Assessing electronic energy loss of heavy ions detected in reflection geometry

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Funding information
Stiftelsen för Strategisk Forskning, Grant/Award Number: RIF14-0053; Vetenskapsrådet, Grant/Award Numbers: 2017-00646_9, 821-2012-5144

We study energy loss spectra of bromine ions scattered from silver thin films in the energy range of 4 to 36 MeV in forward reflection geometry. The different contributions to the energy loss are analyzed by complementary Monte Carlo simulations. We assess the dependence of the scattering yield and nuclear and electronic losses on the penetration depth of detected ions. For scattering from larger depth, we observe decreased elastic losses and increased trajectory length in comparison with predictions from single scattering, with increasing effects for lower energies. To investigate the entanglement of energy loss and depth information, electronic stopping cross sections are deduced from the experimental spectra by two different approaches: (a) assuming a single scattering model and (b) making use of Monte Carlo simulations. The data obtained from the two approaches are compared, and we assess the relative contributions from nuclear stopping and from the effect of multiple scattering on trajectory length. Results of both methods are discussed in the context of composition depth profiling and compared with data from literature and with Stopping and Range of Ions in Matter (SRIM) predictions resulting in good agreement.

KEYWORDS
energy loss, heavy ions, ERDA, Monte Carlo, reflection geometry

1 | INTRODUCTION

Accurate knowledge of the energy deposition by ions in matter is imperative for a wide range of research fields and manifold technological applications including astrophysics,1 medical physics,2 and materials modification.3 Associated with this energy deposition, an energetic ion experiences a decelerating force when it penetrates matter. This force is commonly denoted as stopping power $S$ and is defined as the average loss of kinetic energy per unit distance in the material ($dE/dx$). This specific energy loss can be separated in two contributions: the electronic and nuclear stopping power $S_e$ and $S_n$, resulting from interaction with the electronic system or the atomic nuclei of the target, respectively.4 A quantity commonly used to avoid a dependence of $S$ on the atomic density $N$ of the material is the stopping cross section $\varepsilon = \frac{S}{N}$.

Both $S$ and $\varepsilon$ are characteristics for a given ion-material combination and show a strong dependence on ion energy and charge. At high energies, the dependence of electronic stopping power on the ion energy has been accurately described already in the early days of atomic physics.5,6 At lower energies, the description is complicated by the fact that ions may not be fully stripped, as well as the fact that the quantized nature of electronic states in the solid is limiting excitations to less tightly bound electronic states. In consequence, even for light ions, theoretical predictions by static theories can be inaccurate.7 and
Such predictions of the stopping power of a material for, in particular, heavy ions are essential for predicting particle range in ion implantation and energy deposition in irradiation. In Ion Beam Analysis (IBA), where the energy loss of detected ions is the common observable quantity, the specific energy loss permits to obtain accurate depth perception. Beams of heavy ions are successfully employed in Heavy Ion Elastic Recoil Detection Analysis (HIERDA) to enhance the detection sensitivity for light elements in heavy matrices, while simultaneously providing the total composition of a sample. The accuracy of sample composition depth profiles in HIERDA is, however, hampered by several different factors: commonly, the ions employed in HIERDA have energies, where both predictions from theory and tabulated experimental reference data are scarce. This situation renders predictions based on extrapolation from existing data by Stopping and Range of Ions in Matter (SRIM), the most commonly employed option, despite the associated drawbacks.

The specific relevance of the scarcely available data can be additionally affected by the following complication: stopping power data for heavy ions are commonly obtained in dedicated experiments performed in transmission geometry. In this approach, a (sufficiently thin) self-supporting foil is irradiated by a monoenergetic ion beam, and the energy of the transmitted ions is detected. This method permits a rather straightforward extraction of the electronic stopping, in the absence of large-angle scattering, and thus, nuclear losses. The fact that HIERDA is, however, performed in reflection geometries has several important consequences for the applicability of such energy loss data obtained in transmission: at first, the trajectories of the incident, scattered and recoiled ions in HIERDA with different relative weight of electronic and nuclear losses than previously investigated. Again, energy loss data in HIERDA or for extracting reference stopping power data is affected by the implementation of as a global parameter describing the energy loss along a straight trajectory in the SSM.

