SAND2012-4039 C

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2012 IMOG Gas Technology Meeting

"Unit Cell Expansion in ErT_2 Films"

Mark A. Rodriguez, Jim F. Browning, Gillian Bond, Colleen S. Frazer,

Clark S. Snow, Ralph G. Tissot, and Elaine P. Boespflug

Sandia National Laboratories
Albuquerque, New Mexico 87185-1411

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Overview

- Introduction
- Experimental
- Results
  - Expansion data with time
  - Texture analysis and microstructure
  - Macro-strain
  - Skewed peaks
- Emerging model
- Summary

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Research objectives

- Research goal: to obtain a scientific and technical basis for understanding tritium decay in Er-tritide.
  - ^{3}He Bubble formation and growth
  - Influence of Microstructure on ^{3}He bubble retention
    - Texture
    - Macrostrain
  - Structural analysis of ErT_2 phase
    - Lattice parameter values

Focus of this presentation

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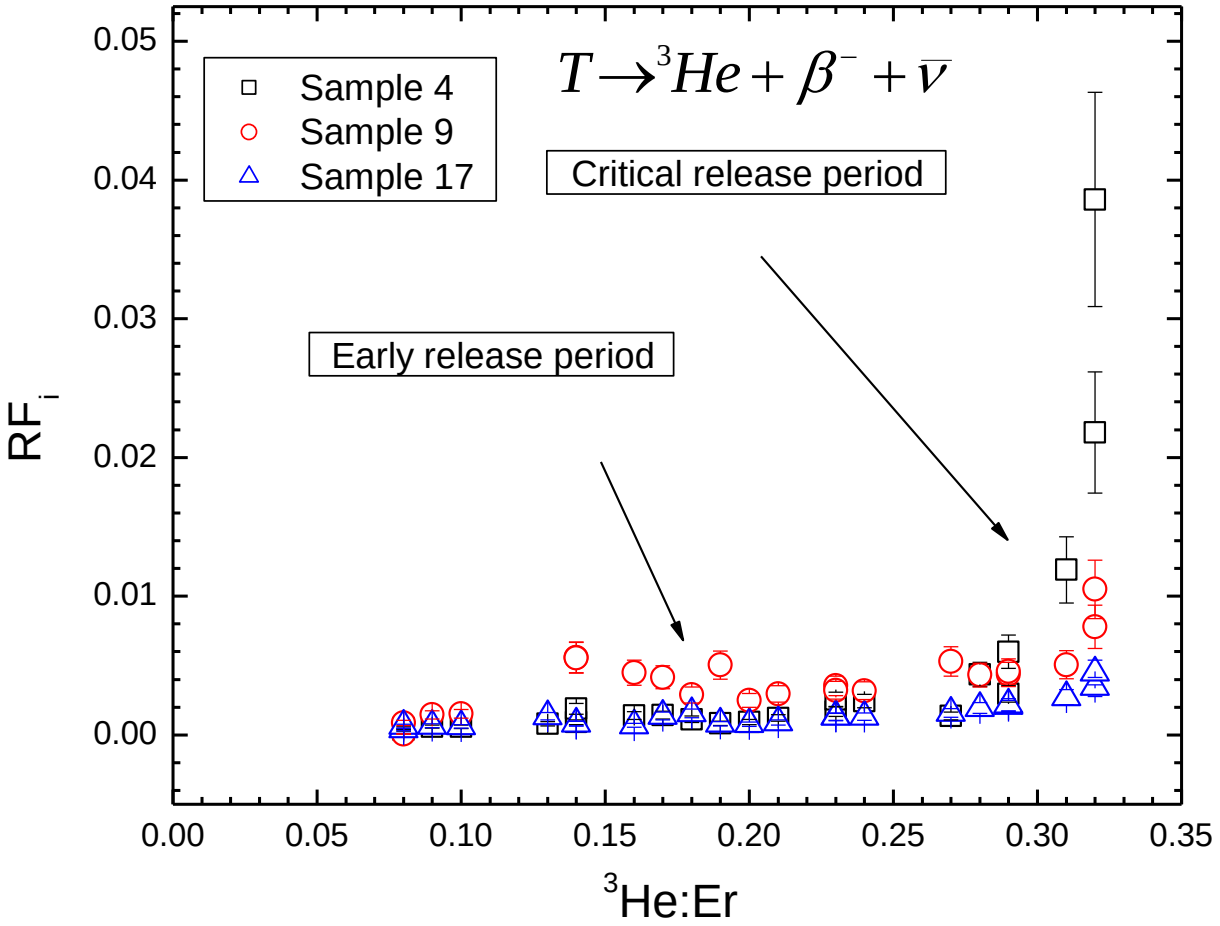
Monitoring of ^{3}He release fraction shows an Early Release and Critical Release period



Typical ^{3}He release characteristics indicate most ^{3}He generated remains in lattice.

Question:

How will the presence of ^{3}He alter the ErT_2 structure?

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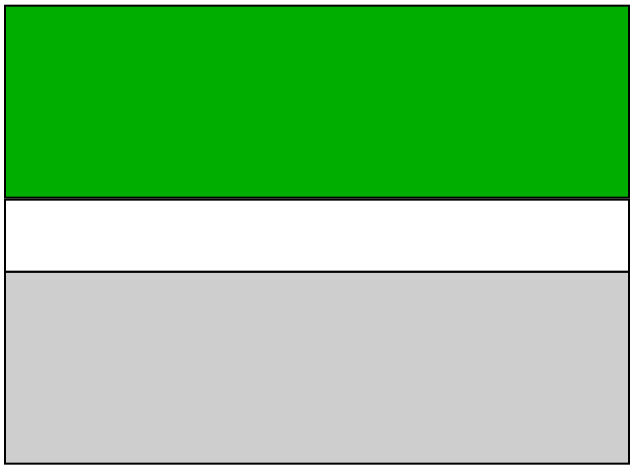
Experimental

- E-beam PVD 5000Å Er/1000Å Mo/Si
- Tritide layer formed by exposure T_2 gas.
- Characterized via IBA, TEM and XRD.



