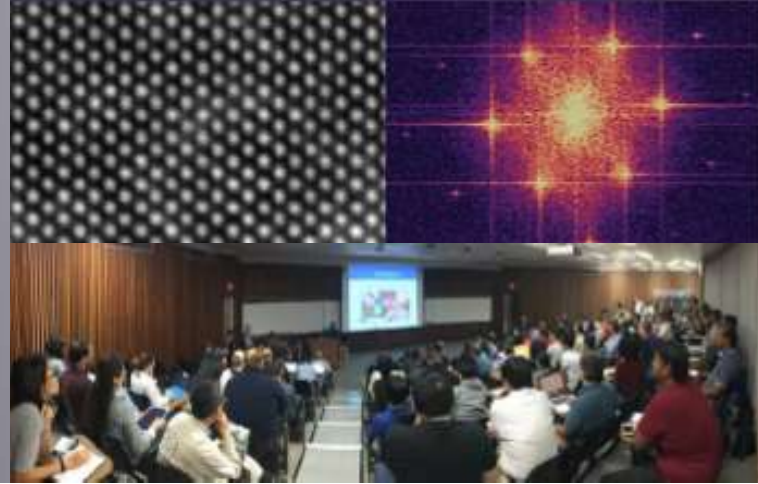


June 4 - 5, 2019

AMC

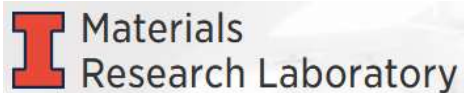
Advanced Materials
Characterization Workshop



Rutherford Backscattering Spectrometry

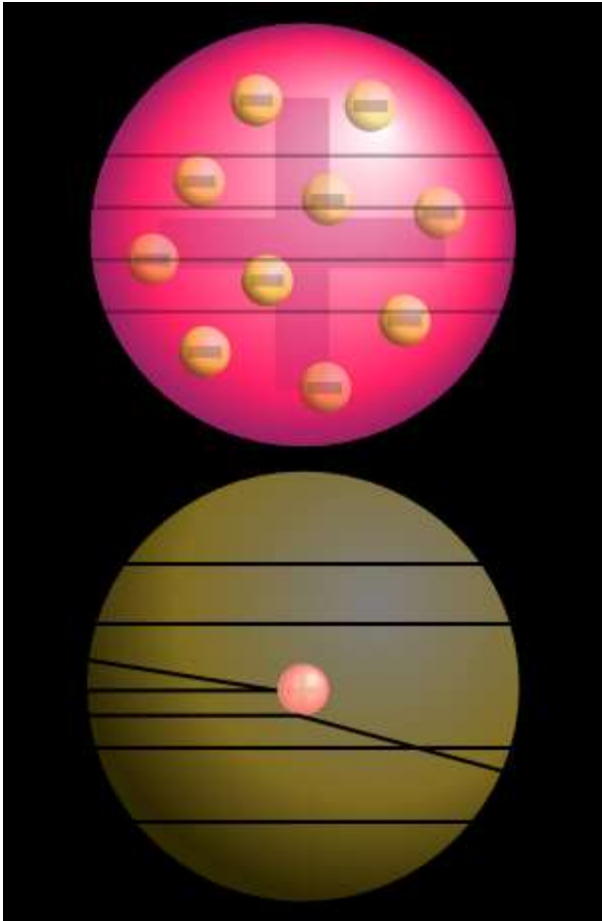
Timothy P. Spila, Ph.D.

Materials Research Laboratory
University of Illinois



amc.mrl.illinois.edu



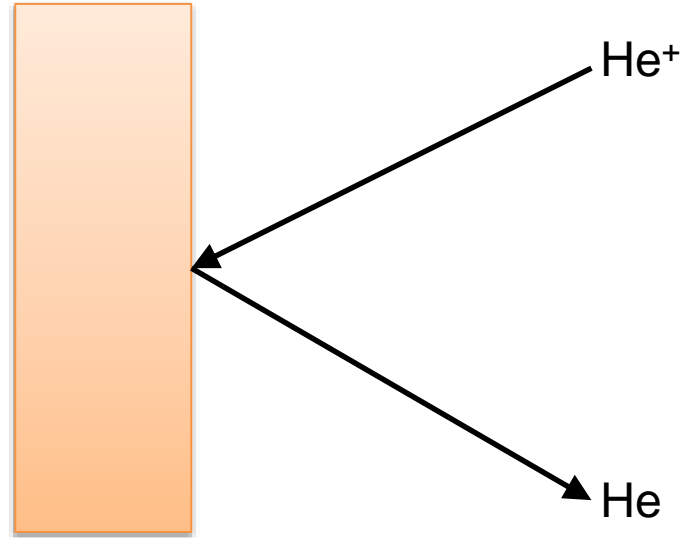


Top: Expected results:
alpha particles passing
through the plum pudding
model of the atom undisturbed.

Bottom: Observed results:
a small portion of the
particles were deflected,
indicating a small,
concentrated positive charge.



Rutherford Backscattering Spectrometry



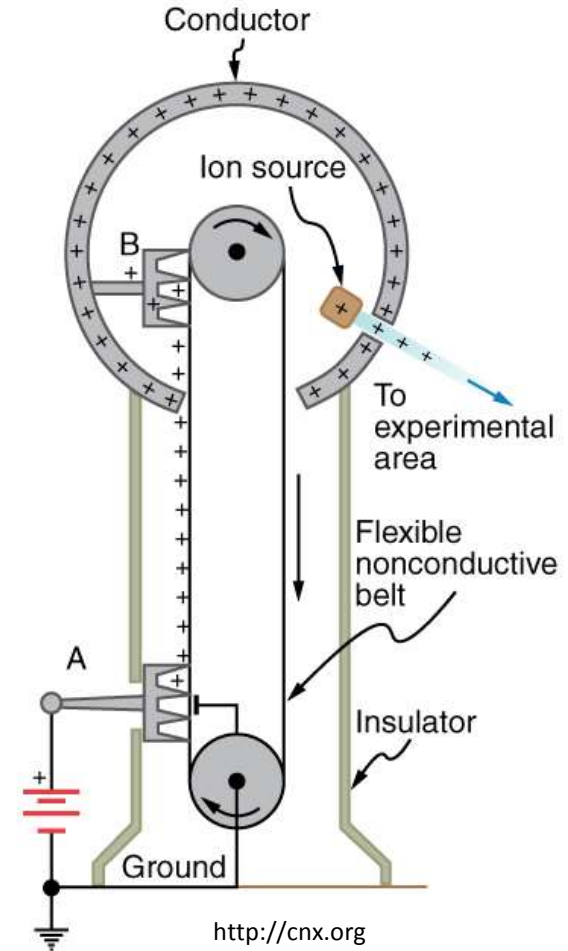
RBS is an analytical technique where high energy ions (~2 MeV) are scattered from atomic nuclei in a sample. The energy of the back-scattered ions can be measured to give information on sample composition as a function of depth.