As indicated, the SSM is commonly employed in calculations of composition depth profiles from energy loss spectra of recoils and scattered particles using analytical tools such as Potku, CONTES, and SIMNRA in HIERDA. Although Monte Carlo (MC) tools have shown to be capable of reproducing experimental spectra with much higher accuracy, these calculations are computationally very expensive. As an alternative, an analytical approximation for the multiple scattering contribution has been developed, which exhibits relatively good agreement with MC data at sufficiently high energies. The need of iterative fitting of simulations to extract energy loss for calculating depth profiles, even nowadays favors approaches using the SSM and analytical expressions for the scattering and recoil yield when running HIERDA as a standard analytical tool. At the same time, sufficiently straight trajectories, which would overcome the above-mentioned potential complications with insignificant multiple and plural scattering contribution, can be only achieved by ion beams in an energy range that is not accessible by most accelerator facilities.

In an earlier study, we started investigating deviations from SSM in the case of iodine in gold, at energies as commonly employed in HIERDA. MC simulations confirmed a significant deviation from a single scattering geometry, in particular for low energies. Nevertheless, values for electronic stopping deduced from experimental energy loss data using predictions for path length and elastic losses from simulations and the single scattering approximation were found in very good agreement. Results were also found comparable with earlier published data obtained in transmission, even for ion energies of less than 100 keV/amu. These results thus indicated an applicability of the single scattering approximation for linking energy loss, stopping power, and depth scales, in HIERDA even at energies, where its basic assumptions are at least partially flawed. Data furthermore indicated that a potential influence of the geometry on electronic stopping cross sections is minor compared with other uncertainties.

In the present study, we aim to further investigate the above-mentioned aspects of electronic energy loss in reflection experiments and their relevance for HIERDA. We performed complementary experiments in reflection geometry for MeV bromine ions in silver thin films, which makes our data cover a wider range of potential projectile ions in HIERDA with different relative weight of electronic and nuclear losses than previously investigated. Again, energy loss data...
are assessed by two evaluation models. We compare stopping power data obtained from the SSM, as employed in common HIERDA experiments, with results of a detailed data evaluation supported by MC simulations. Simulated spectra were employed to extract trajectory length and elastic losses of reflected ions. Finally, we discuss the relevance of the obtained results for accurate composition depth profiling.

2 | EXPERIMENTAL SETUP, MC SIMULATIONS, AND DATA ACQUISITION

The experiments were performed at the 5-MV NEC 15-SDH-2 Pelletron tandem accelerator laboratory at Uppsala University. A thin silver film grown on Si(100) substrate by argon-magnetron sputtering deposition was used as sample. The areal thickness of the film was deduced by Rutherford Backscattering Spectrometry (RBS) using a 2 MeV He⁺ beam (detection angle: 170°). Small angular rotations were applied on the sample during RBS to minimize channeling effects in the substrate. The thickness of the thin film was found to be 80.7 nm. HIERDA performed by 36 MeV 79Br revealed O and C contamination of less than 1.5 at.% in the bulk. The same setup as described below was employed for this analysis.

Energy spectra of scattered ions were measured by the following experimental process. The energy of the incident 79Br ions ranged from 4 to 36 MeV (with an energy step of 2 MeV between 4 and 12 MeV) and directed onto the sample at an incident angle of 67.5° with respect to the surface normal. Scattered ions were detected at 45° with respect to the incident beam direction by a Time of Flight–Energy (ToF-E) telescope. With the experiment being performed in IBM geometry, this corresponds to a symmetric geometry with a detection angle of 67.5° with respect to the surface normal. The telescope consists of two carbon foil timing detectors placed in a distance of 0.4 m and a gas ionization chamber for energy detection. Consecutive measurements of the ToF and the energy of the detected particles allow mass discrimination and the distinction of the scattered primary ions from recoil nuclei originating from the target. As a result, we obtain an energy and a corresponding ToF spectrum. For data evaluation, we used the ToF spectra (time resolution ≈ 200 ps), after their conversion to energy scale, to exploit the higher energy resolution.

For the complementary MC simulations, the experimental geometry was reproduced in TRIM. The collision output data file, which holds the whole particle trajectory for all projectiles, was filtered to select only ions leaving the sample within an angle of 45° ± 2.5° with respect to the incident beam direction in the scattering plane formed by incident beam axis and surface normal. Subsequently, the ion trajectories were analyzed to extract path length and electronic and nuclear energy loss experienced by each ion.