ErT_2 thin film

Molybdenum buffer layer

Si (111) substrate

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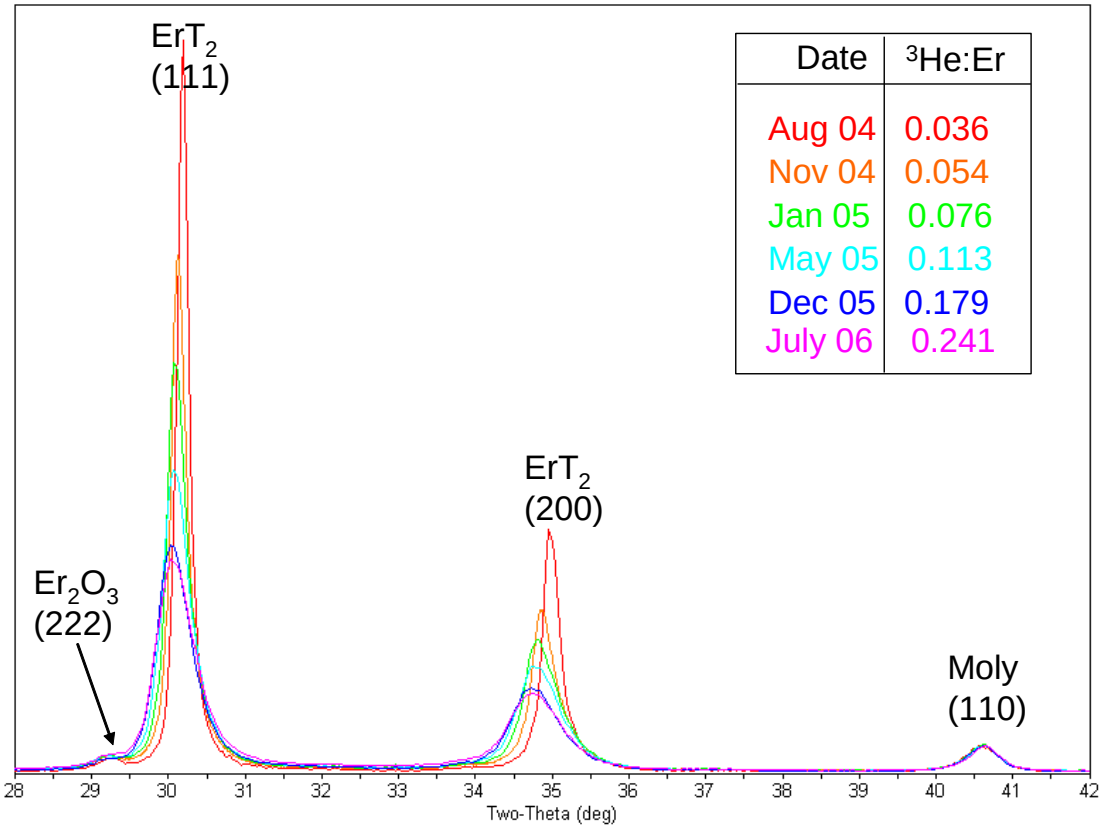
Monitoring ErT_2 films with time shows unit cell expansion for standard (θ–2θ) XRD scans



- **ErT_2 peaks**
  - broaden
  - shift to larger d-spacing
  - show skew
- Moly peaks
  - don't change

