

A Neutron Reflectivity Study of ErT₂ Films

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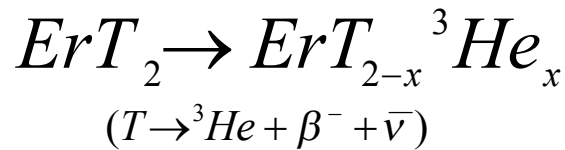
*now at UC Davis

Acknowledgements

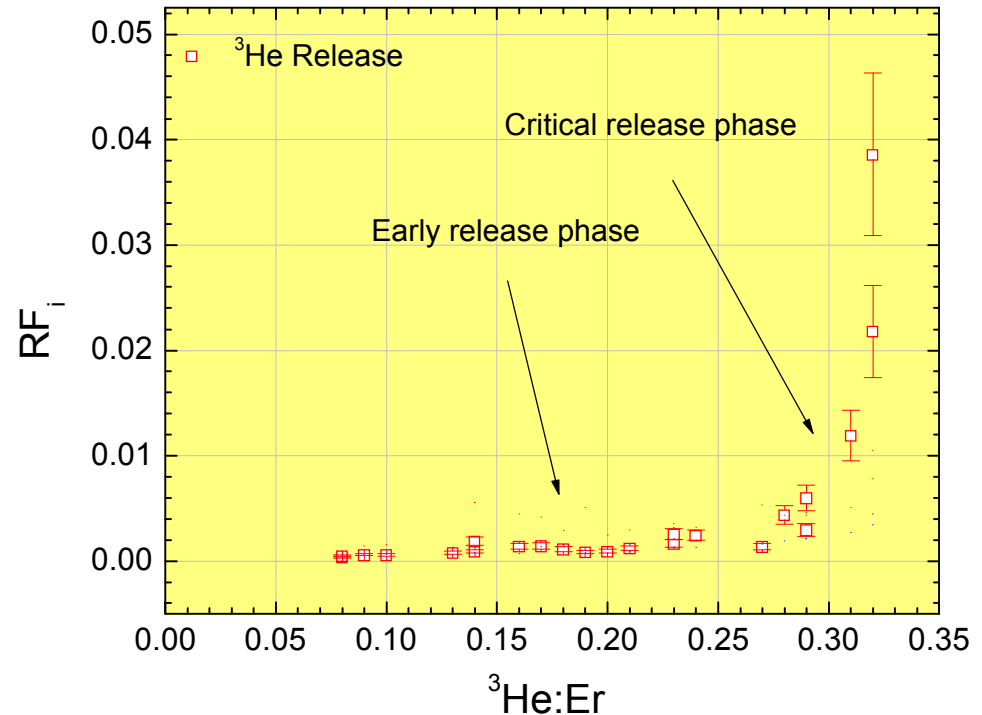
- Erbium and molybdenum film depositions carried out at SNL
 - Dale Blankenship
- Hydriding of the films was carried out at SRNL
 - Kirk Shanahan
 - David Bell
- IBA characterizations performed at SNL
 - Jim Banks
- Neutron scattering experiments performed on SPEAR at LANSCE
 - Lujan Neutron Scattering Center at the Los Alamos Neutron Science Center (LANSCE) is supported under DOE contract W7405-ENG-36 and by the Department of Energy basic Energy Sciences.

Introduction

- ^3He out-gassing in many metal/metal hydride systems is characterized by two distinct release phase regions
- Given that the bulk of the ^3He produced remains in the lattice



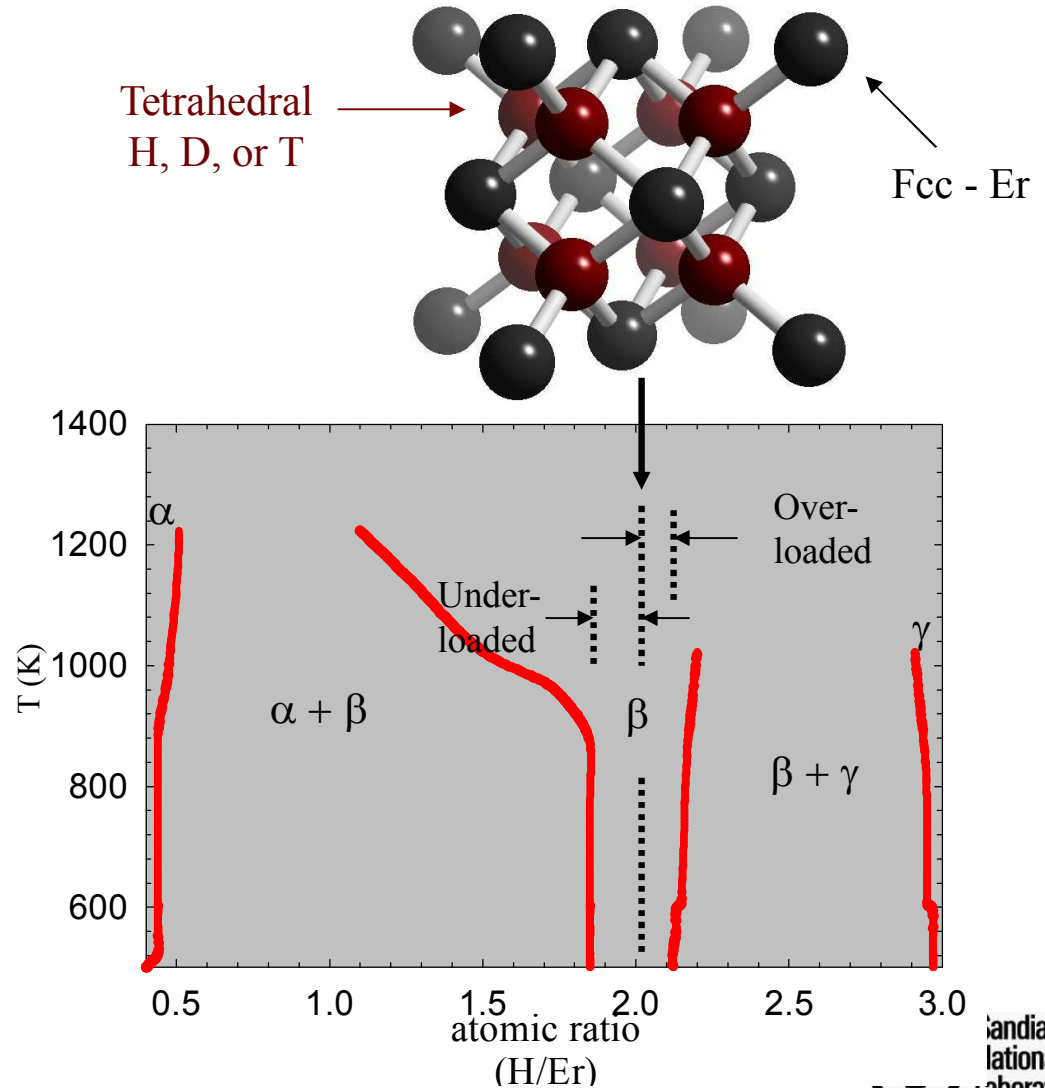
- The intent of this work has been to determine how ErT_2 film structure evolves (interfacial and hydride layer) with increasing ^3He concentration in the lattice



