Surface analysis for students in Nuclear Engineering and Radiological Sciences

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Abstract. Students in Nuclear Engineering and Radiological Sciences at the University of Michigan are required to learn about the various applications of radiation. Because of the broad applicability of accelerators to surface analysis, one of these courses includes a laboratory session on surface analysis techniques such as Rutherford Backscattering Analysis (RBS) and Nuclear Reaction Analysis (NRA). In this laboratory session, the students determine the concentration of nitrogen atoms in various targets using RBS and NRA by way of the $^{14}N(d,\alpha)^{12}C$ reaction. The laboratory is conducted in a hands-on format in which the students conduct the experiment and take the data. This paper describes the approach to teaching the theory and experimental methods behind the techniques, the conduct of the experiment and the analysis of the data.

INTRODUCTION

The intimate connection between the fields of nuclear engineering and materials science was established since the inception of fission reactors. For example, the structure of steels used in the construction of reactors has been the subject of continuous studies in materials laboratories using various techniques [1]. Because of this connection, students at the Nuclear Engineering Department of the University of Michigan have been required to obtain familiarity with materials analysis techniques in one of the undergraduate courses provided by the Department .

An important tool in the field of materials science is surface analysis by non-destructive methods using accelerators. This paper describes a section of this course. The course required hands-on participation by the students in the analysis of a TiN sample using Nuclear Reaction Analysis (NRA).

DESCRIPTION OF THE COURSE

The course consists of the laboratory measurement of the stoichiometry of a TiN sample, preceded by a lecture on the theory of nuclear reaction analysis, an assignment of preliminary work, a study session to review the preliminary work and to tour the facility, and a completed laboratory report RBS and NRA techniques were presented first. The basic theory of nuclear reactions was presented based on the material of [2]. The conservation laws and calculation of the Q-value of reactions were covered and various nuclear reactions for surface analysis were described using the table shown in [3]. The concept of cross-section was introduced. The energy loss of ions in matter, which is a basic element of the RBS technique, was covered extensively using material described in [3]. The depth profile and it's relation to the stopping power was presented and also a discussion of straggling and detector resolution. The codes for performing these analyses (RUMP etc.) were briefly reviewed.

CHOICE OF REACTION

For the laboratory part of the course a nuclear reaction was chosen instead of an RBS analysis for several reasons. First in order to illustrate the fact that the elements of nuclear theory which the students learned in relation to nuclear reactors have application in other areas of science, while RBS is based on Coulomb scattering only. Another reason was to expose the students to a relatively uncommon technique compared to the extensive utilization of RBS and PIXE. We chose the $^{14}N(d,\alpha)^{12}C$ reaction because the analysis of nitrogen has become very popular due to the extensive use of nitrogen implants in materials for modification of surface properties, e.g. increasing the hardness of industrial tools

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and the use of nitrogen in semiconductor applications. TiN coatings on tool surfaces have also been used to achieve tool improvement. This coating, when applied in the 1:1 stoichiometry forms an attractive gold layer, which has also been extensively used on jewelry. TiN can also be used as a coating on glass to reduce infrared transmission and solar load, an attractive feature for automotive application. In a joint collaboration with Ford Motor Company we have studied and produced a modified glass by ion implantation [4]. In that study, Ti and N implants in different stoichiometries were performed on soda lime glass at energies calculated to produce a TiN layer buried just beneath the glass surface. In order to check the stoichiometry, subsequent analyses were performed to obtain the actual implanted doses of Ti and N, using RBS and NRA respectively. Glancing angle x-ray measurements were also done to determine the crystal structure of the compound.

We took one of the samples produced in this work as the study sample for which the students were to verify its stoichiometry.

THE REACTION

The cross section for the $^{14}N(d,\alpha)^{12}C$ reaction was measured by Amsel [5] (see fig1).

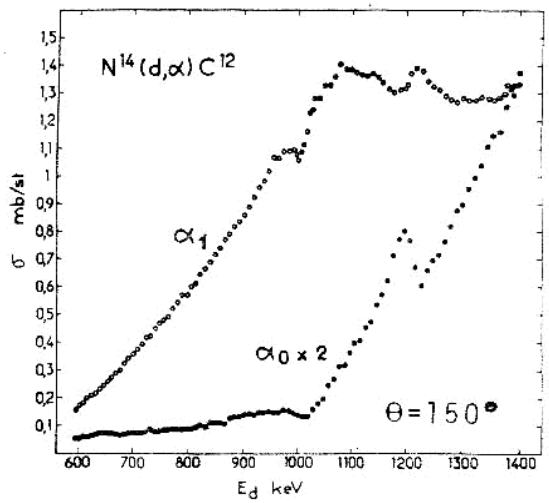

FIGURE 1 Cross section for the $^{14}N(d,\alpha)^{12}C$ reaction [5], © 1971, with permission from Elsevier.

