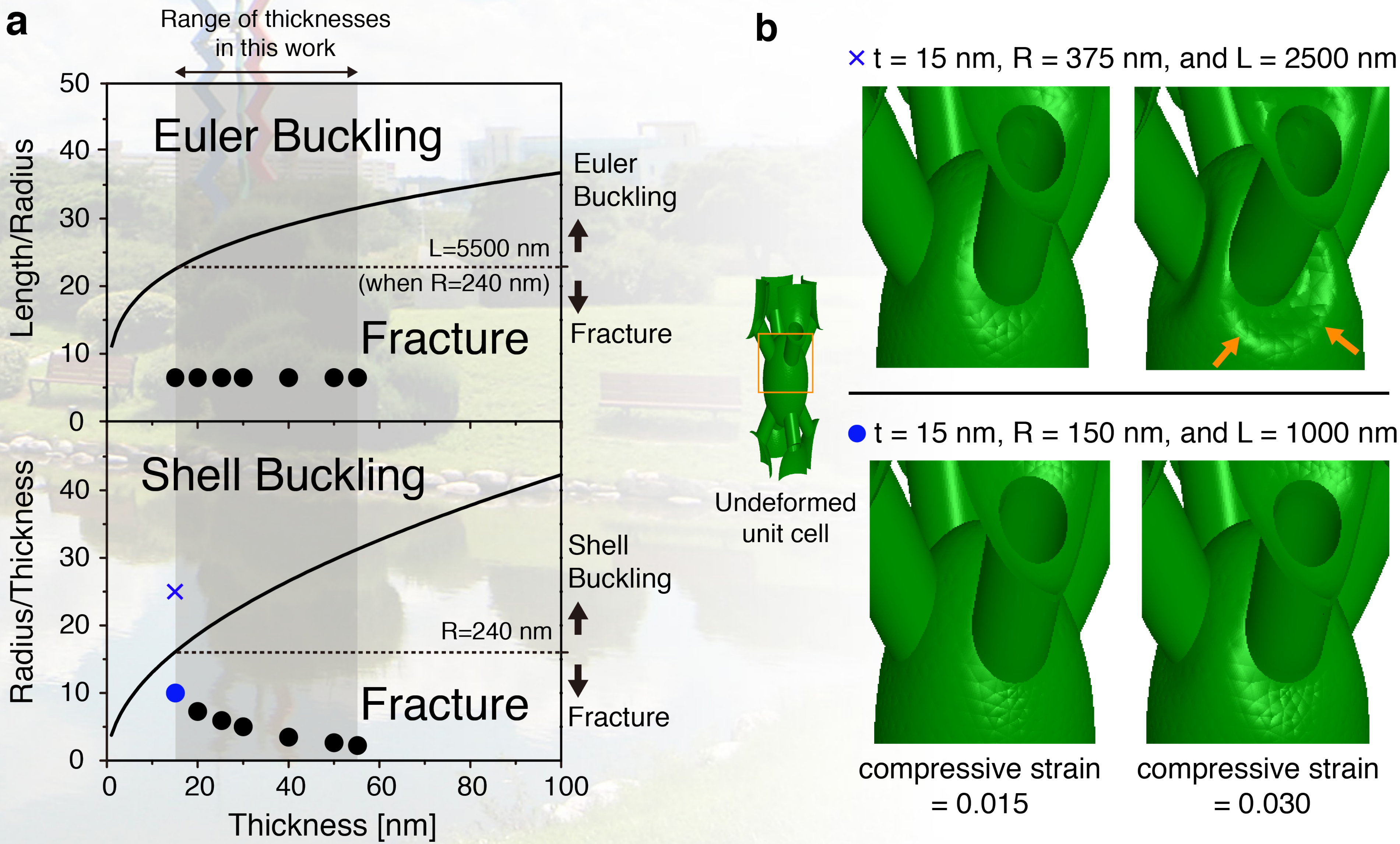


1. **Smaller *t*** is preferred for amplification of properties.
2. Buckling stresses should be higher than fracture strength.
   I. **Smaller *R*** is preferred to suppress shell buckling.
   II. **Smaller *L*** is preferred to suppress Euler buckling.

Geometric Conditions for Buckling Suppression



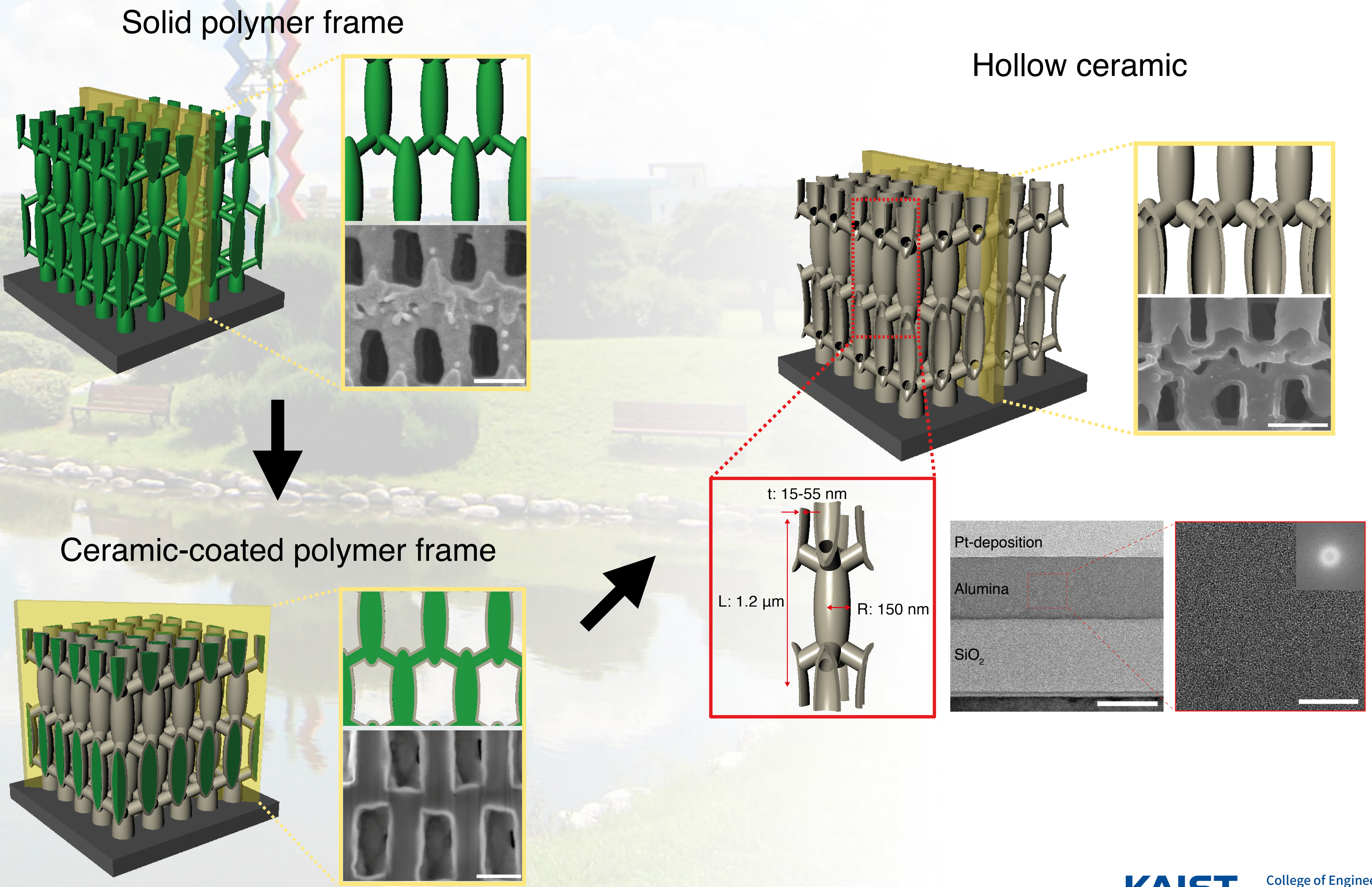
Geometric Conditions for Buckling Suppression



Design Factors to Consider:

4. Existence of Scalable Fabrication Technique

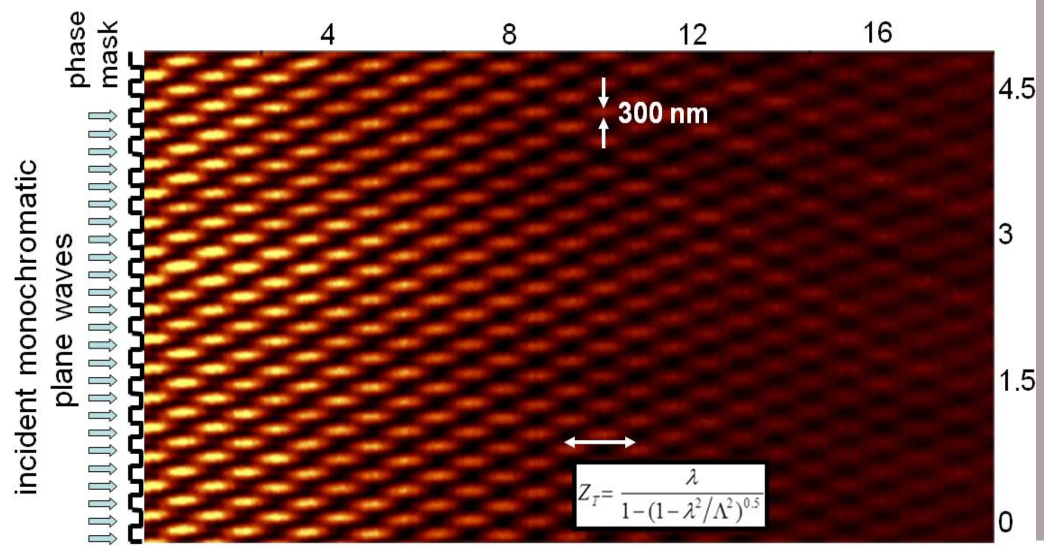
Fabrication Overview

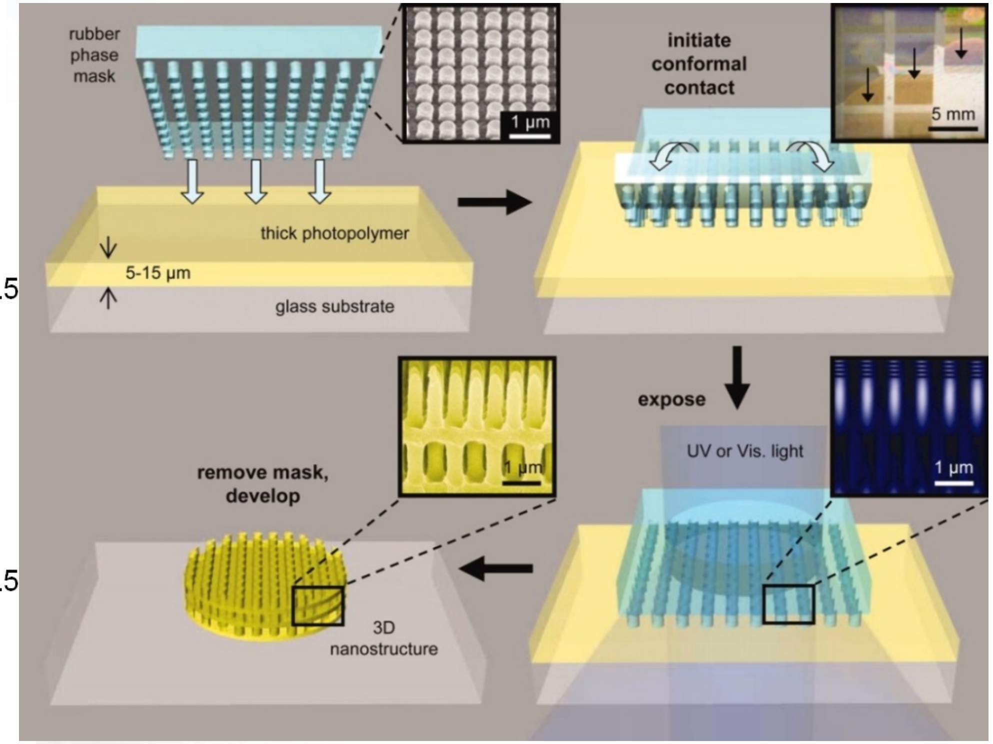


Specimen Fabrication Process

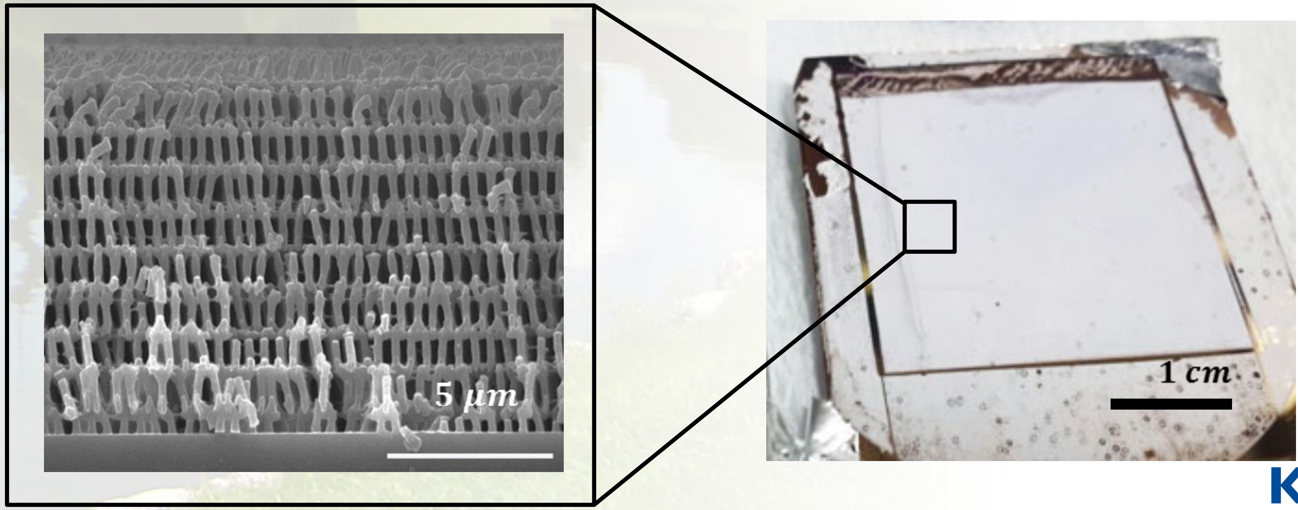
Talbot Effect

:Near-field diffraction effect

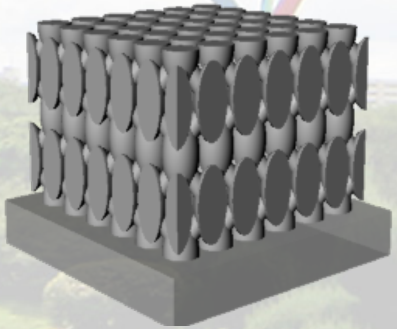


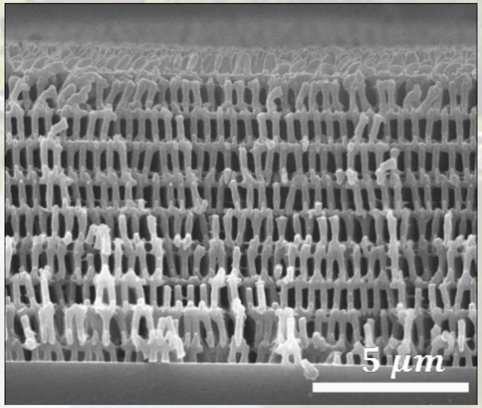


(Shir et al. 2007)



Specimen Fabrication Process



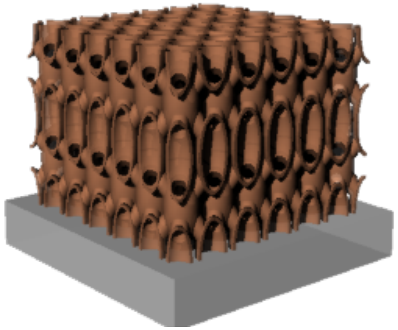


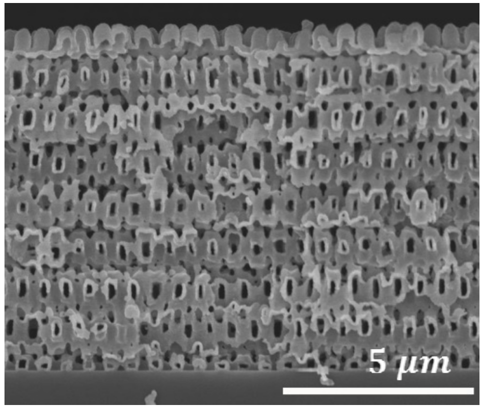
ALD (Atomic layer deposition)

