

In-situ measurement of low-Z material coating thickness on high Z substrate for tokamaks^{*,a)}

D. Mueller^{1,b)}, A.L. Roquemore¹, M. Jaworski¹, C.H. Skinner¹, J. Miller¹, A. Creely¹, P. Raman² and D. Ruzic²

¹Princeton Plasma Physics Laboratory, Princeton, New Jersey 08543, USA

²University of Illinois, Champaign, IL, USA

(Presented XXXXX; received XXXXX; accepted XXXXX; published online XXXXX)

Rutherford backscattering (RBS) of energetic particles can be used to determine the thickness of a coating of a low-Z material over a heavier substrate. Simulations indicate that 5 MeV alpha particles from an ²⁴¹Am source can be used to measure the thickness of a Li coating on Mo tiles between 0.5 and 15 μm thick. Using a 0.1 mCi source, a thickness measurement can be accomplished in 2 hours of counting. This technique could be used to measure any thin, low-Z material coating (up to 1 mg/cm^2 thick) on a high-Z substrate, such as Be on W, B on Mo, or Li on Mo. By inserting a source and detector on a moveable probe, this technique could be used to provide an in situ measurement of the thickness of Li coating on NSTX-U Mo tiles. A test stand with an alpha source and an annular solid-state detector was used to investigate the measurable range of low-Z material thicknesses on Mo tiles.

I. INTRODUCTION AND DESCRIPTION

Generally, Rutherford backscattering (RBS) of energetic particles is used as a technique to measure near surface concentrations of high Z atoms. It can also be used to determine the thickness of a coating of a low-Z material over a heavier substrate. Simulations made using the SIMNRA program¹ indicate that 5 MeV alpha particles from an ²⁴¹Am source can measure the thickness of a Li coating on Mo tiles of between 0.5 and 15 μm . A Monte Carlo code that takes into account the wide angular and energy distribution of the alpha particles from a source close to the scattering surface and the detector was written to model the results more realistically than can be easily done with SIMNRA that assumes a well-collimated incident beam with a small energy spread. We first attempted this measurement using a source from a smoke detector to prove the concept and were able to measure the thickness of a thin (0.3 mg/cm^2) mylar film over a Mo substrate in 2 four day counting periods. Using a 0.1 mCi source, a thickness measurement can be accomplished in 2 hours of counting. This technique could be used to measure any thin, low-Z material coating (up to 1 mg/cm^2 thick) high-Z substrate, such as Be on W, B on Mo, or Li on Mo. By inserting a source and detector on a moveable probe able to reach the desired surfaces, this technique could be used to provide an in situ measurement of the thickness of Li coating on NSTX-U Mo tiles overnight, or in the case of dedicated experiments between shots. It may be feasible to detect the presence of an O impurity on the surface by measuring for an extended time if the amount of O is equivalent to about 0.2 μm (40 atomic percent of a 0.5 μm thick Li coating) but poor statistics due to the small cross-section for scattering from low-Z materials will greatly limit the utility of this technique as a means to

measure impurities in the Li coating. Furthermore, the absolute amount of O is difficult to measure due to the presence of resonant nuclear reactions between the alpha particles and O. A test stand with an appropriately collimated alpha source and an annular solid-state detector was used to investigate the detection count rate and the measurable range of low-Z material thicknesses on Mo tiles. Note, because the alpha particles lose energy primarily in electron collisions, the areal density of electrons is what is determined by this energy loss technique. For low-Z materials with 2 nucleons per electron, the areal mass density (the product of thickness and material density) is directly proportional to the electron areal density.