$$\text{RF}_i = \frac{-\frac{d}{dt} N(t)}{G(t)}$$

Structure of erbium hydride

- The beta phase of erbium hydride assumes the fluorite structure
- The hydrogen-to-erbium atomic ratio in the fluorite structure extends from about 1.85 to 2.15

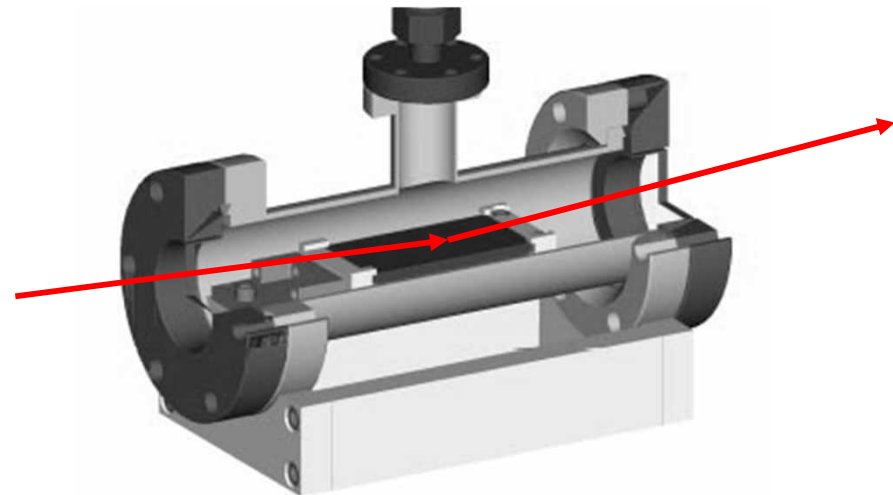


Experiment

- Sample configuration:
 - Si <111> substrate
 - Deposition
 - 1000 Å Mo (to prevent formation of Er-Si compounds) deposited by e-beam PVD
 - 1500 Å of Er deposited by e-beam PVD
 - rate of 10 Å/s
 - Substrate temperature = 450°C
 - Hydriding
 - 450 °C at ~ 200 Torr
 - SS reactor
- Scattering Chamber:
 - Modified Nor-Cal 6" tee
 - Swagelok BW series valve
 - Sapphire windows
 - Sample sits on BN
 - Chamber evacuated ~ 1×10^{-7} Torr



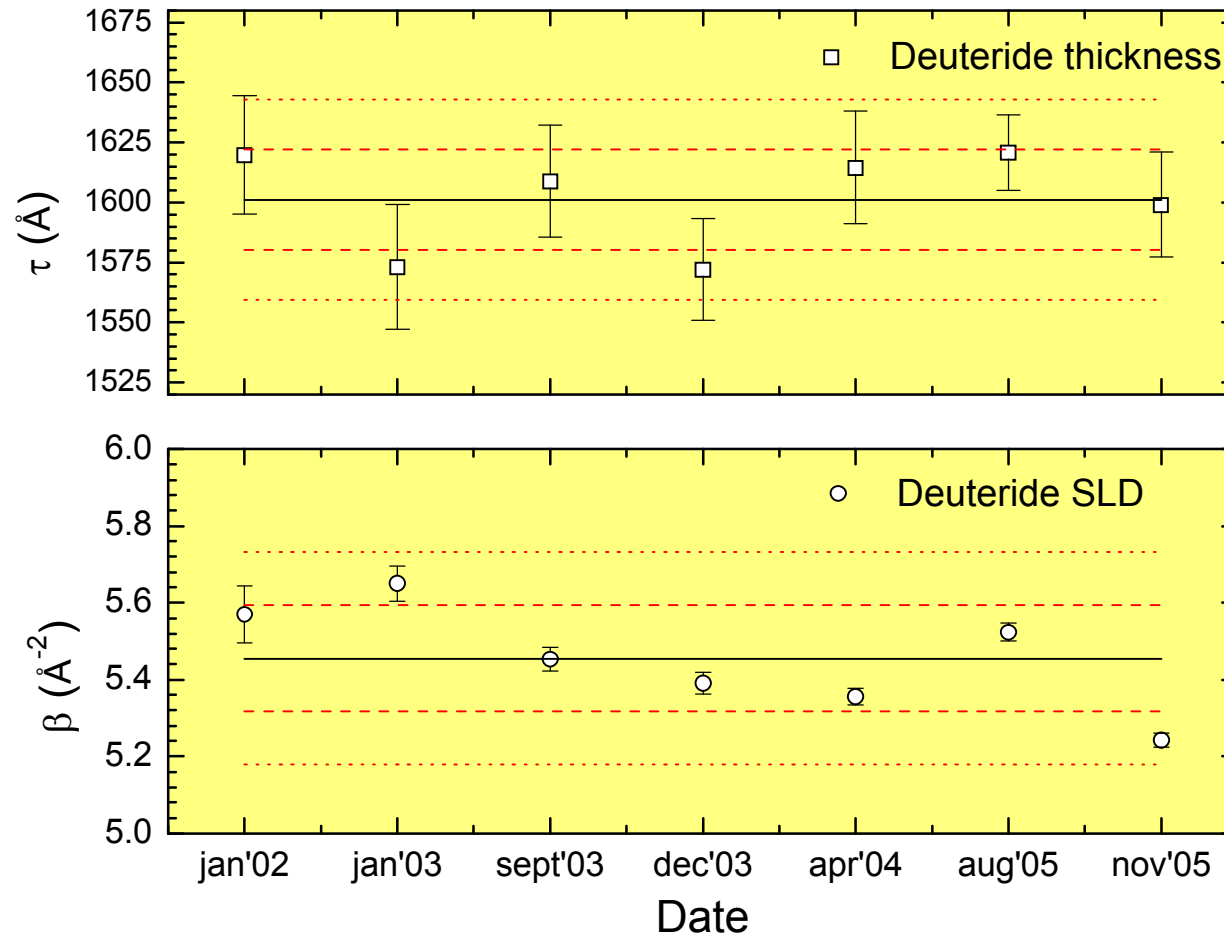
Si dimensions:
2.54 cm x 5.08 cm x 0.635 cm