3 | DATA EVALUATION

One important question to be addressed in the present work is to what extent, and for which range of primary energies the SSM can be applied to extract reliable stopping cross sections for heavy ions in reflection geometry. Two ToF-to-energy converted spectra of scattered bromine ions with significantly different primary energies are shown in Figure 1 (black solid line). In the case of 36 MeV 79Br presented in panel (a), one can see that the spectrum exhibits a well-defined plateau and width ΔE. In fact, the shape of the peak can be reproduced sufficiently well by analytical modeling using SIMNRA as shown for comparison (red solid line). The analytical models for multiple and dual scattering are selected in SIMNRA but prove insufficient to reproduce the observed background at lower energies. The red dashed line corresponds to a SIMNRA spectrum with the employed stopping power adjusted to the width of the experimental spectrum by using a correction factor.

The shape of the acquired spectra is severely modified at lower primary energies. As illustrated in Figure 1B, a 6 MeV bromine spectrum only rudimentarily resembles an RBS spectrum. While an edge from single scattering from the surface still can be identified, the falling edge of the spectrum is severely affected by plural and multiple scattering. Also, at energies exceeding the kinematic limit for single scattering kE0, dual scattering leads to a pronounced intensity. Both SIMNRA simulations with (red dotted line) and without (red solid and dashed lines) multiple and dual scattering corrections are clearly unable to reproduce the experimental results. In the former case, the low-energy tail is reproduced in contrast to the height of the spectrum and the high energy tail. A much more accurate reproduction of the experimental spectrum, fitting width, and intensity distribution within statistics is achieved by the data obtained from the MC simulation (blue solid line), that is, TRIM, which however requires much more extensive computing.

As a first step in the analysis, one can try to test the capability of extracting stopping cross sections by making use of SSM. In this case, the width of the experimental spectrum ΔE is used for the calculation of the electronic stopping cross section via [ε]out = ΔE/Ns. Here, Ns denotes the areal density of the film, and [ε] denotes the stopping cross-section factor, defined as

\[ [\epsilon] = \left[ \frac{1}{\cos \alpha \epsilon_{\text{in}}} + \frac{1}{\cos \beta \epsilon_{\text{out}}} \right], \]

where εin is the stopping cross section of the ions entering the material and εout is the stopping cross section after scattering and on their way out towards the detector. The incident and exit angle are indicated by α and β, respectively, in our case α = β = 67.5°, and k is the kinematic factor of the 45° scattering. By assuming the mean energy approximation, SRIM values were used for the calculation of a theoretical value of the stopping cross-section factor [ε]SRIM. Because SRIM prediction is expected to scale reasonably with energy, we made use of a scale factor c = k [ε]SRIM to deduce a pair of electronic stopping crossing-section values (εin and εout) for each primary beam energy. Note that for a sufficiently high number of datapoints, this procedure can be also abandoned if abstraction from any a priori scaling is desired.
Obviously, assessing the width of the spectra at lower energies comes with a significantly increased uncertainty as the background contribution due to plural scattering has to be estimated. More specifically, the determination of the exact position of the low-energy edge \( (E_{\text{min}}) \) comes with an almost constant uncertainty of at least 100–200 keV, which drastically increases the total uncertainty of the deduced data towards lower energies. As a result, the uncertainty in \( \Delta E \) should be expected to range from 3% to 10% depending on the primary energy. Additionally, a systematic uncertainty of the thin film thickness (≈3%) and random uncertainties due to experimental statistics (≈5%) are considered. Therefore, the total uncertainty of the final deduced stopping data is estimated to be within 6–23%.

Due to the increasing contribution from plural and multiple scattering, the assumptions of well-defined path length and elastic losses for particles scattered from the backside interface of the film made in the SSM obviously have only limited validity. To help understanding the specific impact of these simplifications in the single scattering assumption, ion trajectories were calculated by TRIM and yield additional information on the typical scattering processes experienced by the ions at each energy.