- Al_2O_3 deposition (15 - 55 nm)
- TMA + H_2O precursors

Furnace

- Burning out polymer frame
- 350°C for 5hr, 500°C for 2 hr



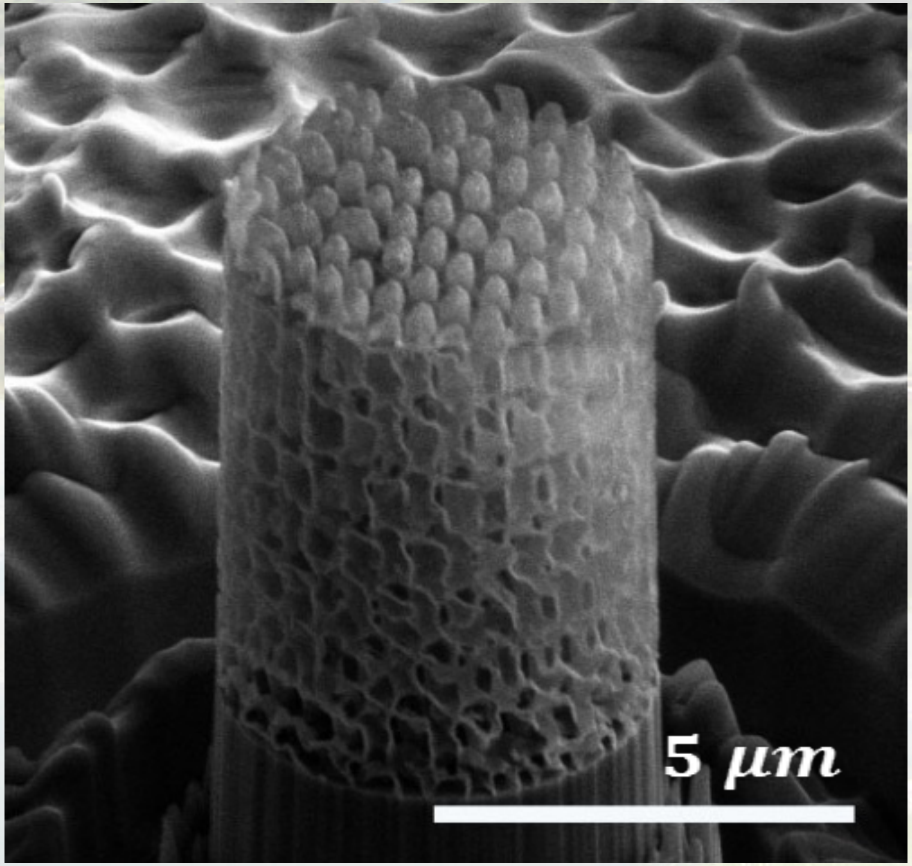


Mechanical Characterizations

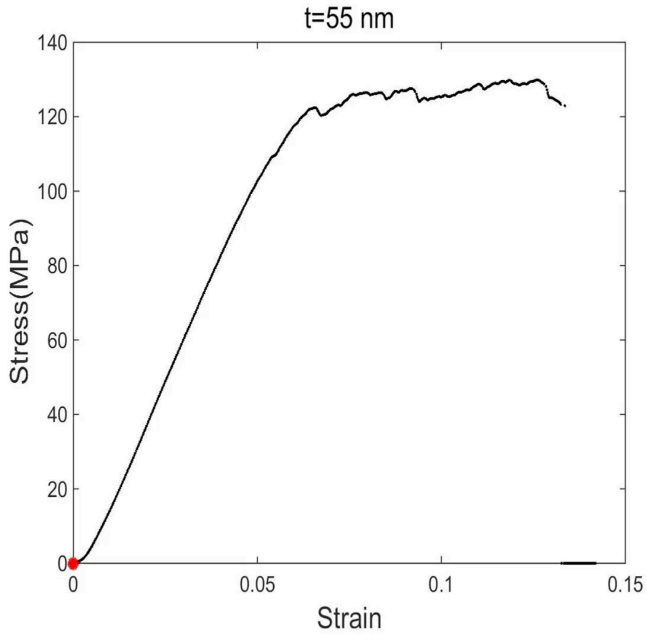
Mechanical Characterization of Ceramic Nano-Architectures

Pillar-shaped samples fabrication by Focused Ion Beam (FIB)

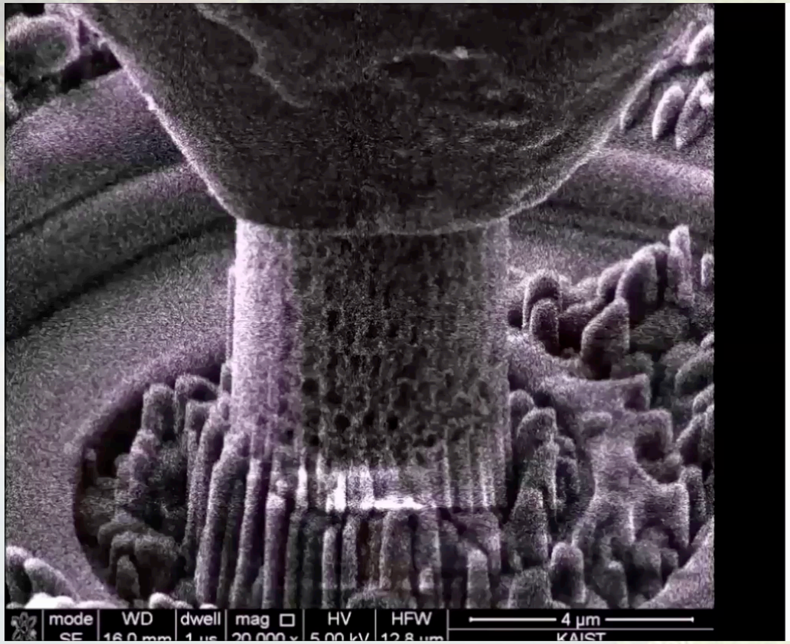
- 5 micron diameter
- 8 micron height

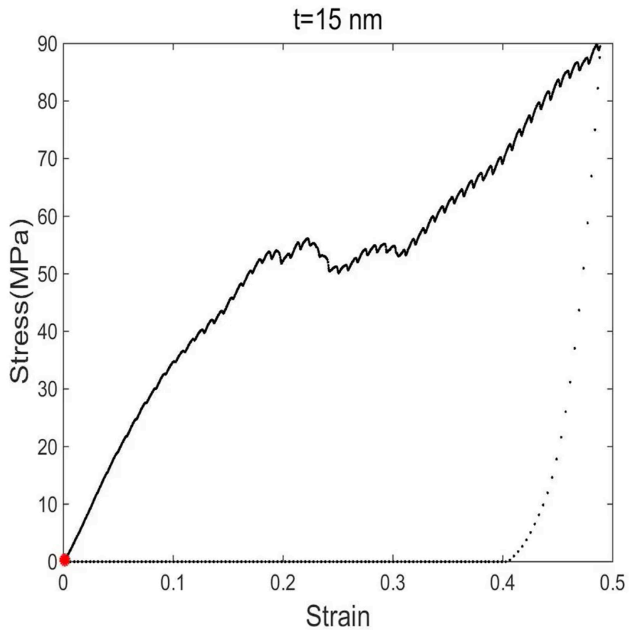


ALD cycles	Thickness	Density	Relative Density
75	15 nm	105 ± 6.59 kg/m³	0.036
100	20 nm	123 ± 1.50 kg/m³	0.042
130	25 nm	172 ± 7.41 kg/m³	0.059
160	30 nm	228 ± 19.6 kg/m³	0.079
220	40 nm	287 ± 14.4 kg/m³	0.099
300	50 nm	377 ± 6.10 kg/m³	0.130
350	55 nm	451 ± 14.7 kg/m³	0.155


