



Backscattered helium spectroscopy in the helium ion microscope: Principles, resolution and applications



R. van Gastel^{1,*}, G. Hlawacek^{1,2}, S. Dutta^{1,3}, B. Poelsema

Physics of Interfaces and Nanomaterials, MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands

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ABSTRACT

We demonstrate the possibilities and limitations for microstructure characterization using backscattered particles from a sharply focused helium ion beam. The interaction of helium ions with matter enables the imaging, spectroscopic characterization, as well as the nanometer scale modification of samples. The contrast that is seen in helium ion microscopy (HIM) images differs from that in scanning electron microscopy (SEM) and is generally a result of the higher surface sensitivity of the method. It allows, for instance, a much better visualization of low-Z materials as a result of the small secondary electron escape depth. However, the same differences in beam interaction that give HIM an edge over other imaging techniques, also impose limitations for spectroscopic applications using backscattered particles. Here we quantify those limitations and discuss opportunities to further improve the technique.

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1. Introduction

The realization of a practical gas field ion source by Ward and co-workers in 2006 [1] enabled the introduction of helium ion microscopy (HIM) as a technique to characterize materials. Since then approximately thirty HIM instruments have been installed worldwide and a new generation of focused ion beam workstations based on gas field helium and neon ion sources is about to hit the market. HIM is an excellent technique for the characterization of the microstructure of materials [2,3]. From a practical point of view it possesses the same imaging capabilities as a scanning electron microscope (SEM), where the incident beam also generates secondary electrons that are used for image formation, but yields different contrast and enhanced surface sensitivity [4]. It complements those imaging capabilities with the use of backscattered helium for image formation and the spectroscopic characterization of a sample. Because the incident beam is composed of ions rather than electrons, the information that can be obtained is akin to what is measured in Rutherford Backscattering Spectroscopy (RBS) experiments. In previous publications, we have highlighted the use of backscattered helium for crystallography [5] and the imaging of subsurface structures [6]. In this manuscript, we will focus on

the analytical possibilities, added value, and limitations of HIM as a characterization technique when performing backscattered helium spectroscopy. It has previously been suggested as a tool for elemental analysis of bulk samples and thin film metrology [7]. Because of the inherent limitation posed by the lateral spreading of the incident particles over the interaction volume, as well as the sample modification caused by the incident beam [2,7], we focus here exclusively on planar samples.

2. Backscattering basics

A description of backscattered helium spectroscopy is typically given in terms of the kinematic scattering factor and the stopping power of the sample. The conservation laws for binary collisions state that:

$$E_1 = E_0 \left(\frac{M_1}{M_1 + M_2} \right)^2 \left(\cos \theta \pm \sqrt{\left(\frac{M_2}{M_1} \right)^2 - \sin^2 \theta} \right)^2 = KE_0$$

where E_0 and E_1 are the energy of the helium before and after the collision, and M_1 and M_2 are the masses of the helium and specimen, respectively. The average energy loss due to a succession of binary collisions per unit travel length are commonly referred to as the nuclear stopping power of the specimen. They cause scattering of the incident helium over an angle θ , and provide a physical basis for interpreting backscattered helium spectra. The simplest interpretation of the spectra makes the assumption of single scattering events, which is not valid in the energy range in which a

* Corresponding author.

¹ All three authors contributed equally to this manuscript.

² Present address: Helmholtz-Zentrum Dresden-Rossendorf, Bautzner Landstrasse 400, 01328 Dresden, Germany.

³ Present address: IMEC, Kapeldreef 75, 3001 Leuven, Belgium.

HIM operates, typically around 30 keV. For helium that is backscattered in the first few nanometers of the near surface region, that assumption can however be used to analyze the planar structure of the sample. The position of edges and the onset of scattering in energy spectra can be used as relatively safe indicators of the composition and structure of the sample at the surface or at deeper interfaces, as was e.g. demonstrated for the analysis of bulk materials [7]. However, it is important to note that depth and compositional information are intrinsically intertwined, complicating the interpretation of spectra. It is the electronic stopping power that results from the interaction of the incident helium with the electrons present in the sample that causes a slow, but gradual loss of energy of the helium projectile. This effect broadens the backscattered helium spectra and leads to a loss of compositional and structural resolution as a function of depth. To make matters worse, the reduced accuracy of the information that can be obtained as a function of depth is further washed out by the finite energy resolution of the available detector units [8]. Here, we performed SRIM [9] simulations to establish the precise vertical resolution that can be obtained in backscattered helium experiments, complemented with SIMNRA [10] and ResolNRA [11] calculations. We illustrate our results with an experimental characterization of the simulated structure.