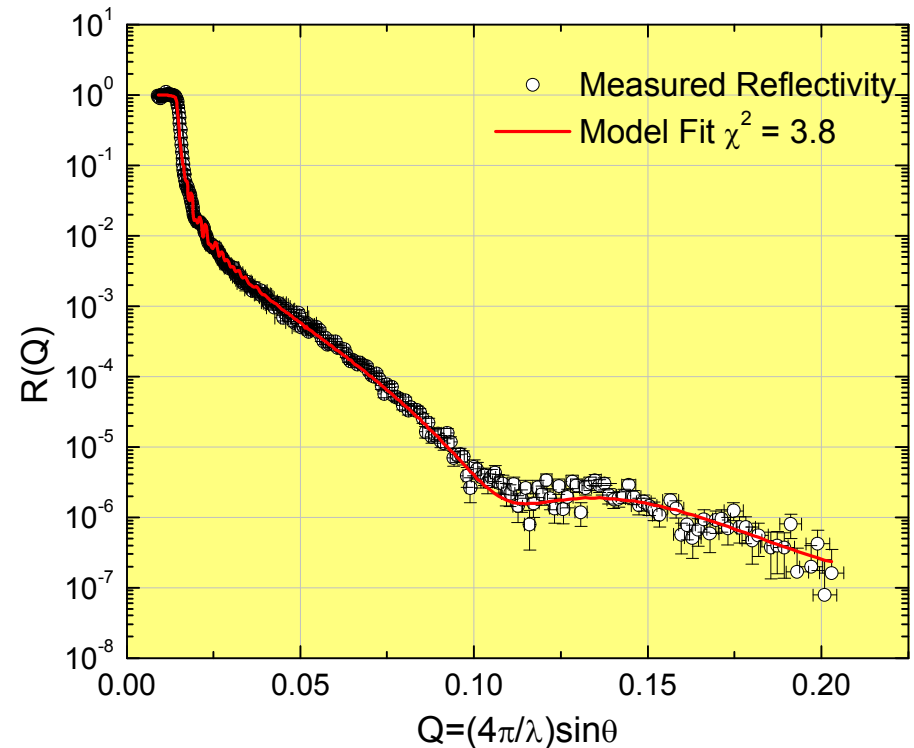
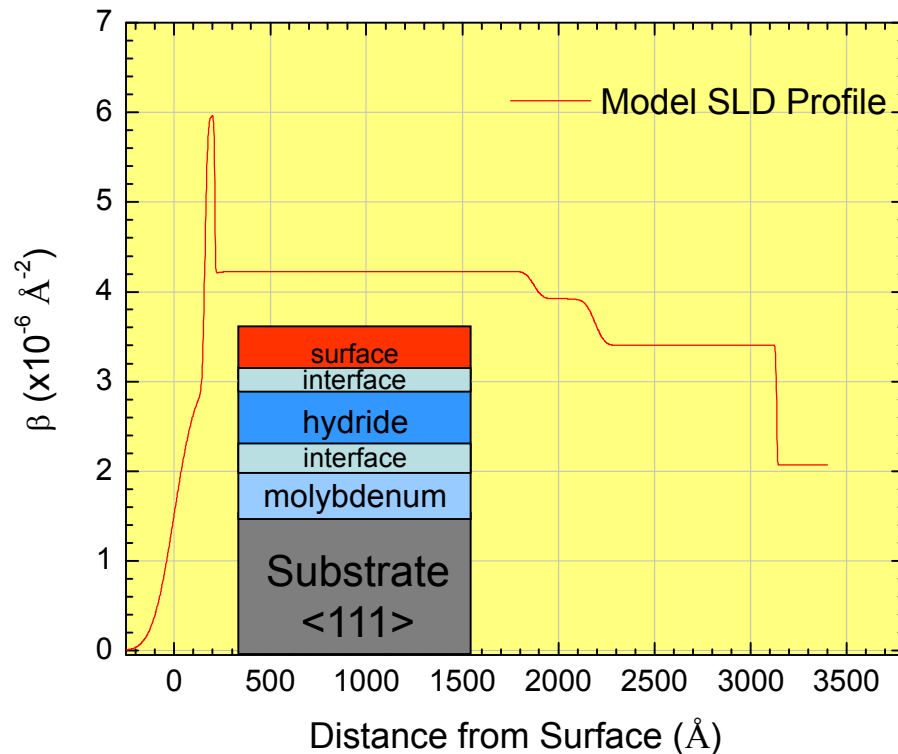
IBA characterization of tritide films

Atom	Sample 1 (atoms/cm ²)	Sample 2 (atoms/cm ²)
H	1.65x10 ¹⁷	2.31x10 ¹⁷
D	4.54x10 ¹⁵	4.89x10 ¹⁵
T	7.59x10 ¹⁷	7.14x10 ¹⁷
Er	4.86x10 ¹⁷	4.48x10 ¹⁷
(HDT)/Er	1.91	2.08