Van de Graaff accelerator



http://archive.thedailystar.net/newDesign/print_news.php?nid=73473



3 MeV Van de Graaff accelerator



beam size ϕ 1-3 mm
flat sample
can be rotated

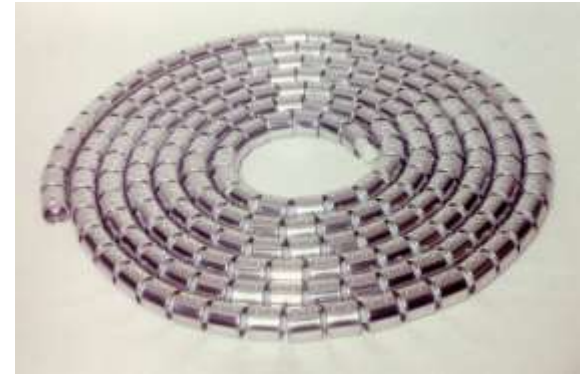


Rutherford Backscattering Spectrometry

3 MeV Pelletron accelerator



beam size ϕ 1-3 mm
flat sample
can be rotated



Pelletron system consists of

- **Ionization chamber**
- Acceleration tube
- Focusing quadrupole
- Steering magnet
- RBS end station



Pelletron system consists of

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Pelletron system consists of

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Pelletron system consists of

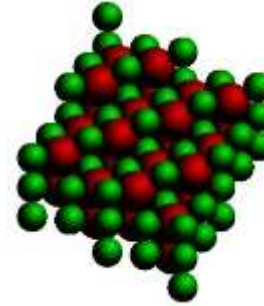
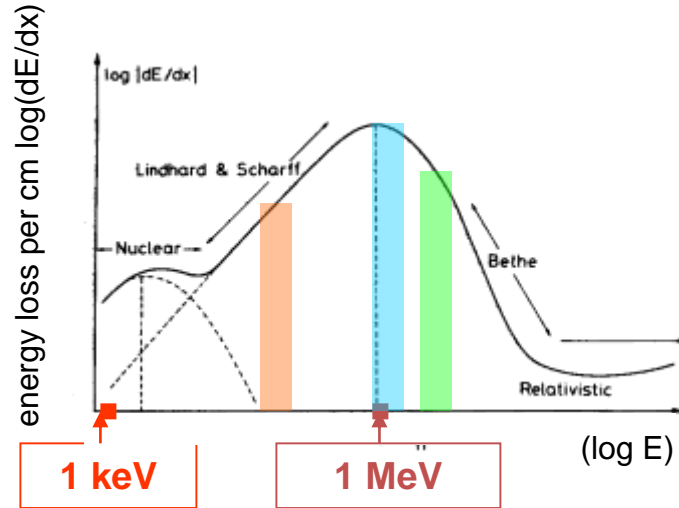
- Ionization chamber
- Acceleration tube
- **Focusing quadrupole**
- **Steering magnet**
- RBS end station



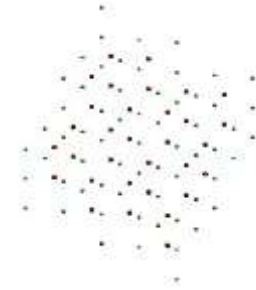
Pelletron system consists of

- Ionization chamber
- Acceleration tube
- Focusing quadrupole
- Steering magnet
- **RBS end station**





1 keV



1 MeV

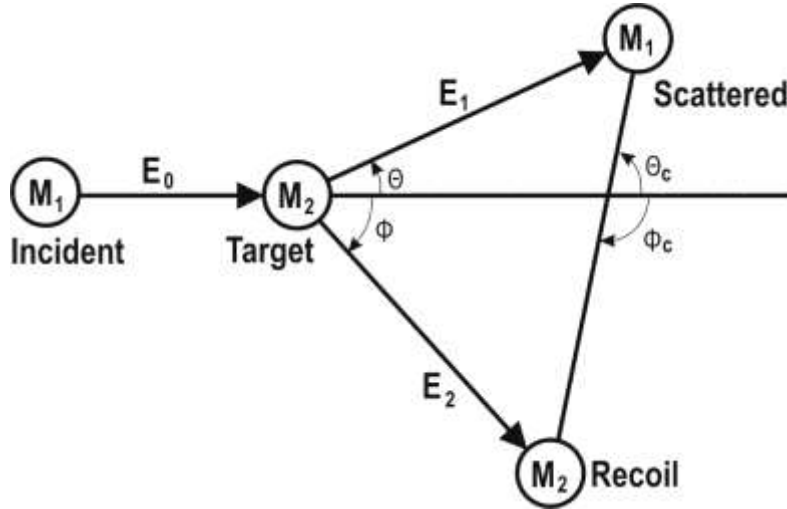
thin film projected on to a plane: **atoms/cm²**

$$(Nt)[\text{at}/\text{cm}^2] = N[\text{at}/\text{cm}^3] * t[\text{cm}]$$

Figure after W.-K. Chu, J. W. Mayer, and M.-A. Nicolet, *Backscattering Spectrometry* (Academic Press, New York, 1978).



