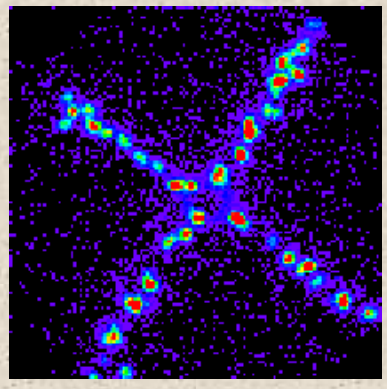
Hydrogen and Helium Isotopes in Materials Conference
Albuquerque, NM **February 6 & 7, 2007**



^{3}He Concentration Profiles

by the

Neutron Depth Profiling (NDP) Technique

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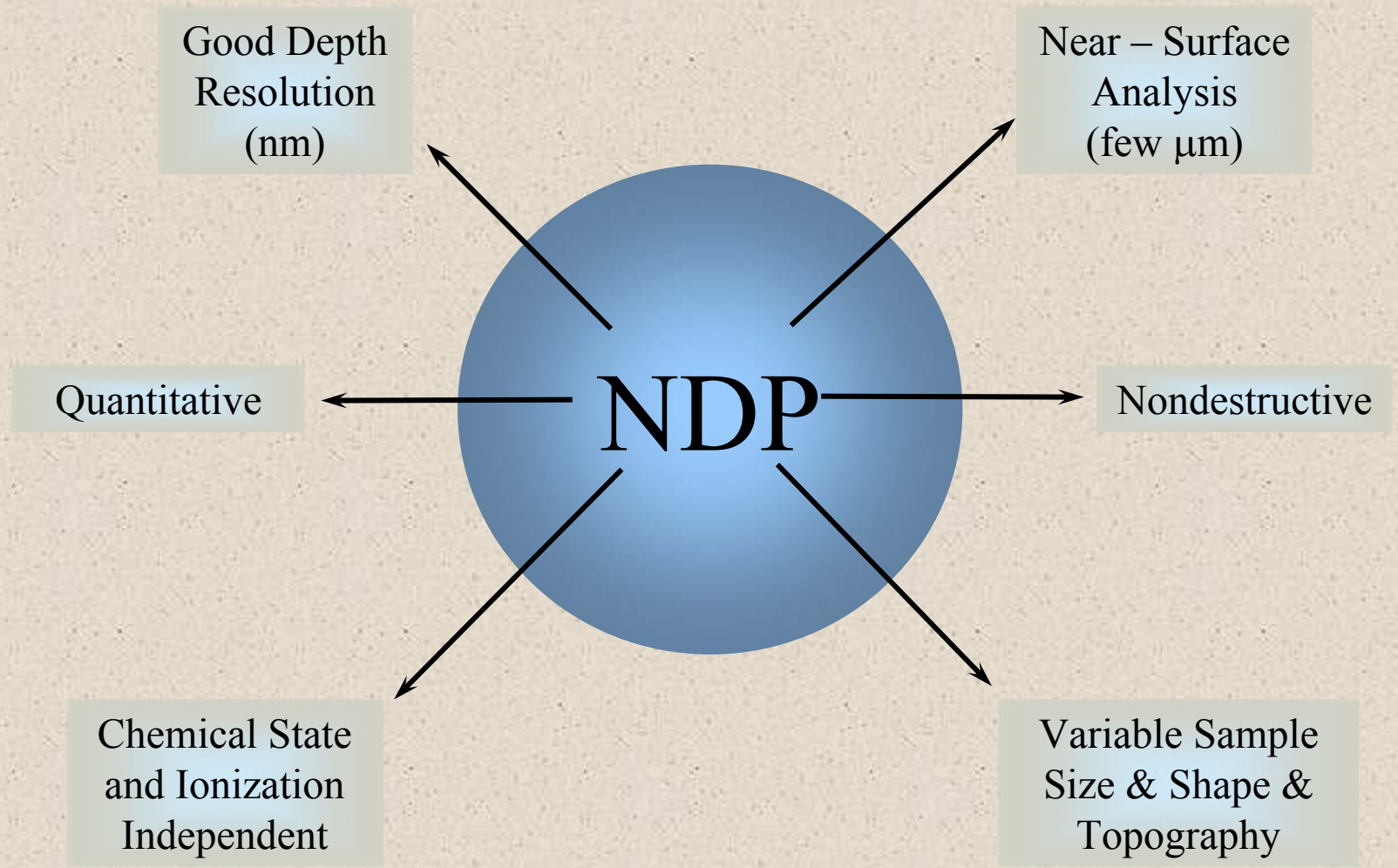
Metrological Attributes of NDP



Prompt Neutron Reactions for Neutron Depth Profiling

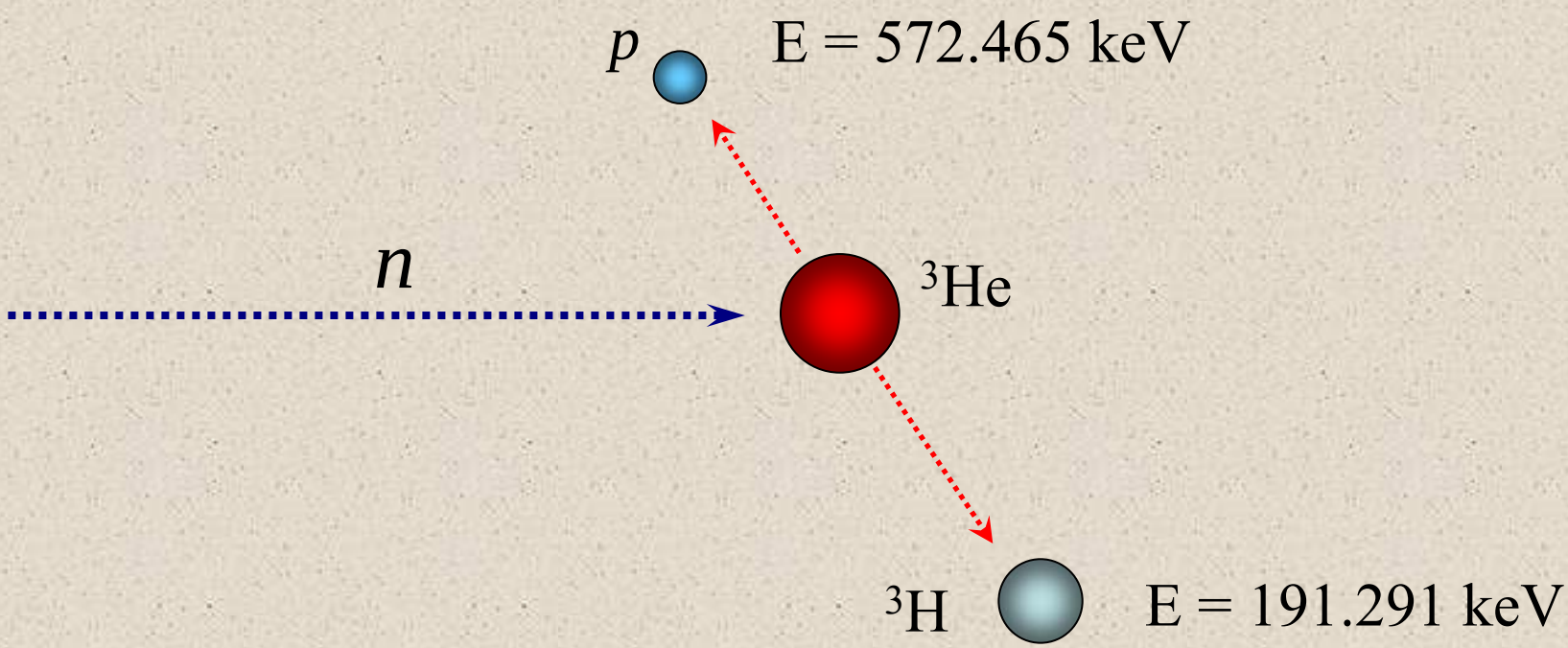
At. No.	Element	Reaction	Cross section (barns)‡	Abundance (or at/mCi)	$T_{1/2}$	Recoil Mass Energy (keV)	Particle Energy (keV)	Branch Ratio
3	**He**	$^{3}He(n,p)^{3}H$	5331	1.37E-06	-	191.291	572.465	
6	**Li**	$^{6}Li(n,\alpha)^{3}H$	940.3	0.075	-	2727.877	2055.515	
10	**B***	$^{10}B(n,\alpha)^{7}Li$	3837	0.199	-	1013.126	1775.868	6.28%
10	**B***	$^{10}B(n,\alpha)^{7}Li$	3837	0.199	-	839.635	1471.763	93.70%
14	N	$^{14}N(n,p)^{14}C$	1.83	0.99634	-	42.02	583.851	
17	O	$^{17}O(n,\alpha)^{14}C$	0.24(?)	3.80E-04	-	404.064	1413.632	
33	S	$^{33}S(n,\alpha)^{30}Si$	0.1686	0.0075	-	411.534	3081.804	
35	Cl	$^{35}Cl(n,p)^{35}S$	0.489	0.7577	-	17.233	597.931	
40	K	$^{40}K(n,p)^{40}Ar$	4.4 (0.39 α)	1.17E-04	1.277 E9 y	56.259	2230.781	
7	Be	$^{7}Be(n,p)^{7}Li$	~ 48000	2.45E+14	53.12 d	206.523	1437.718	
22	Na†	$^{22}Na(n,p)^{22}Ne$	27800	4.38E+15	2.6019 y	158.831	3465.797	0.84%
22	Na†	$^{22}Na(n,p)^{22}Ne$	27800	4.38E+15	2.6019 y	102.979	2247.069	99.10%
59	Ni	$^{59}Ni(n,\alpha)^{56}Fe$	~ 12.3	1.28E+20	76000 y	340.316	4755.791	
210	Po	$^{210}Po \rightarrow \alpha \rightarrow ^{206}Pb$	---	6.38E+14	138.376 d	103.077	5304.381	