The National Spherical Torus experiment (NSTX) has used Li coatings of the divertor and wall surfaces to investigate the recycling characteristics and energy confinement with and without Li coatings, applied with various techniques.^{2,3,4} The range of deuterium atoms from the plasma edge into a Li coating is about 250 nm.² The thickness of the Li coatings on interior surfaces has not been measured directly so the relation between Li thickness on any surface to changes in recycling and confinement is based on modeling of the Li deposition. Furthermore, erosion and deposition redistribute the Li from its initial location and change the Li depth. Analysis of the surfaces after the experimental run with the usual array of techniques⁵ is not possible because upon venting, the Li is oxidized and forms a coating of Li_2CO_3 of the entire interior of the device, the Li having been redistributed by the plasma from its initial deposition area.³ The National Spherical Torus experiment Upgrade (NSTX-U) will have a Material Analysis Particle Probe (MAPP) to permit a sample to be withdrawn in-vacuo between shots for surface analysis⁶. Presently there is no system capable of measuring the Li distribution in situ, nor

^{a)}Contributed paper published as part of the Proceedings of the 20th Topical Conference on High-Temperature Plasma Diagnostics, Atlanta, Georgia, June, 2014.

^{b)}Author to whom correspondence should be addressed: dmuller@pppl.gov

of investigating the impurity content of the Li, save for the MAPP location. JET with its ITER like Wall has observed the erosion of Be and redistribution of Be unto the W surfaces of the divertor during limited operation and subsequent erosion from the divertor during diverted operation.⁷ At present there is no measurement technique in place to measure the thickness of the Be coating in situ over time. Alcator C-Mod has proposed using an energetic ion beam to map the thickness of Boron films on its Mo walls with nuclear reaction analysis.⁸ Here it is proposed to use a simple technique that, overnight, could measure the thickness of low Z materials deposited on metal surfaces at several locations using a system mounted on a moveable probe head. That technique is measurement of the energy loss of alpha particles from Rutherford backscattering using a 0.1 mCi ²⁴¹Am alpha particle source at a single point in as little as 2 hours. This could make it feasible to measure the change in low Z coating thickness at up to several locations overnight. Although it is not the focus of this paper, this technique could be used to measure the areal density of a high Z material on a graphite surface, the usefulness of technique would be limited by the count rate to surfaces with reasonably high Z content. It would, however, rely upon prior knowledge of the impurity's identity since it would be difficult to identify the high Z element unambiguously using the broad energy spectrum from a thick alpha source.

II. EXPERIMENTAL SETUP

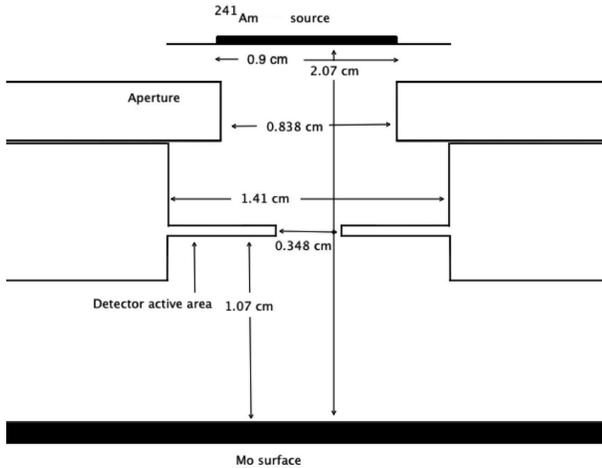


FIG. 1. The low intensity of the source and the small cross section dictate the geometry of source, detector and Mo surface must minimize the distances and maximize the solid angle acceptance.

The cross section for RBS is small. The differential Rutherford cross-section is given by

$$d\sigma / d\Omega = \left(Z_1 Z_2 / \pi \epsilon_0 E^2 \right)^2 \left[\sin^{-4} (\theta / 2) \right]. \quad (1)$$