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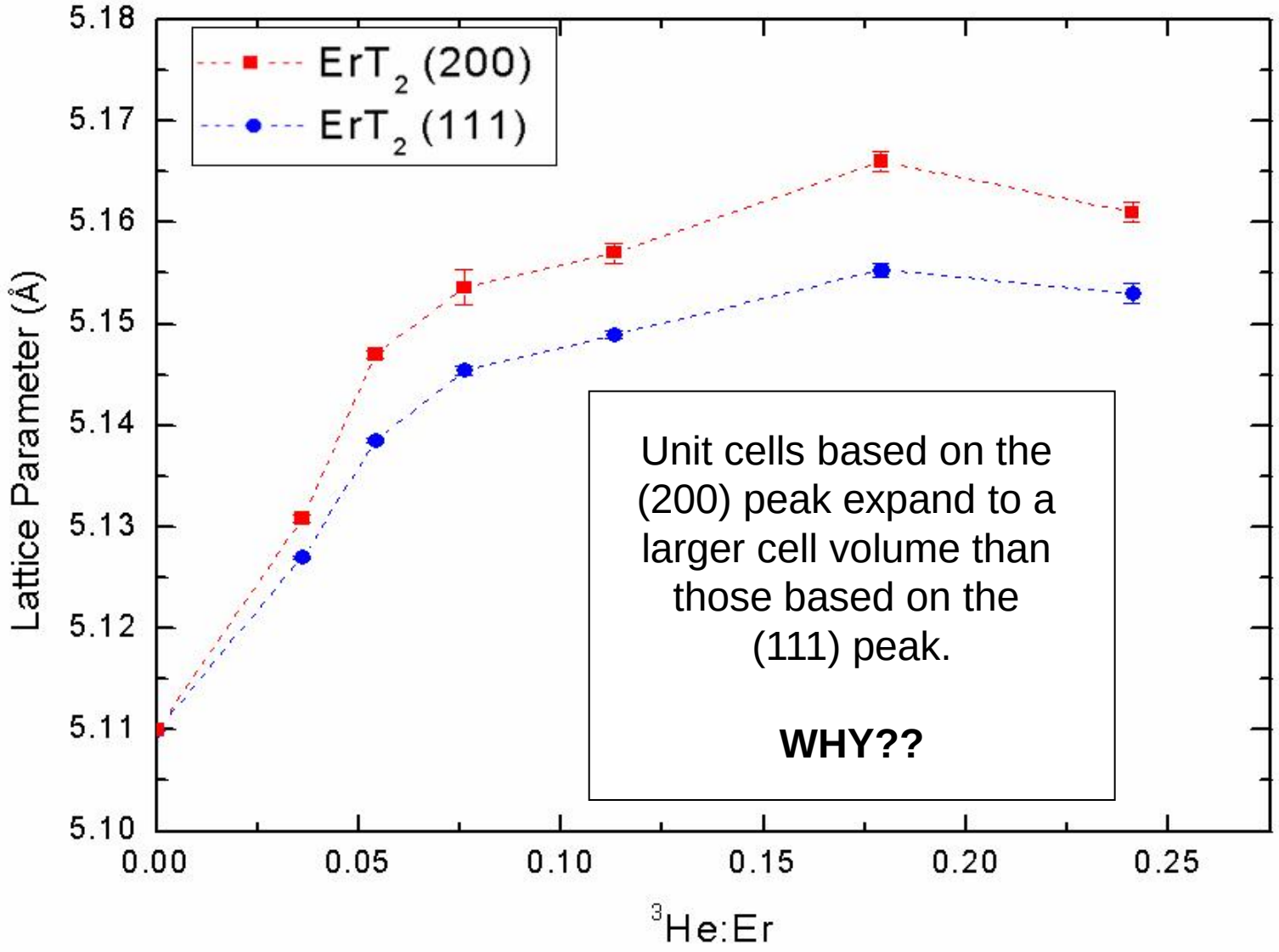
The unit cell lattice parameter for ErT_2 displays an ***hkl dependence*** of expansion with time



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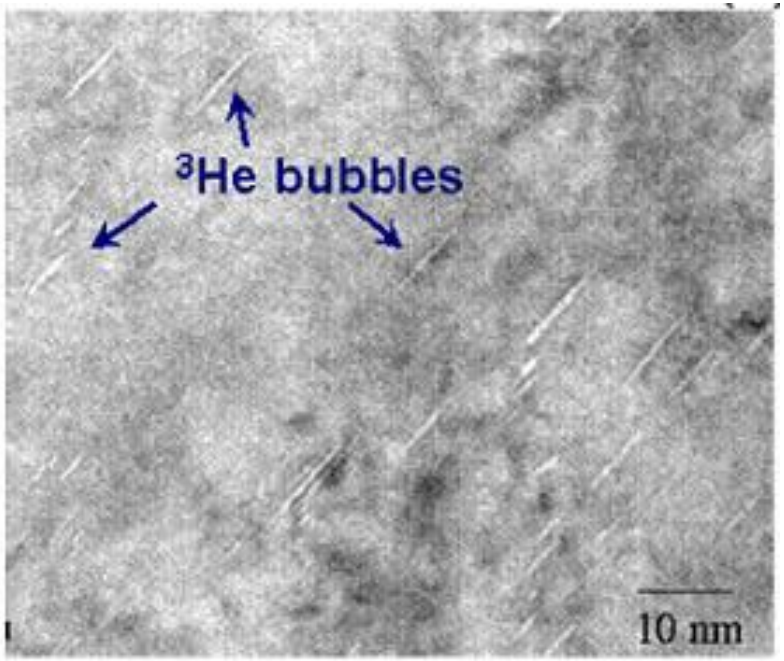
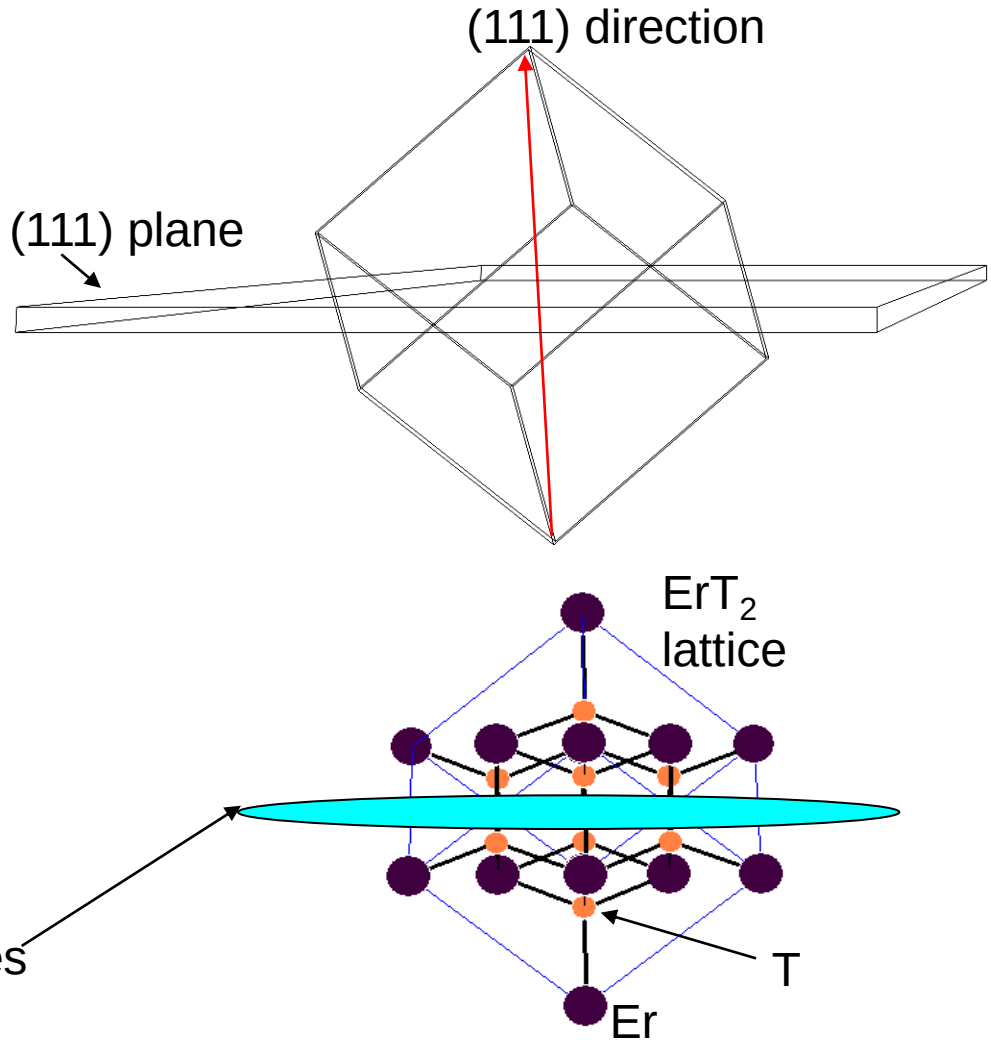
TEM analysis has demonstrated that ^{3}He bubbles form along the (111) planes in the fluorite structure





