Evolution of the mechanical properties of aging erbium tritide films

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Metal tritide films

- Metal tritide films are essential for applications such as neutron generators, but the property changes as the T decays lead to problems.
- We have been following the mechanical properties of Erbium Tritide films as they age. We found that the films first strengthened, then softened.
- The two regimes can be explained by dispersion strengthening combined with a simple elastic softening due to the bubble growth.

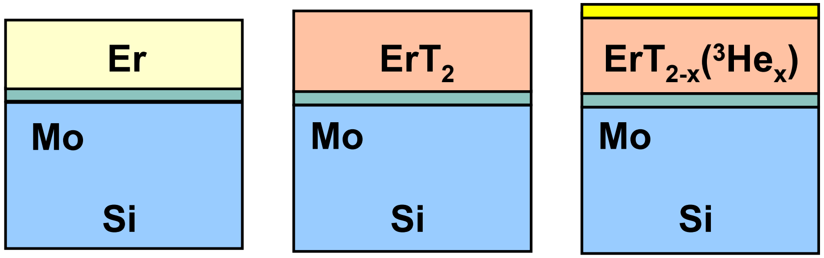
Samples: ErT_2 layers on Mo/Si

Sample preparation

- 500 nm Er
95 nm Mo
- Silicon

- Hydrided with 100% Tritium

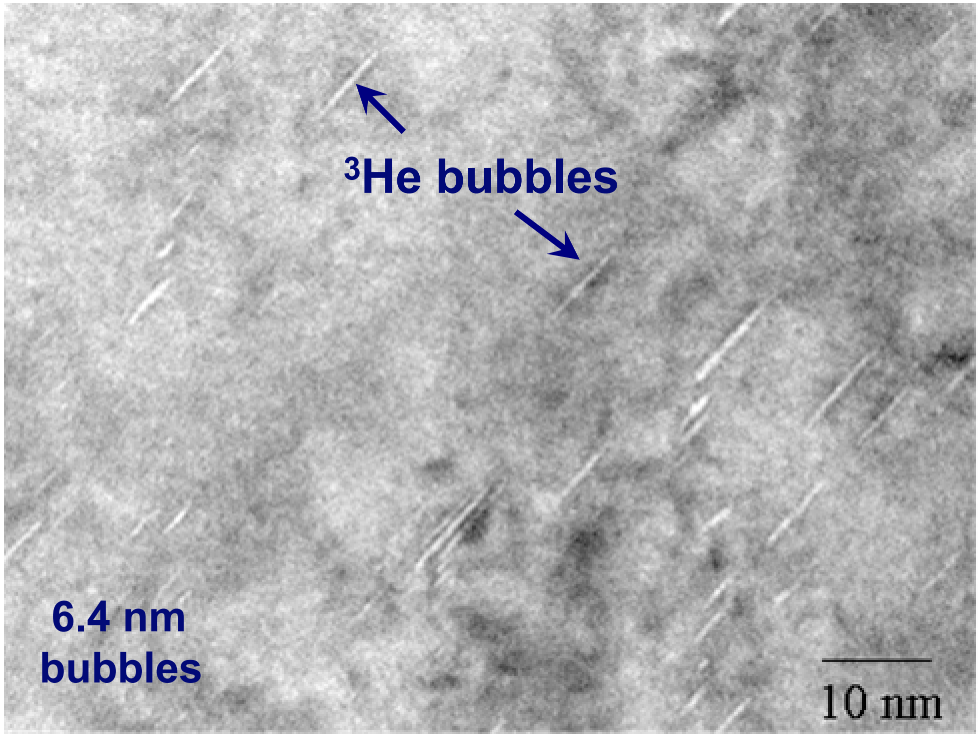
- Aged in vacuum



Oxide forms during hydriding and upon air exposure.

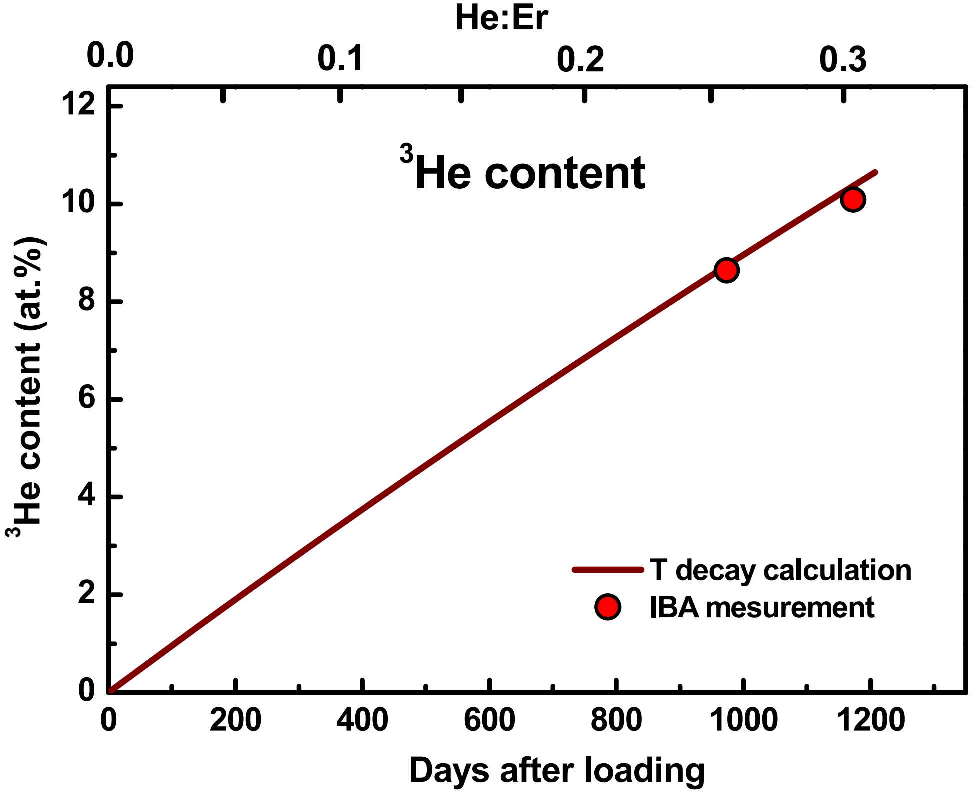
Tritium decays into ^3He, forming platelet-like bubbles on (111) planes.