* A 477.595 (3) keV (IAEA) γ emission occurs in 93.9 % of the ^{10}B reactions

† A 1274.58 keV γ emission occurs in XX % of the ^{22}Na reactions

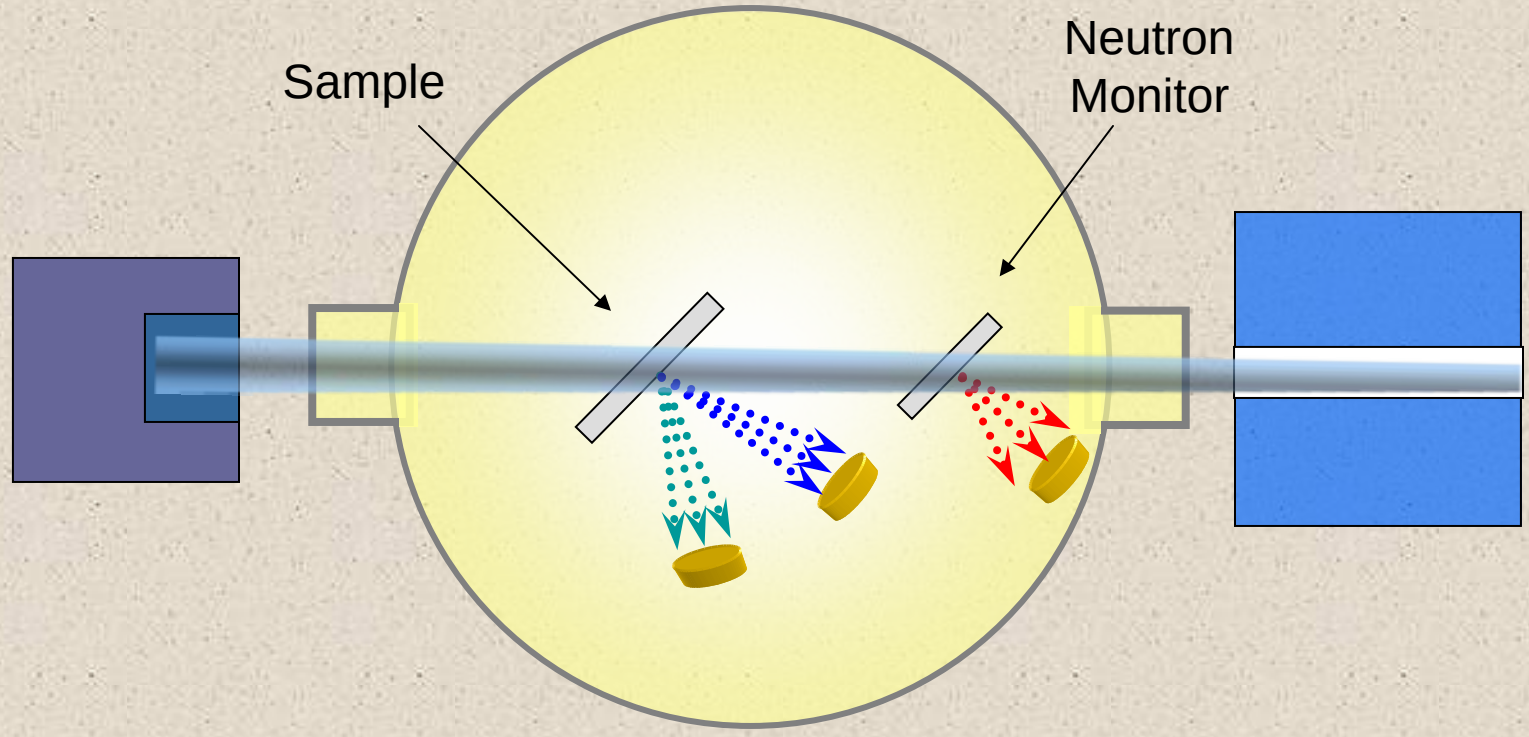
NDP: Physical Principle

$^{3}He(n,p)^{3}H$



Particle emission is always diametrical

NDP Instrument

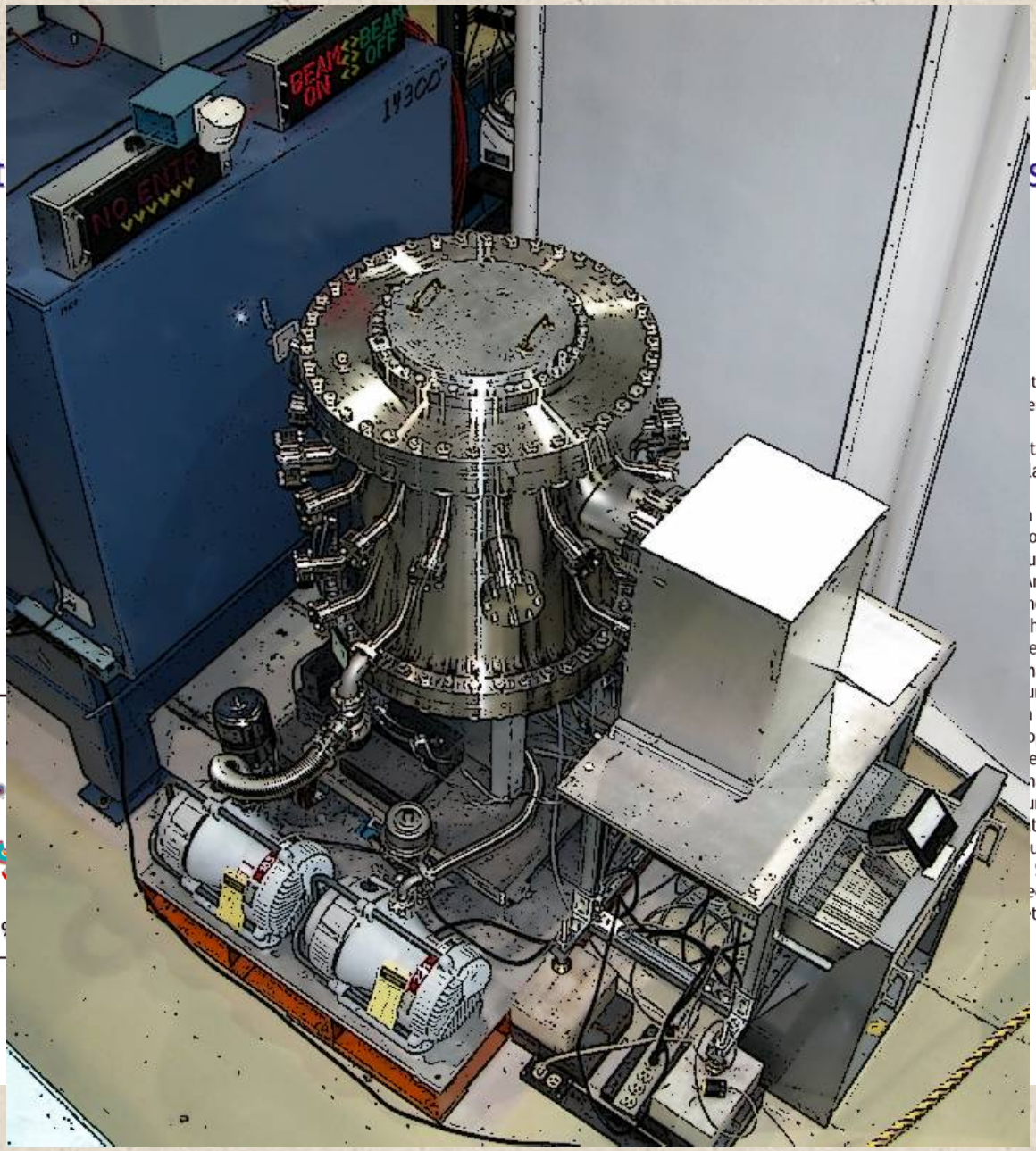


- Determine **concentration vs depth profiles** through the first few micrometers of surface
- **Few nanometer depth resolution** depending on the depth, reaction, and material

National Institut search

Thermal Neutron Instruments
in Confinement Building

1) NG-0 cold neutron Multi-Analyzer Crystal Spectrometer
2) BT-7 thermal Triple Axis Spectrometer with double-focusing monochromator
3) BT-8 Residual Stress Diffractometer
4) BT-9 thermal Triple Axis Spectrometer
5) BT-1 Powder Diffractometer
6) BT-2 Neutron Imaging Facility
7) BT-4 Filter Analyzer Spectrometer
8) BT-5 Perfect Crystal Small-Angle Neutron Scattering Diffractometer
9) (from top) VT-5 thermal neutron capture Prompt Gamma-ray Activation Analysis



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Interferometry and
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analysis
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hopper Time-of-Flight
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nall Angle Neutron Scat-
ument
Physics Station
opper Time-of-Flight
er
nall Angle Neutron Scat-
ument
ttering Spectrometer
utron Depth Profiling
Sample Reflectometer
ed Neutron
ter/Reflectometer

Neutron Depth Profiling: Boron

$^{10}_{5}B + ^{1}_{0}n$

94%

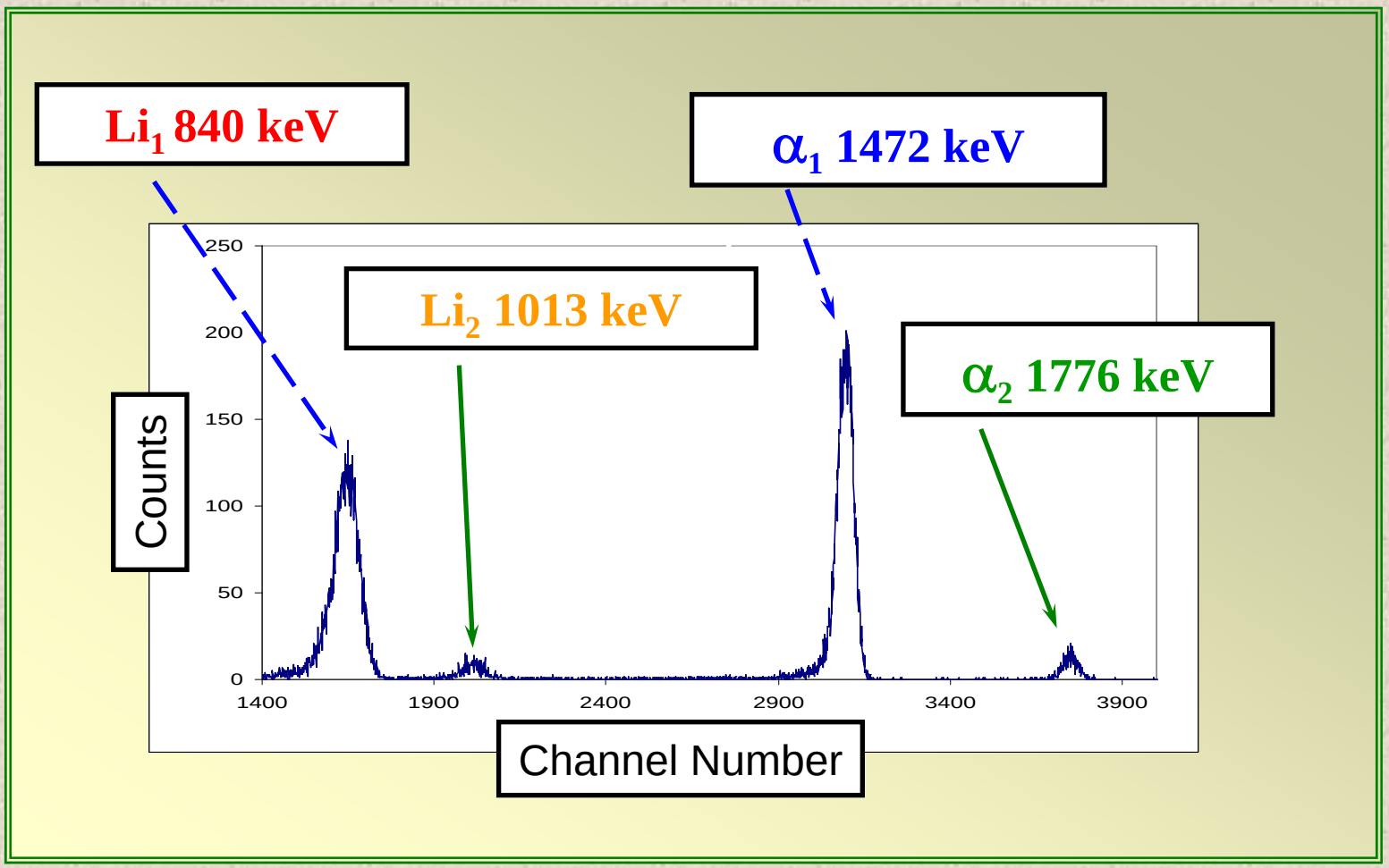
$^{7}_{3}Li^{*}$ (0.84 MeV) + $^{4}_{2}He$ (1.47 MeV)

