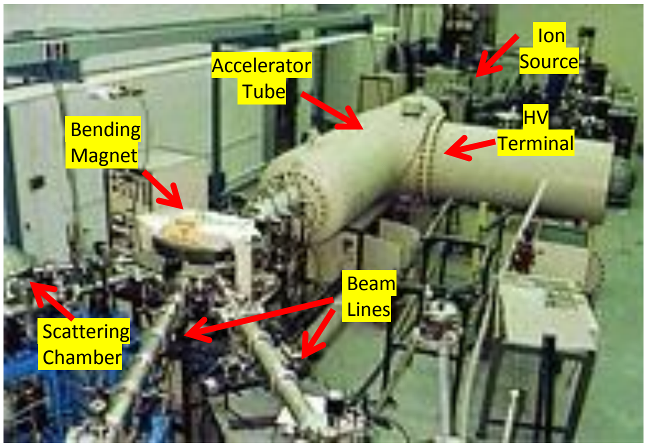
P627/Chem 542 Surface and Interface Science Spring 2013

Rutherford Backscattering Spectrometry (RBS) Lab Session

Purpose: In lecture, we discussed the use of Rutherford backscattering spectrometry (RBS) to perform quantitative elemental depth profiling at surfaces and for thin films. In this lab we will use the 1.7-MV tandetron accelerator in the Ion Scattering Facility of the Laboratory for Surface Modification to acquire RBS spectra from several samples, and use these spectra to determine elemental composition and film thickness. In addition, you will be given several previously–acquired spectra for similar analysis using commercially available software known as SIMNRA. The objective is for you to become familiar with the components of the ion beam system, experience the procedures needed to produce a beam, steer it onto a sample and acquire a spectrum, and ultimately to analyze the spectrum to obtain useful information regarding the composition and depth distribution of elements in the near surface region of the sample.

Introduction: Rutherford backscattering spectroscopy uses a well collimated, monoenergetic beam of ions (typically α-particles or protons) that is directed onto the sample to be analyzed. The typical energy of the beam is between 1 MeV and 2 MeV. The incident ions scatter from the nucleus of the atoms in the sample (called the target nucleus) and are backscattered owing to the coulomb repulsion with the positively charged target nucleus. Owing to the kinematics of the collision, the backscattered ion has less energy than it had prior to the collision. The precise change in energy (Δ) depends upon the masses of the incident ion (M_1) and target nucleus (M_2) as well as the scattering angle (Θ), which is the angle between the velocity vector of the incident ion and that of the scattered ion. In most cases, the scattering angle is close to 180°, so the detector is placed near the incident beam. In addition to energy lost upon scattering, the incident (and backscattered) ions lose energy to electrons as they pass through the film (both on the way in, and on the way out). This latter process is what give the technique its depth sensitivity. By monitoring the number of backscattered ions as a function of energy, the elemental composition and depth distribution of elements can be determined.

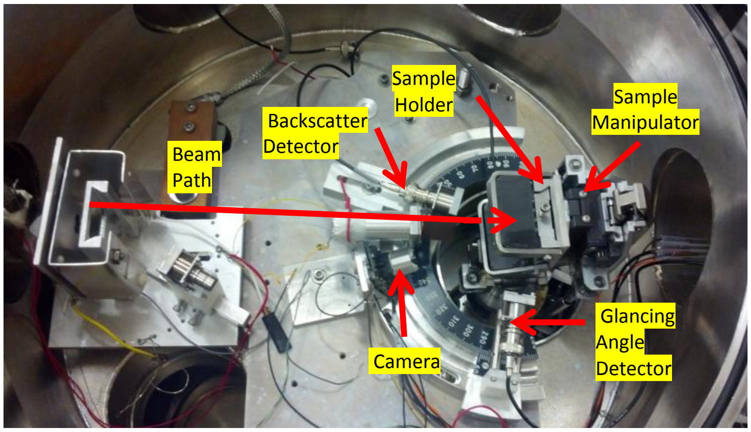


1.7 Mev Tandetron Ion Acceleration Facility at the Laboratory for Surface Modification

Outline of the experiment:

During the lab session, you will see three samples mounted on the standard sample holder. The sample holder will be place on a goniometer that allows the sample to be positioned in the beam. During this step, Dr. Wielunski will point out different components of the apparatus that are in the experimental chamber. Once the samples are loaded, the chamber will be closed and pumped out to a pressure below 10^{-6} torr (no, this is not an ultrahigh vacuum technique). While the chamber is pumping, different parts or the ion source, acceleration system, beam steering system and electronics will be described. You will take part in the acquisition of RBS spectra from three samples, two that are SnS films, and a third that is a film with a more complicated composition. Once the data is acquired, Dr. Wielunski will demonstrate the SIMNRA software used to analyze the spectrum of one of the SnS films. You will then be given the data electronically so that you can, as part of the lab report, can perform the same analysis for all three spectra and draw some specific conclusions regarding the nature of the films.

Your lab report will include the answers to the questions below as well as plots of your raw data, reduced data, your analysis and results, as well as a discussion of how the results compare to an idealized version of the sample, and what may have caused any deviations from ideal conditions.



Overhead view inside scattering chamber

The Scattering Chamber: A view inside the scattering chamber is shown at the left, with the key components labeled. The top of the sample holder is the wedge shaped piece of metal mounted in the manipulator. The manipulator is a goniometer with translational and rotational degrees of freedom. The samples are mounted on a metal plate, held vertical in the manipulator, between the two dark shields. The manipulator can be translated along three axes to align the sample. The goniometer rotates the sample holder about the axis of the beam. Two silicon detectors are shown. The Backscattering detector is mounted at a scattering angle of about 165 deg. The Glancing Angle Detector is mounted at a scattering angle of about 100 deg.