In Figure 2, the path length distribution of the ions contributing to the spectrum depicted in Figure 1B is shown. We have also filtered data in order to plot separately the distributions of the ions that reached a depth close to the backside of the thin film (i.e., penetrated at least 90% of the film thickness) and those that did not reach deeper than 10% from the surface. Based on the film thickness, the maximum path length predicted by the SSM is 4218 Å (for ions scattered from the interface of the film to the substrate) and is indicated by the vertical black dashed line. However, as seen in Figure 2, the majority of the ions experience a significantly longer trajectory. The distribution
of the ions that reached the edge of the film and were expected to be close the SSM maximum confirms the deviation from SSM, with less than the 10% of the ions featuring a trajectory length in accordance with the predictions for single scattering. Also, the fraction of ions scattered from the deepest depth slap is exceeding the number of ions scattered from an equally thick surface slap by a factor of $\approx 7$, which explains the only minor expression of the single scattering edge in Figure 1B.

At the same time, it is shown in Figure 3 that the total elastic losses are found to be—on average—significantly lower than what is expected by the SSM. The kinematic factor of bromine scattered from silver by $45^\circ$ is 0.63, which—for a primary energy of 6 MeV—is translated to an elastic energy loss between $\sim 1850$ and $2227$ keV, depending on the specific depth at which the scattering takes place. This range of nuclear losses expected by SSM is indicated by the shadowed region. However, the vast majority of the ions experience much lower nuclear losses despite the fact that they undergo many interactions with the target nuclei. A straightforward explanation to this effect is that there are more favorable ways in terms of energy and more probable trajectories for an ion to be deflected into a certain angle than trajectories containing a single large-angle scattering event. For example, the inset in Figure 3 depicts a typical trajectory of an ion penetrated deep in the film, which exhibits a total nuclear loss of $937$ keV (close to the maximum of the distribution). One large scattering event at $29.5^\circ$ takes place which decreases the ion energy by $884$ keV, and the final deflection of $45^\circ$ degrees is the result of plural scattering. Amongst ions reaching our virtual detector, such trajectories are clearly overrepresented compared with trajectories with scattering angles slightly larger than necessary for direct scattering into the detector.

Also, ions scattered from the near-surface region reveal a trajectory selectivity effect, which is rooted in the limited ways of an ion to become detectable after interacting with a sufficiently thin film. Thus, single scattering processes are favored in these cases, and as a result, the nuclear losses of the involved ions are scattered around to SSM prediction. Altogether, these facts lead to the situation that ions scattered from deeper layers, despite experiencing additional nuclear losses along their longer trajectory, feature lower total elastic losses. For comparison, we have also shown the expected integral contribution from nuclear losses according to SRIM for a single scattering trajectory from the backside of the thin film.

The results from these observations were used in a more sophisticated evaluation model to extract electronic stopping cross-section values. The model was first tested for limited values in Kantre et al.\textsuperscript{32} As mentioned earlier, these calculations are found to be computationally expensive and for lower scattering cross sections, that is, ions of higher energy, it is hard to obtain sufficiently good statistics, as CPU times of several days are necessary. Therefore, we limit our calculations to low energies, where the discrepancy between experiments and the SSM is expected to be largest.

First, we focus on the ions that experience the maximum energy loss, that is, those that penetrated deep in the film. These ions are determined experimentally by the left edge of the spectrum as denoted in Figure 1 ($E_{\text{min}}$), and their path length and nuclear loss distributions are plotted in Figures 1 and 3, respectively (light blue histograms). The electronic stopping power can be therefore calculated by

$$S_e(E) = \frac{(E_0 - E_{\text{min}}) - E_{\text{nuc, TRIM}}}{x_{\text{TRIM}}},$$

where $E_0 - E_{\text{min}}$ is the experimentally measured total energy loss of these ions. Average nuclear loss calculated by TRIM ($E_{\text{nuc, TRIM}}$) is

![Figure 3](image-url)
subtracted to estimate the electronic energy loss. This value, divided by the distance traveled in the material, that is, average path length \( x_{\text{TRIM}} \), defines the electronic stopping power \( S_e \), which is assigned to \( \varepsilon = \frac{E_{\text{in}} - E_{\text{out}}}{x_{\text{TRIM}}} \).

In addition to the uncertainties from extracting experimental energy loss data, in this approach, we have to take the statistical fluctuation of data obtained from the MC simulations into account. This contribution of this uncertainty on the deduced energy loss is estimated to range from \( \sim 4\% \) at low energies to \( \sim 10\% \) at higher energies.