γ (0.48 MeV)

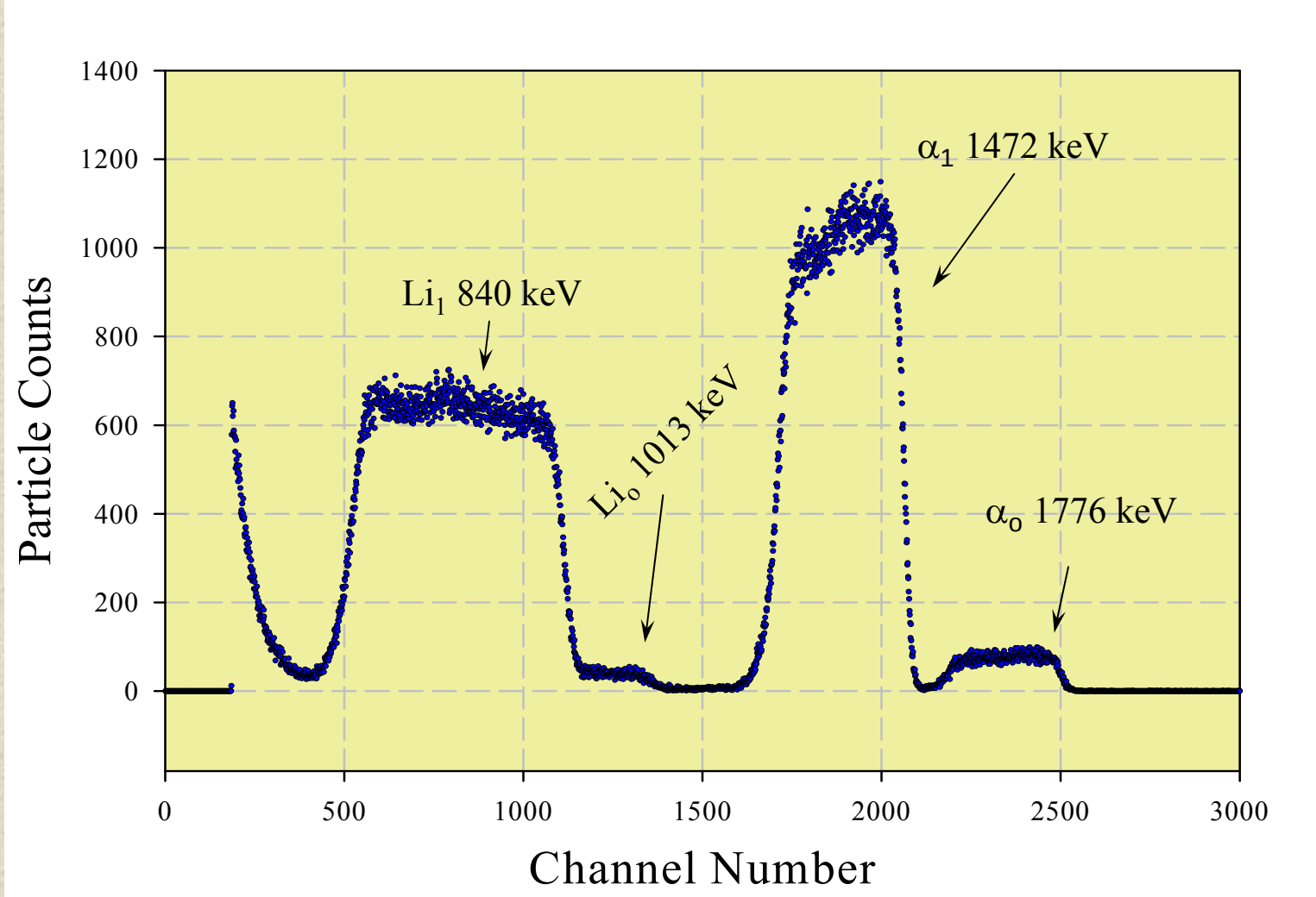
6%

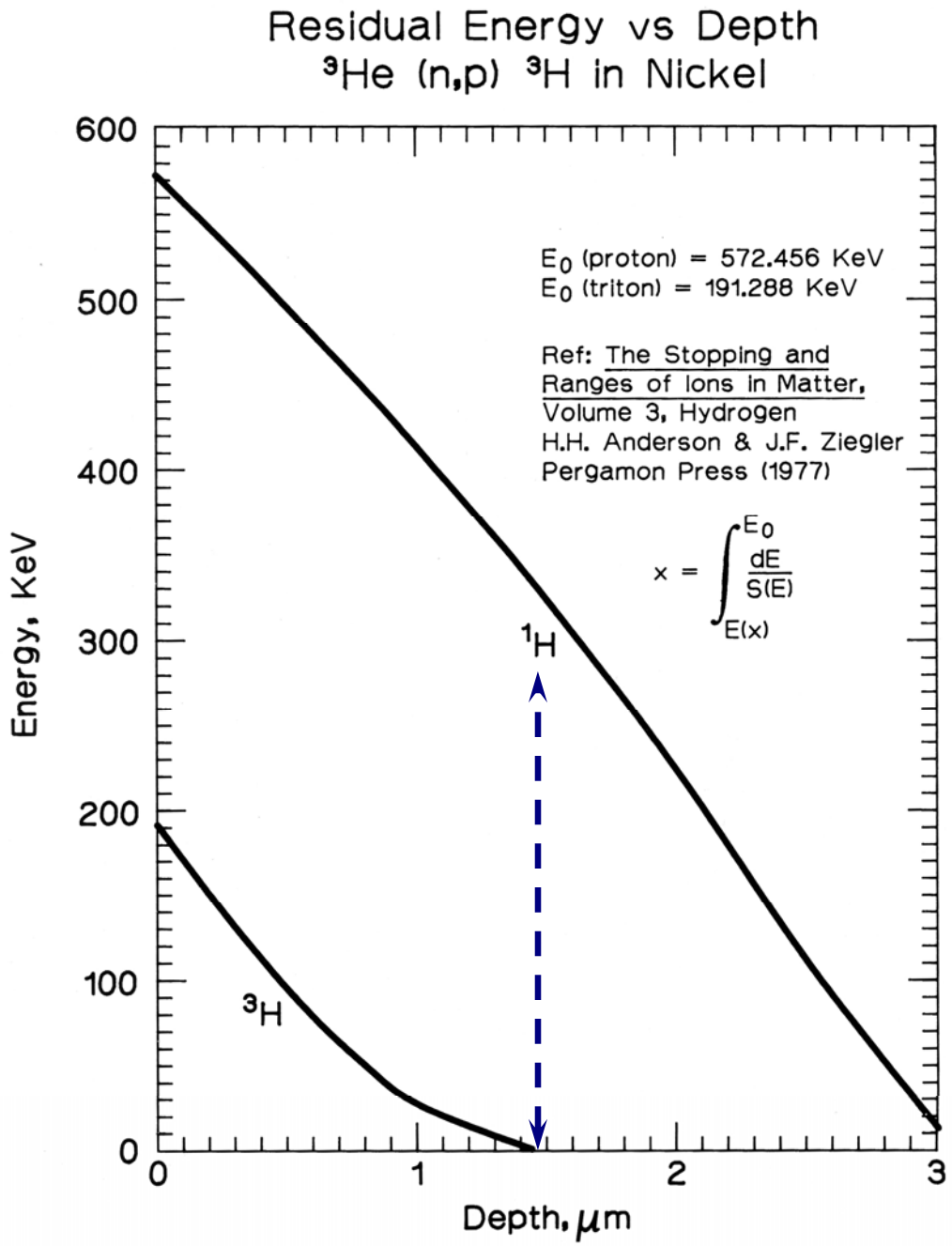
$^{7}_{3}Li$ (1.01 MeV) + $^{4}_{2}He$ (1.77 MeV)