TEM cross-section bright-field, ~{110} zone 62 days after hydriding.

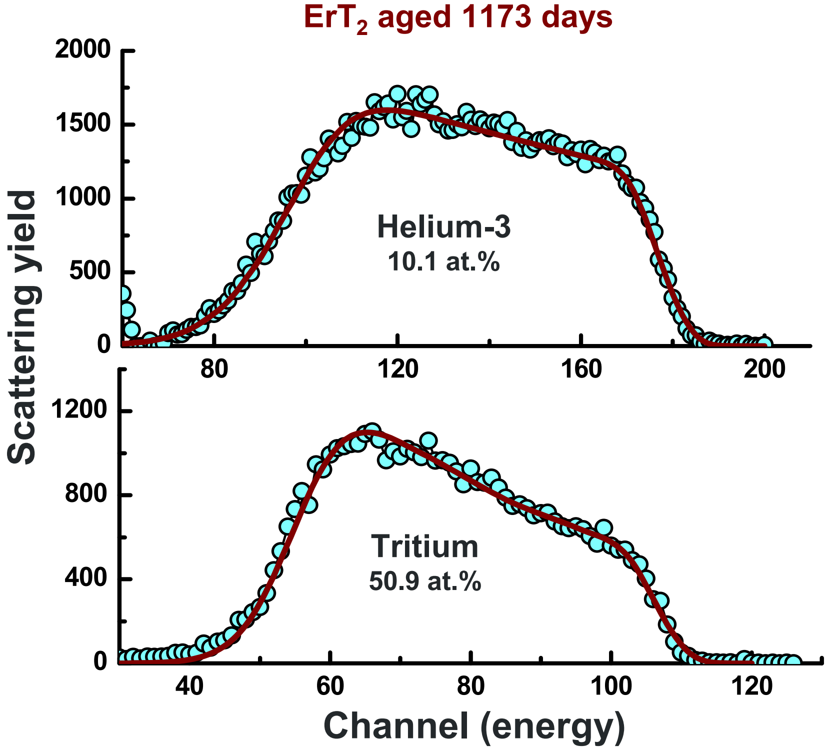


Tritium decay and ^{3}He content

Calculated buildup of ^{3}He for 100% T-loaded ErT_2



IBA of aged ErT_2 shows expected levels of T, ^{3}He

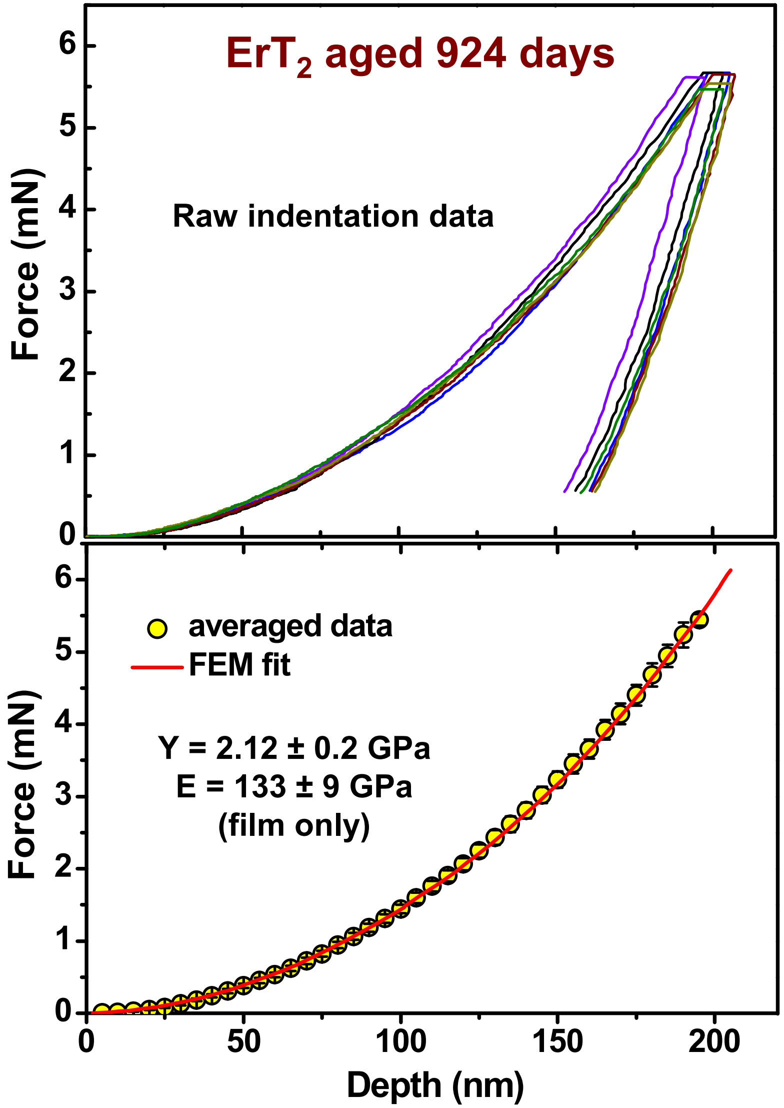


Nanoindentation of tritiated films

- Containment procedures allowed use of indenter outside T envelope.
- Only known nanoindentation of tritiated thin films.
- Finite-element modeling* is used to determine film yield strength and Young's modulus separate from the substrate:

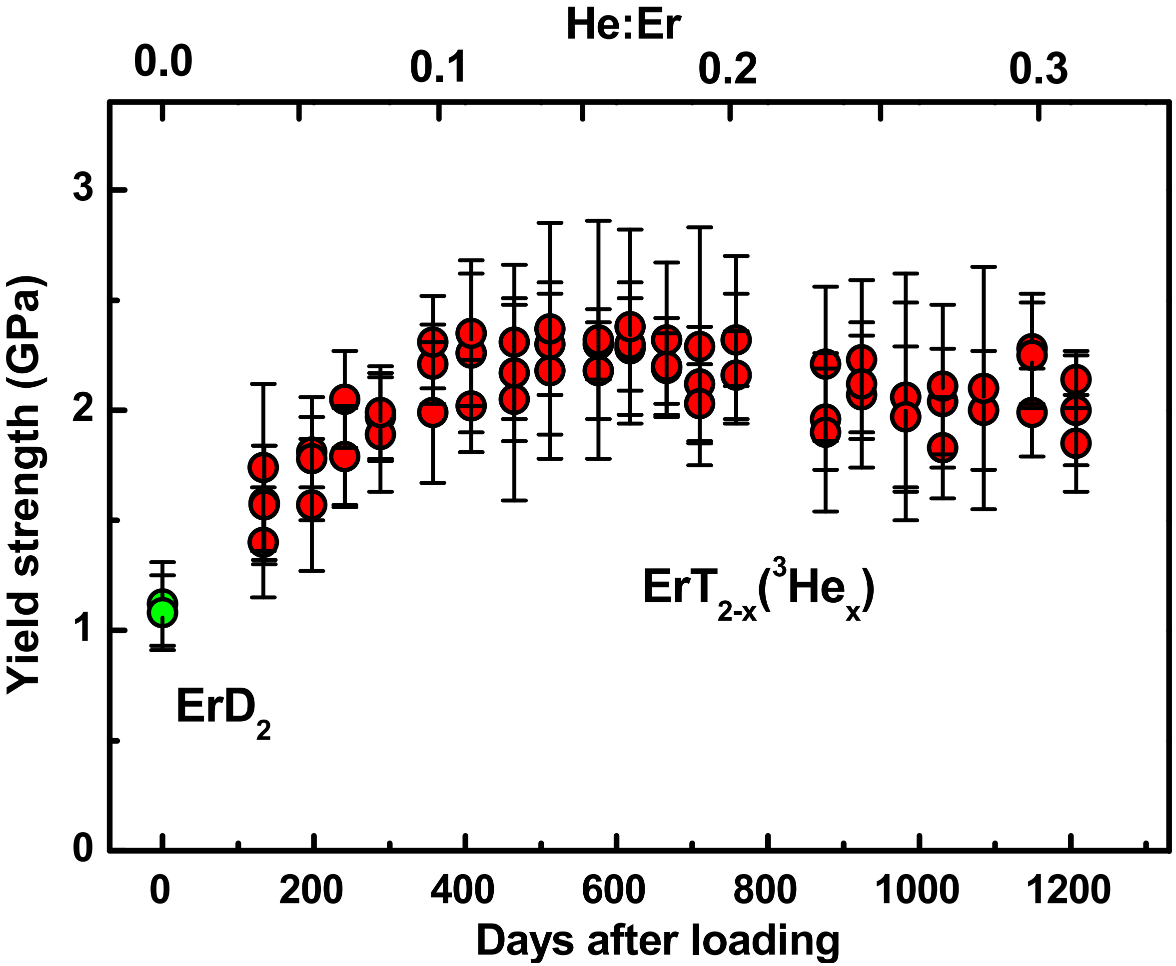
  - Properties of the indenter and underlying layers and substrate are fixed at known values.
  - Properties of the layer are varied until a good fit to experiment is obtained.
  - Tip yielding, stress, friction are all modeled.

*J.A. Knapp, et al., JAP, 85 (1999) p.1460.



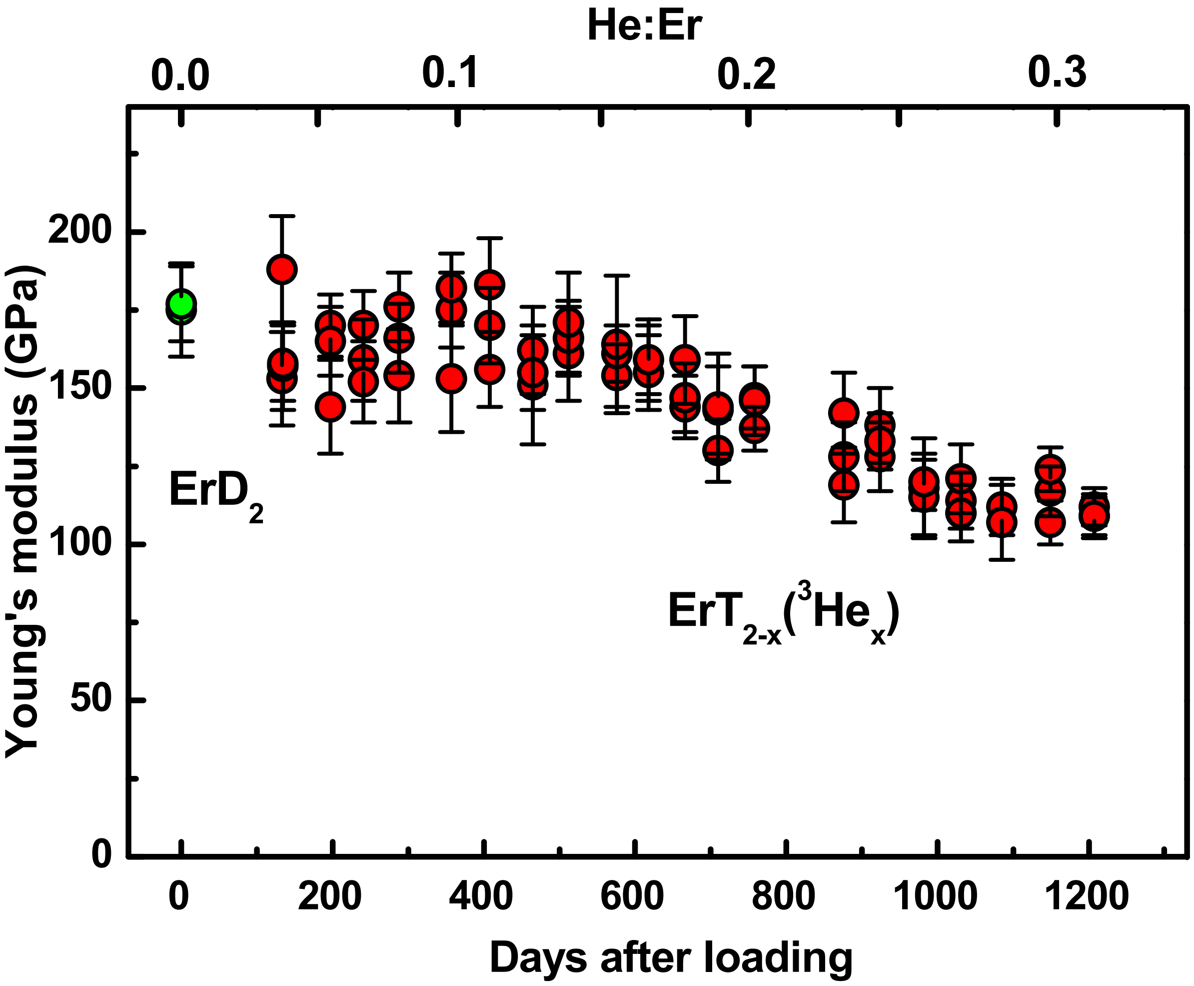
Nanoindentation: yield strength

Yield strength increased as ^{3}He bubbles formed and grew. After an initial increase, the hardness of the films leveled off and then decreased as the films aged.