^{3}He bubbles are long thin plates that propagate along the (111) planes in the Fluorite-type ErT_2 lattice

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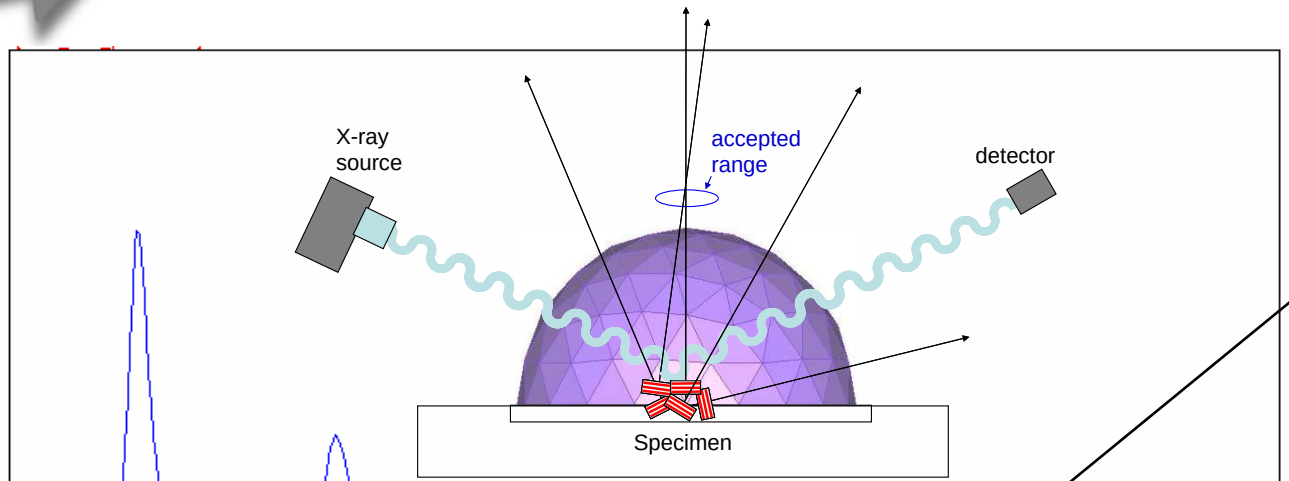
Standard θ–2θ XRD analysis only measures diffraction planes that are in the plane of the film



Peak intensity of ErT_2 (220) in observed pattern is suppressed: **Texture effect**