Intro questions.

1) How is a tandem ion accelerator different from a so-called van der Graaf accelerator, and what are some of the advantages (and drawbacks) of the tandem design?

2) Suppose you have two samples comprised of a thin film of Au on a Si substrate. One sample has a carbon film on top of the gold, and the other does not. RBS spectra are taken from each sample under identical scattering conditions. How can I look and the two RBS spectra and, without doing any modeling, tell which sample has the carbon film on the Au?

3) Suppose I scatter 2 MeV protons from a thin Au film on top of Si, and measure the backscattered protons. In one case my detector is at a scattering angle of 180°, in the other it's at 125°. In which spectrum will the Au peak be at higher energy? Justify your answer.

4) Why is it easy to use RBS to measure the thickness of a gold film on a Si substrate, but hard to measure the thickness of a Si film on a gold substrate?

5) How can a tandem accelerator with a terminal voltage of 1 MV be used to create a beam of ions that have an energy of 3 MeV when they hit the target?.

6) Suppose you try to deflect a 1 MeV proton beam using a uniform magnetic field oriented perpendicular to the path of the beam. What must the strength of the field be (in Tesla) so

that you deflect the beam by 90° over a distance of 1 m? (Note: Similar fields are used in our apparatus, but only a deflection of about 30° is needed, so the bending magnet is NOT 1 m in length.)

Analyzing the data.
Download SIMNRA from the following website: http://home.rzg.mpg.de/~mam/

The data files are in an ASCII format with some appropriate header. SIMNRA has many tabs allowing the user considerable flexibility for analysis of different forms of ion-solid interactions (including nuclear reactions). We will confine our attention to RBS on a well-defined target of an Sn_xS_y film (with a small oxygen content) on a Si substrate (which has a thin SiN layer at its surface). The key operations are contained in the tabs TARGET, CALCULATE, TOOLS, and PLOT. In TARGET we will enter our best guess for the sample (e.g., film on a substrate, elemental composition, and description of target either in terms of atomic concentration or areal density). Using CALCULATE, we will create a simulated spectrum to match the experimental data and determine elemental composition, thickness, and (if relevant) depth profile. From TOOLS, we will use the INTEGRATE SPECTRUM option to find the total counts under the elemental peaks in both the experimental and simulated spectra and use this info to refine our description of the target. PLOT allows us to change in the way the data is displayed. We will of course also use FILE to load data and candidate simulations.

Your fist task will be to simulate an experimental RBS spectrum from one of the SnS film as described above, where you are given a first guess simulation. Load the data SnS_example into SIMNRA.

QUESTION: Identify which elements are associated with the peaks and onset near channel 850 in the simulated spectrum. What is the reason the guess got the right edge of the two peaks correct, but the left edge of each is wrong. What is the reason that the simulation shows a large onset at channel ~ 850, while the data has an onset near channel 750? Explain why this is consistent with the way in which it missed the description of the peaks.

Now, using the intuition you have from the qualitative way in which the simulation misses the data, change the assumptions for the SnS film in the target and try to get the best possible fit. The recommended steps is to first try to get the thickness of the SnS film correct, then adjust the composition, and then iterate to get a good fit. Perform the following steps (NOTE: DO NOT VARY THE OXYGEN CONTENT OF THE FILM, ONLY CHANGE Sn and S.):

a) Keeping the target window in the “Concentration” mode, adjust the thickness in an attempt to improve the fit.
b) After you choose a new thickness, go to the CALCULATE tab and select “calculate spectrum.”
c) Repeat this process until you believe you have the correct film thickness (I suggest using Sn peak for this purpose).
d) Now, use the “integrate spectrum” function from the TOOLS tab to compare the integrated counts in the right peak in the experimental and simulated spectra. Get the ratio.
e) Now, using this ratio, go to the target window in the “areal density” mode, update the areal density for Sn.
f) Recalculate the spectrum as see how it works.
g) Now, returning to the “concentration” mode, change the thickness to best fit the Sn peak.

h) Repeat the spectrum integration (now for both the Sn and S peaks) and repeat the process until you get a fit that agrees both in peak widths and integrated intensity.

This will be your determination of the film thickness and elemental concentration.
Note that SIMNRA gives the thickness of the film in units of 10^{15} atoms/cm^2. Given the density of SnS is 5.08 g/cm^3, and a mole of SnS is 150.78 g, calculate the molecular volume, and then, using Avogadro's number, the atomic density in Atoms/cm^3. From this you should be able to calculate the thickness of the film in Angstroms.

Now, having analyzed this sample spectrum, analyze the other two experimental spectra you acquired in the lab.

Note that the third film is comprised of La, Fe, and a third element that you need to identify. The film is on a thick MgO substrate.

Data-based Lab questions.

1) Use SIMNRA to analyze each spectrum measured in the Lab. What is the composition and thickness of each film? Include a printout of your SIMNRA fit along with your lab report.
2) One of your spectra is complicated by the fact that the film is sufficiently thick that backscattered intensity from heavier target nuclei overlap intensity backscattered from lighter ions. Use SIMNRA to estimate the maximum SnS film that would give an RBS spectrum where the signal from Sn and that from S *do not* overlap.
3) In the initial guess for the example spectrum, the simulation looks like two sharp peaks, rather than the experimental data, which looks more like two plateaus. Why is that? Simulate a SnS spectrum for a film with a smaller thickness than that of the original guess. Explain why the simulated spectrum changes the way it does.
4) Once you have determined the elemental composition of the third film, and identified the mystery element, simulate spectra using the elements to the right and to the left of the mystery element in the periodic table, and show how neither of these simulations gives a good fit to the data.
5) For the third film, explain why the area of each peak is approximately the same, even though the concentration of each element may be considerably different.