Elastic Two-Body Collision



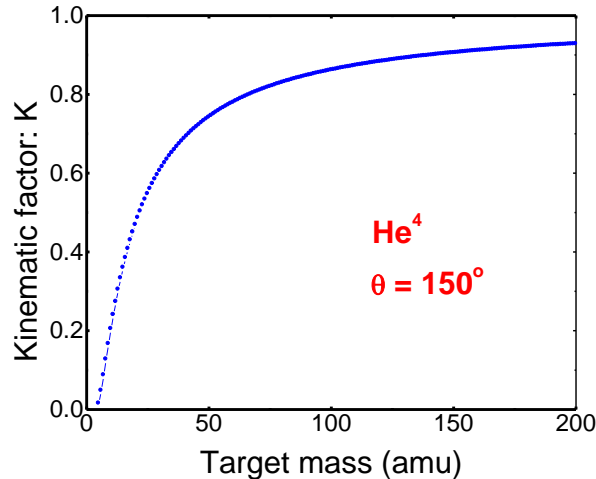
Elastic Scattering

$$M_1 v_0^2 = M_1 v_1^2 + M_2 v_2^2$$

$$M_1 \vec{v}_0 = M_1 \vec{v}_1 + M_2 \vec{v}_2$$

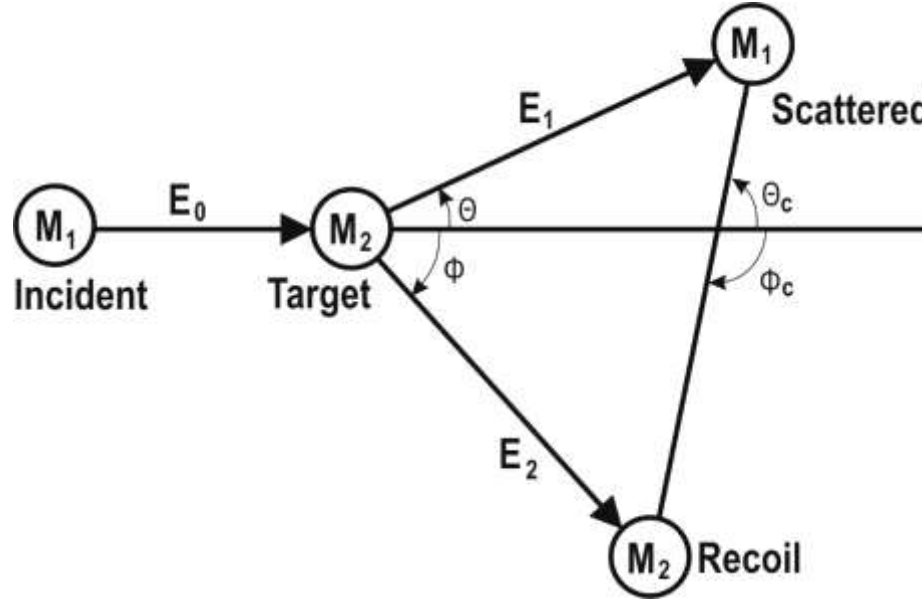
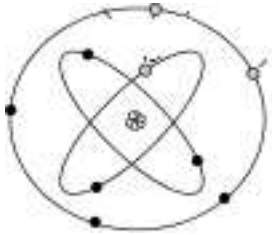
$$E_1 = KE_0$$

$$K = \left(\frac{\sqrt{M_i^2 - M_i^2 \sin^2 \theta} + M_i \cos \theta}{M_i + M_t} \right)^2$$



$M_1 < M_2, 0 \leq \theta \leq 180^\circ$
 $0 \leq \phi \leq 90^\circ$

RBS: He backscatters
 from $M_2 > 4$



Coulomb interaction between the nuclei:
 exact expression -> quantitative method

$$\sigma_R(E, \theta) \propto \left(\frac{Z_1 Z_2}{4E} \right)^2 \left[\sin^{-4} \left(\frac{\theta}{2} \right) - 2 \left(\frac{M_1}{M_2} \right) \right] \propto \left(\frac{Z_2}{E} \right)^2$$

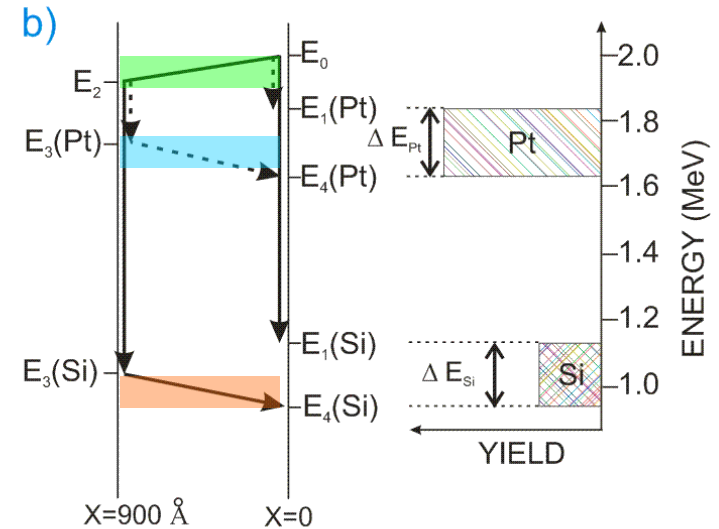
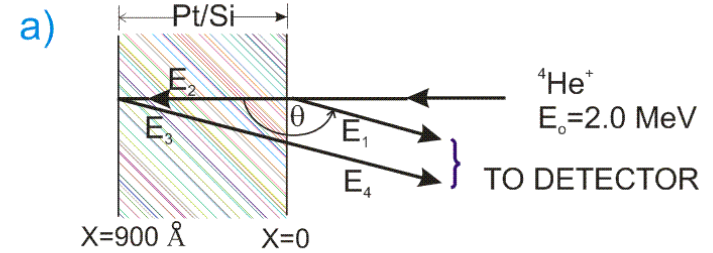
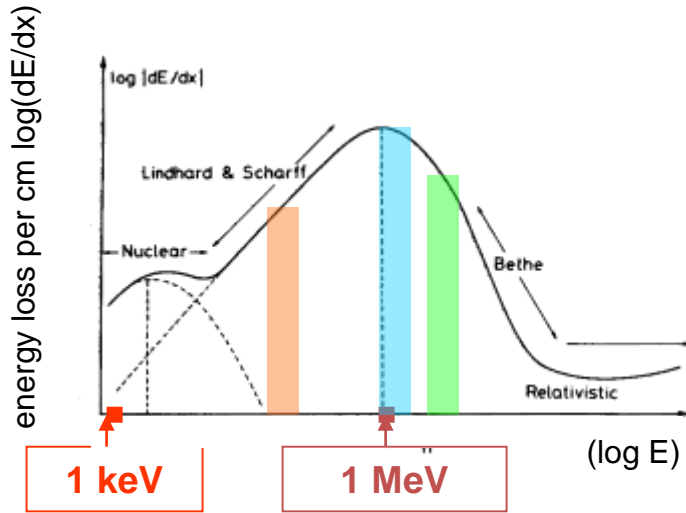


Figure after W.-K. Chu, J. W. Mayer, and M.-A. Nicolet, *Backscattering Spectrometry* (Academic Press, New York, 1978).