For scattering of alpha particles from Mo this is

$$9.15 \times 10^{-24} \left[\sin^{-4} (\theta / 2) \right] E^{-2} \text{cm}^2 / \text{sr}, \quad (2)$$

where E is the alpha particle energy in MeV. The intensity of the collimated source of alpha particles is low (8×10^5 particles/s compared to the intensity (10^{10} to 10^{16} particles/s) of accelerated beams commonly used in RBS measurements. Together, these require that the source and detector be close to the surface to be measured and that a wide angular acceptance must be allowed. The physical size and intensity of commercially available ²⁴¹Am sources, about 1cm circle and up to 0.1 mCi respectively dictate the geometry that can be used effectively. Figure 1 illustrates the setup used in the test stand. Increasing the distances used decreased the count rate substantially with little or no improvement in the resolution of the observed scattered spectra. The detector used was a Si surface barrier detector and associated electronics whose energy resolution are insufficient to resolve the 3 closely spaced peaks from a 1 nCi source, but whose energy resolution and linearity are more than adequate for our purpose. The three energies and their percentages are 5486 keV (85%), 5443 keV (13%), and 5388 keV (1%). The energy resolution and the calibration of the energy of the alpha particles emitted by the 0.1 mCi ²⁴¹Am source are shown in Figure 2.

III. RESULTS

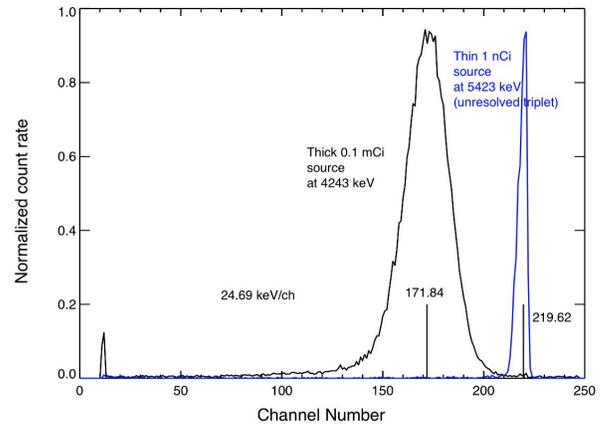


FIG. 2. The energy spectra of the alpha particles emitted from a thin, 1 nCi ²⁴¹Am source which has negligible energy loss in the source and that from the 0.1 mCi source used in the RBS measurements which has substantial energy loss in the gold and Am matrix of the source as well as in the thin gold coating.

A source and detector assembly shown in figure 1 was used to measure the backscattered alpha particles from a Mo surface and from a Mo surface covered by one, two and three layers of Mylar ($C_{10}H_8O_4$)_n. The thickness, 0.3

mg/cm² (2.1 μm), of the Mylar layers was measured by weighing a sample and by the energy loss of alpha particles from the 1 nCi ²⁴¹Am source with the Mylar placed directly between the source and the detector. The two methods agree to better than 2%.

The backscattered alpha particles lose energy passing through the Mylar coating both before and after scattering from the Mo. The end points of the energy spectra determined by a linear fit to the high energy edge of the spectra are 3639, 3094, 2514 and 1756 keV. The differences in the endpoint energies are primarily due to

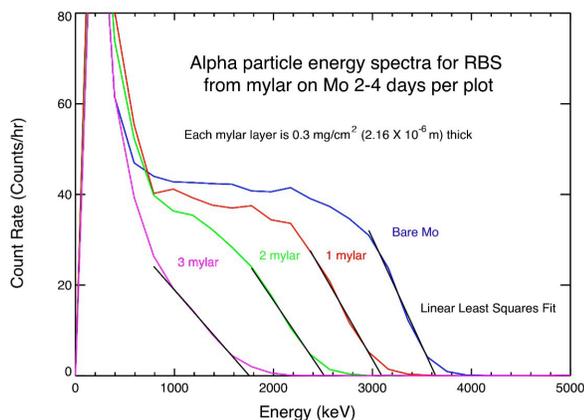


FIG. 3. The energy spectra of alpha particles from the thick 0.1 mCi source that have undergone RBS from a Molybdenum substrate that is bare (blue), and covered by 0.3 (red), 0.6 (green) and 0.9 (cyan) mg/cm² of Mylar and accumulated for 2 to 4 days for each. The black lines are fits to the data for the purpose of determining the end points of the energy spectra. Note the data is binned into 200 keV wide bins.