The feature which makes this the preferred reaction for N analysis is its high positive Q-value which causes the α particles to have energies very much higher than signals from elastic scattering, providing a "clean" spectrum that is free of background. The decay of the compound nucleus ^{16}O leaves the residual ^{12}C either in its ground state, with the corresponding emission of an α particle of about 10 MeV or the first excited state, emitting an α particle of about 7 MeV (see fig.2). Even though the elastically scattered deuterons are of lower energy, they consume a large fraction of the electronic acquisition circuit time. To circumvent this problem, an absorbing mylar foil is interposed between the target and detector, calculated to completely stop the elastic events and allow only the higher energy particles from the reaction through albeit with some energy loss. When other elements are present in the target, some other nuclear reactions of positive Q-value can produce reaction products in the energy region of the spectrum around the 7 MeV α . Even so the higher energy α can still be seen in a background free region, facilitating the analysis.

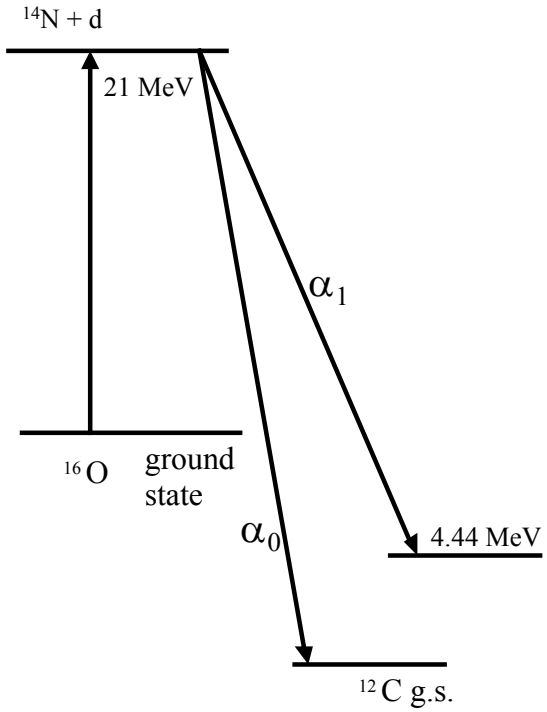

FIGURE 2 Energy diagram for the $^{14}N(d,\alpha)^{12}C$ reaction .

LABORATORY PREPARATION

A session was needed to familiarize the students with the equipment. A primer on accelerators was given. A preliminary description of the vacuum chamber, detector and electronics was also given. Later in the laboratory the students were shown the procedure for loading a sample. Special emphasis was given to charge collection. Most students are familiar with data acquisition techniques using computers and MCA's. However the concept of measuring the incident number of particles on target using a charge integrator is new to most. The operation of the instrument was described in some detail. Quite unexpectedly, students in previous years' classes had great difficulty in understanding the charge integrator. Also the production of secondary electrons, its effect on charge collection and solutions to the problem were presented.

In preparation for the analysis of the experiment the students were asked to calculate the energy loss of the α particles from the reaction after passing through the mylar absorber and estimate the position of the peaks in the spectrum. They were also asked to choose a deuteron energy based on an examination of the cross section curve. The cross section has a plateau at low energies, which makes it useful for analyzing thick samples without the use of deconvolution techniques. Another advantage of using low energies is the lower overall background radiation, making the presence of personnel in the vicinity of the acquisition chamber safe. However, at these energies the cross section is low making data collection lengthy. At higher energies the cross section increases but varies with energy, limiting the thickness of samples which can be treated without requiring deconvolution techniques. Also at higher beam energies the overall background radiation also increases creating safety concerns. A representative of the Radiation Safety Services Department was present at all stages in order to insure that the maximum permissible radiation level was never exceeded.

PROCEDURE

To calculate the stoichiometry of the TiN sample requires not only the number of N atoms/cm^2, the area density of nitrogen atoms, but also that for Ti. The Ti information was obtained by conventional RBS. Since it would be very difficult to do both experiments in the single available day for the lab work, the tabulated RBS spectrum in MS Excel format and parameters of the measurement, charge, detector solid angle, were provided so the students could calculate this quantity separately. Since most students do