4 | RESULTS AND DISCUSSION

The electronic stopping cross section of bromine in silver as obtained by both methods is plotted in Figure 4. Data are compared with SRIM predictions for electronic (solid line) and nuclear (dash line) stopping and literature data\(^{36}\) (open squares). Stopping cross sections calculated by SSM are indicated by black solid squares, whereas data from MC-supported analysis are represented by red solid squares.

At high energies, SSM appears to be approximately 8% higher than SRIM values but in good agreement with data from literature.\(^{36}\) A possible systematic deviation to be expected for stopping cross-section data obtained in reflection geometry compared with data obtained in transmission would favor slightly higher data obtained in reflection for the reasons stated in the introductions. As shown by the investigation performed in the previous section, the single scattering scenario is a good approximation in this energy range, and the probability for one large scattering close to 45° is still high. The latter implies very small impact parameters that, in turn, lead to perturbation of the charge state equilibrium effectively resulting in higher electronic energy loss. Note, however, that even an excess energy loss of several tens of keV for the given film thicknesses would only induce an increase of \( \varepsilon \) on the order of 1%, that is, much lower than the uncertainty of the deduced data.

At low energies, one would expect from the SSM and the corresponding assumption of a trivial linear dependence between energy loss, thickness, and electronic stopping to be unable to result in reasonable stopping cross-section data. However, the obtained values scale well with SRIM predictions and literature data. At the same time, results evaluated by the alternative method exhibit good agreement with SRIM, in the absence of literature data at such low energies, further corroborating the correct magnitude and energy scaling of the deduced electronic stopping cross sections.

Even the distorted spectra obtained at low energies (see, e.g., Figure 1B) can be employed to deduce reasonable stopping cross-section values by the SSM evaluation even though the basic assumptions have been proven invalid. In consequence, also the linear relation between energy loss and depth of interaction is still valid in good approximation. An explanation can be given by looking at the MC simulations. The observed increase of multiple and plural scattering has two main consequences with opposite effects on the energy loss: first, nuclear losses are smaller than in SSM prediction, when scattering from subsurface layers. However, at the same time, ions deviate more significantly from the straight trajectory assumed in the SSM. As a result, the path lengths are significantly increased which leads to higher electronic energy loss. Therefore, the low-energy edge of the experimental spectrum is the outcome of two counteracting processes that result in a spectrum width not much different than expected in a single scattering scenario.

This observation has also implications regarding the validity of data analysis and depth profiling performed by HIERDA. As mentioned above, the analysis is commonly performed assuming the SSM, even at energies and ion-material combinations for which the model is an oversimplification. The results above imply that the energy loss histograms employed for calculation of depth profiles are affected by three effects: (a) an increased path length of the primary particle reaching a target nucleus at a given depth, (b) a consequently on average reduced elastic energy transfer for subsurface created recoils, and (c) an increased resulting path length of the recoil, with respect to the specific depth of the collision. Although the detrimental effects on the energy of a scattered particle are counteracting each other, the effective reduced elastic energy transfers in particular to heavy recoils can well explain the commonly observed gradients in the depth profiles deduced for homogeneous compound materials with heavy and light constituents observed in HIERDA.

5 | SUMMARY

Energy loss spectra in reflection forward scattering geometry were measured for MeV bromine ions scattered from silver thin films. In addition, simulations in the experimental geometry were performed using TRIM and the obtained MC data were used to extract information on elastic energy losses and the path length distribution of
ACKNOWLEDGMENTS

The authors would like to acknowledge financial support from Vetenskapsrådet (contracts 821-2012-5144 and 2017-00646_9) and the Stiftelsen för Strategisk Forskning (SSF, contract RIF14-0053) also for energies at which the basic assumptions of the model scales from energy loss spectra as recorded, for example, in HIERDA simulations. These simulations permit to extract average path length and elastic losses for a given energy loss spectrum. Results indicate that the single scattering approximation can yield reasonable depth scales from energy loss spectra as recorded, for example, in HIERDA also for energies at which the basic assumptions of the model are flawed.

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How to cite this article: Kantre K-A, Moro MV, Paneta V, Primetzhofer D. Assessing electronic energy loss of heavy ions detected in reflection geometry. Surf Interface Anal. 2021;53:650–657. https://doi.org/10.1002/sia.6951