A lithium depth-marker technique for rapid erosion and deposition measurements

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ABSTRACT

A novel, high-resolution technique has been developed for the measurement of erosion and deposition in solid material surfaces. The technique uses a combination of nuclear reaction analysis (NRA) and Rutherford backscattering spectrometry (RBS) to determine the change in depth of a previously implanted marker layer consisting of ^{7}Li. A scoping study shows that ^{7}Li is an ideal marker candidate due to a high Q ($\sim$18 MeV) nuclear reaction, ^{7}Li$(p,\alpha)^4$He. Net erosion or deposition is measured by NRA of modified alpha energy passing through the bulk material. The reaction's high cross-section provides for the fast time resolution needed to measure erosion from high flux plasmas, and a highly penetrating proton beam provides for a large range of erosion/deposition measurements. Additionally, the implantation of low-Z Li leads to relatively low vacancy concentrations in the solid material due to implantation. This technique thus provides greater assurance that the measured erosion rate is indicative of the solid material: due to both the low vacancy production and the fact that no films or deposits are involved. Validation was performed by comparing the measured and predicted amount of erosion based on previously measured sputtering yields; the two were found to agree, within the uncertainty of the experiment. The depth resolution of the techniques is $\sim$60 nm at a net erosion depth of about 1 μm. The benefits of this technique are summarized as: short time scales (minutes) to obtain results, the marker layer can be used in any solid material, greater assurance that the measured erosion is indicative of the unperturbed solid material, and the continuous monitoring of the surface composition for contaminants and/or identification of deposited species using RBS simultaneous with the NRA.

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1. Introduction

The study of fundamental plasma-material interactions is critically important for the development of the next generation of plasma facing components for devices such as fusion reactors and plasma thrusters. In particular, processes which can affect performance and lifetime are a major concern. The erosion of material from surfaces is one such process, as it can cause perturbation to the adjacent plasma within a device, and can obviously limit the lifetime of the device if engineered PFCs are fully eroded through.

Several techniques exist for measuring erosion. The most straightforward procedure is to measure the change in mass of a sample before and after erosion. Surface profilometry offers an alternative method for directly measuring erosion. These techniques are not without disadvantages, however. For example, both can require plasma exposure time scales on the order of hours, in order to erode away enough material (several μm) to be within the resolution of the technique [1–4]. Additionally, neither of these

approaches is sensitive to surface contamination due to the transport of sputtered material from elsewhere in the device, and can only account for erosion or deposition of pure material from a surface.

Other techniques that have been developed to measure erosion have made use of a "layer cake" method, in which a sample coupon is built up by depositing thin layers of different materials onto a substrate. One such technique makes use of spectroscopy to determine how long it takes for each layer of material in the stack to be eroded through [5]. Another technique uses RBS to quantify the erosion of the top layer of material [6–9]. Both of these methods are high resolution, and generally sensitive to erosion depths on the scale of micrometers. The RBS-based technique is also sensitive to surface contamination. Nonetheless, both approaches rely on the creation of a sample coupon by layer deposition. However, there is no guarantee that a deposited film layer has the same erosion/thermal properties of the solid material. In fact, one expects that fundamental properties such as grain size and orientation (for metals), thermal conductivity, and defect concentration are different in the film. Therefore film techniques, while useful, leave questions regarding their representation of bulk material erosion/deposition, particularly in applications such as fusion and plasma

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thrusters where the environment is quite hostile to surfaces (e.g. situations with both continuous and transient high local heat loads).

This paper describes the development of an alternative ion beam analysis-based technique for obtaining erosion and deposition rates. In this technique, a "depth-marker" is implanted at a given distance from the surface using a mono-energetic ion beam. The basic principle is that as the background "solid" material is eroded or built-up through deposition, ion beam analysis will be able to track these changes by modification in the slowing-down of scattered particles and nuclear reaction products from the non-intrinsic marker. In this way, our technique builds on previous work for providing erosion/deposition in fusion devices by pre-implanting mono-energetic silicon in carbon and using RBS of the implanted Si [10]. However, in this new technique a high-Q nuclear reaction is used as the "scattering" event from the marker rather than elastic backscattering.

After implantation, initial NRA and RBS pulse-height spectra are measured simultaneously with independent charge-particle detectors. After the surface is exposed to a plasma, post-exposure NRA and RBS traces are taken, and compared to the initial traces. The RBS profiles are used to determine whether any unexpected changes to the surface composition have occurred, while the NRA profiles determine the net erosion or deposition of material from the surface. There are several benefits to using a "dual" NRA/RBS-based approach. First, the technique is rapid, requiring plasma exposure times on the order of minutes to obtain measurements that fall within the resolution of the method. Secondly, by using an inelastic scattering event from the marker, it opens the possibility of using a light-mass marker in a heavy substrate; a situation very unfavorable for RBS-only techniques due to the Z^2 dependence for Rutherford backscattering. This is desirable in fusion for example to examine heavy metal erosion (e.g. tungsten).