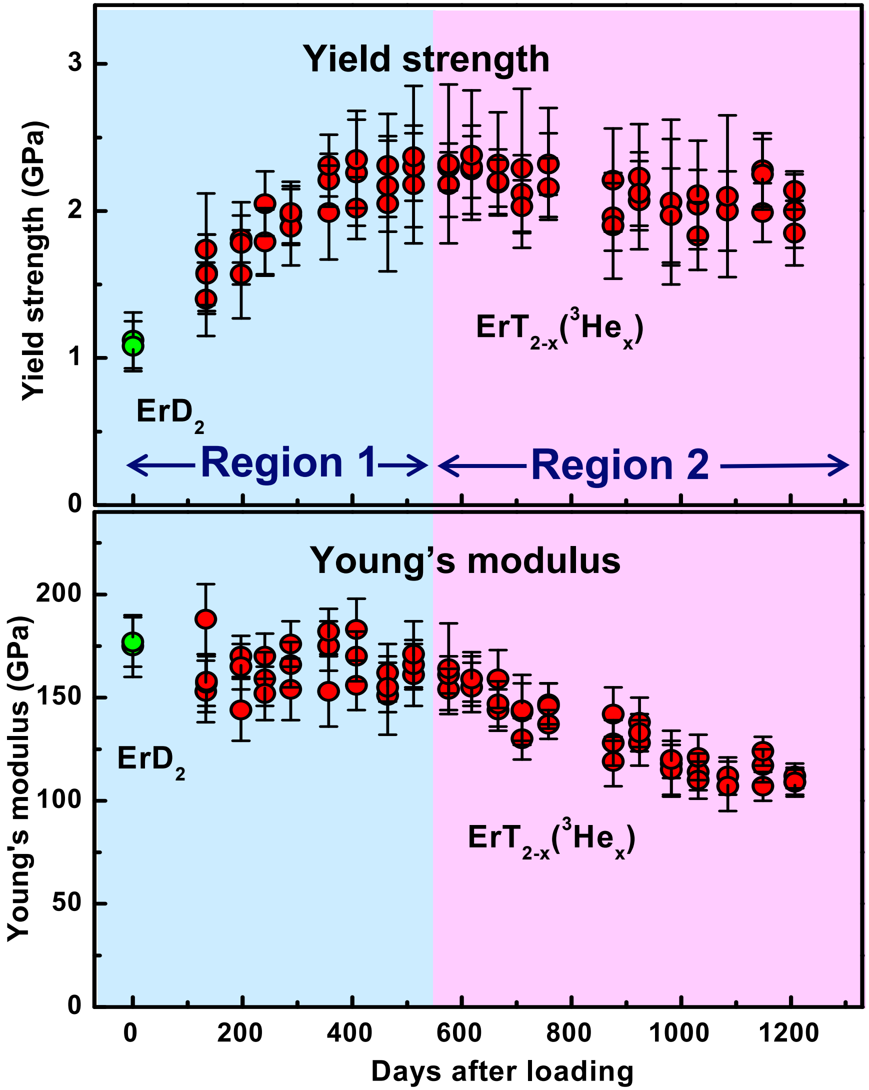
Nanoindentation: Young's modulus

<u>**Young's modulus**</u> **initially stayed constant and then decreased at about the same time as the yield started to decrease.**



Mechanical properties: two regions

- **Region 1**
  - increasing yield strength with bubble growth
  - nearly constant or decreasing elastic properties
- Region 2
  - decreasing yield strength
  - decreasing elastic properties



Region 1 - dispersion hardening

- **Bubbles pin dislocations**
  - **study in Ni(He) shows Orowan-type strengthening <u>even though bubbles are shearable</u>**
  - **implants with 1 to 10 at.%: spherical bubbles from 1 to 6.4nm**

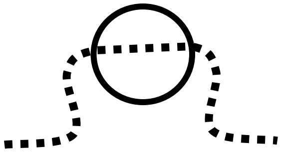
- **Calculation confirms pinning[*]**

Three effects provide binding:

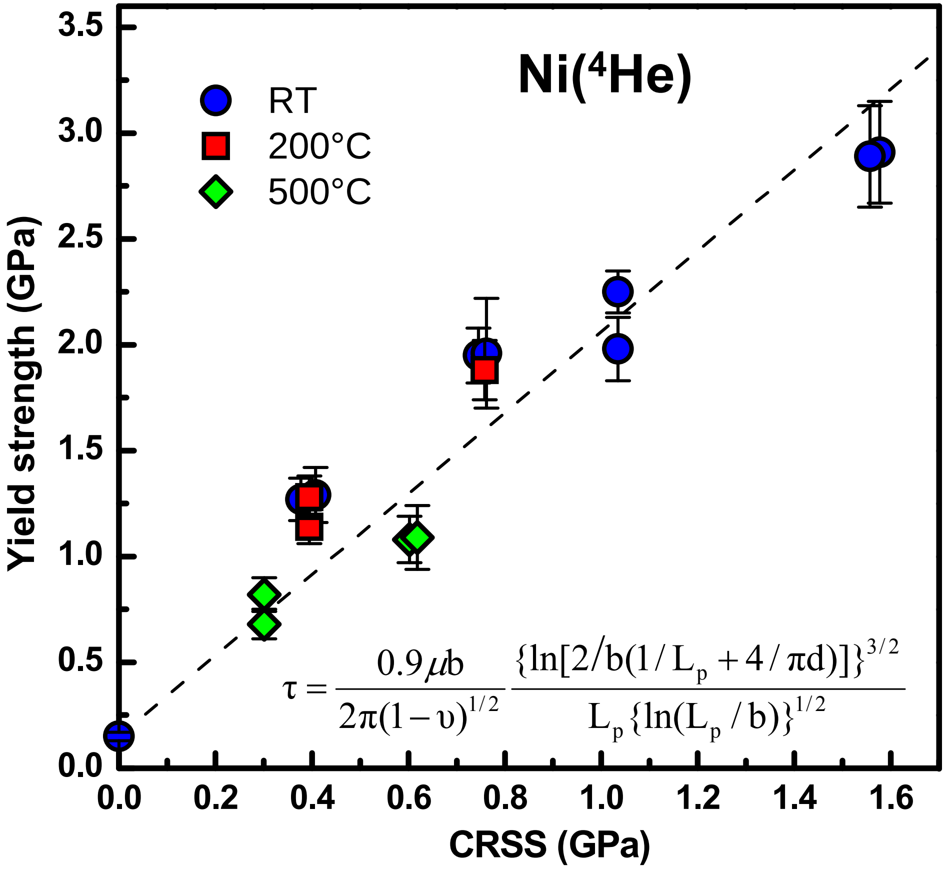
1) reduction in dislocation strain energy