Observed ErT_2 XRD (Dec '05) $^3He:Er = 0.179$

Simulated ErT_2 XRD pattern

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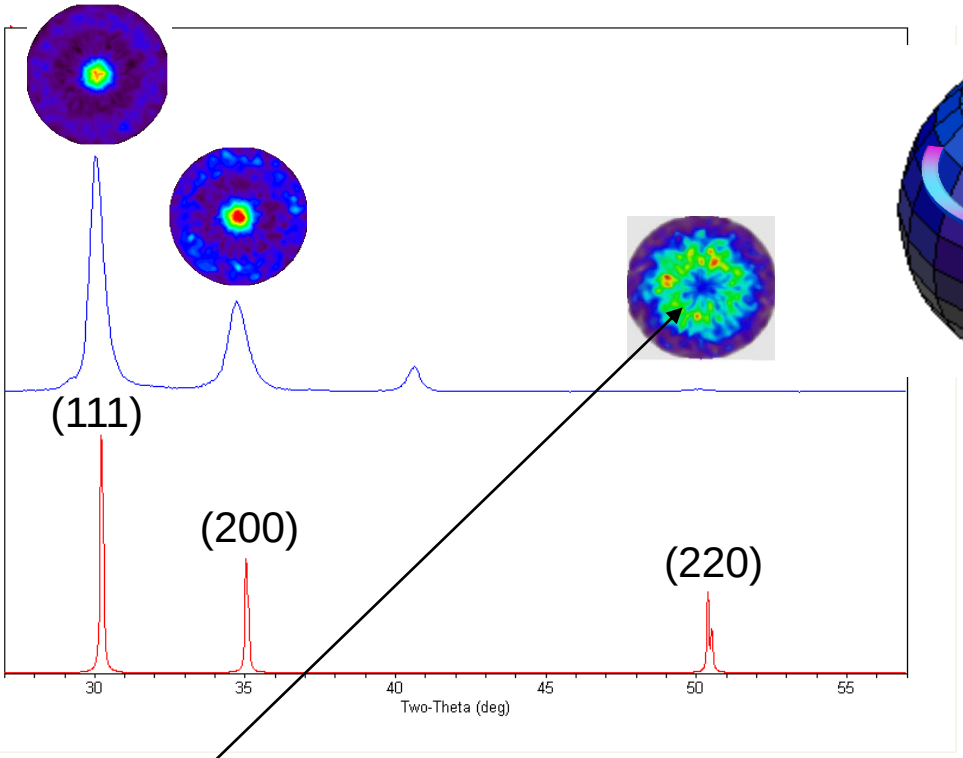
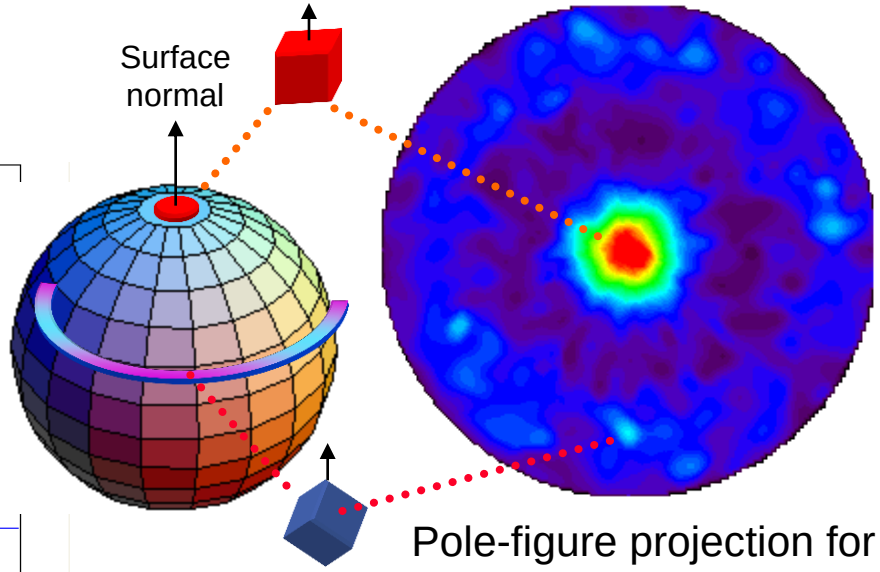
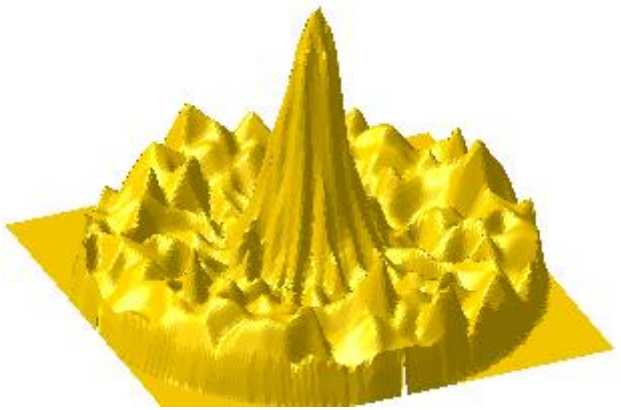
Pole-figure analysis confirms bi-modal (111)/(200) out-of-plane texture, in-plane fiber texture





Pole-figure projection for
ErT_2 (200) hkl



Ring of intensity in the ErT_2 (220) Pole-figure is critical for accurate diagnosis of texture

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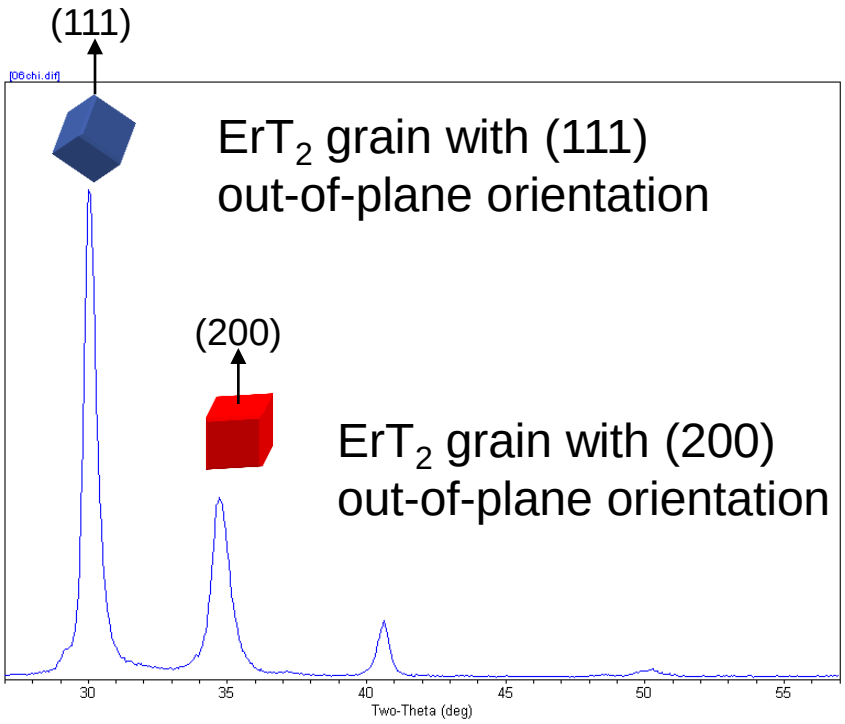
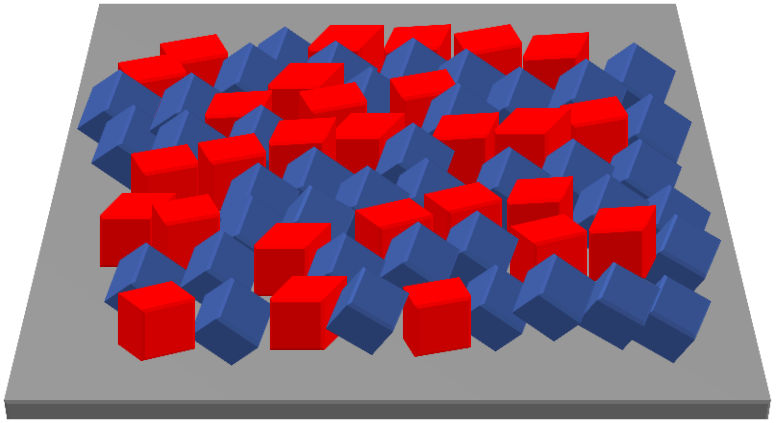
Texture analysis yields an important picture about the microstructure of the ErT_2 films