Deuteride layer thickness and SLD as measured over the duration of the study

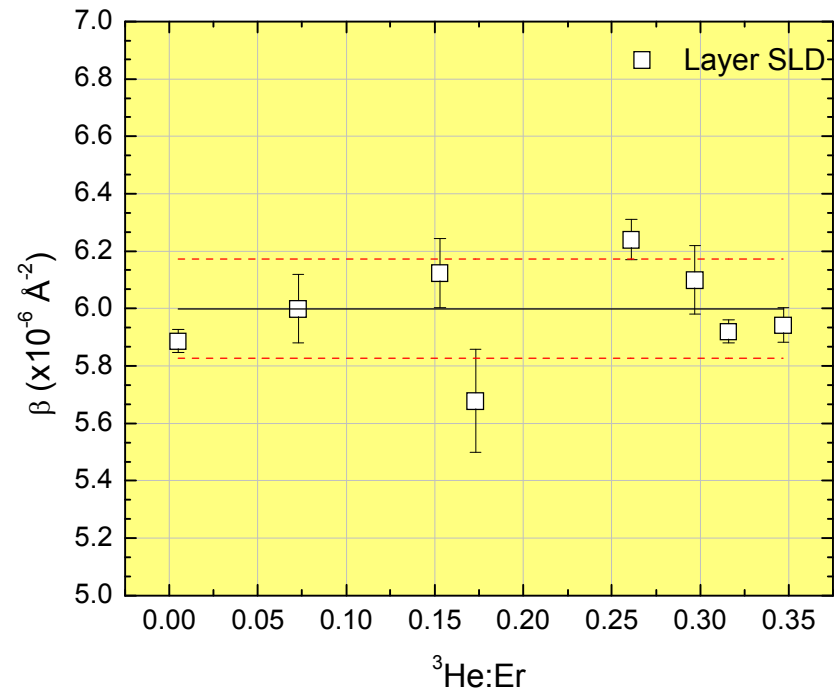
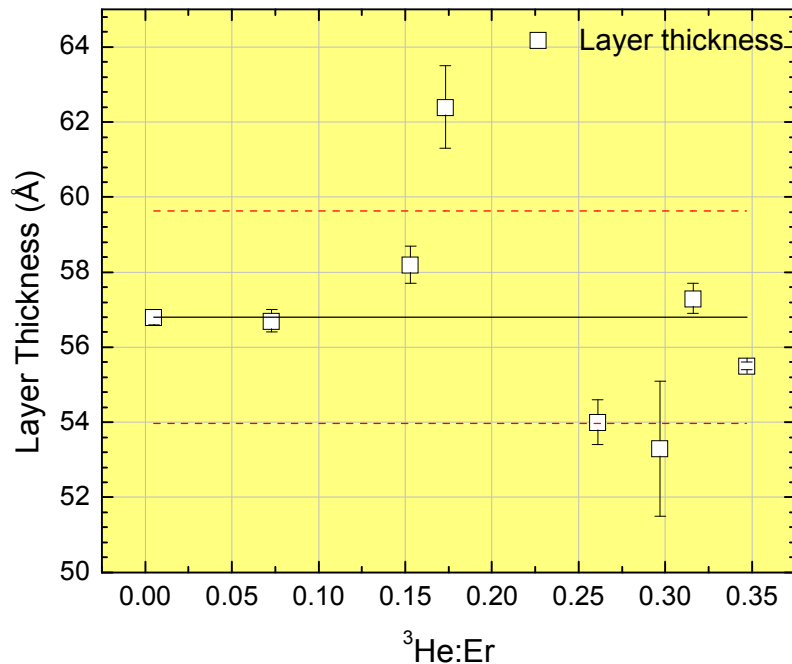


Example of experimental results and subsequent model fit to data

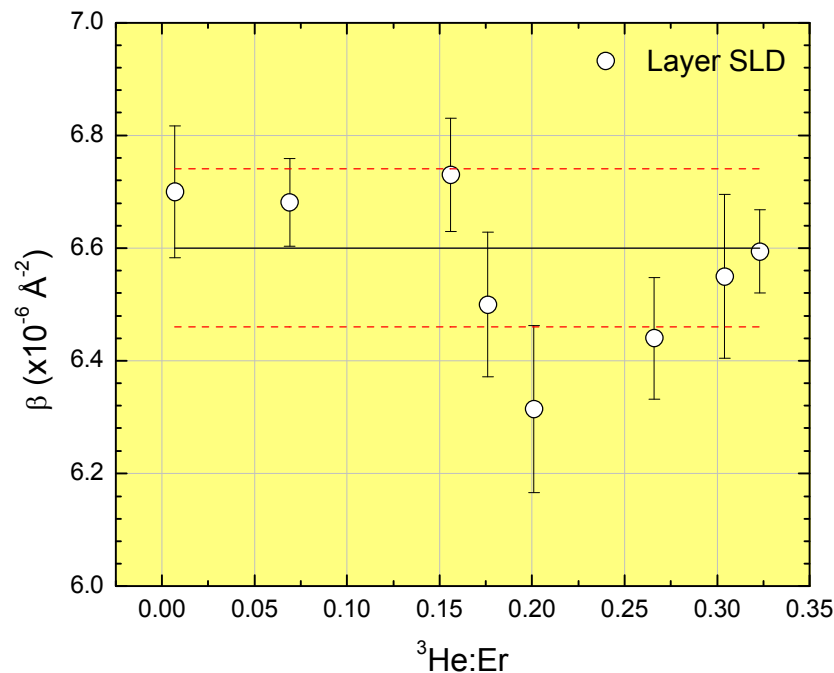
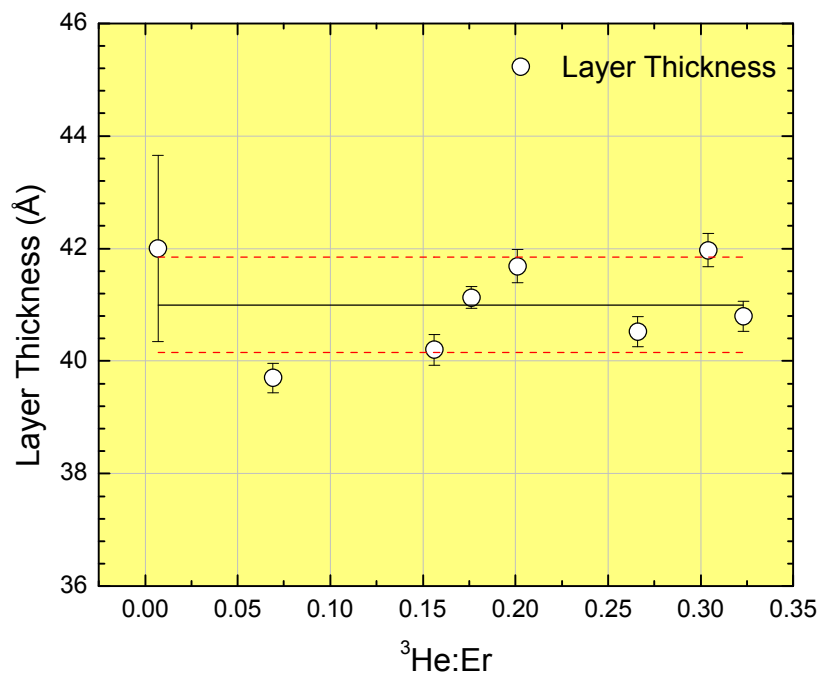