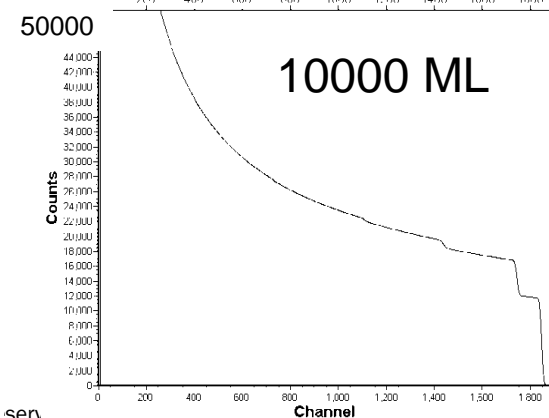
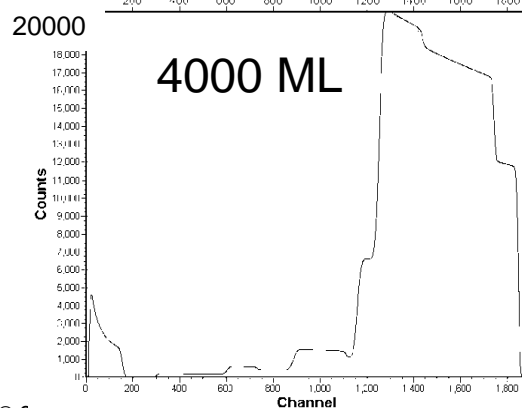
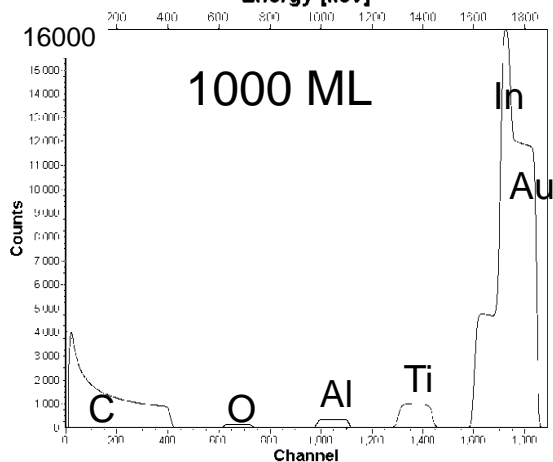
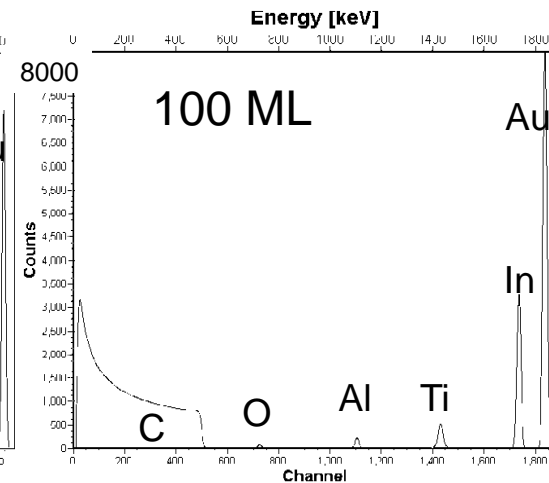
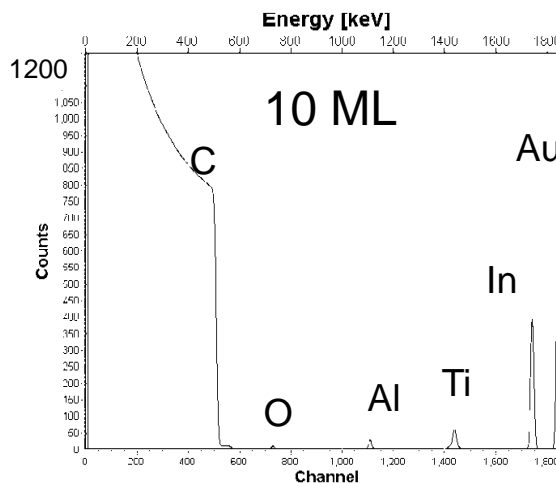
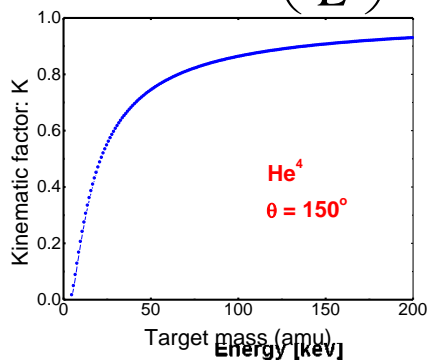


RBS – Simulated Spectra

hypothetical alloy $\text{Au}_{0.2}\text{In}_{0.2}\text{Ti}_{0.2}\text{Al}_{0.2}\text{O}_{0.2}/\text{C}$

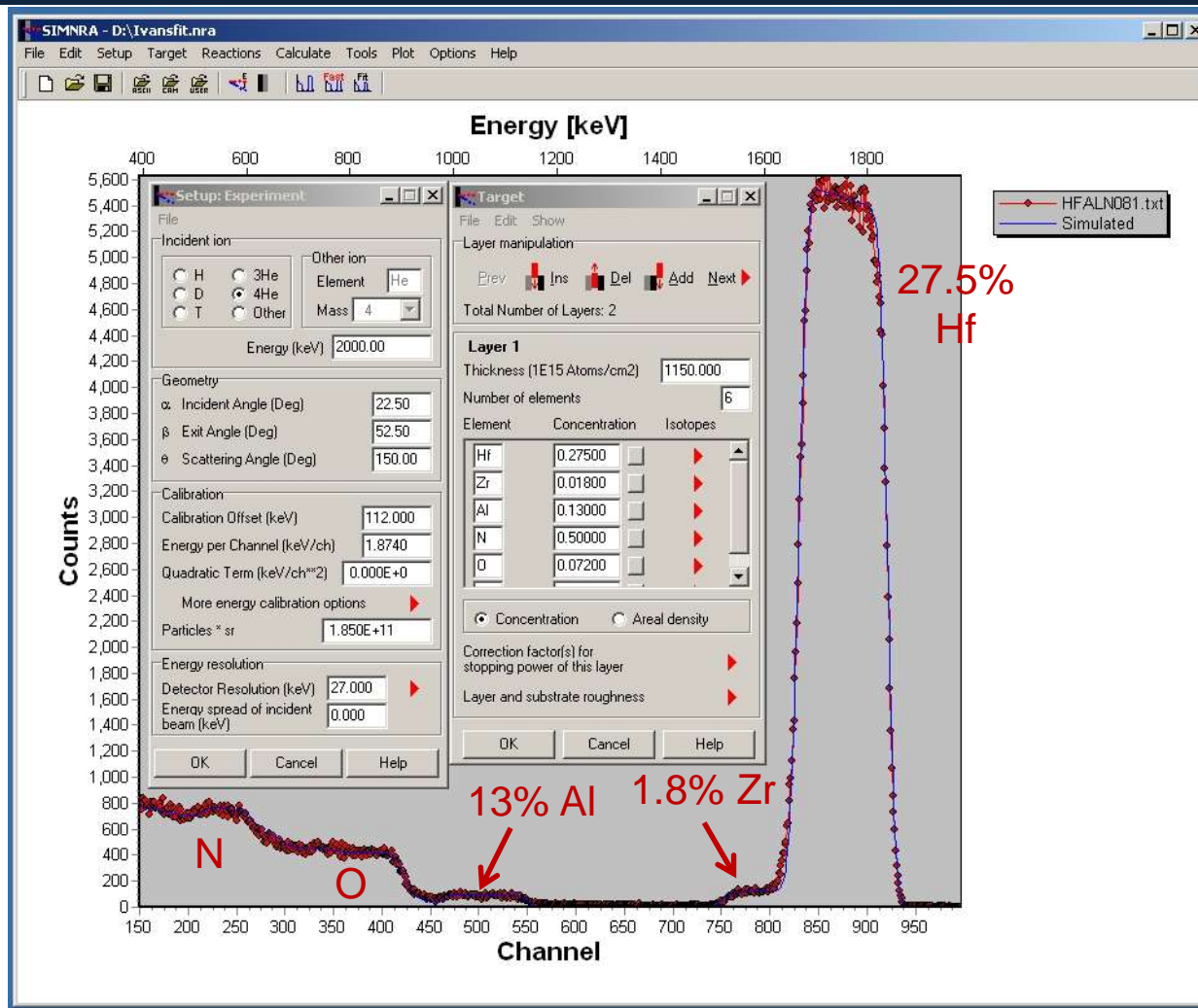
Element (Z,M): O(8,16), Al(13,27), Ti(22,48), In(49,115), Au(79,197)