$^{10}B(n,\alpha)^{7}Li$ Charged-Particle Energy Spectrum

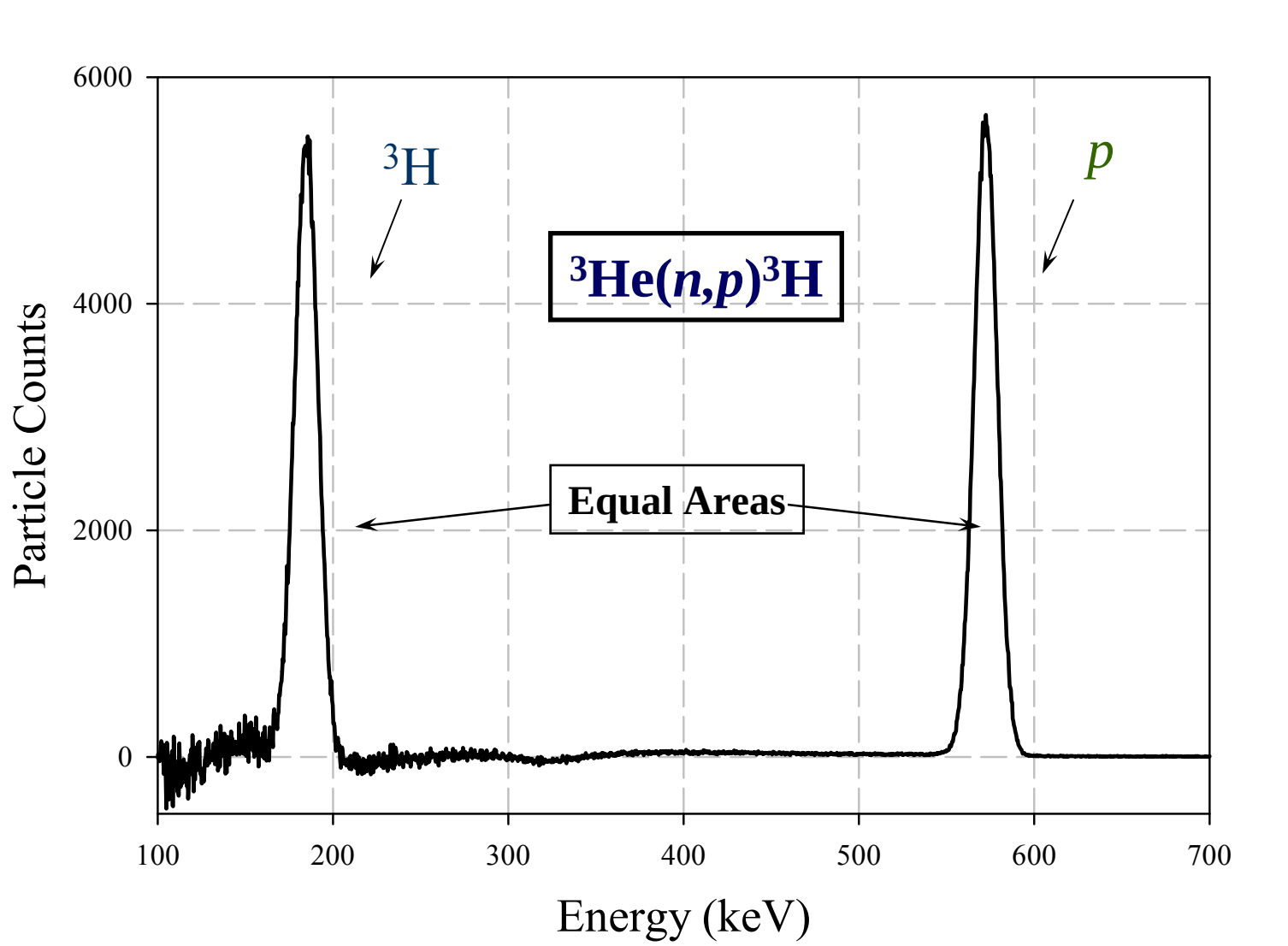


NDP Spectrum of Thin Boron Film



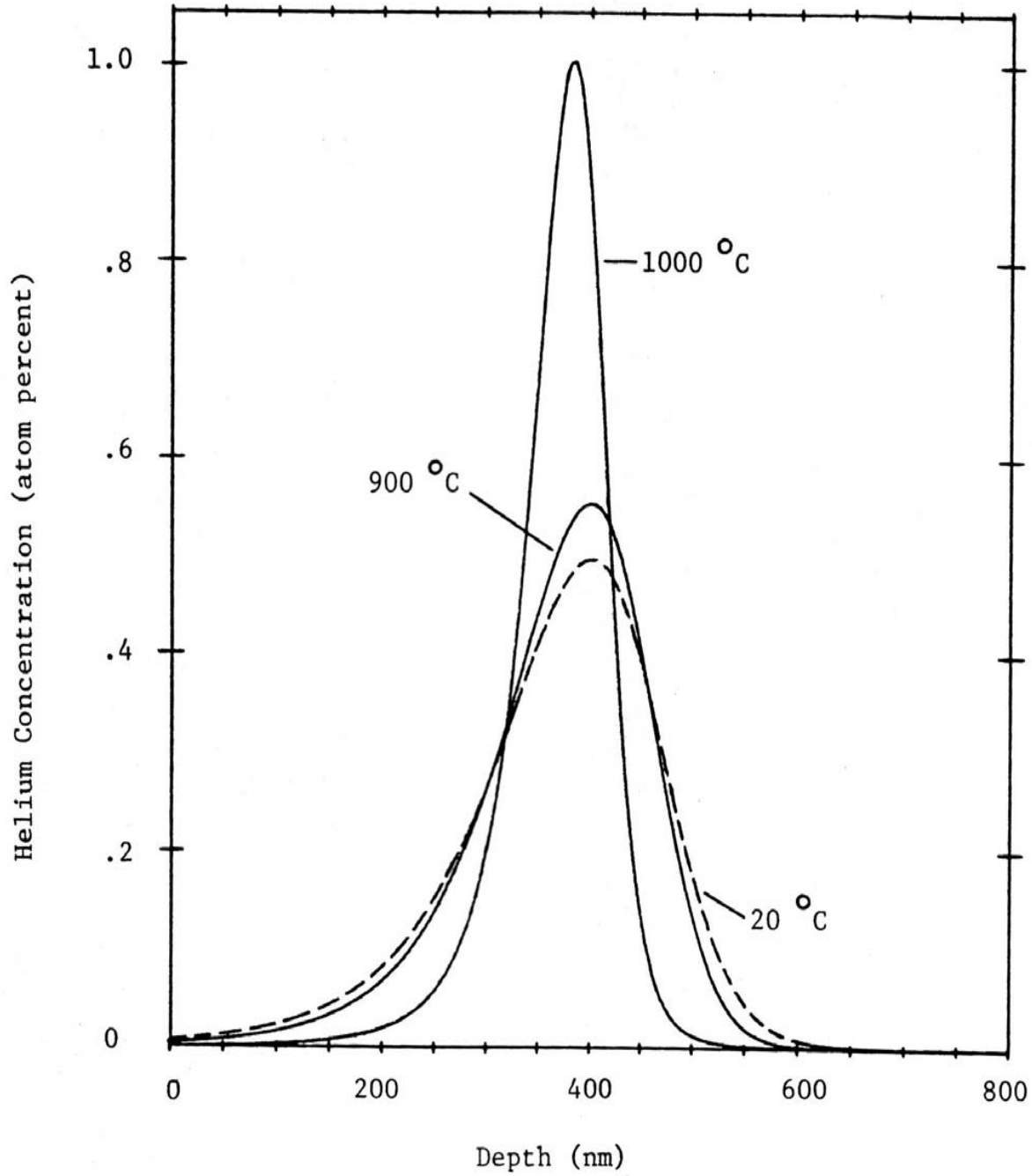

Residual Energy vs Depth
^{3}He (n,p) ^{3}H in Nickel
E_0 (proton) = 572.456 KeV
E_0 (triton) = 191.288 KeV
Ref: The Stopping and Ranges of Ions in Matter, Volume 3, Hydrogen
H.H. Anderson & J.F. Ziegler
Pergamon Press (1977)
$x = \int_{E(x)}^{E_0} \frac{dE}{S(E)}$
^{1}H
^{3}H
Energy, KeV
Depth, μm
600
500
400
300
200
100
0
0
1
2
3

Charged Particle Spectrum - Surface ^{3}He Implant



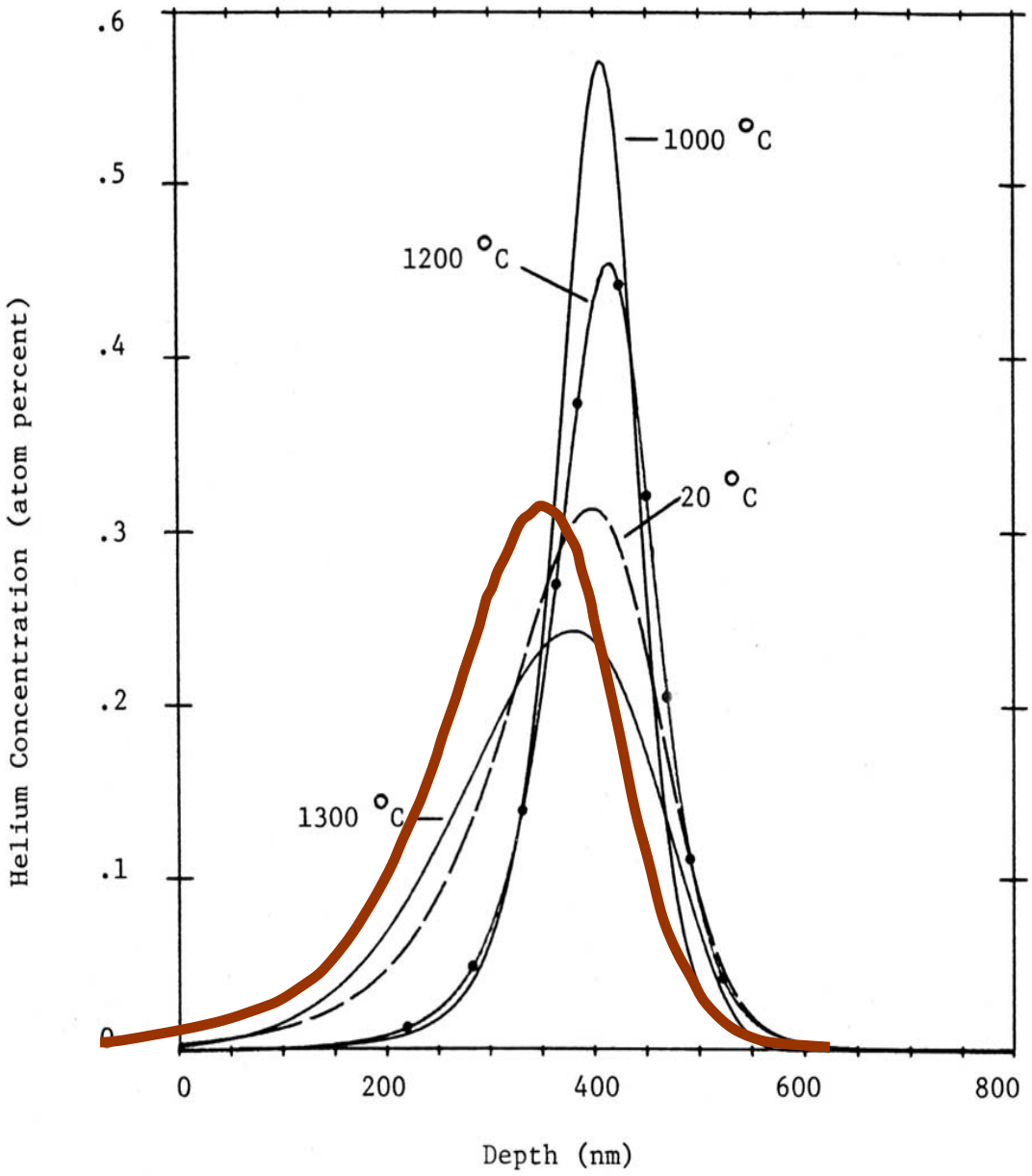
NDP Determination of ^{3}He in Single Crystal Ni

- 1E16/cm^2 implant
- Series of two high temp. anneals



NDP Determination of ^{3}He in Single Crystal Ni

- 1E16/cm^2 implant
- Series of three high temp. anneals



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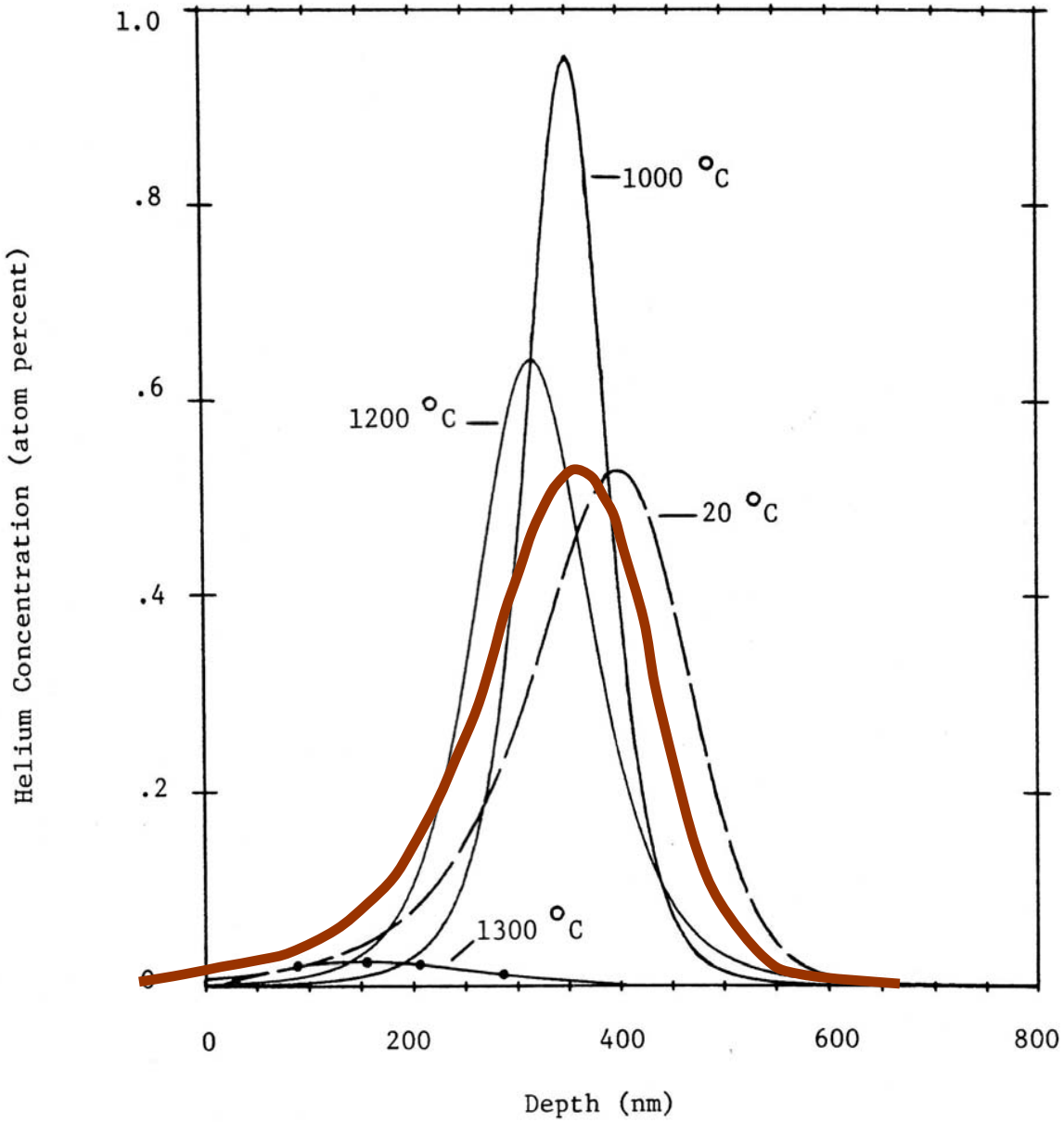