Sample 1
 $^3\text{He:Er} = 0.073$

Sample 1: layer thickness and SLD of near surface region as a function of ^3He concentration

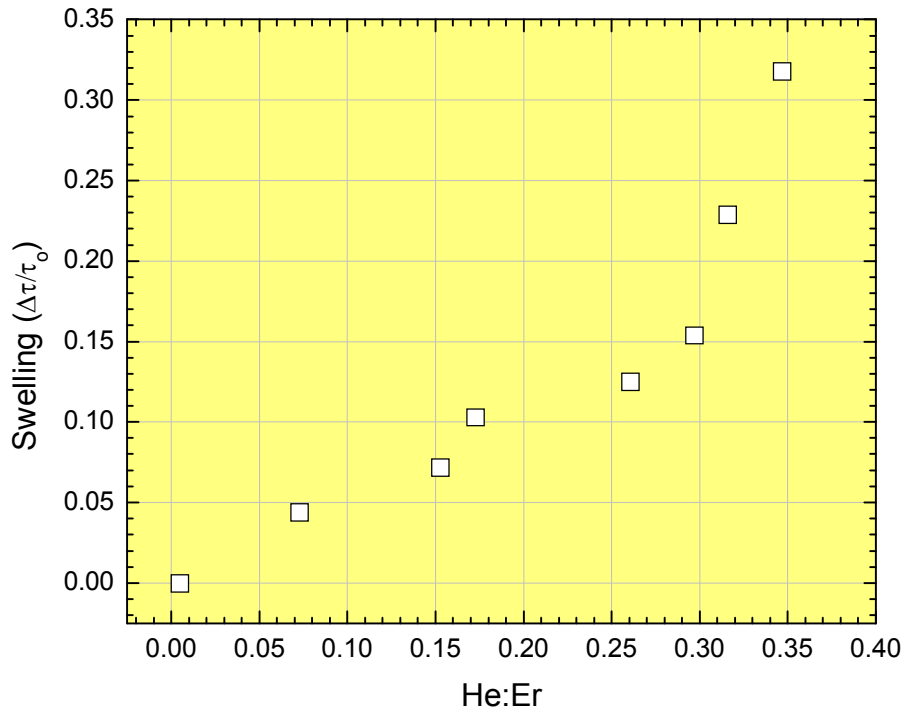


Sample 2: layer thickness and SLD of near surface region as a function of ^3He concentration



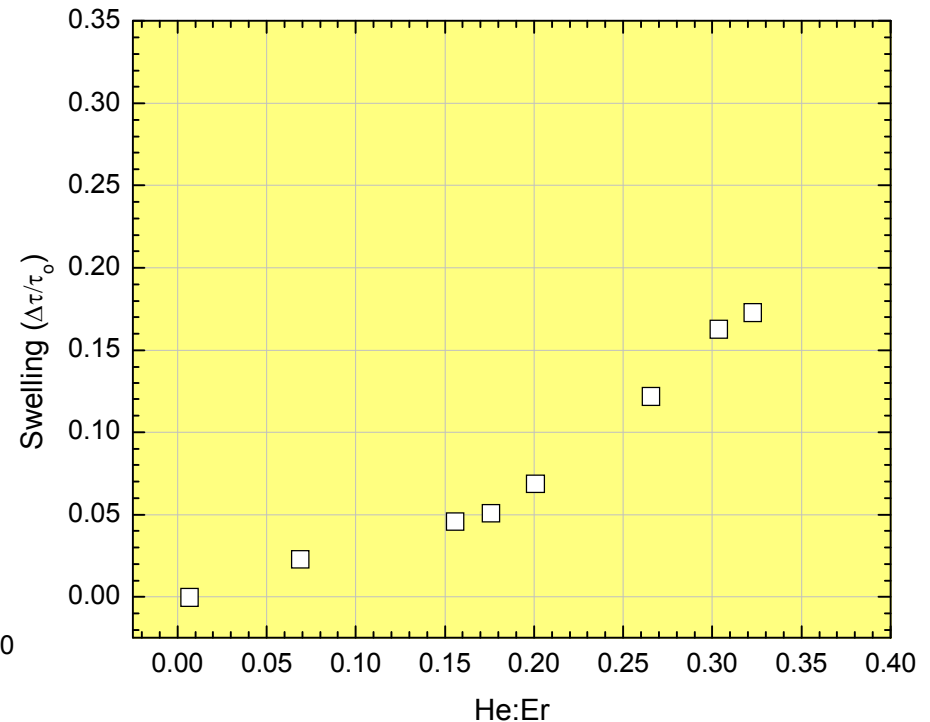
Tritide layer swelling as a function of ^3He concentration