t=55 nm
Stress(MPa)
Strain
0
20
40
60
80
100
120
140
0
0.05
0.1
0.15

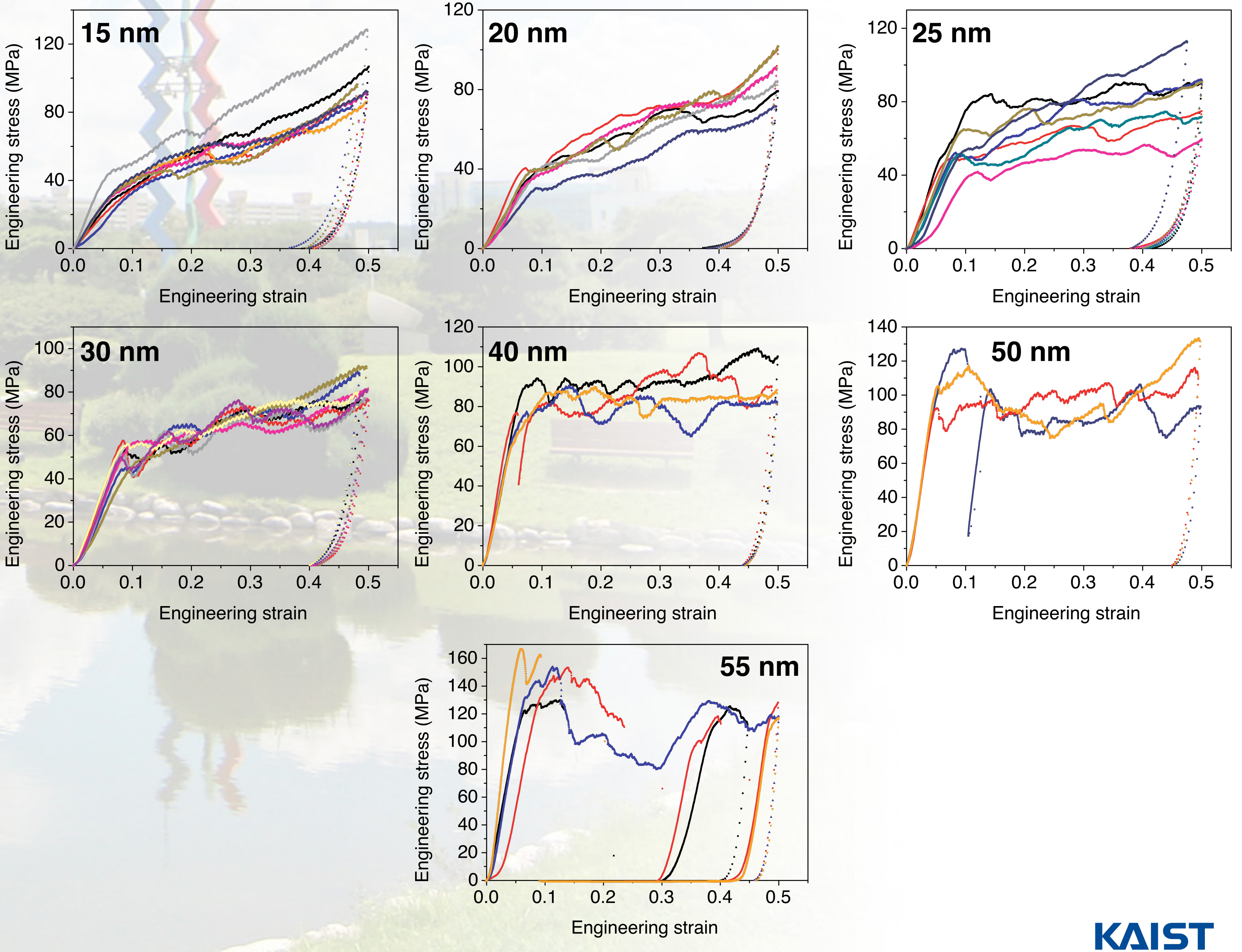
t = 55 nm


mode
WD
dwell
mag
HV
HFW
4 μm

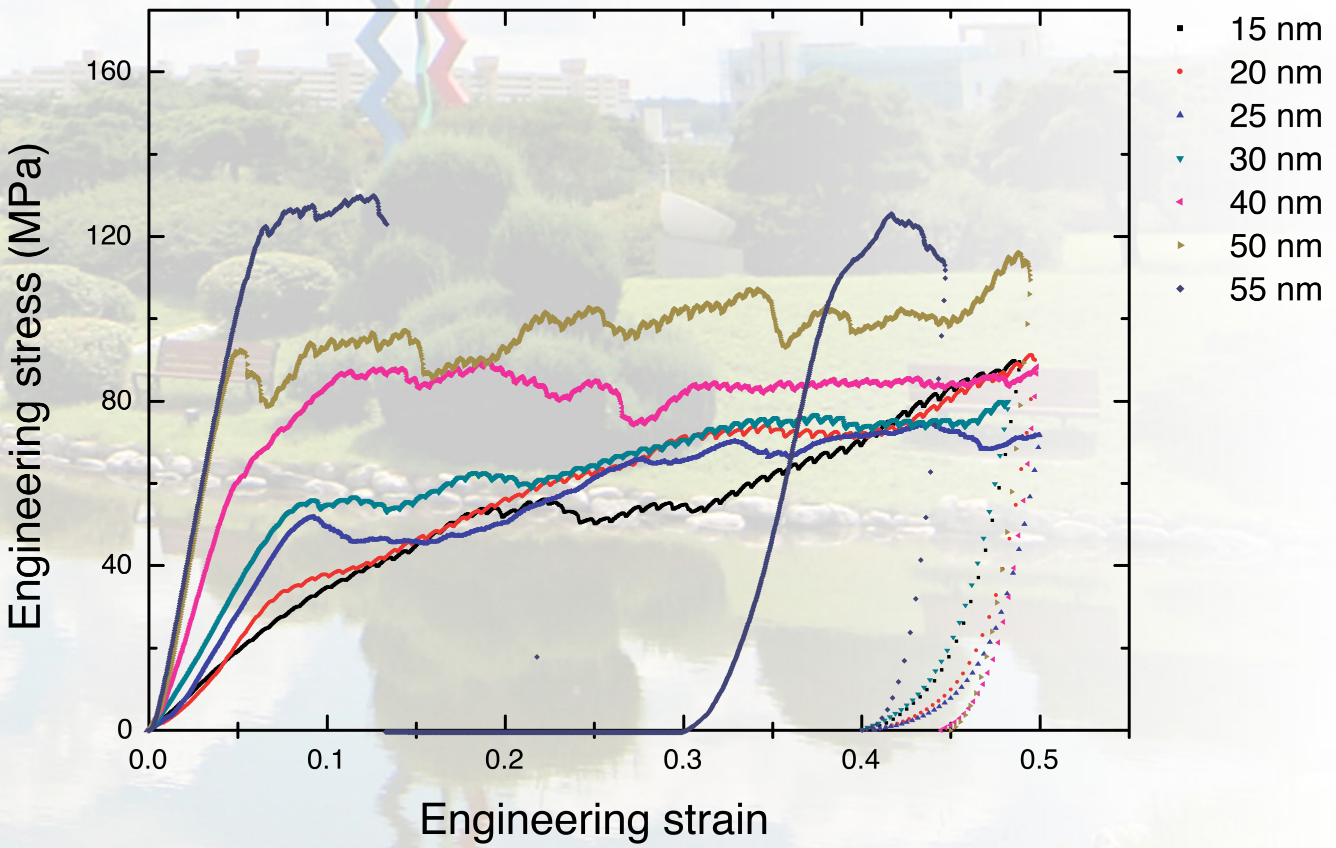

t=15 nm
Stress(MPa)
Strain
0
10
20
30
40
50
60
70
80
90
0
0.1
0.2
0.3
0.4
0.5

t = 15 nm

Mechanical Characterization of Ceramic Nano-Architectures

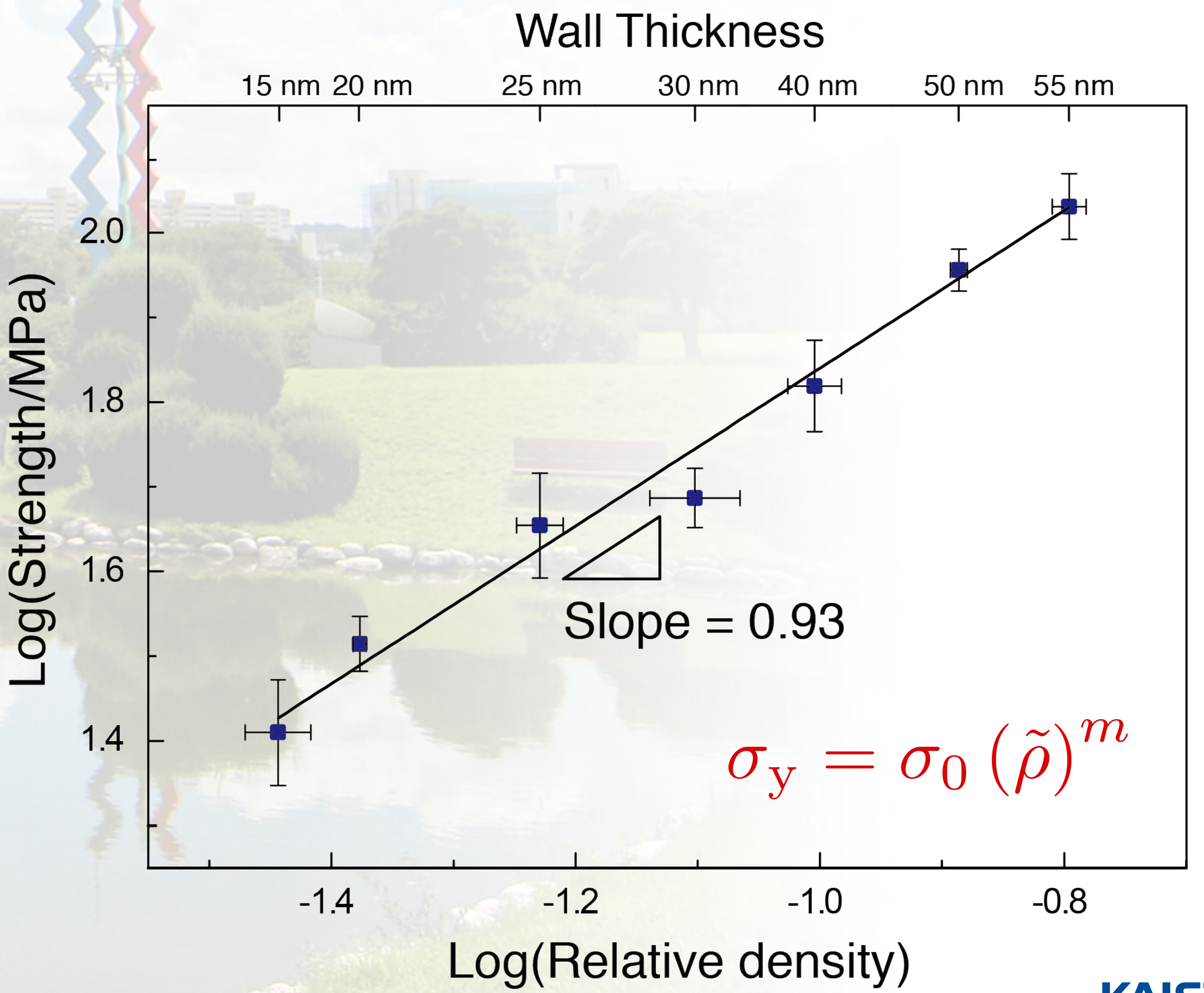


Mechanical Characterization of Ceramic Nano-Architectures

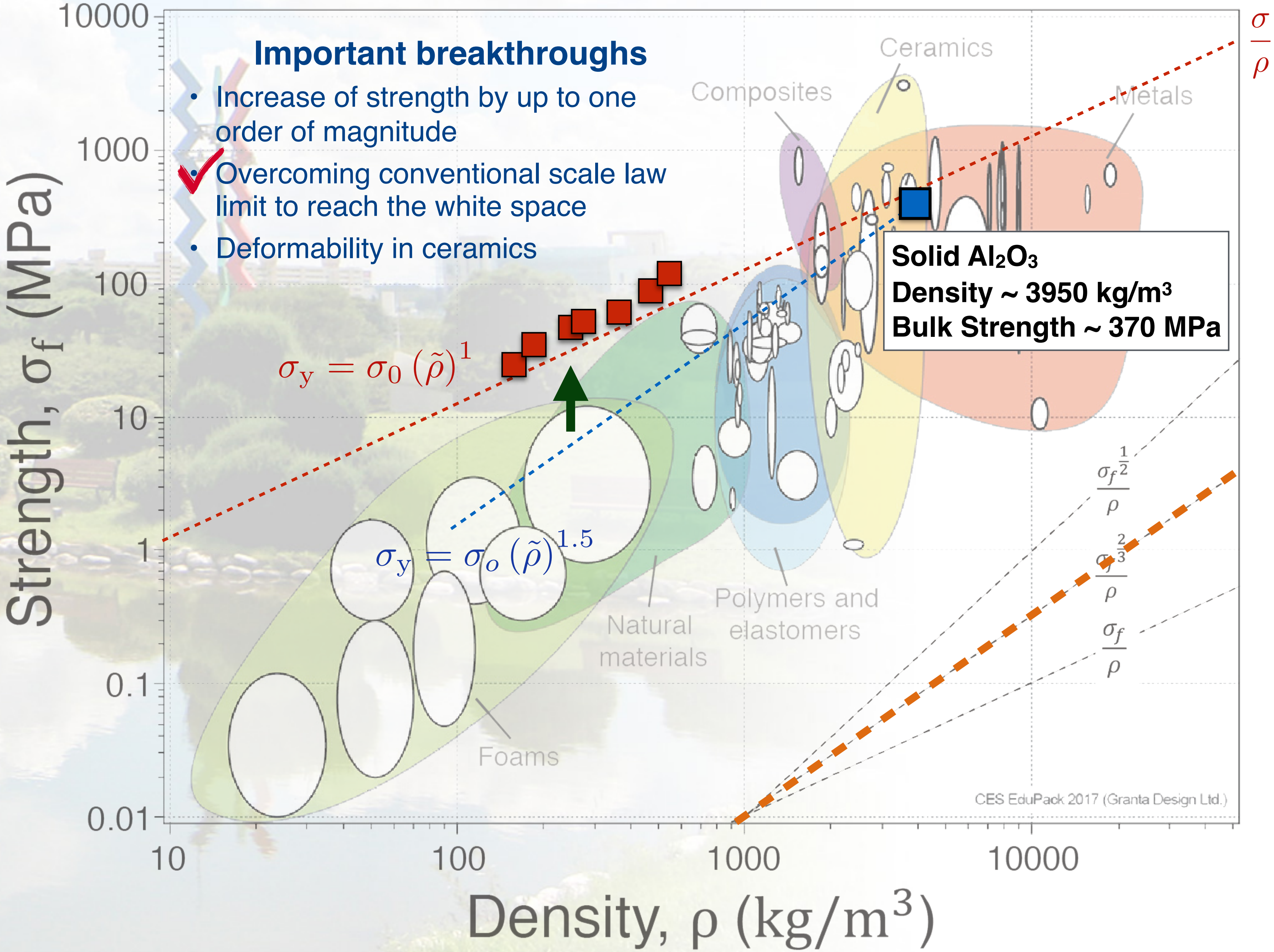


Thickness	Strength
15 nm	25.7 ± 3.7 MPa
20 nm	32.7 ± 2.5 MPa
25 nm	45.1 ± 6.4 MPa
30 nm	48.6 ± 3.9 MPa
40 nm	65.9 ± 8.2 MPa
50 nm	90.3 ± 5.2 MPa
55 nm	110.9 ± 9.9 MPa

Mechanical Characterization of Ceramic Nano-Architectures