Inertial Fusion Energy Reaction Chamber

Helium flux and temperature conditions

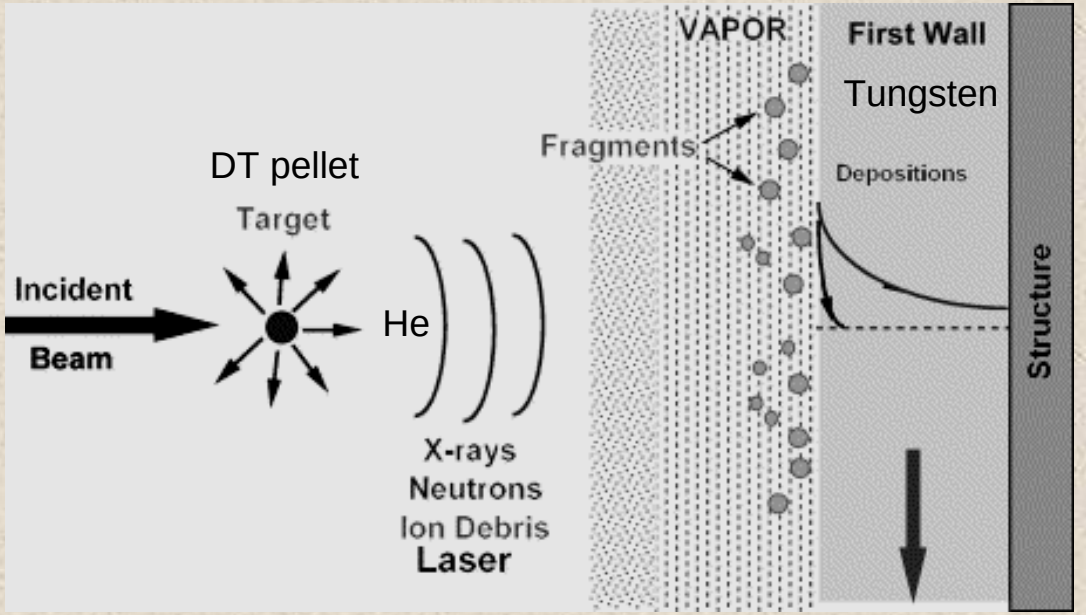
Helium flux: 5 x 10^{22} He/m^2/day

Implantation pulse width: ~2 μsec

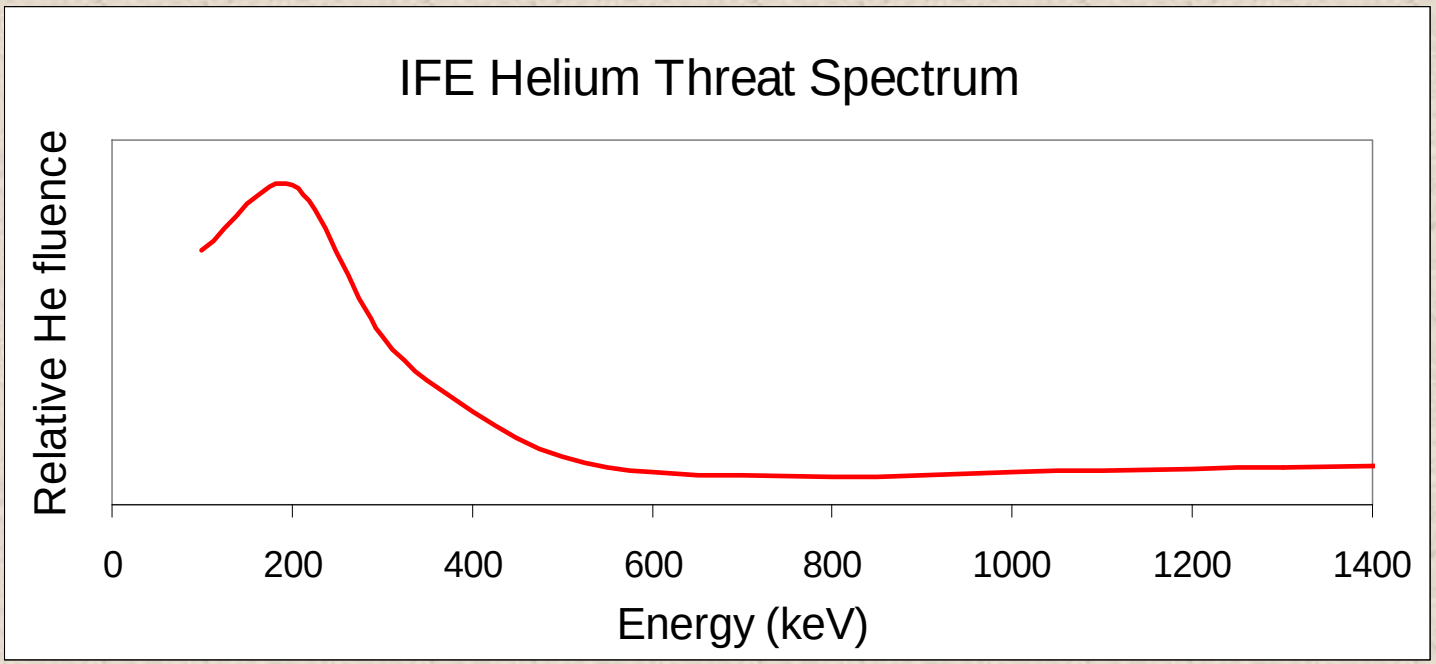
Helium spectrum: 50 – 500 keV

Wall temperature: 600 – 800°C with 2 μsec spikes to ~2400°C

Rep rate: 5 Hz **10^{17} He/m^2/shot**

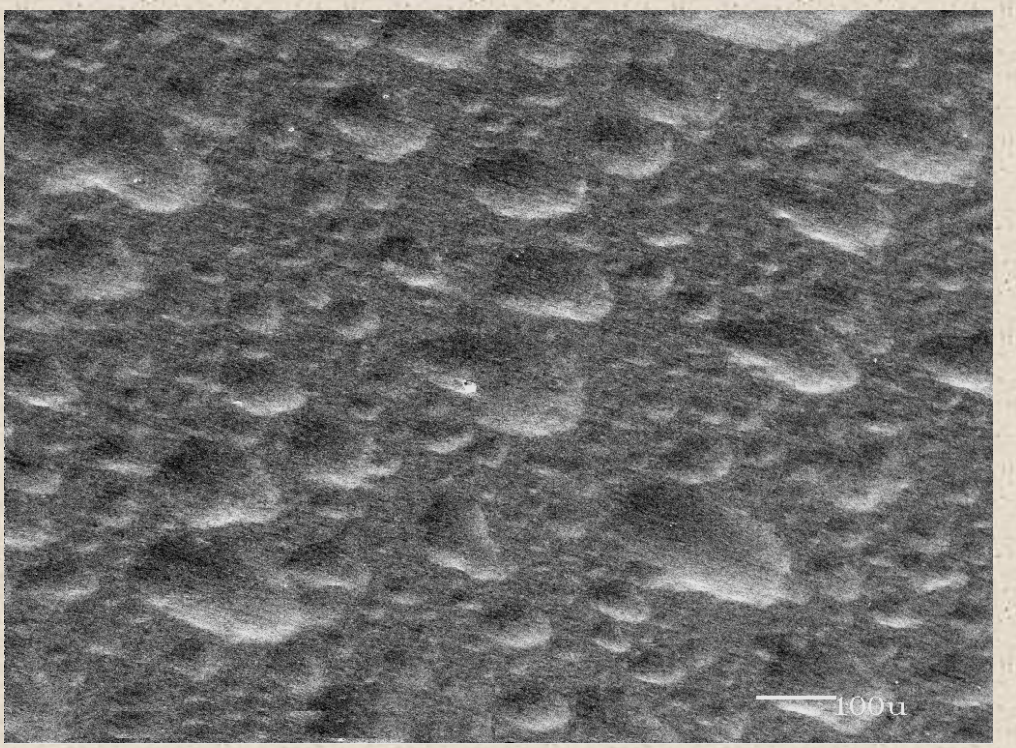


Total Helium Threat Spectrum

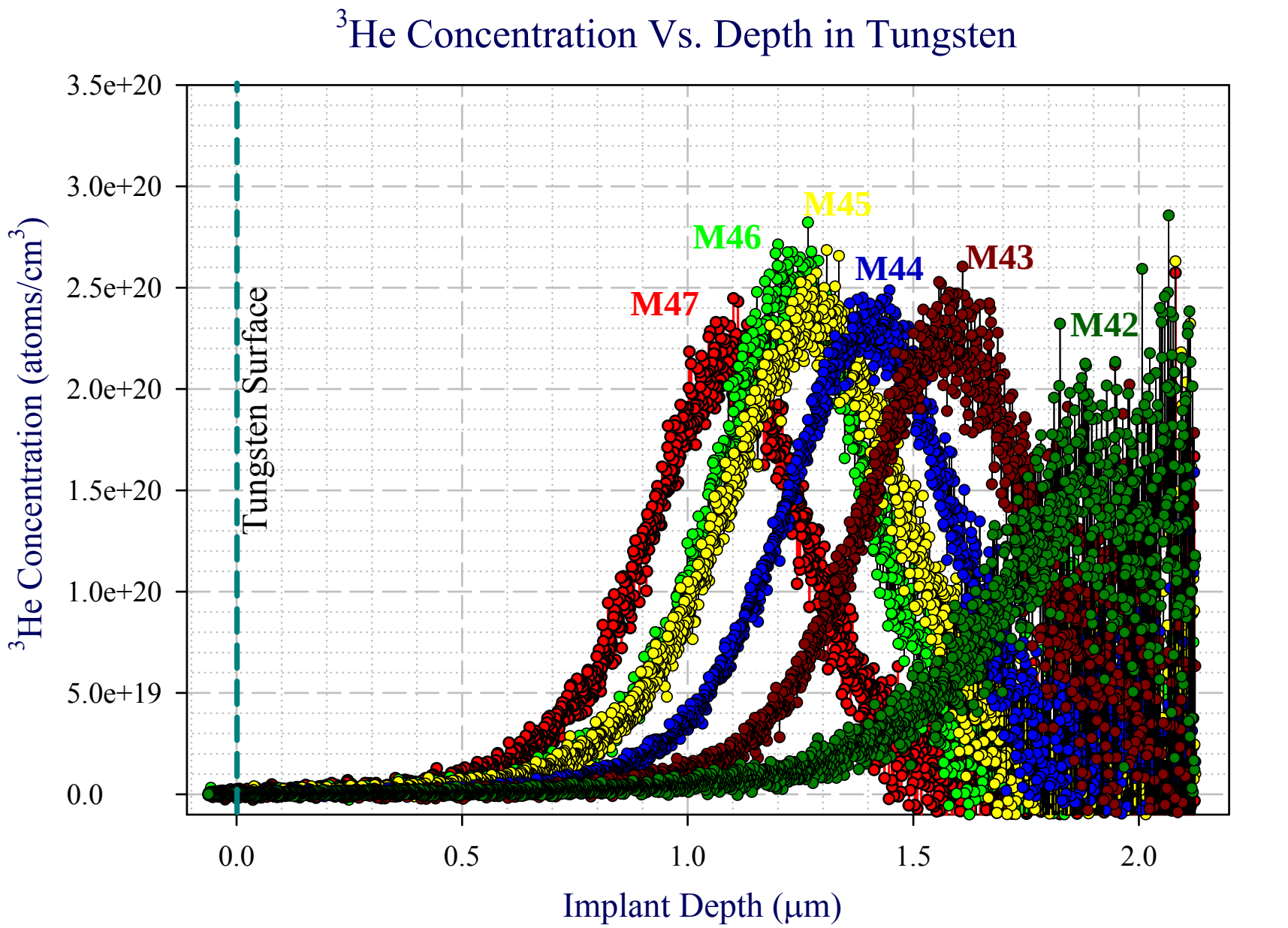


- **Bulk of helium spectrum 100 – 500 keV**

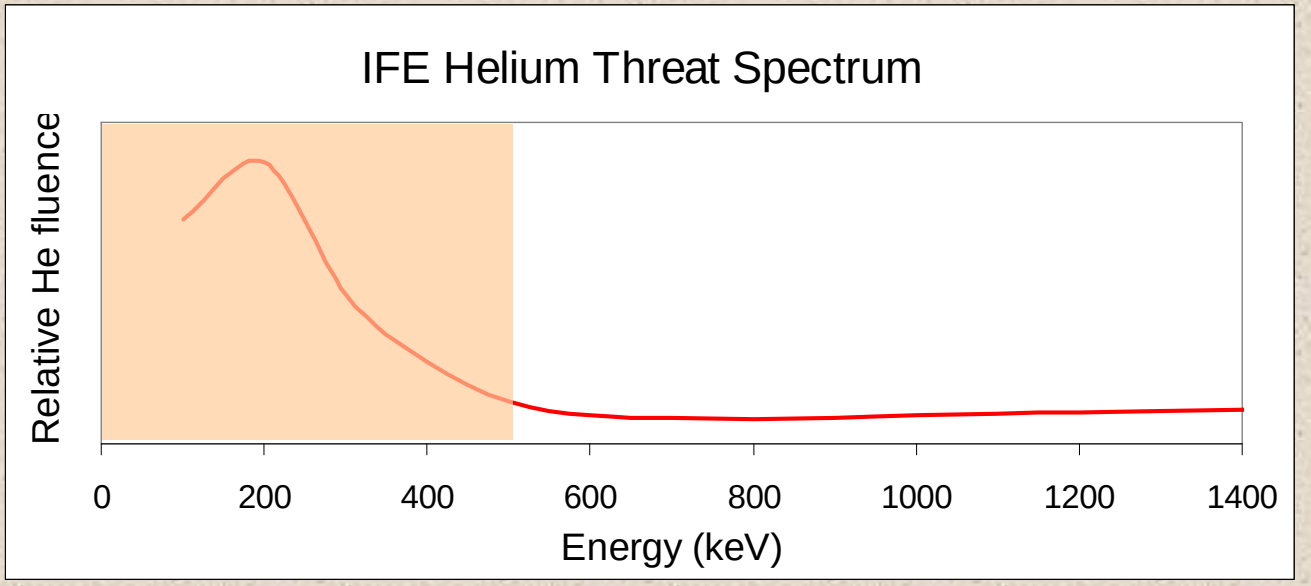
He Irradiation Results in Blistering



- SEM of polycrystalline W
- 1.3 MeV ^{3}He implanted to 2 x 10^{21} He/m^{2} at 800°C
- Flash annealed at 2000°C