2) absence of core energy

3) barrier to surface step formation in bubble



Ni implanted with 1-10 at% ^{4}He



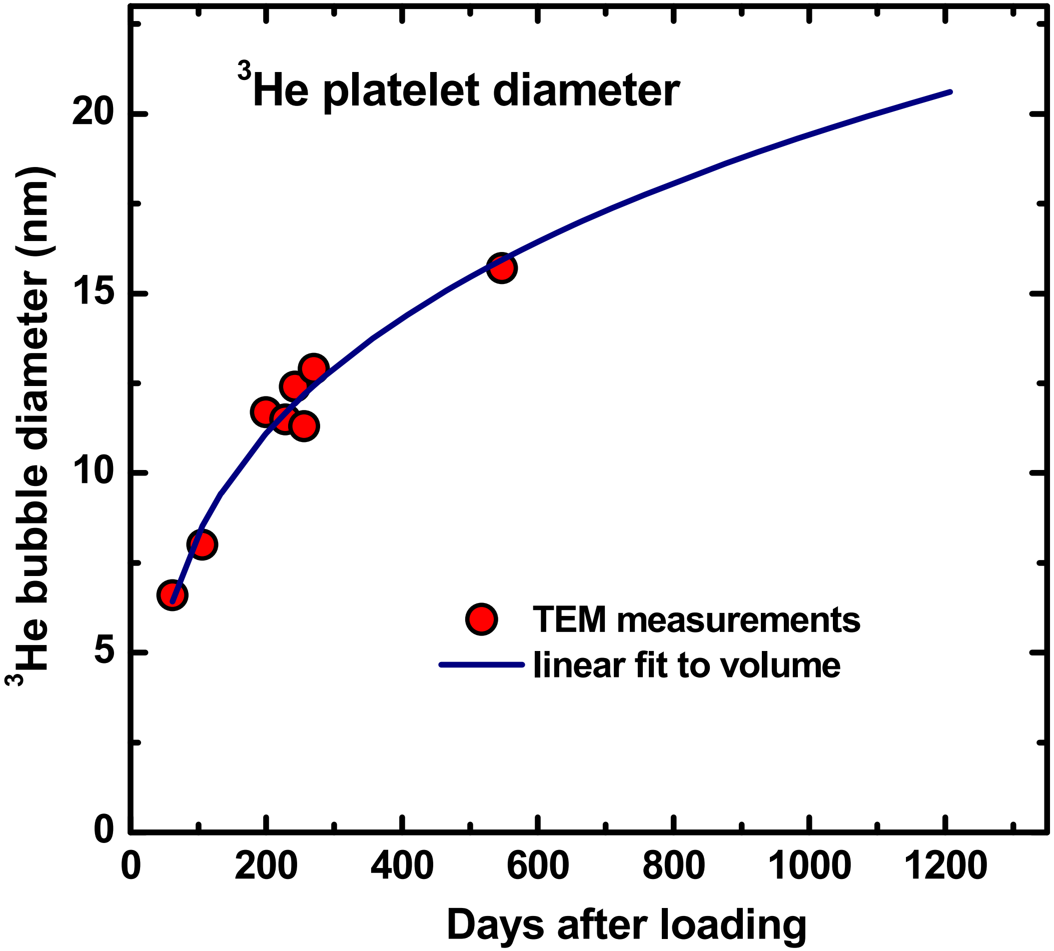
$$\tau = \frac{0.9\mu b}{2\pi(1-\upsilon)^{1/2}} \frac{\{\ln[2/b(1/L_p + 4/\pi d)]\}^{3/2}}{L_p\{\ln(L_p/b)\}^{1/2}}$$

[*] **calculation by S.M. Myers**

J.A. Knapp, et al., JAP, <u>103</u>, 013518 (2008)

Bubble sizes

- **TEM measurements**
  - sizes measured up 547 days: 15.7 nm
  - sizes consistent with no new nucleation after the first few weeks
- **Linear fit to volume**
  - constant thickness
  - assumes no nucleation after the first few weeks



Bubble pressure

- **Bubble pressure is needed to deduce number density**
  - **Since these are platelets we use Cowgill's formula for p_{de}, calculated pressure for dipole expansion.[1]**

$$p_{de} = \left(\frac{2\gamma}{s}\right)\left[\frac{(2r+b+s)}{(2r+b)}\right] + \frac{\mu d}{(2r+b)}$$

d = {111} interplanar spacing
s = platelet thickness ($2d<s<3d$)
r = platelet radius
γ = surface energy ($0.637\ J/m^2$)
b = burgher's vector (0.3623 nm)
μ = shear modulus (70 GPa)

  - **The He equation of state by Kortbeek & Schouten[2] then provides the He density in the bubbles, giving the number density.**

[1] **Don Cowgill, HHIMC presentation, 2005.**

[2] **P.J. Kortbeek and J.A. Shouten, J. Chem. Phys. 95 (1991) p.4519.**

Orowan-type strengthening for platelets

- **For platelets the calculation of Critical Resolved Shear Stress τ is more complicated than for spheres or rods.**
  - **We use a formula computationally derived by Zhu and Starke[1]:**

$$\tau = 0.12\mu \frac{b}{(D_p t_p)^{1/2}} \left[f_v^{1/2} + 0.70 f_v \left(D_p t_p\right)^{1/2} + 0.12 f_v^{3/2} \frac{D_p}{t_p} \right] \ln\left(\frac{0.079 D_p}{r_0} \right)$$

t_p = *platelet thickness*
D_p = *platelet diameter*
f_v = *volume fraction*
b = *burgher's vector (0.3623 nm)*
μ = *shear modulus (70 GPa)*
r_0 ~ *burgher's vector (used ½b)*