G:M=1.91



Sample 1

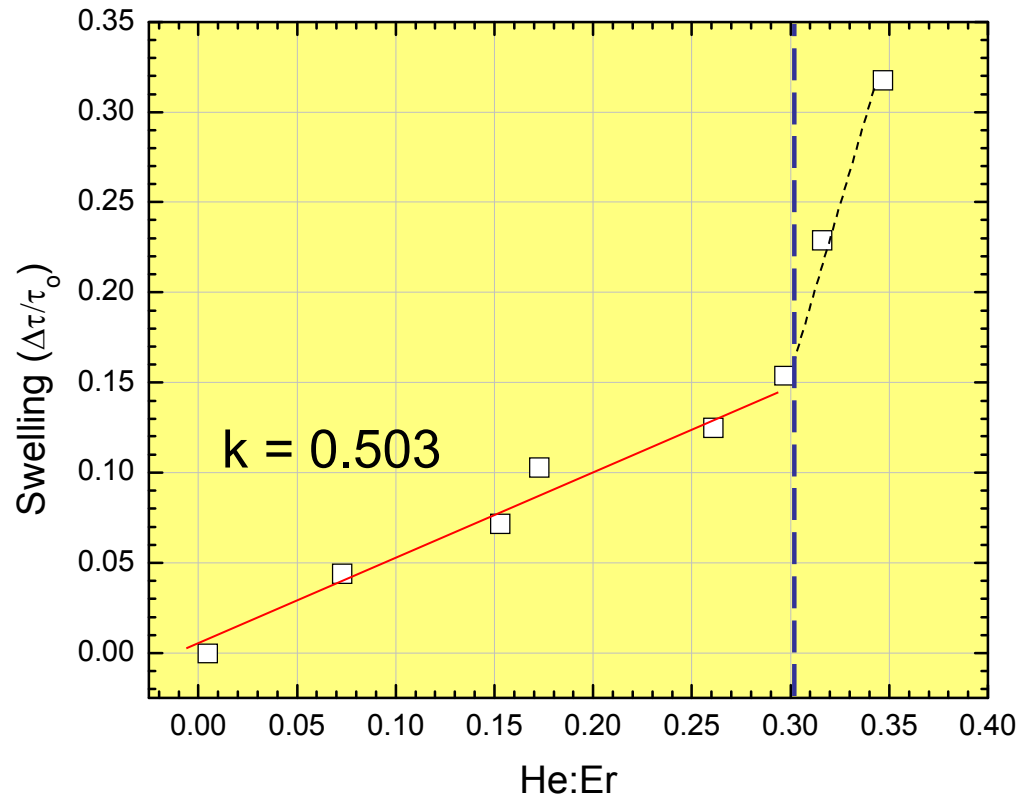
G:M=2.08



Sample 2

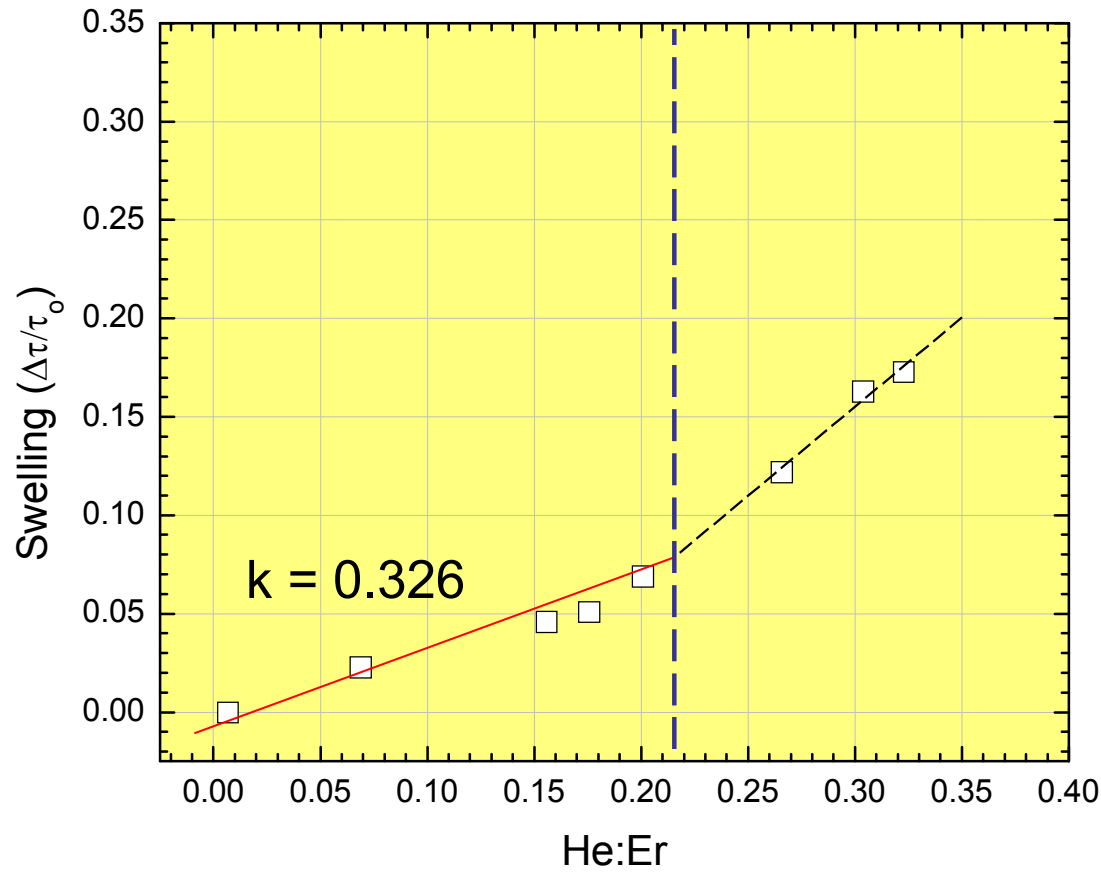
A closer look at the swelling data

Sample 1



A closer look at the swelling data

Sample 2



Relationship between tritide swelling and ^3He atomic volume

$$\frac{\Delta\tau}{\tau_o} = \underbrace{\left(\frac{v_{\text{He}}}{v_{\text{M}_\text{H}}} \right)}_k \left(\frac{^3\text{He}}{\text{Er}} \right)$$

(Shobert, et al., Phys. Rev B, 31(11), 7109, 1985.
Shobert, et al., Phys. Rev B, 40(2), 1277, 1989.
Cowgill, SAND 2004-1739, 2004.)