For simplicity, let us say that the grain morphology is the same shape as the unit cell symmetry (i.e. cubic).





Schematic showing a *bi-modal* (111) and (200) out-of-plane preferred orientation for ErT_2 grains.

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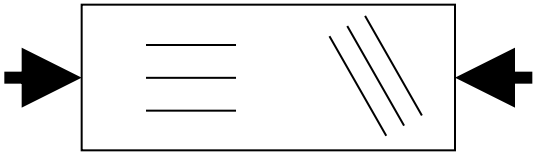
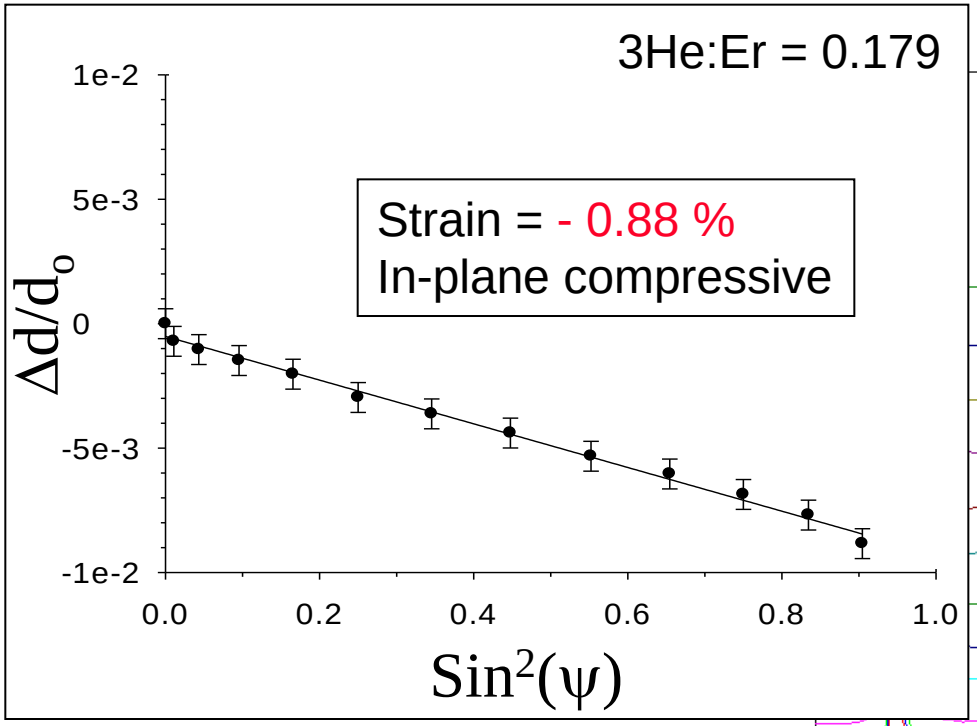
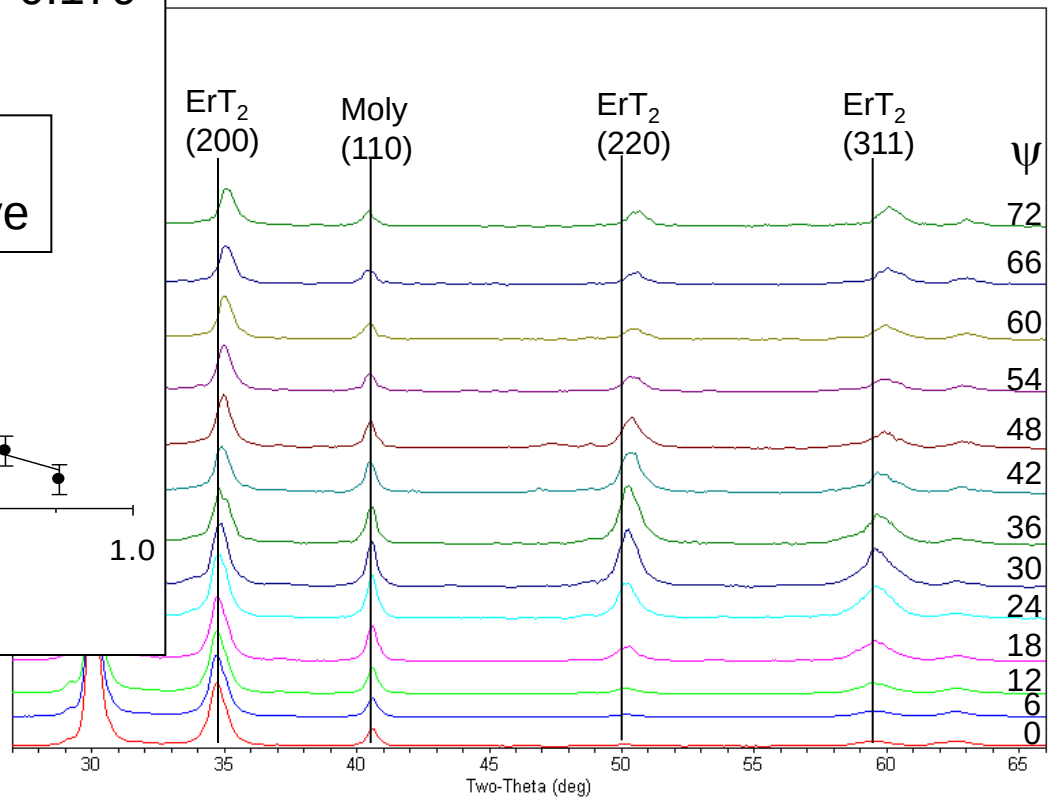
$Sin^2\psi$ analysis revealed significant in-plane strain in the Dec '05 film