2. Method

2.1. Development of the implantation procedure

The development of the proposed depth-marker method faces several design challenges. First of all, the marker must be detectable using ion beam analysis methods. Secondly, the marker should not fundamentally change the material surface as it passes through it on the way to being implanted. Analysis of a variety of different depth marker candidates was conducted, and ultimately ^{7}Li was chosen.

Regarding the first criterion outlined above, various other experiments have determined depth profiles of Li concentrations in a material by making use of the nuclear reaction ^{7}Li$(p,\alpha)^4$He [11–14]. This reaction between a proton and a ^{7}Li atom has a high Q-value of 17.34 MeV, and produces two ~9 MeV alpha particles that can easily be measured using a charged particle detector. Even more important, the alpha energies are high compared to the ion beam energies typically used for IBA (<3 MeV), so that they are easily distinguished from elastic scattering. Thus, even a small atomic concentration of Li (i.e. <5%) in the implanted layer can be easily seen experimentally. The probing beam to trigger the nuclear reaction is protons, which is also advantageous, since high-current proton beams are readily available using accelerators. Also, the protons are also highly penetrating and require only moderate energies (>1 MeV) to overcome the Li coulomb barrier and trigger the nuclear reaction [15]. This ability to measure the marker at significant depths provides both a larger dynamic range to the erosion techniques and the possibility of applying the depth marker in high-Z materials with large stopping power.

In terms of the second criterion, ^{7}Li was selected as a candidate for implantation due to its low atomic number, which means that it causes a relatively low number of lattice vacancies as it passes through a material. The number of vacancies per atom, as predicted by a SRIM ("Stopping and Range of Ions in Matter") [16,17] simulation for a beams of ^{7}Li ions on Al and W, is shown in Fig. 1. The goal of the analysis shown in Fig. 1 is to find conditions for which the number of vacancies per atom is considerably less than one. If this criterion is met in a region on top of the implanted layer, then one can expect to erode material from this region without the erosion process being unduly impacted by structural changes in the Al surface.

In Fig. 1, the simulated fluence of Li ions on Al is 7.90×10^{16} ions/cm^2, which is the fluence needed to produce an atomic concentration of ~4% Li. The Li beam energies range from 1.50 to 3.00 MeV. For the Li on W cases, the fluence is 3.95×10^{16} ions/cm^2 (the fluence needed to produce an atomic concentration of 2% Li), and the beam energies range from 3.0 to 6.0 MeV. Different beam energies and fluences were used in the W case, due to the differences in stopping power and the number of vacancies per atom generated in Al versus W. If the same beam energy and fluence as the Al case is used in the W simulation, the Li layer is implanted at a much shallower depth, and the number of vacancies per atom is greater than one in the region on top of the implanted layer.

Looking at an example from Fig. 1, for the case of a 2.25 MeV Li beam on Al, the number of vacancies per atom is significantly less
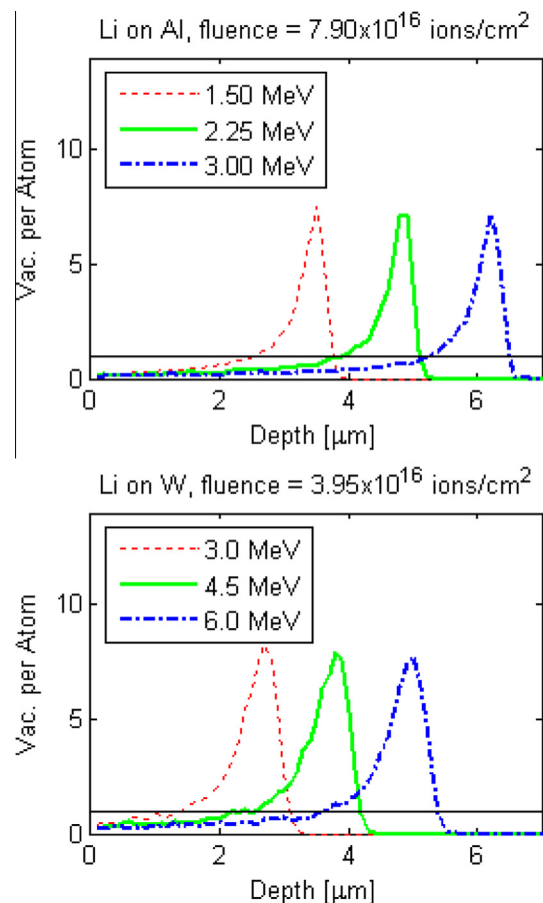

Fig. 1. Vacancies per atom due to ^{7}Li beams of various energy on an Al surface (top), and on a W surface (bottom). The total fluence was 7.9×10^{16} ions/cm^2 in the Al case, and 3.95×10^{16} ions/cm^2 in the W case. The black horizontal line on each plot shows the 1 vac. per atom cut-off.