3. Simulations

We have performed simulations on two distinctly different planar configurations: a light-on-heavy stack (a material with a lower atomic number on top of a heavy material) and a heavy-on-light-stack (a material with a higher atomic number on top of a light material). The materials chosen for these two configurations were silicon and hafnium oxide (HfO), as schematically depicted in Fig. 1. The results from SRIM are presented in Fig. 2. A contrasting trend in the number of backscattered ions as a function of top layer thickness is visible for the two configurations. In the case of the light-on-heavy stack, a decrease in the number of backscattered ions occurs with increasing thickness of the top layer, whereas an increase occurs for the heavy-on-light stack. Both trends can be understood in terms of the scattering cross section of the top layer, which is roughly proportional to Z^2 [12]. In the case of the light-on-heavy stack, the majority of the helium can pass through the light silicon top layer without much resistance, only undergoing a limited number of small angle collisions and also losing energy as a result of the energy dependent electronic stopping power (which is most effective for faster ions). They then enter into the heavy substrate layer where the scattering probability is much larger. Furthermore, as the thickness of the top layer is increased, more ions travel deeper into the stack, thereby decreasing the likelihood that they are scattered out of the sample and detected. For the heavy-on-light stack, the top layer has a large cross sectional area for scattering. Increasing the top layer thickness therefore increases the probability that the ions are scattered out of the sample.

These qualitative arguments are further corroborated by the data presented in Fig. 2, which plots the number of ions scattered by each layer. To separate the contribution from each individual



Fig. 1. Schematic image of the light-on-heavy (left) and heavy-on-light (right) configurations that were used in our simulations. The thickness of the top layer was varied and the thickness of the substrate layer was chosen to be much larger than the interaction volume of the helium beam.

backscattered ion, they were assigned to different layers based on the deepest point they reached during their trajectory through the sample. Although this is a crude approximation that ignores other mechanisms through which an ion loses energy in the sample, it clearly visualizes the trends that occur in the backscatter yield with increasing top layer thickness. It can be seen that, for the same thickness of the top layer, heavy-on-light backscatters a higher fraction of ions than light-on-heavy. Also, the fraction of backscattered ions scattered from the top layer shows an increasing trend in both cases, but the rate of increase is much higher in the case of the heavy-on-light stack as a result of the relatively high cross-sectional area of the top layer. To further understand the different contributions to the backscattered helium spectra, SIMNRA was used to calculate the total energy spectra [10].

4. Discussion

The spectra for the five different layer thicknesses for the light-on-heavy configuration are plotted in Fig. 3. Since hafnium is the heaviest element, its energy spectrum spans almost the entire energy range in Fig. 3(a). The oxygen contribution to the spectrum is not plotted because the low Z number and small kinematic factor for oxygen yields only a negligible contribution at the lower side of the spectrum. The top silicon layer is very thin (~ 2 nm) and only a minute fraction of ions (0.02%) is scattered from that layer.

Almost all the backscattered helium (99.98%) originates from the thick underlying layer of HfO and undergoes various scattering events, resulting in the broad spectral contribution from hafnium. In Fig. 3(b), which shows the spectrum for a Si layer thickness of ~ 10 nm, a distinct silicon contribution becomes discernible around 19 keV, in accordance with its kinematic factor of 0.56 and the primary energy of 35 keV. In Fig. 3(c–e) we observe that the silicon contribution to the spectrum progressively gets bigger as a result of the increasing thickness of the top silicon layer. Simultaneously, the spectrum of hafnium diminishes as a result of the shielding by the thicker Si layer. A gradual shift of the center of the Si spectrum towards lower energies also occurs, which can be explained by the stopping power the ions experience, as well as multiple scattering events of ions. However, the leading edge of the spectrum does not move, since it is the intrinsic contribution from helium that is immediately scattered at the surface of the Si layer.

The spectra plotted in Fig. 3 allow us to establish the accuracy with which the position of the interface between Si and HfO can be established. The energy spread of the ions as they leave the surface of the sample provides a direct measure for this number and can be inferred from the graphs of Fig. 3 by examining only those ions that are scattered from a small region around the interface. The final resolution is then established by convoluting this number with the detector resolution, which is 4.2 keV for the energy range that we are concerned with here. The resulting vertical resolution as a function of sample depth is plotted in Fig. 4 for the smallest and largest layer thicknesses that were investigated. The resolution and depth are plotted in terms of atomic densities of the two materials, where 1 nm corresponds to $4.98 \cdot 10^{15} \text{ cm}^{-2}$ for Si and $5.95 \cdot 10^{15} \text{ cm}^{-2}$ for HfO. We can see that the total depth resolution for all elements degrades with increasing depth. If the detector resolution is included, the final depth resolution is further lowered by two orders of magnitude. This trend is the same for all thicknesses of the Si layer. One noteworthy fact is that the depth resolution in the Si layer is approximately 20% for moderate thicknesses up to around 75–90 nm. The depth resolution at the same depth, but including detector resolution, produces an uncertainty of approximately 66%. This means that with a better detector resolution, the intrinsic experimental resolution with an error of 20% can be approached. Also, at the same depth, hafnium has a better resolu-

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