An in-plane compressive force develops because ErT_2 grains wish to expand but the film geometry limits this. Out-of-plane expansion is not likewise hindered.





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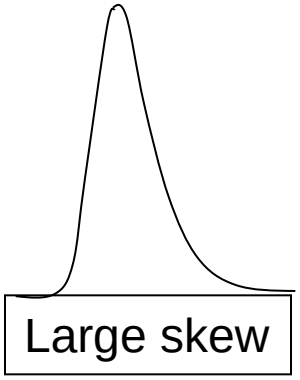
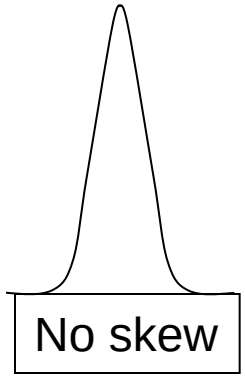
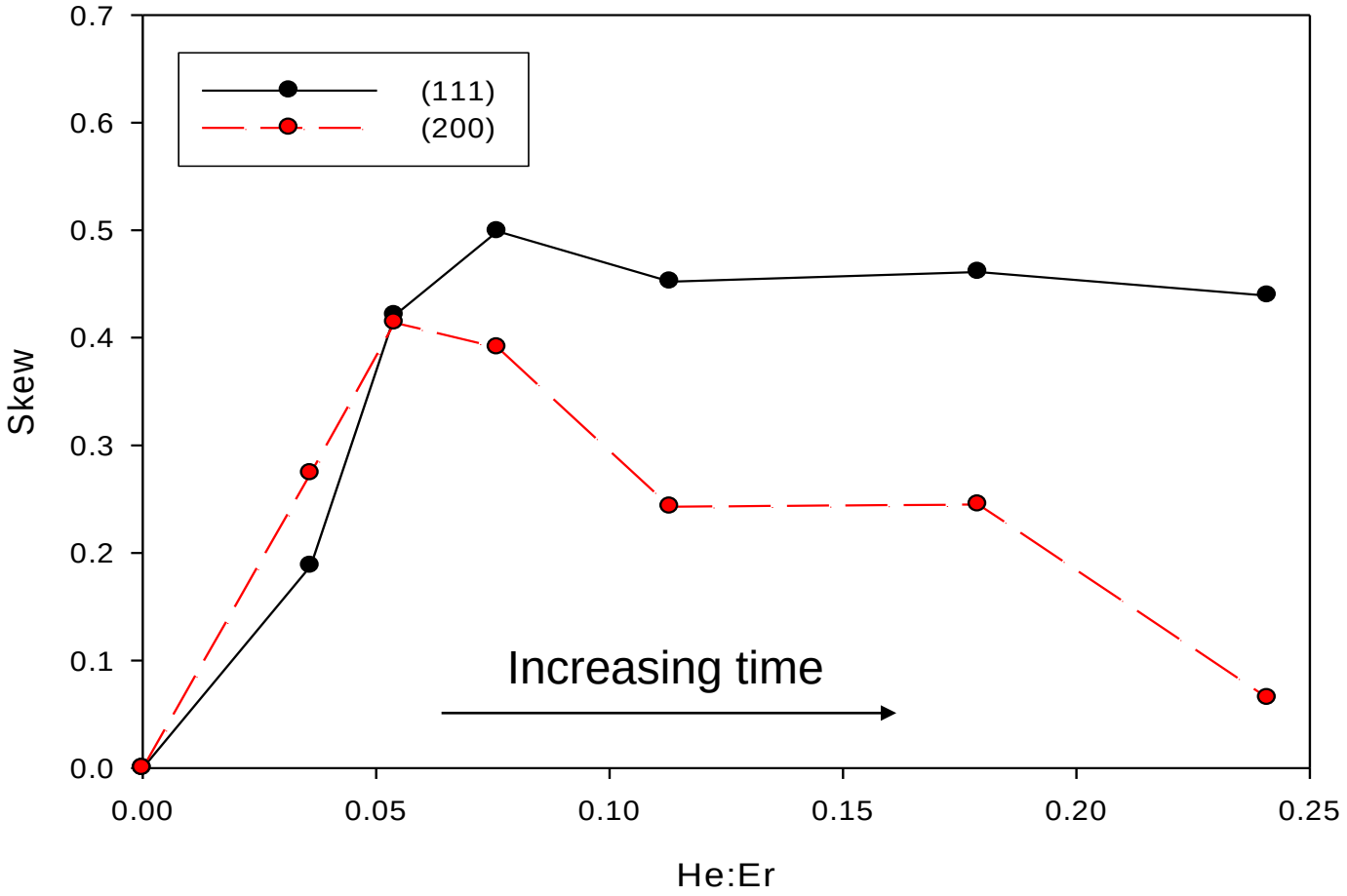
(111) oriented out-of-plane grains saturate at a high skew value, while the (200) out-of-plane grains re-normalize to a more symmetric peak profile.