Ceramics Nano-architectures



Overcoming Conventional Scaling Law Limit

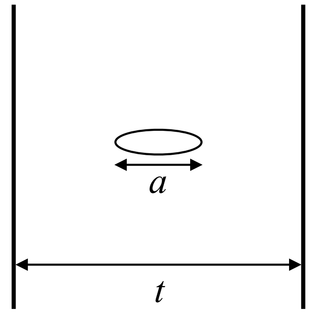
Size-dependent Fracture Strengths of Base Material Based on Griffith Theory

$$\sigma_f = \frac{K_I}{\sqrt{\pi a}} \quad \text{when } t \gg a$$

$$\sigma_f = \frac{K_I}{\sqrt{\pi a}} \cdot \frac{1}{F(\phi)} \quad \text{for finite } t$$

$\phi = \frac{a}{t}$

$F(\phi)>1$: Geometric function



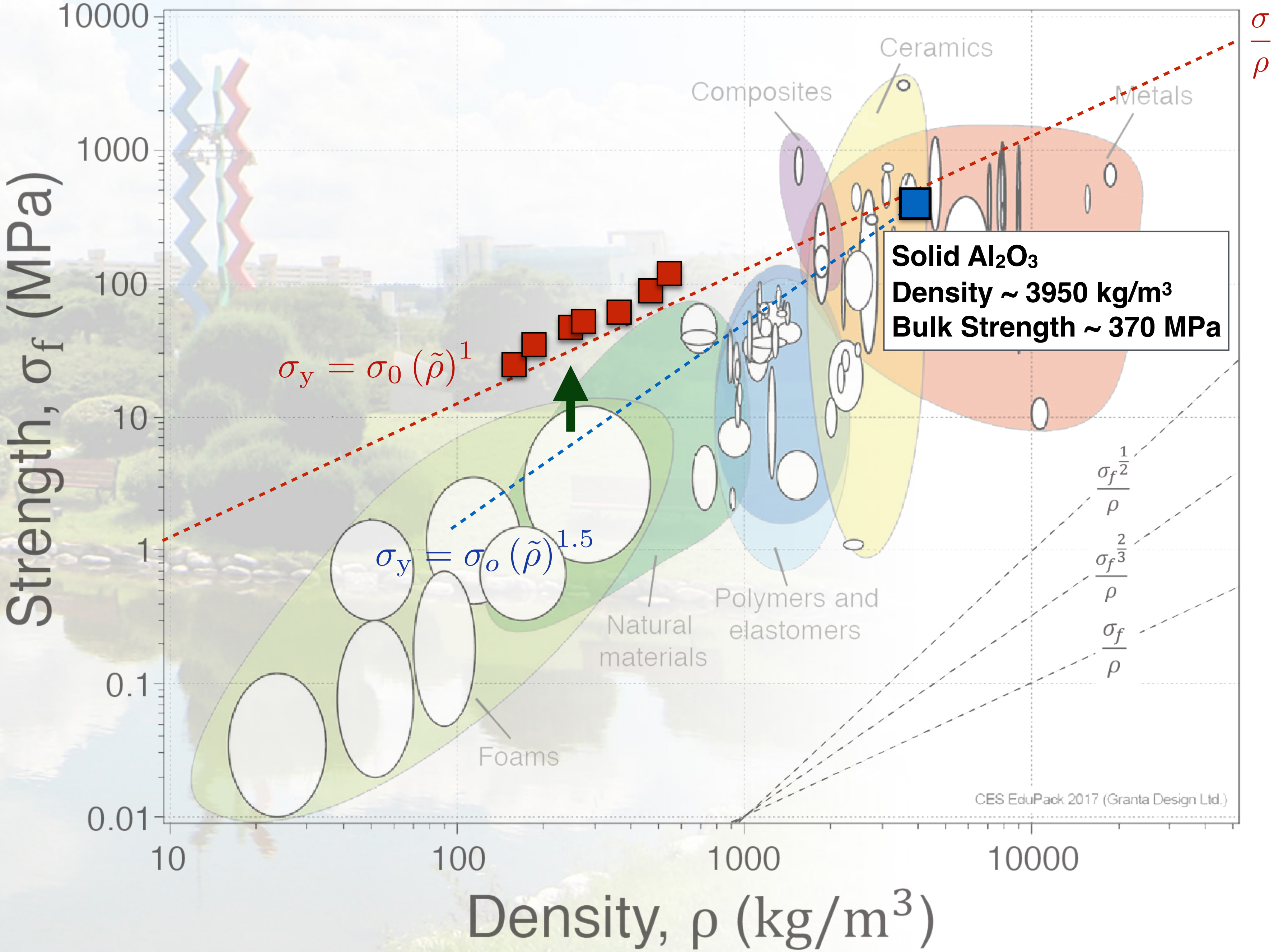
$$\bar{\sigma} = \frac{K_{\mathrm{IC}}}{\sqrt{\pi \bar{a}}} F\left(\bar{\phi}\right) = \frac{G(\beta, \nu) F(\bar{\phi})}{\sqrt{\alpha \Gamma\left(1-\frac{2}{m}\right)}} \frac{K_{\mathrm{IC}}}{\sqrt{\pi t}} \left(\frac{A_0}{A}\right)^{\frac{1}{m}} \approx \alpha \sqrt{\frac{E\gamma}{t}}$$