^{3}He Concentration Vs. Depth in Tungsten
^{3}He Concentration (atoms/cm^3)
3.5e+20
3.0e+20
2.5e+20
2.0e+20
1.5e+20
1.0e+20
5.0e+19
0.0
Tungsten Surface
M47
M46
M45
M44
M43
M42
0.0
0.5
1.0
1.5
2.0
Implant Depth (μm)

Threat Spectrum Implantation Conditions

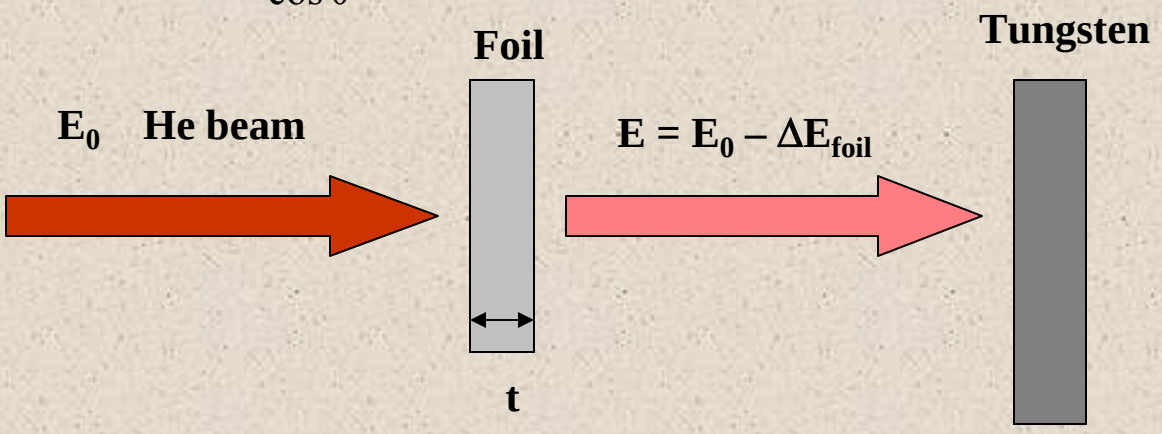


- **Implantation at 850°C with flash heating to 2000°C between implant steps**
- **Total helium dose implant divided by the no. of steps as a threat profile**
  Sample heating 850°C → 2000°C → 850°C per step

Helium Threat Spectrum

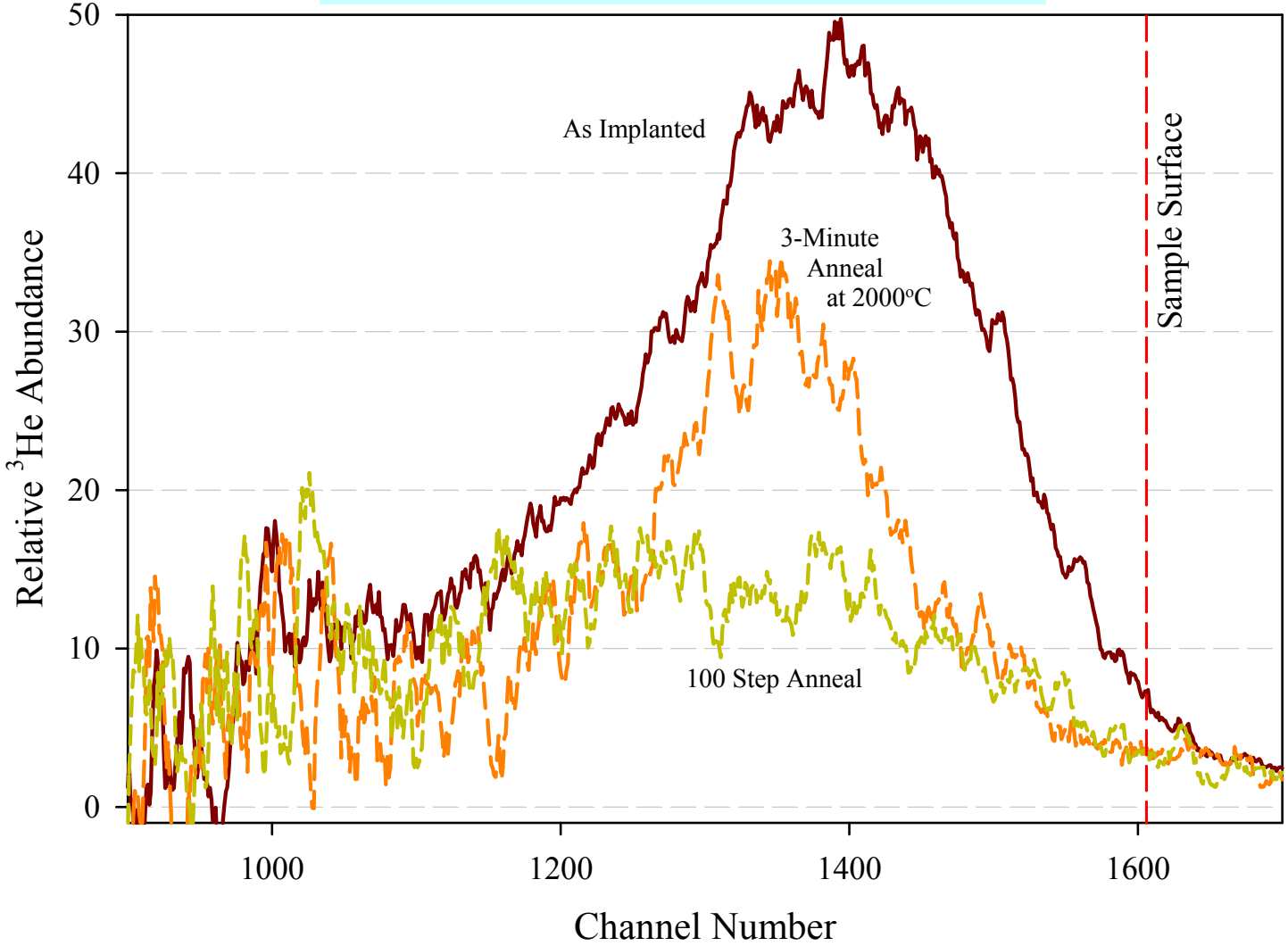
- **Degrade the monoenergetic beam by transmission through a thin Al foil**
- **Tilting the foil provides a range of degraded energies by varying the path length d through the foil**