[1] **A.W. Zhu and E.A. Starke, Jr., Acta. Mater. 47 (1999) p.3263.**

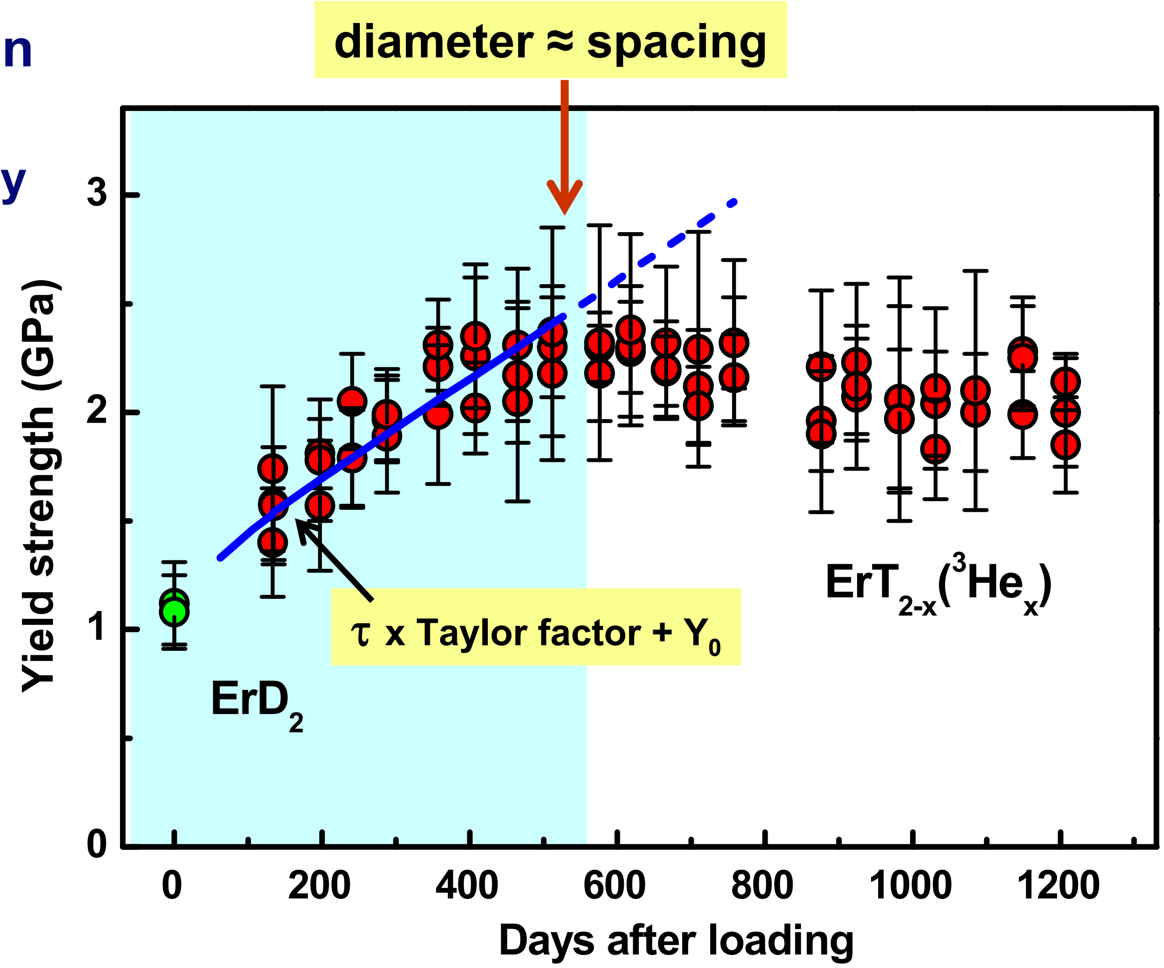
Increase in yield strength

Yield strength increases in proportion to τ (CRSS)

- constant of proportionality is the Taylor Factor (typically 2-3)
- here we use 2

Calculation of τ diverges when platelet diameters are equal to the average center-to-center spacing

- estimate of CRSS becomes invalid when bubbles nearly overlap.