Large skew



No skew



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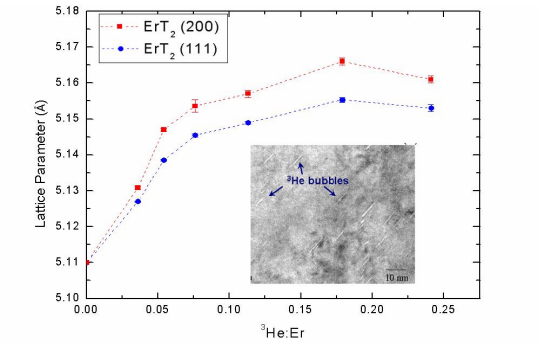
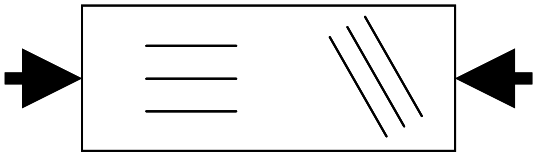
Summary of observations for our analysis: what does it tell us?



hkl-dependent
out-of-plane expansion



In-plane compression

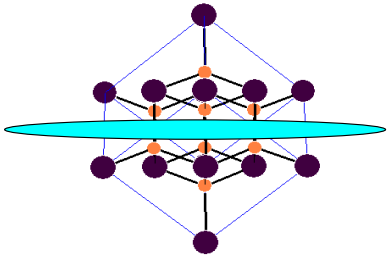
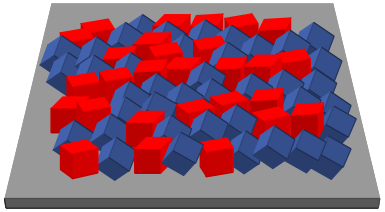
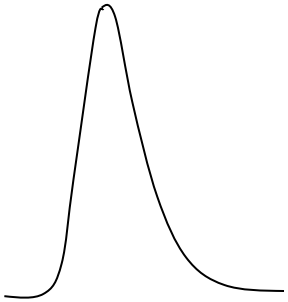


Plate-like ^{3}He bubbles
along ErT_2 (111) plane



Bi-modal texture



hkl-dependent
skew behavior

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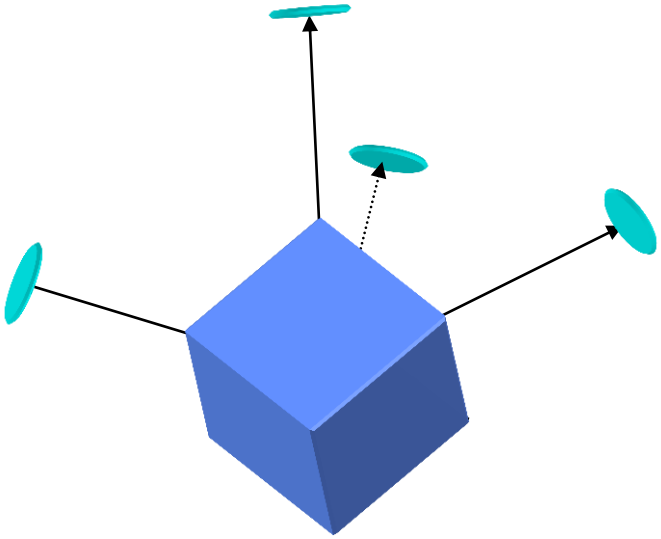
Our developing theory

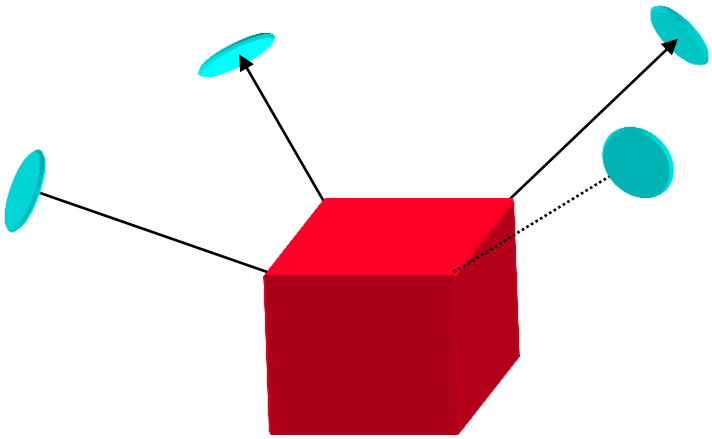
(111) out-of-plane grains

One (111) direction out-of-plane
-expands easily
Three additional (111) directions just 20^{o} from in-plane
-hindered expansion



(200) out-of-plane grains

All four (111) directions have ***equivalent components*** both in and out-of-plane
-more uniform expansion
- less internal opposition from other (111) directions



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Summary

- Different expansion rates and magnitudes are observed in the (200) out-of-plane grains as compared to (111).
- Our ErT_2 films showed a bimodal (111)/(200) out-of-plane texture.
- In-plane compressive strain exists in ErT_2 films with significant Tritium decay.
- Peak skew with T decay is markedly different for (111) and (200) out-of-plane grains.
- Observations can be explained in terms of ^3He bubble expansion along (111) planes in the ErT_2 lattice.
- This effect might result in different ^3He release characteristics for the differing grain orientations.

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