$$\sigma_R(E, \theta) \propto \left(\frac{Z_2}{E} \right)^2$$



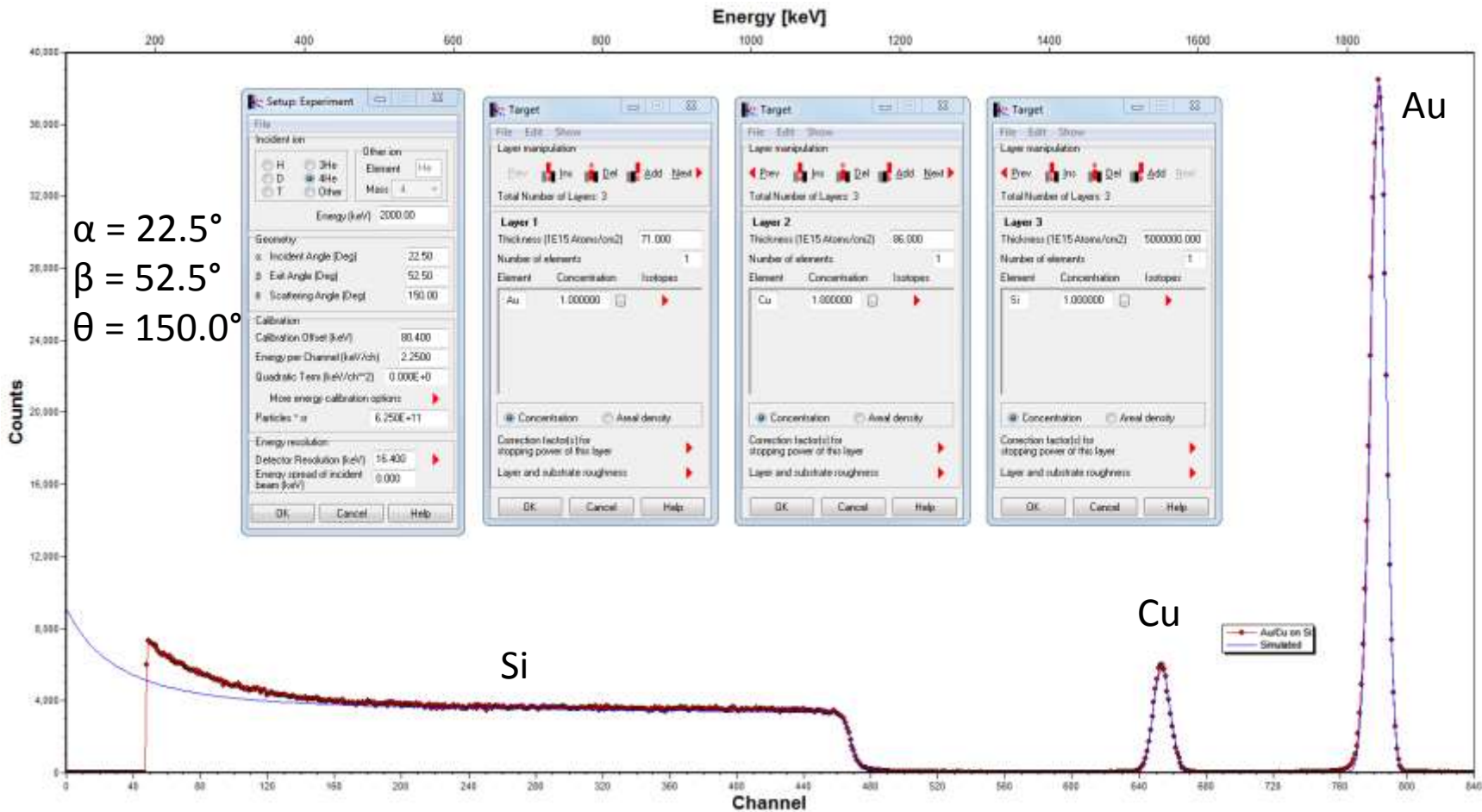


SIMNRA Simulation Program for RBS and ERD



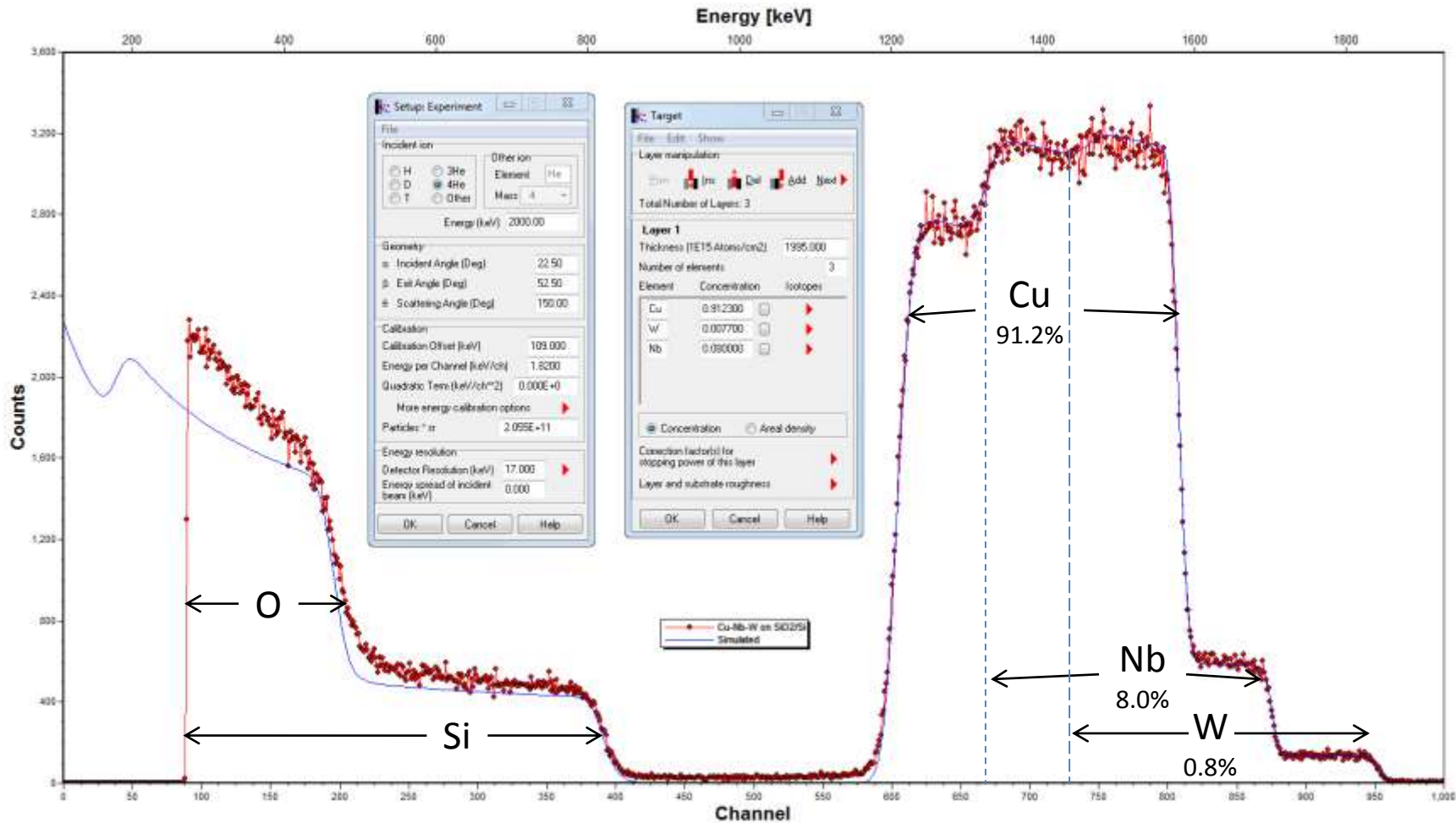


Calibration Sample





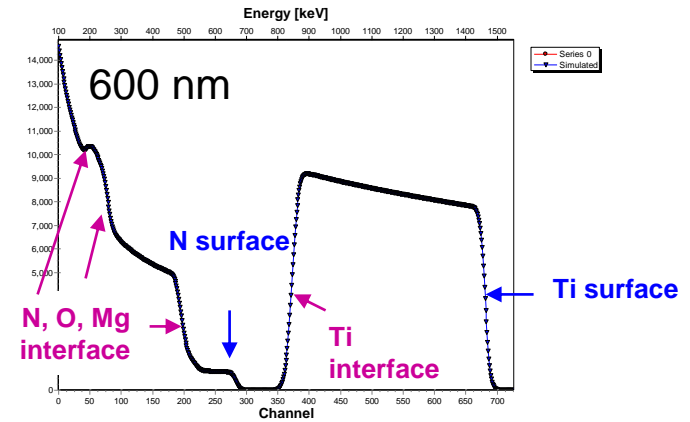
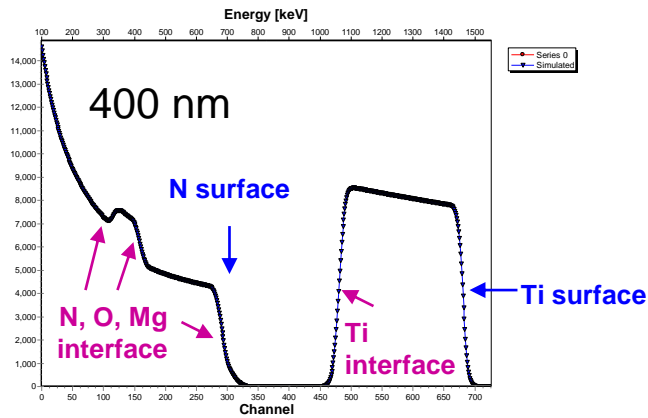