$$\sigma_{\mathrm{y,nano}} = \sigma_{\mathrm{o,nano}} (\tilde{\rho})^m = \alpha \sqrt{\frac{E\gamma}{t}} (\tilde{\rho})^m$$

$$\approx \alpha \sqrt{\frac{A}{V_{\mathrm{ext}}}} \sqrt{E\gamma} (\tilde{\rho})^{m-\frac{1}{2}}$$

A: Surface area of structure

V_{ext}: Volume of whole sample



Strength, σ_f (MPa)
Density, ρ (kg/m^3)
10000
1000
100
10
1
0.1
0.01
10
100
1000
10000
Ceramics
Composites
Metals
Natural materials
Polymers and elastomers
Foams
$\frac{\sigma}{\rho}$
$\sigma_y = \sigma_0 (\tilde{\rho})^1$
$\sigma_y = \sigma_o (\tilde{\rho})^{1.5}$
Solid Al_2O_3
Density ~ 3950 kg/m³
Bulk Strength ~ 370 MPa
$\frac{\sigma_f^{\frac{1}{2}}}{\rho}$
$\frac{\sigma_f^{\frac{2}{3}}}{\rho}$
$\frac{\sigma_f}{\rho}$
CES EduPack 2017 (Granta Design Ltd.)

How much can we improve further?

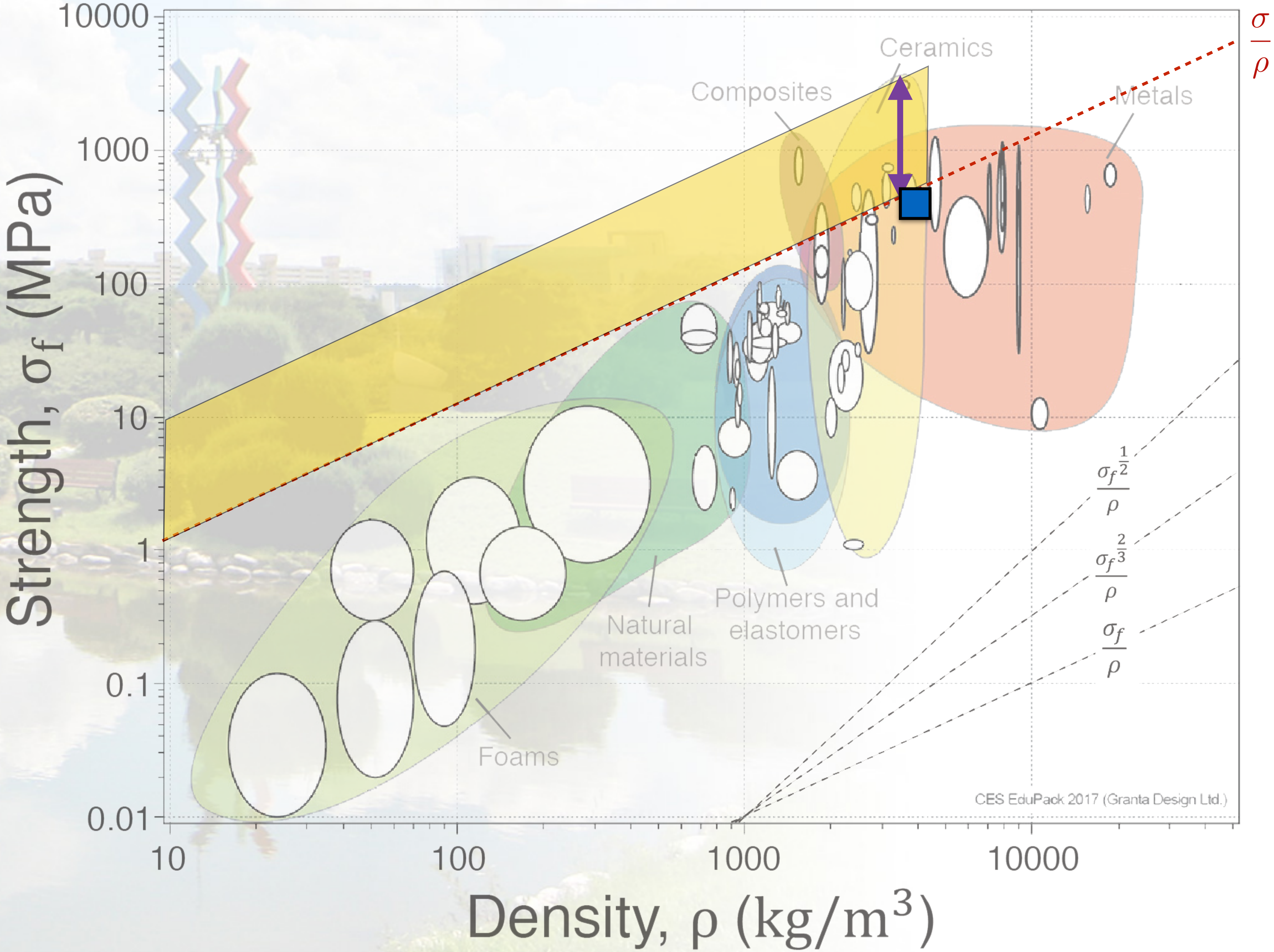
Design Factors

1. Integration of Scaling Laws
2. Selection of Base **Material**
3. Determination of **Architectures** & Dimensions
4. Existence of Scalable Fabrication Technique

Things to Improve:

1.Base Materials

Further Works



Things to Improve:

2. Architectural Scaling Law

Bending- vs. Stretching-dominated Architectures

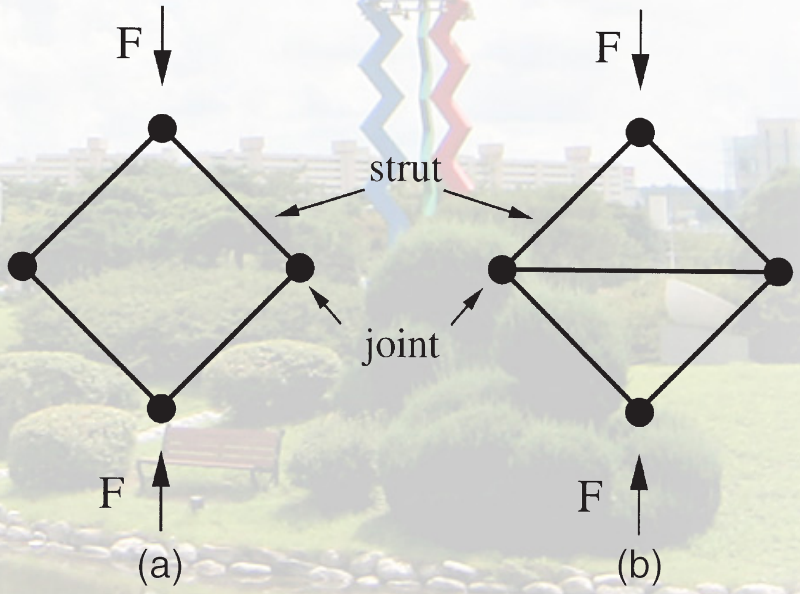


Fig. 1. (a) A mechanism; (b) a structure.