$$d = \frac{t}{\cos\theta}$$ **where θ = 0° is normal incidence**

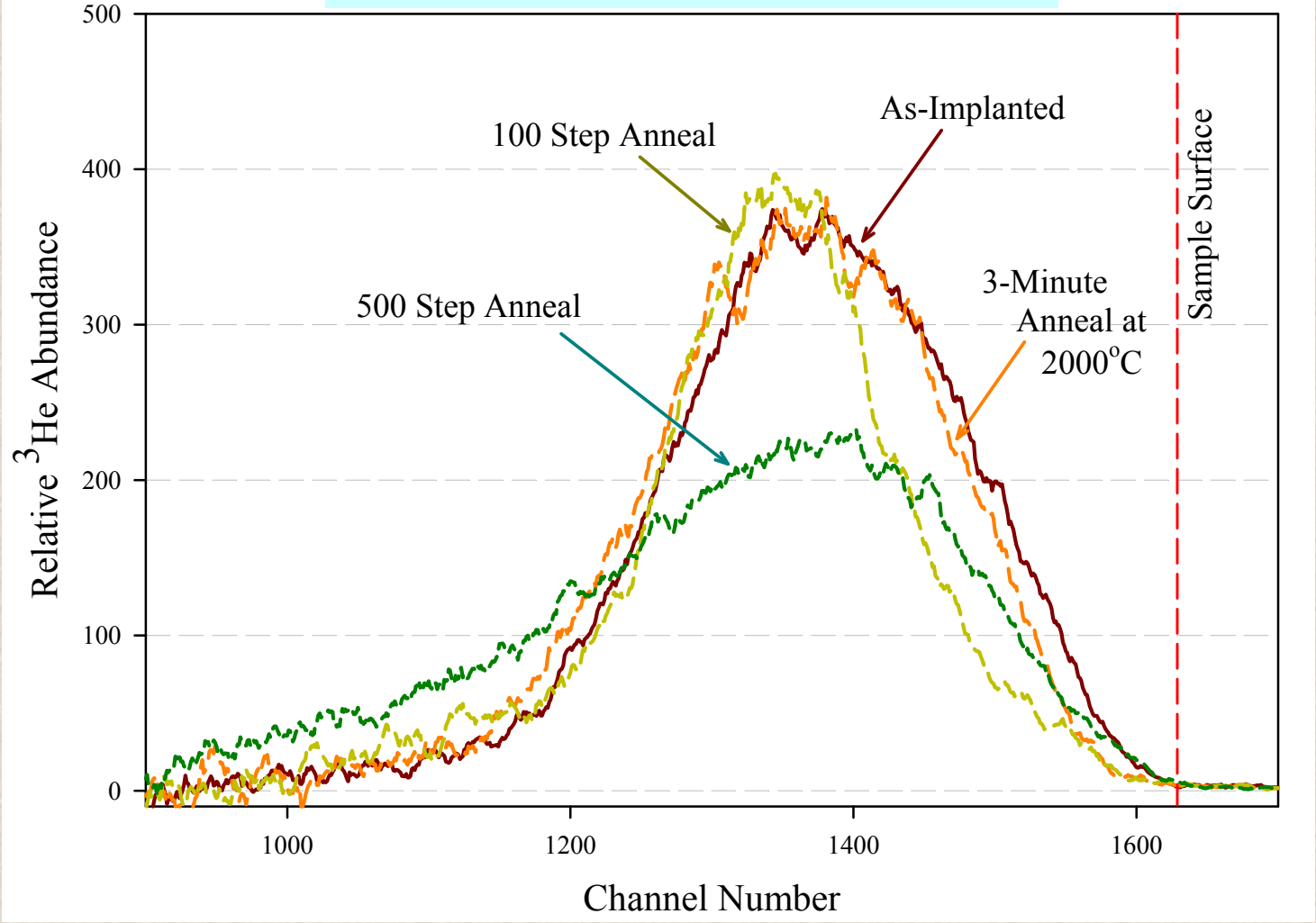


- **Al stopping power: ~330 keV/micron**
- **900 keV ^{3}He beam through a 1.5 micron Al foil tilted 0 – 60°**
- **Degraded energies: 100 – 500 keV**

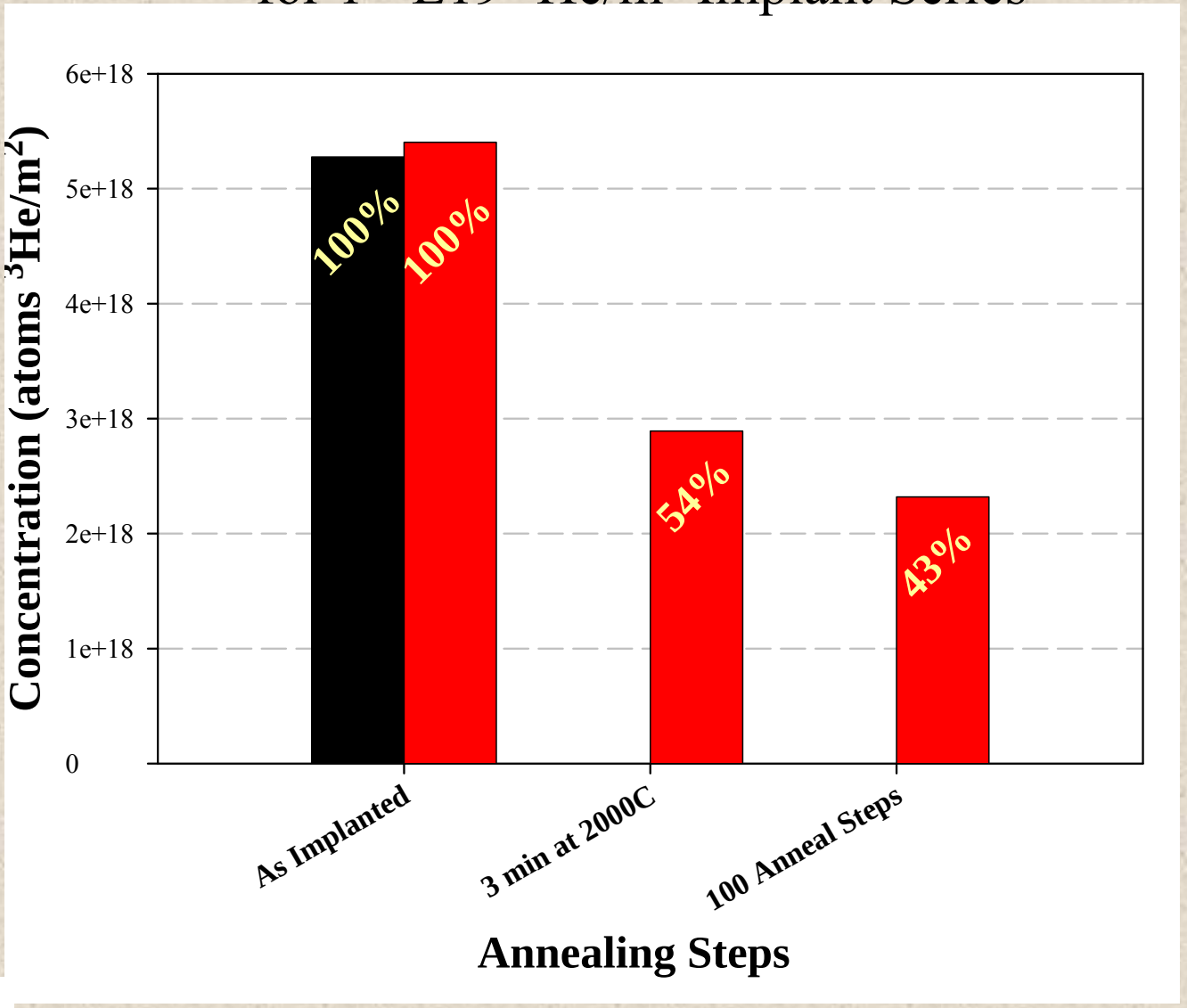
Implant – Anneal Sequence
1 +E19 $^3He/m^2$ Implant Series



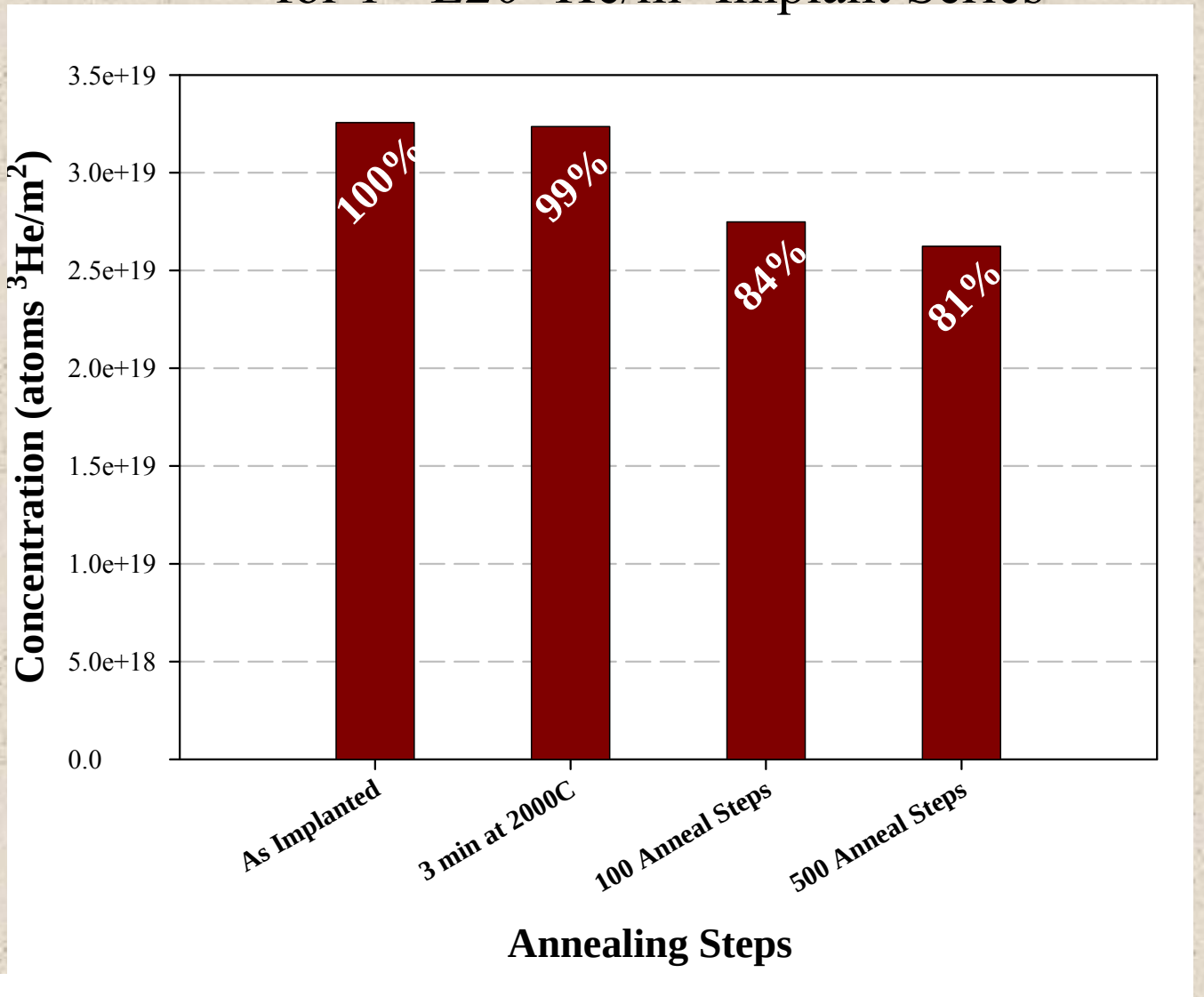
Implant – Anneal Sequence
1 +E20 $^3He/m^2$ Implant Series