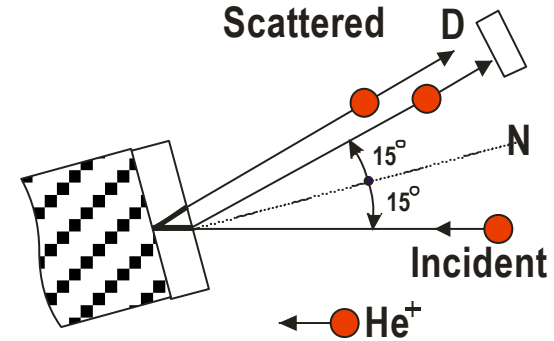
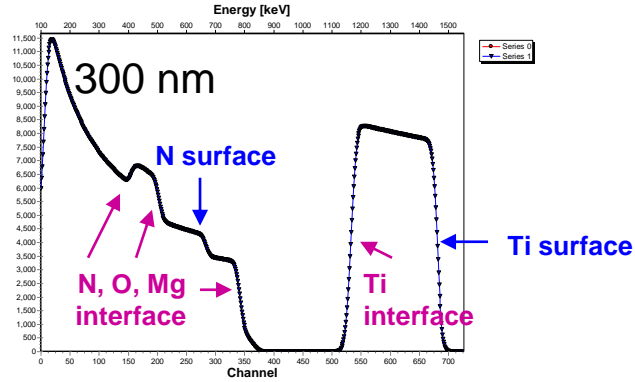
Cu-Nb-W Alloy on SiO₂/Si





Thickness Effects

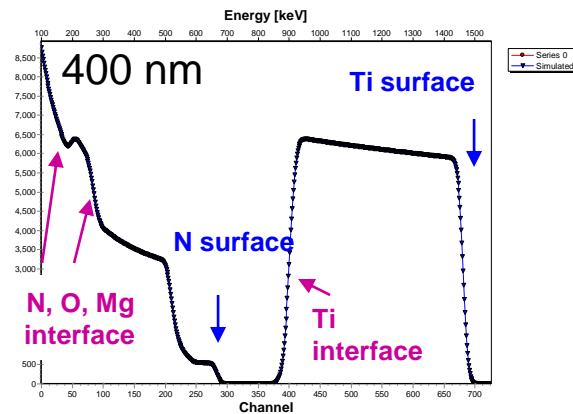
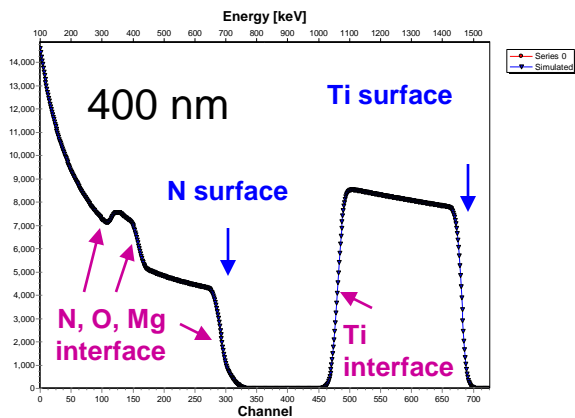
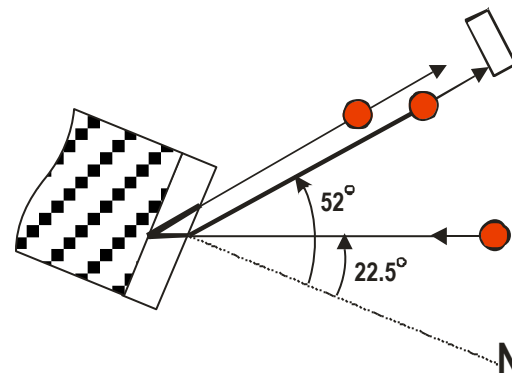
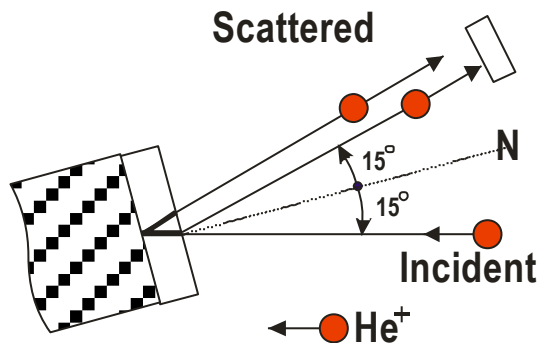
TiN/MgO