Mechanical properties: two regions

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- **Region 2**
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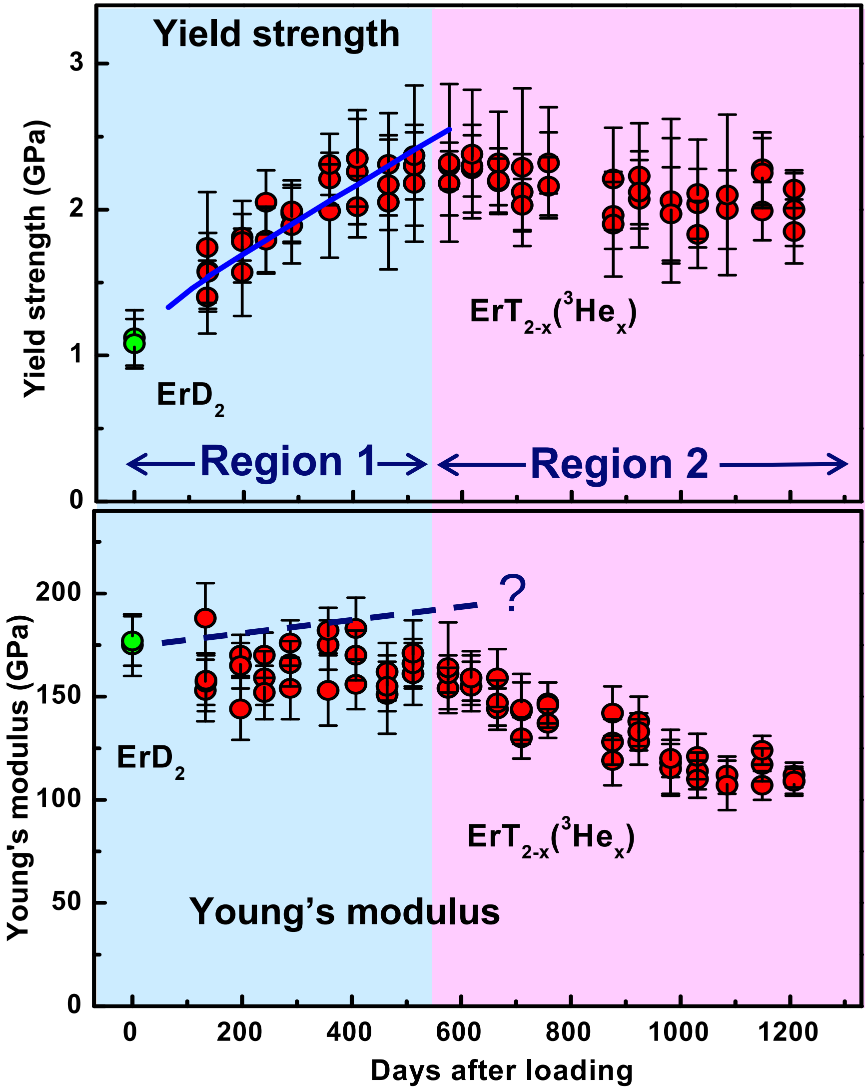
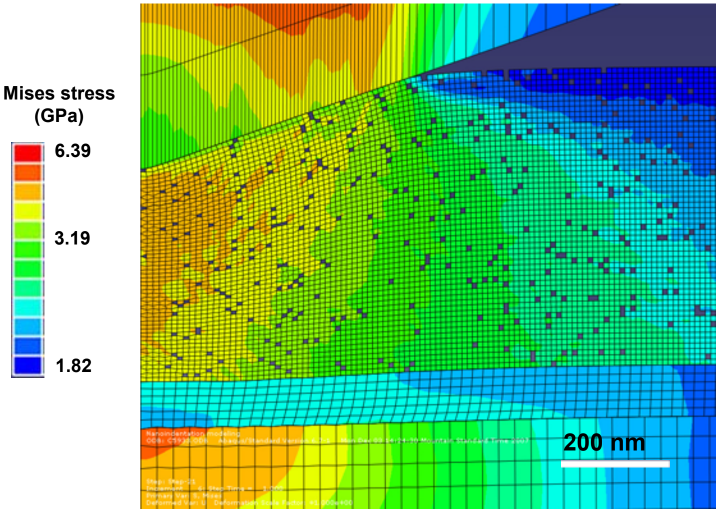
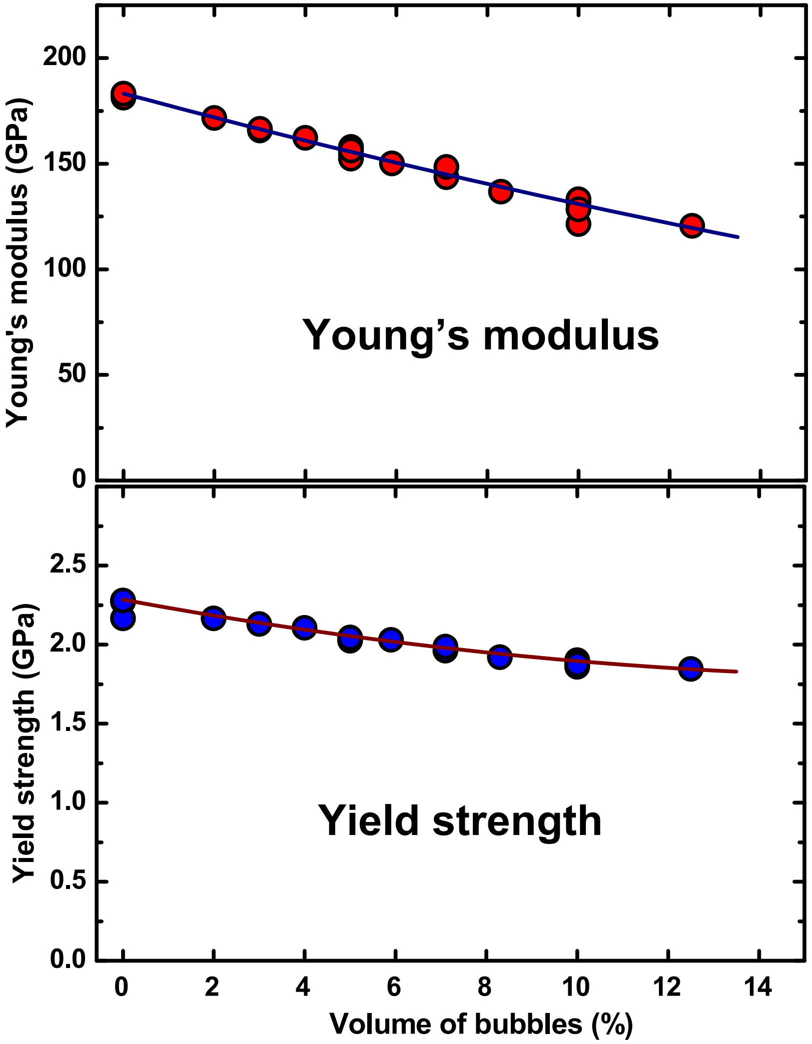


Region 2 - elastic softening

- **Bubbles lower the average elasticity and strength**
  - as the bubble volume fraction increases, elasticity and yield strength decrease.
  - FEM simulations using 2-12 vol.% bubbles, with varying sizes and shapes, quantify the effect.