the energy loss in the Mylar (as well as a modest decrease in the energy loss to recoil energy of the Mo). Using the stopping powers of alpha particles in Mylar from the ASTAR database⁹ and the endpoint from the bare Mo case, the expected endpoint energies for the 3 mylar cases are 3074, 2424 and 1660 keV respectively. This agrees with the measured end points well and represents 3%, 7% and 5% more energy loss from this simple model than from the experiment. This accuracy is sufficient for measuring the thickness of light Z coatings on a high Z substrate. In order to determine, if this technique can be used in a shorter time, the experiment was repeated with 2 hour counting periods for the Mylar covered cases. Figure 4 shows the results of those experiments compared to a long exposure to the bare Mo surface. Despite the 200 keV bin size used, the 2-hour data is limited by statistical noise, nevertheless the endpoints (3189, 2492, and 1572 keV) indicate energy losses less than 15% different than that for the long exposures. This demonstrates that this technique could be useful to determine the thickness of low Z coatings on Mo or W walls in situ in an experimental device in measurements during a halt in operations

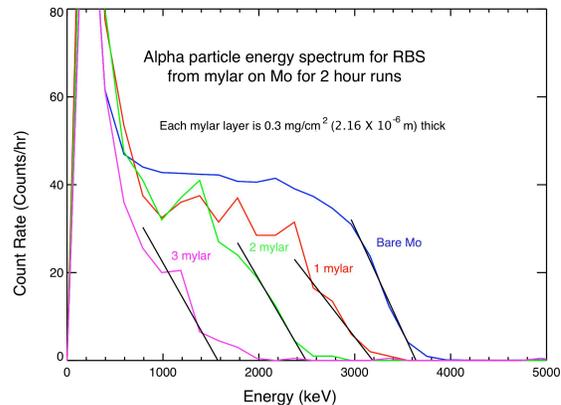


FIG. 4. The same energy spectra from the thick 0.1 mCi source shown in Fig. 3 with the data for the Mylar coated runs accumulated in only 2 hrs.

overnight. The thickness of Li coatings on Mo could be measured to better than 10% accuracy over the range of 0.05 to 19 μm in a few hours per point using this technique with a suitable probe to position the detector assembly in vacuum.

* Work supported by U.S.D.O.E. Contract No. DE-AC02-09CH11466

¹ M. Mayer, SIMNRA Report IPP 9/113, Garching, 1997; <http://www2.if.usp.br/~lamfi/guia-simnra.pdf>

² H.W. Kugel, M.G. Bell, J.P. Allain, et al., *Journal of Nuclear Materials* **415** (2011) S400–S404; <http://dx.doi.org/10.1016/j.jnucmat.2010.12.016>

³ H.W. Kugel, M.G. Bell, H. Schneider, et al., *Fusion Engineering and Design* **85** (2010) 865–873; <http://dx.doi.org/10.1016/j.fusengdes.2010.04.004>

⁴ H.W. Kugel, J.P. Allain, M.G. Bell, et al., *Fusion Engineering and Design* **87** (2012) 1724–1731; <http://dx.doi.org/10.1016/j.fusengdes.2011.07.010>

⁵ W.R. Wampler, *Nuclear Instruments and Methods in Physics Research B* **219–220** (2004) 836–845; <http://dx.doi.org/10.1016/j.nimb.2004.01.173>

⁶ C.N. Taylor, B. Heim, S. Gonderman, et al., *Rev. Sci. Instrum.* **83**, 10D703 (2012); <http://dx.doi.org/10.1063/1.4729262>

⁷ K. Krieger, S. Brezinsek, J.W. Coenen, et al., “Beryllium migration and evolution of first wall surface composition in the JET ILW configuration”, to be published in the *Journal of Nuclear Material*; <http://dx.doi.org/10.1016/j.jnucmat.2013.01.042>

⁸ Z. S. Hartwig and D. G. Whyte, *Rev. Sci. Instrum.* **81**, 10E106 (2010); <http://dx.doi.org/10.1063/1.3478634>

⁹ ASTAR database; <http://physics.nist.gov/PhysRefData/Star/Text/programs.html>