- Where
 - $v_{\text{He}} \equiv$ helium atomic volume
 - $v_{\text{MH}} \equiv$ erbium atomic volume in the hydride

- Given

$$v_{\text{MH}} \approx 135 \text{\AA}^3$$

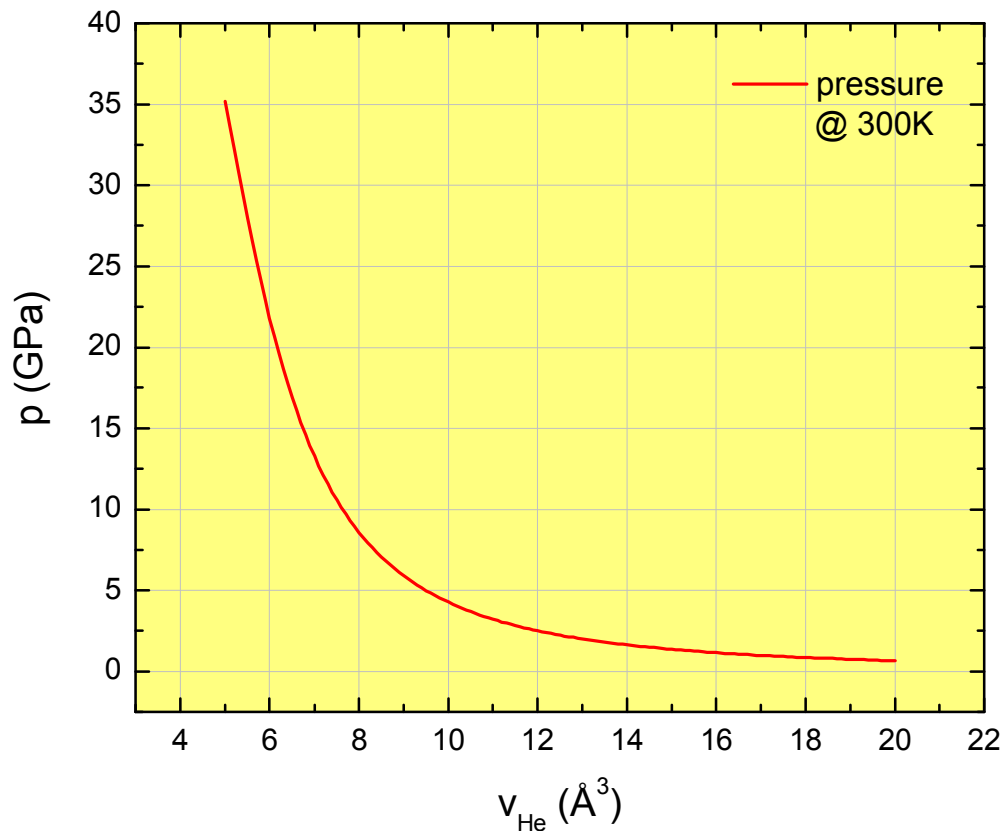
- Then

$$\text{Sample1: } v_{\text{He}} = 17.2 \text{\AA}^3$$

$$\text{Sample2: } v_{\text{He}} = 11.1 \text{\AA}^3$$

Bubble pressure determined by use of Mill's EOS

$$p^{-1} = 0.1196 - 0.04801v_{\text{He}} + 0.005955v_{\text{He}}^2$$



$$\Rightarrow p_{\text{sample1}} = 0.95 \text{ GPa}$$

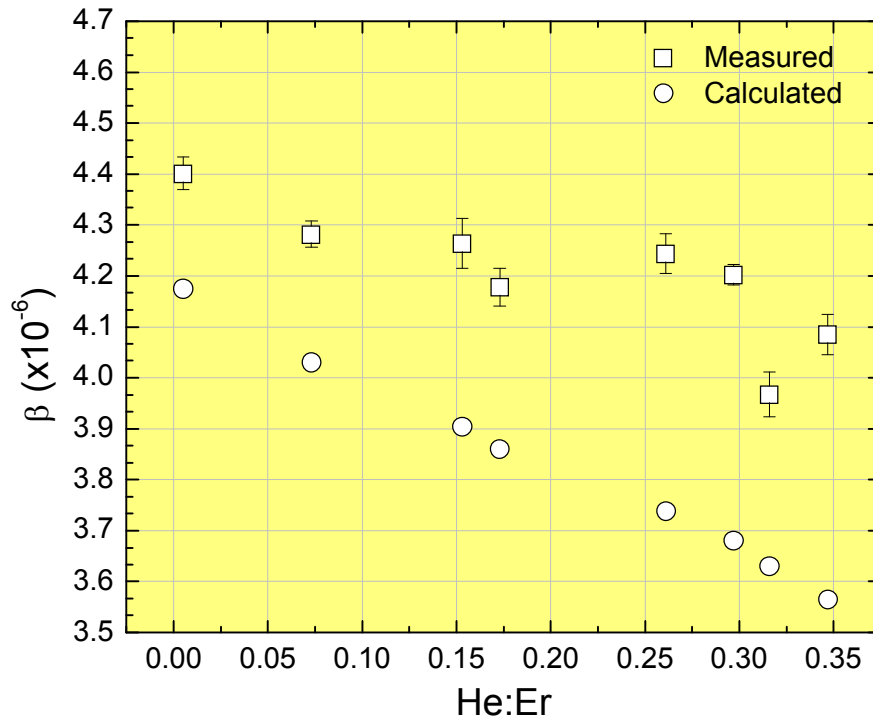
$$\Rightarrow p_{\text{sample2}} = 3.12 \text{ GPa}$$