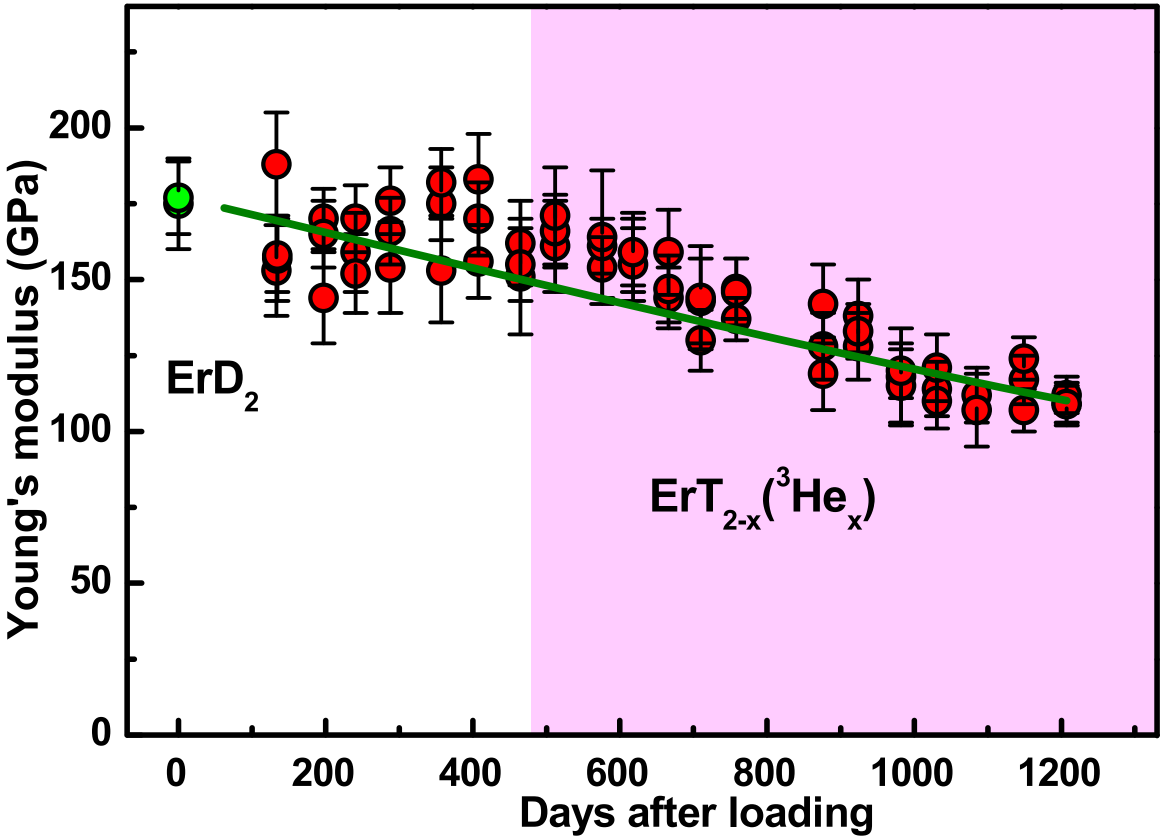
Mises stress for nanoindentation of ErT_x with 5 vol.% He



Decrease in elasticity

Elastic modulus decreases as bubble volume density increases.

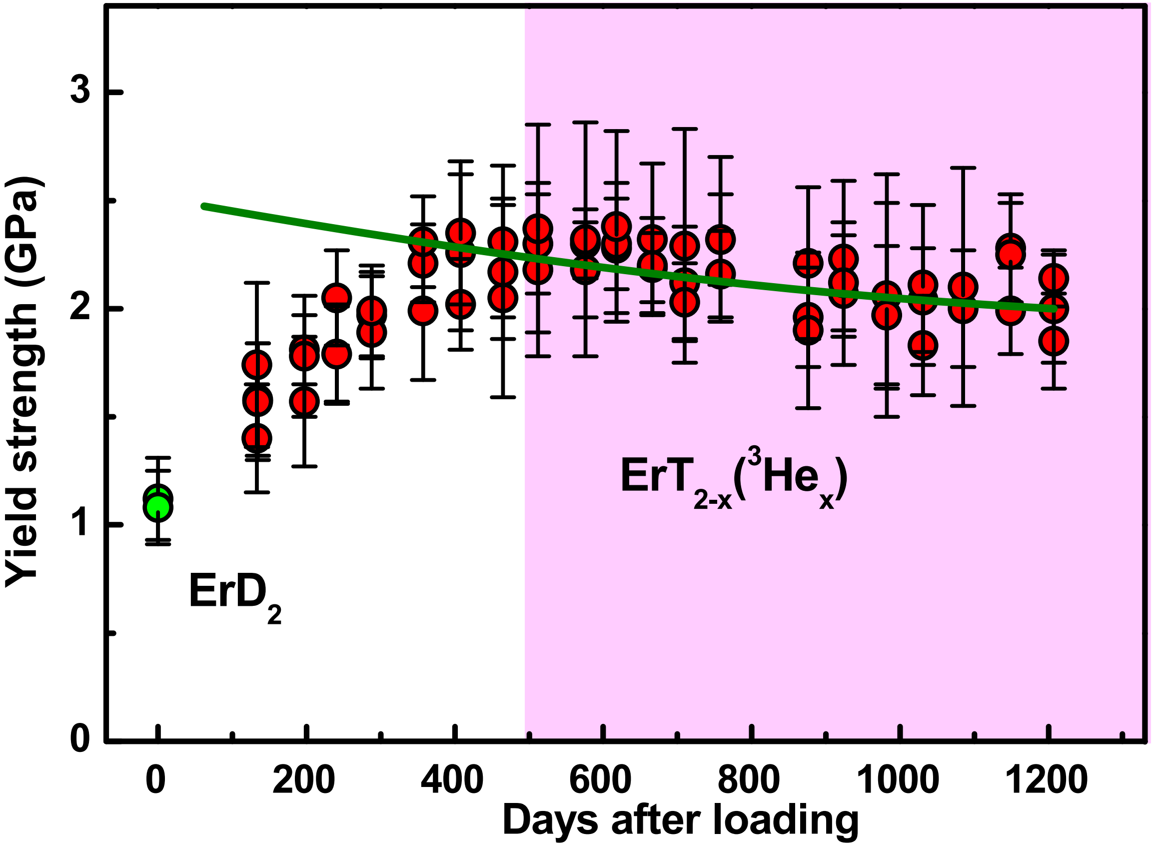
- might be expected from "rule-of-mixtures"
- FEM calculation is not scaled



Decrease in yield strength

Yield strength also decreases as bubble volume density increases.

- Not expected from simple "rule-of-mixtures" - FEM simulations required.
- effect is clear in region 2, where dispersion hardening is no longer effective.



Summary

- **Region 1**
  - **increasing yield strength with bubble growth**
  - **nearly constant or decreasing elastic properties**
  - **dispersion hardening**

- **Region 2**
  - **decreasing yield strength**
  - **decreasing elastic properties**
  - **elastic softening**