Implant – Anneal Sequence for 1 +E19 $^3He/m^2$ Implant Series

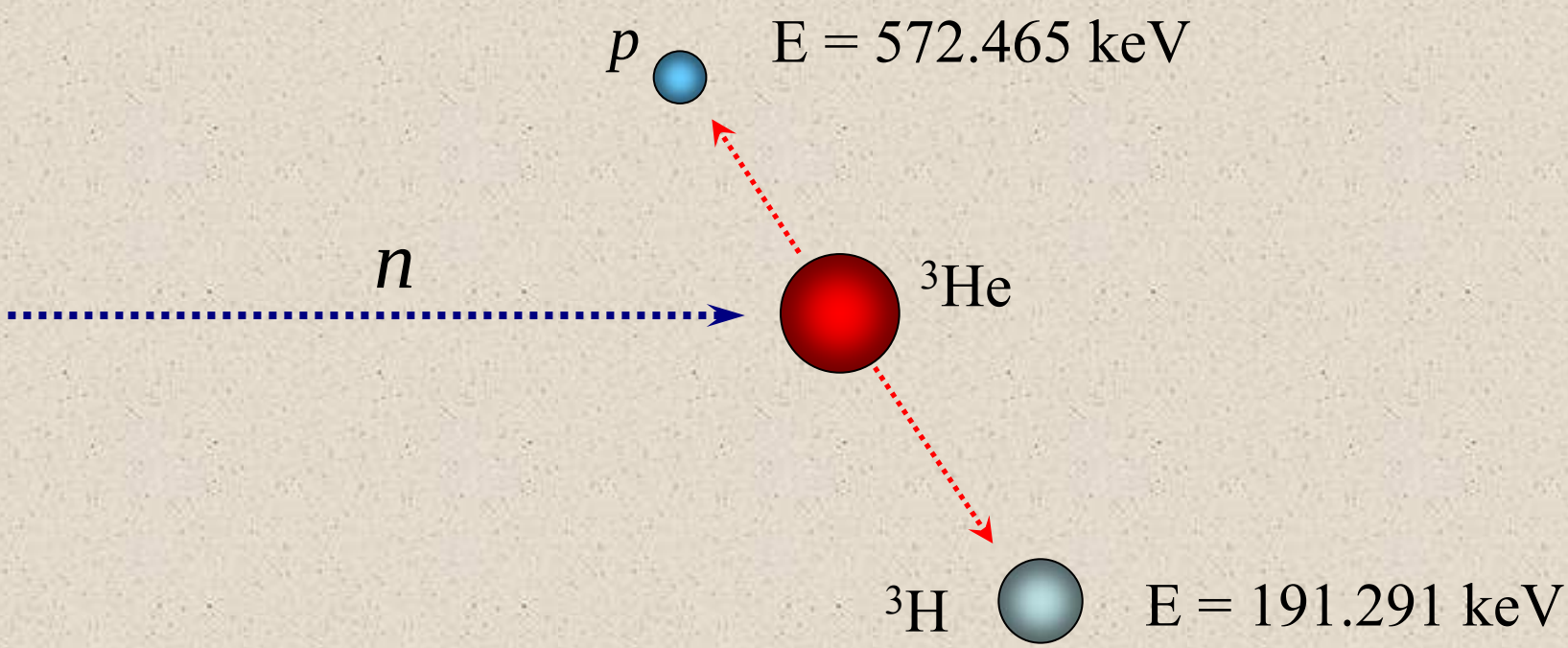


Implant – Anneal Sequence for 1 +E20 $^3He/m^2$ Implant Series



NDP: Physical Principle

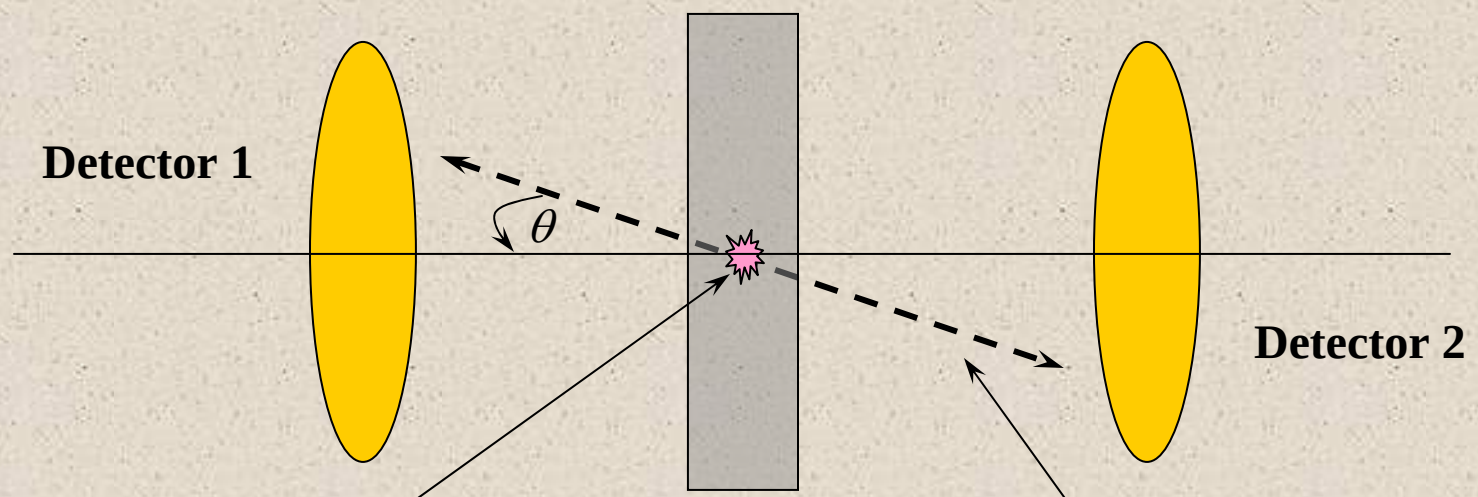
$^{3}He(n,p)^{3}H$



Particle emission is always diametrical

Improved Depth Resolution *and* Detection Efficiency Using Coincidence NDP

For ***THIN*** samples -- ***both*** reaction particles can escape the sample:

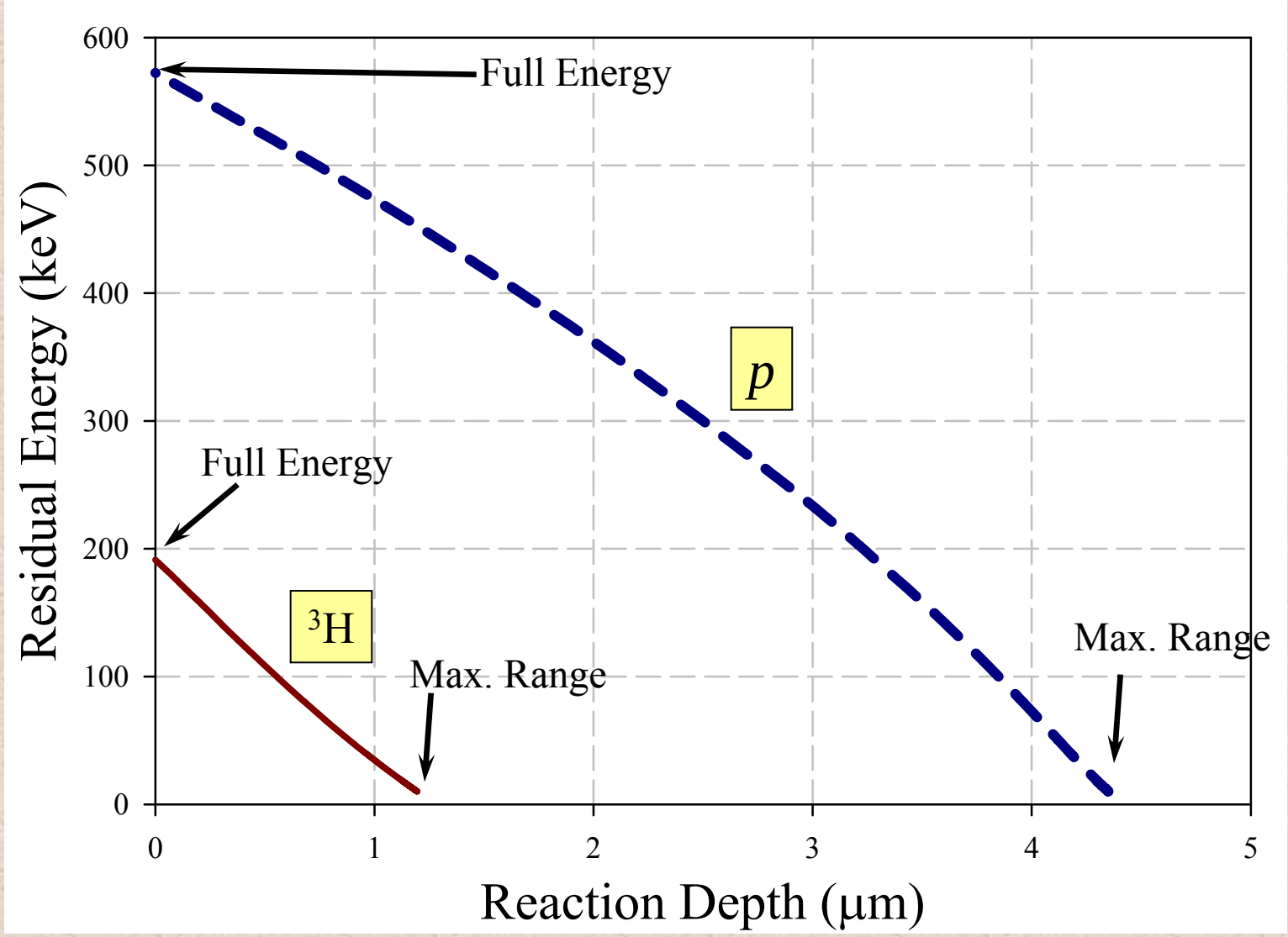


We know: $\varepsilon_{1\text{-initial}}$ & $\varepsilon_{2\text{-initial}}$

We measure: $\varepsilon_{1\text{-final}}$ & $\varepsilon_{2\text{-final}}$

Using a Jacobian Transformation, we calculate: $\varepsilon_{1\text{-better}}$, $\varepsilon_{2\text{-better}}$, & θ

Particle Ranges for $^3He(n,p)^3H$ in ErT_2



Concepts to Take Away:

1. Neutron Depth Profiling (NDP) is a quantitative, nondestructive technique for determining ^{3}He profiles.
2. ErT_2 Profiles can be determined to depths of about 4 μm with resolutions of nanometers.
3. For thin films, NDP can be developed to profile ^{3}He with resolutions of Angstroms using co-incidence detection methods.