Summary of tritide layer swelling data

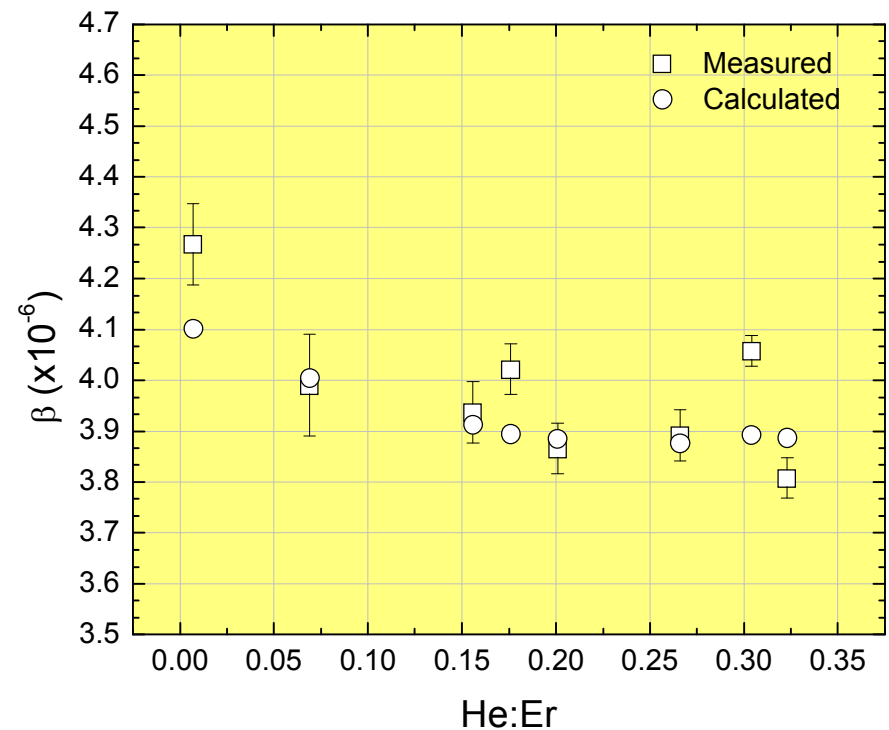
	Sample 1	sample 2
HDT:Er	1.91	2.08
T:Er	1.56	1.59
Slope	0.503	0.326
$^3\text{He:Er}$	0.297	0.201
$\Delta\tau/\tau_0$	0.15	0.11
$v_{\text{He}} (\text{\AA}^3)$	17.2	11.1
P (GPa)	0.95	3.12

Measured verses predicted SLD

$$\beta_{\text{layer}} = x\beta_{\text{ErT}_2} + (1-x)\beta_{\text{bubbles}}$$



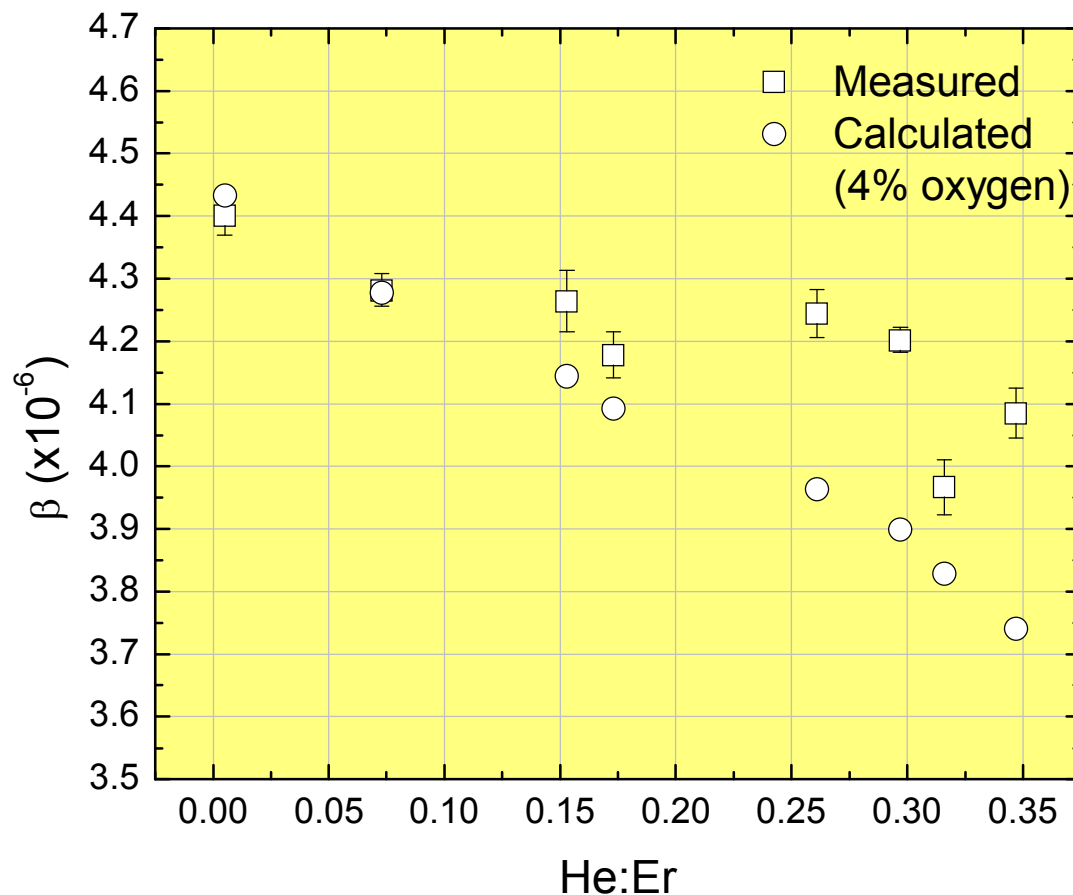
Sample 1



Sample 2

Measured verses predicted SLD

(corrected for oxygen)



Summary

- The data indicate the existence of a near surface region with a length scale of $\sim 50 \text{ \AA}$ and of high scattering length density (consistent with high oxygen/tritium concentration (perhaps ErOOT or $\text{Er}(\text{OT})_3$)).
- Tritide layer expansion yields information on helium atomic volume within a bubble.
- The helium atomic volume obtained in the experiment is used with Mill's helium EOS to estimate pressure within a bubble during the constant expansion stage.
- The sample with higher hydrogen isotope-to-erbium ratio shows a higher bubble pressure and indicates a transition to higher helium atomic volume within a bubble occurs at a lower $^3\text{He}:\text{Er}$ ratio.