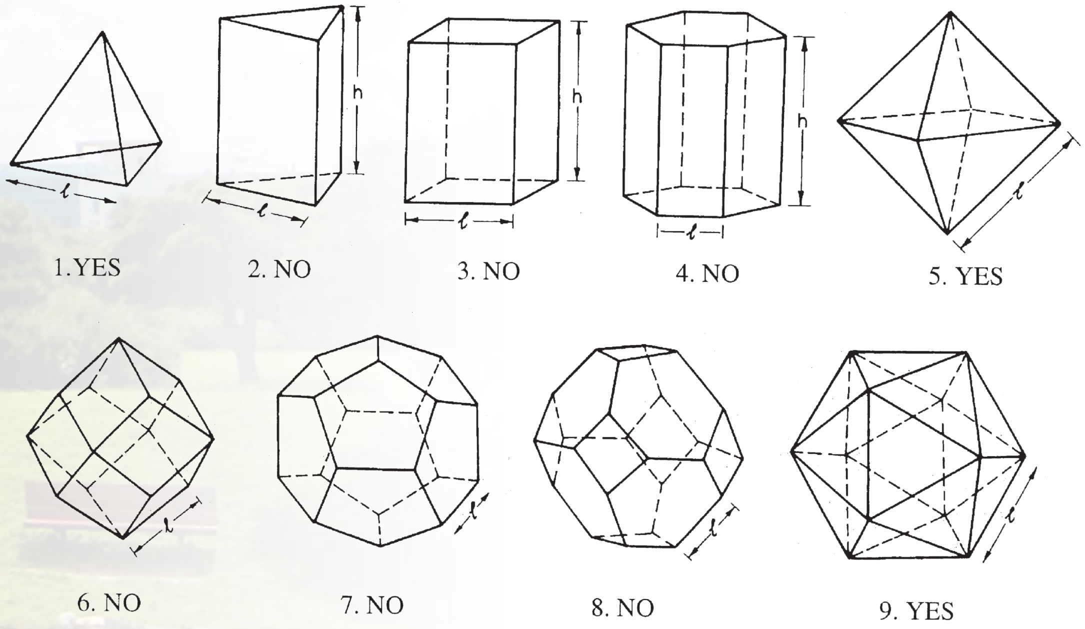


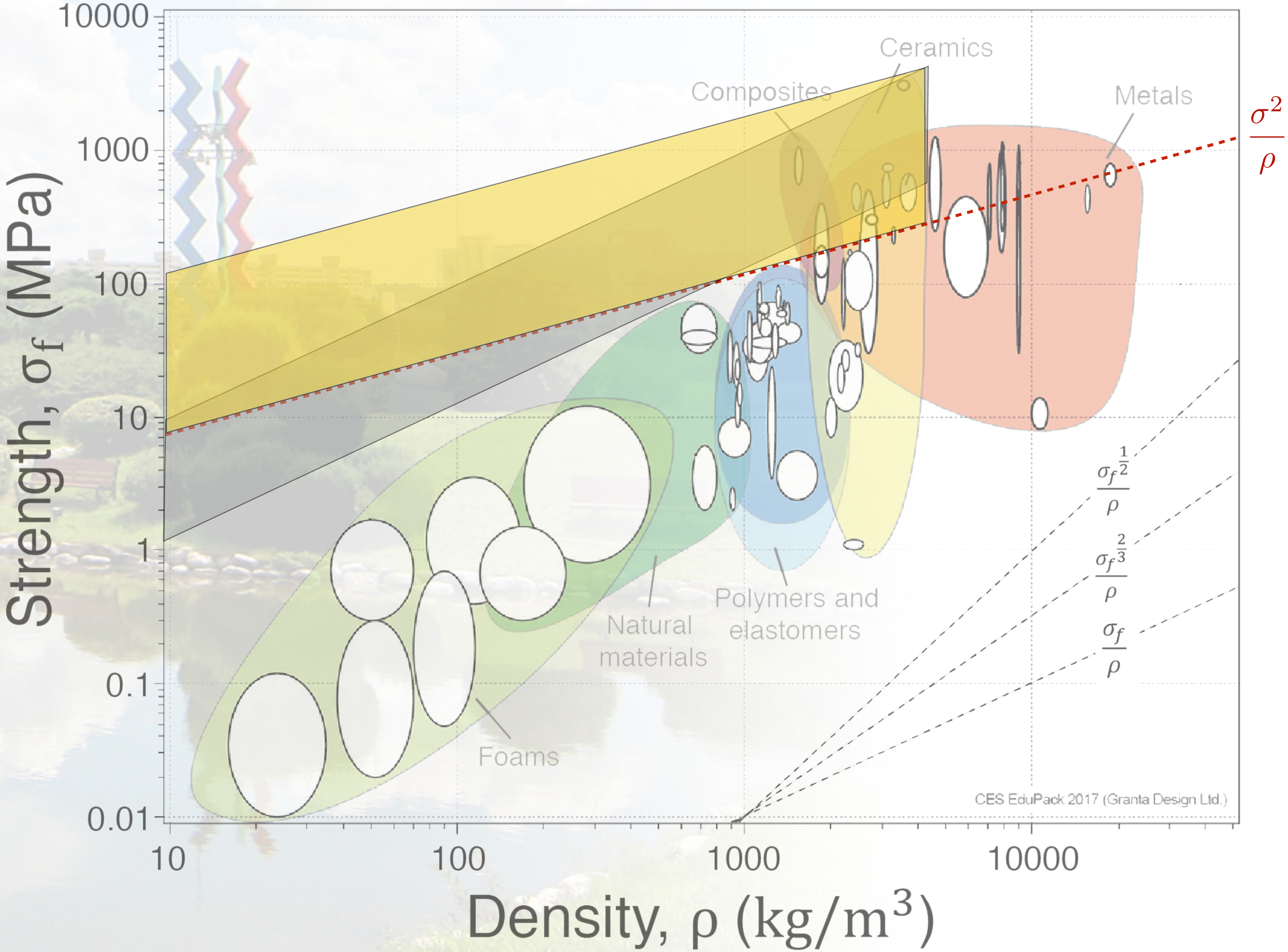
Fig. 4. Three-dimensional polyhedral cells that do, or do not, satisfy the Maxwell criterion.

Deshpande VS, Ashby MF, Fleck NA. Foam topology: bending versus stretching dominated architectures. Acta Mater. 2001;49(6):1035-1040.

$$\sigma_{\mathrm{y}} = \sigma_0 \left(\tilde{\rho}\right)^1$$

$$\sigma_{\mathrm{y,nano}} \propto \left(\tilde{\rho}\right)^{m-\frac{1}{2}} = \left(\tilde{\rho}\right)^{\frac{1}{2}}$$

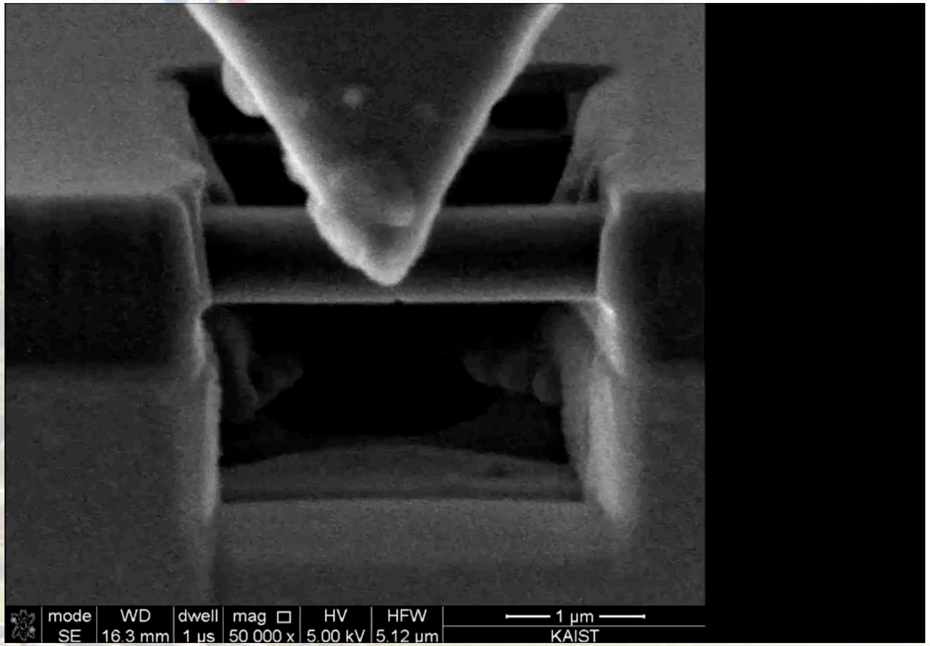
Further Works

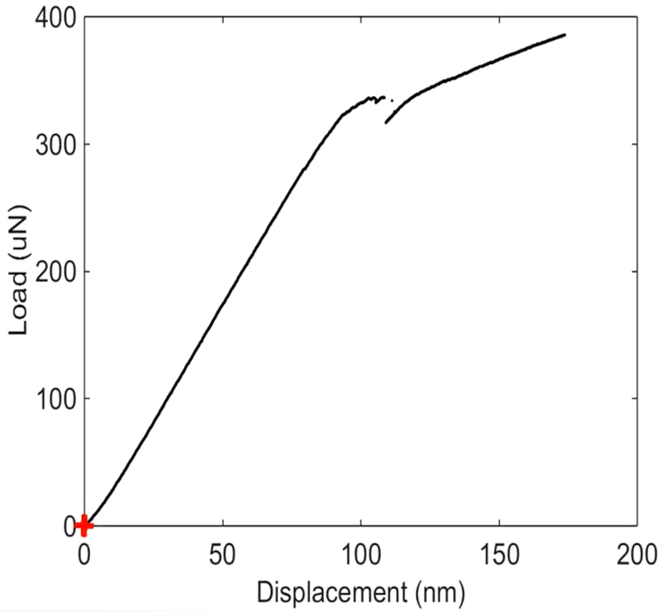


Things to Improve:

3. Materials Scaling Law

Intrinsic Toughening Mechanisms at Nanoscale





- Increased crack stability
- Confinement of stress field

→ Effects of plastic zone increases →

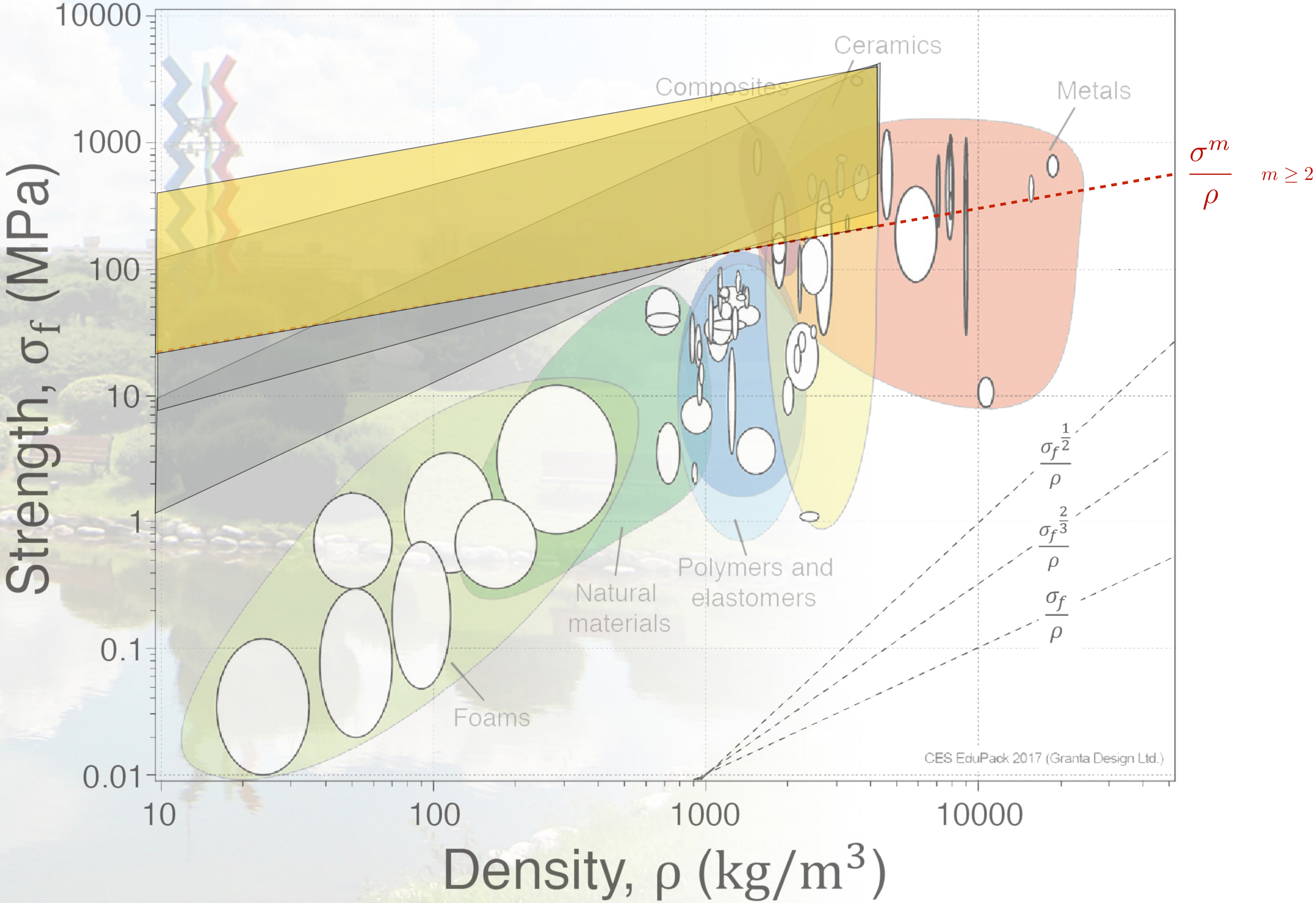
$$\sigma_f = \sqrt{\frac{2E\left(\gamma_s + \gamma_p\left(t\right)\right)}{t}} \frac{1}{\sqrt{\pi\phi}} \frac{1}{F\left(\phi\right)}$$

$$\sigma_f \propto \left(\frac{1}{t}\right)^{\frac{1}{2}+n}$$

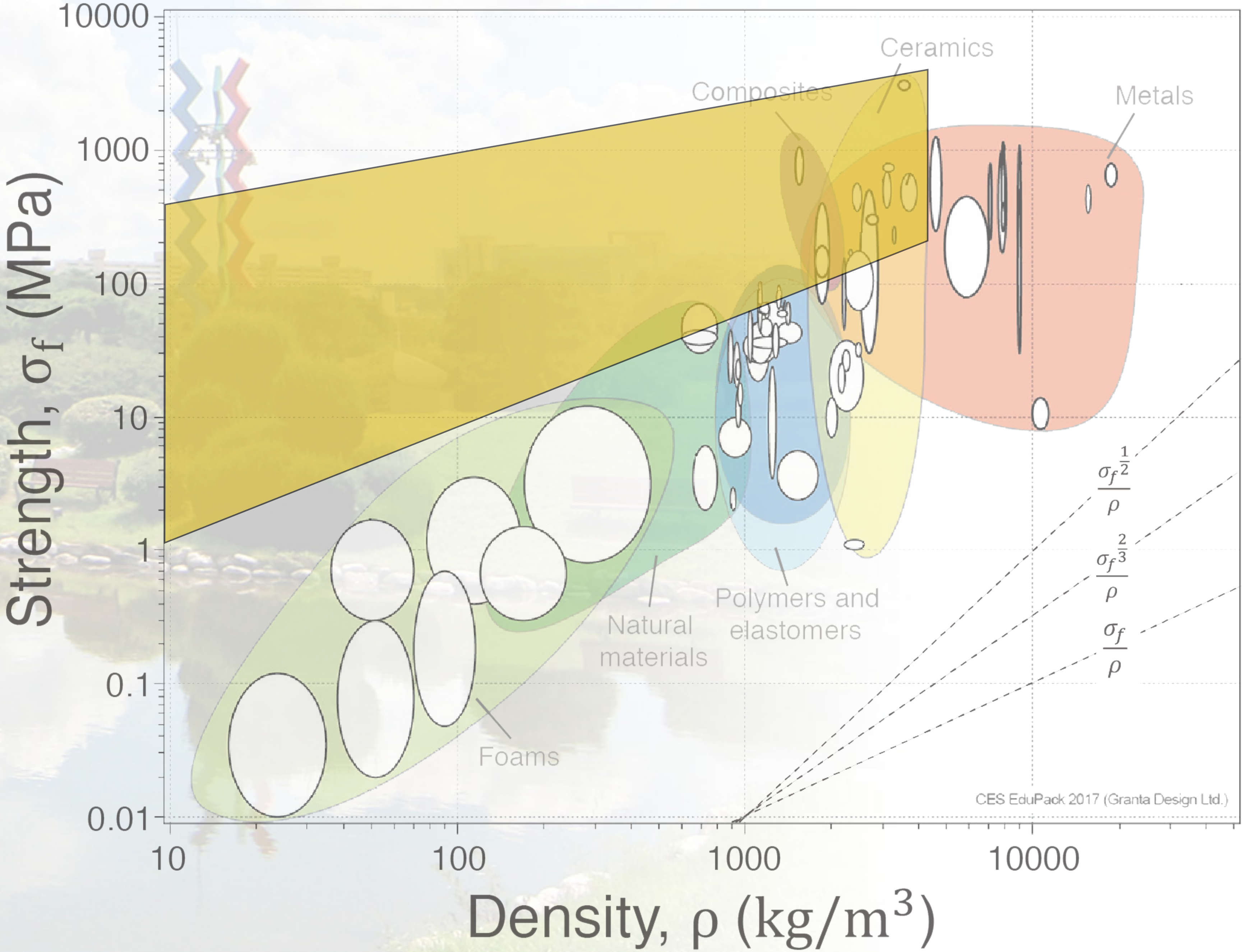
→

$$\sigma_{\mathrm{y,nano}} \propto \left(\tilde{\rho}\right)^{m-\left(\frac{1}{2}+n\right)}$$

Further Works



Further Works



Summary

- Using nanomechanical principles, it is possible
  - to simultaneously impart high strength and flexibility to ceramic materials
  - to overcome the limit of conventional scaling law in porous ceramics.
- This is just a beginning. There are a lot more things to do

Thanks for your attention !