Incident Angle Effects

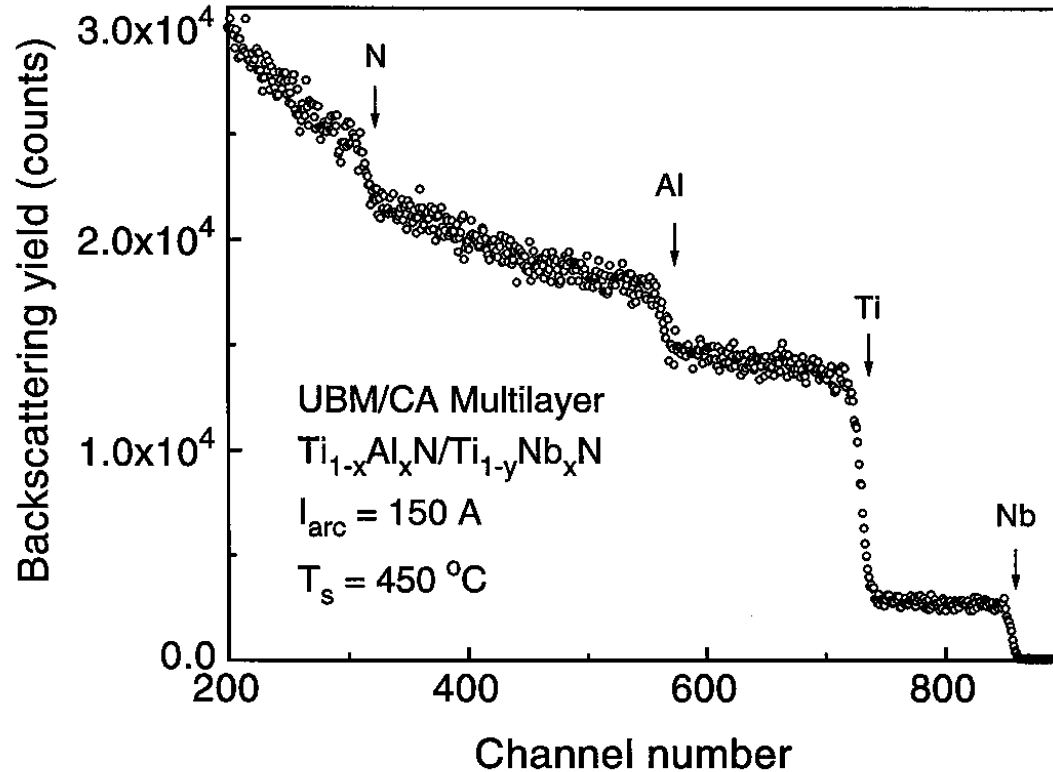
TiN/MgO



Surface peaks do not change position with incident angle



Example: Average Composition



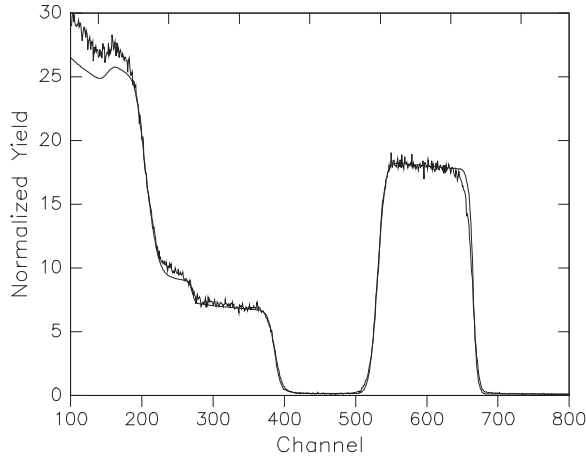
I. Petrov, P. Losbichler, J. E. Greene, W.-D. Münz, T. Hurkmans, and T. Trinh, *Thin Solid Films*, 302 179 (1997)