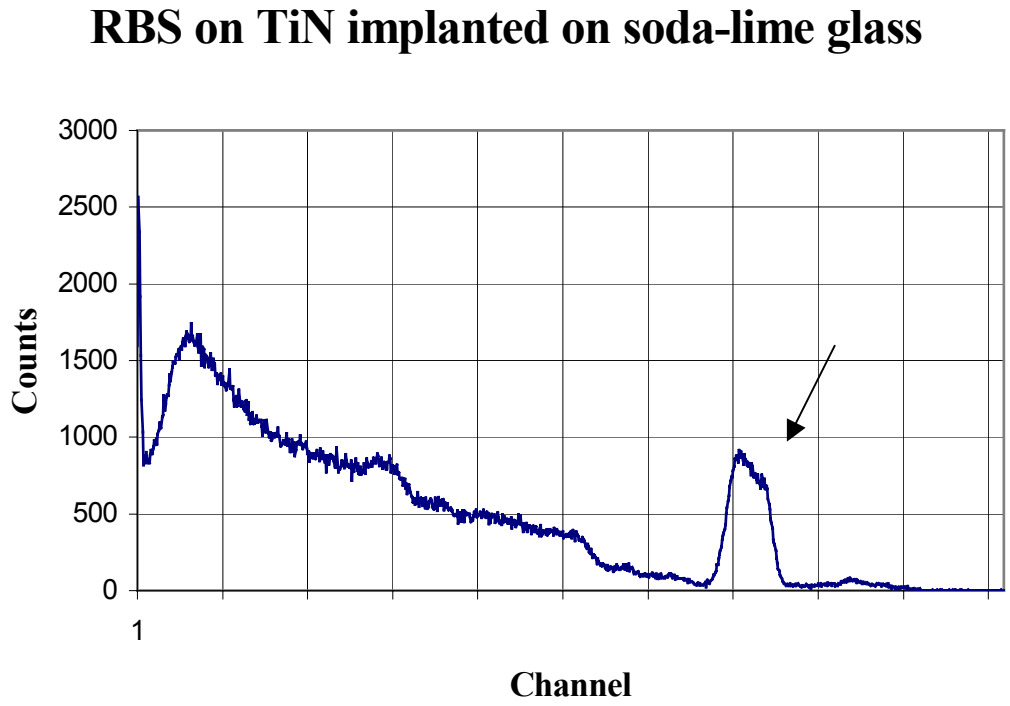

FIGURE 3 RBS spectrum of a TiN implant on soda lime glass. This is a reformatted spectrum in MS Excel format for easy manipulation by the students. The scattering from Ti is shown by the arrow.

not have access to data collection software used in the laboratory, the MS Excel format was used to manipulate spectra. The authors wrote special software that can replicate the data in this format. Figure 3 shows the RBS spectrum for the TiN sample used in the experiment.

The N measurement in the laboratory class was planned so that the time involved would be long enough to stimulate discussions during data collection but not so long as to be tedious. The students were divided into 2 groups and the whole lab class was conducted in one half day. The data acquisition time used per group was about 40 minutes, which we found ideal for these purposes. Figure 4 shows the NRA spectrum obtained by one of the groups.

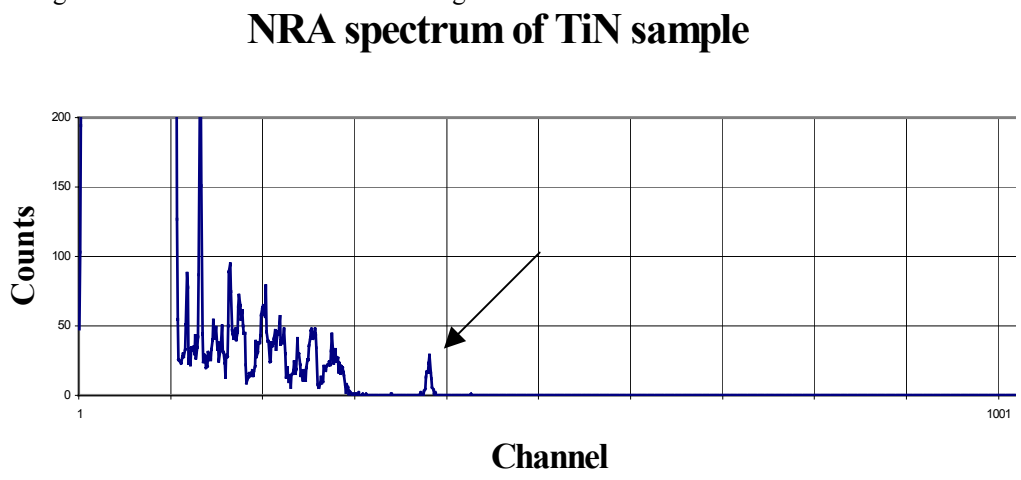

FIGURE 4 Same as figure 3 for the NRA spectrum from TiN implant on soda lime glass. This spectrum was obtained by the students in the laboratory. The peak of interest is indicated by the arrow.

STUDENT REPORTS

As part of their lab report, students were also asked to identify and assess the various sources of error in the experimental procedure and finally, determine the counting statistics needed to achieve various levels of uncertainty in measured stoichiometry. This statistical aspect of the experiment appears to have been more problematic to the students than concepts with NRA itself, since, the most frequent questions pertained to error propagation and determining the uncertainty of the measured stoichiometry.

CONCLUSIONS

The students put in a substantial amount of work on this class and generally felt very satisfied with this experiment and their work. Their overall performance was excellent. The success of this lab was attributed to several aspects that have been developed over the years. First and foremost, the students were able to conduct the lab with their own hands. Second, the combination of a theory lecture, followed by a preliminary assignment testing their understanding of the concepts in the lab, and then a discussion and a tour of the facility and instrumentation, in total, provided the background and familiarity necessary to make the lab session a success. Upon completion of the course, several commented that the real-world application of this particular sample and analysis was an important part of the success of this laboratory experiment.

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