RBS: Oxidation Behavior

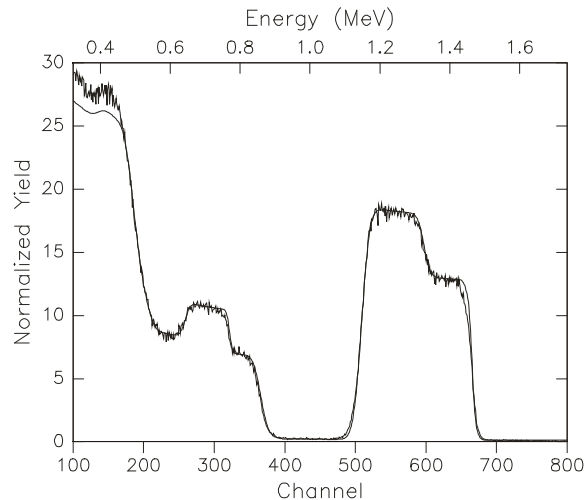
TiN/SiO₂

As-deposited



Experimental spectra and simulated spectra by RUMP

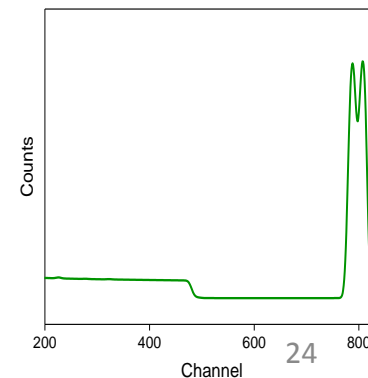
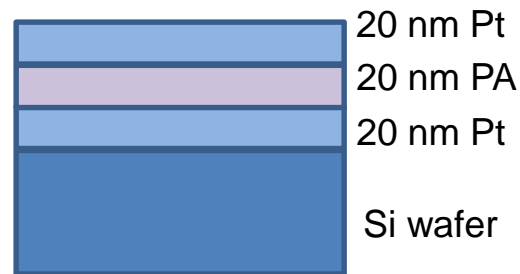
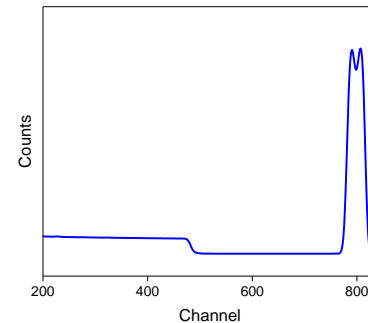
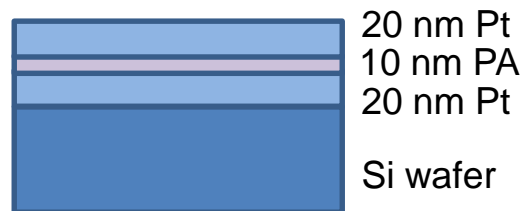
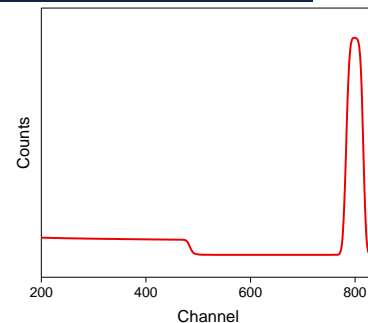
Annealed in atmosphere for 12 min at $T_a = 600$ °C





Areal mass density by RBS

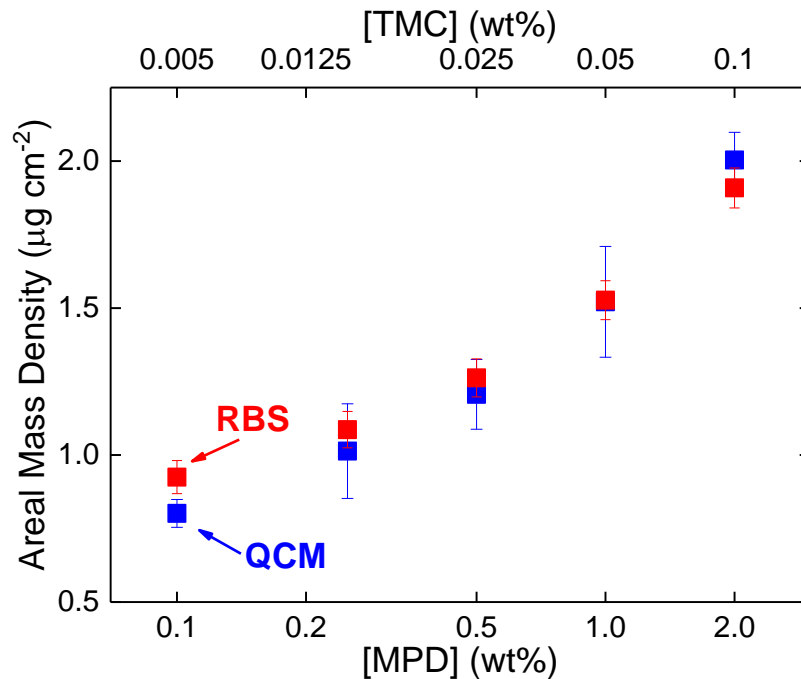
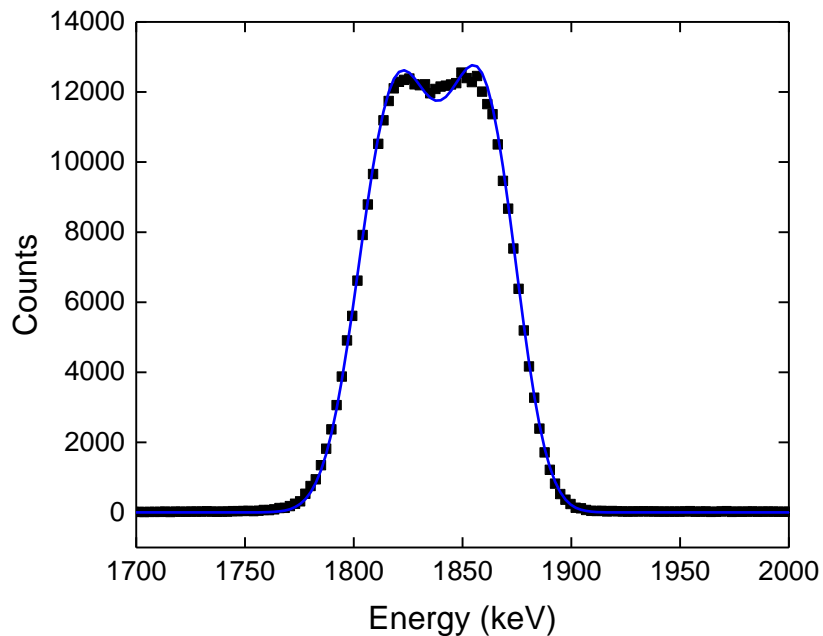
- Free-standing polyamide films are too thin to give sufficient signal in the RBS.
- Use the added stopping power of the polymer to split the Pt peak in the RBS spectrum.

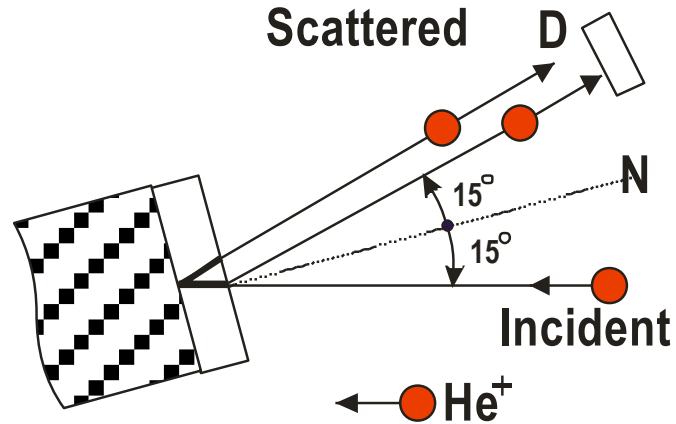




Areal mass density by RBS

0.005 wt% TMC, 0.1 wt % MPD





- Quantitative technique for elemental composition
- Requires flat samples; beam size $\Phi 1\text{-}3$ mm
- Non-destructive
- Detection limit varies from 0.1 to 10^{-6} , depending on Z
 - optimum for heavy elements in/on light matrix, e.g. Ta/Si, Au/C...
- Depth information from monolayers to $1\ \mu\text{m}$



Optimizing Simultaneous PIXE and RBS Capabilities

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Pollock@pelletron.com

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Robert.White@nrel.gov



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