Multi-Beam Ion Microscopy

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Introduction

Over the past fifty years the scanning electron microscope (SEM) has established itself as the most versatile and productive tool for imaging and microanalysis in many areas of science and technology, and some seventy-thousand instruments generate millions of micrographs every day. Scanning electron microscopes do, however, have one fundamental limitation in that the only experimental variable available to the operator is the choice of the accelerating voltage. Although the ability to vary beam energy is both necessary and important, it is an unfortunate fact that changing the beam energy also alters many aspects of performance: imaging resolution, relative strength of different signal components, depth of beam penetration, capabilities of the various analytical systems, and the severity of charging and beam-induced damage. This makes it difficult or impossible to optimize the interaction of interest.

The Zeiss helium ion microscope (HIM) (Figure 1) is closely related to the SEM in its functions and capabilities and can similarly operate over a range of beam energies [1]. In its current form, it may now have the capability to generate high-quality beams not only of helium but also of several other elemental ions as well. Because the details of ion-solid interactions at a given beam energy vary considerably depending on the type of ion used and the properties of target material, access to a "multi-beam microscope" can provide the experimental flexibility that is absent on the SEM. This article examines how the ability to select from among a number of different ion beams, as and when desired, could enhance imaging and analysis and provide valuable additional operating flexibility.

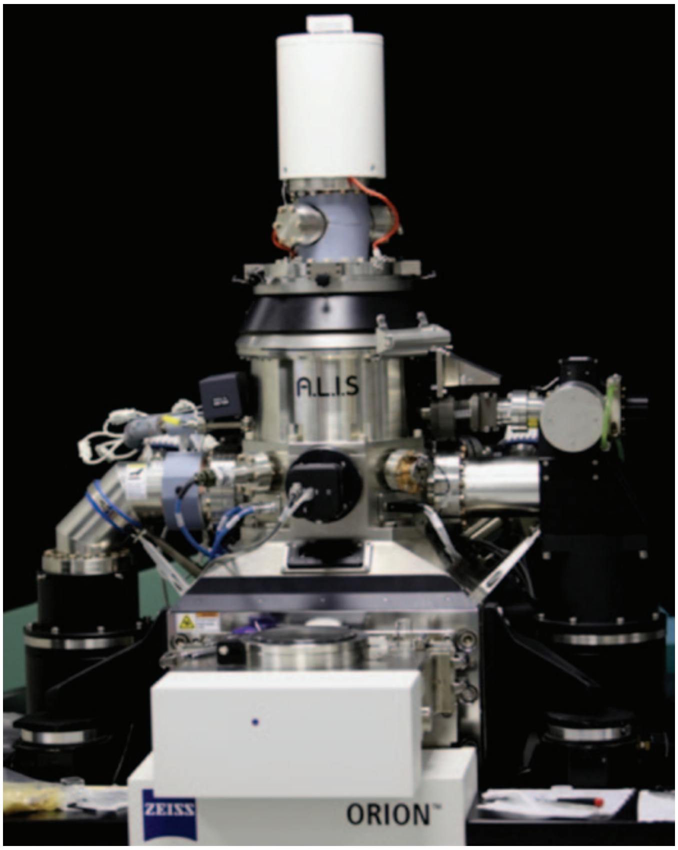

Figure 1: Zeiss ORION helium ion microscope (HIM) showing the gun and specimen chamber areas. (Picture is courtesy of Carl Zeiss NTS.)

Which Ion Beams to Use?

The ion source employed in the Zeiss ORION HIM [2] operates by the ionization of gas molecules in the vicinity of an atomically sharp emitter tip placed within the gun chamber (Figure 2). Helium is an excellent and convenient choice for ion microscopy, but a wide variety of other gaseous ions could, in principle, also be employed provided that they satisfy two basic requirements: they must be chemically pure, and they must only exhibit a single stable ionization state. If the gas can support multiple ionization states, then the final beam will be a mixture of several components of widely differing wavelengths, and this would make high-resolution operation impossible. In general, ions of low atomic number will be more suitable for imaging than heavier ions because they permit superior performance to be obtained at lower beam energies than the more massive ions, and the ions that best satisfy these requirements are H+, He+, Ne+, and Ar+. In addition, because Ga+ is already widely used in conventional focused ion beam tools, it will also be considered here for comparison purposes. At present there is very little comparative experimental data available on the absolute performance of, and the problems associated with, the various ion beams and of their interactions with materials. So for the purposes of this article, the examples illustrating the behavior of various ions have been simulated using an enhanced version of the IONiSE Monte Carlo program [3].

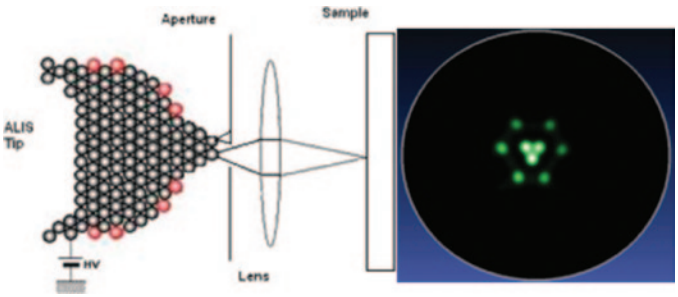

Figure 2: Schematic drawing of the emitting ALIS tip, 3 atoms in size. (Image is courtesy of Carl Zeiss NTS.)

Where Do Ion Images Come From?

The first task in microscopy is always to determine from where within the sample the imaging information is coming. In the HIM it is the range of the incident ions that will determine what can be observed and from how far beneath the incident surface a signal can be retrieved. Range data, determined from the IONiSE simulation, has been found to fit a form of the well-known Kanaya-Okayama expression where the range R (in nanometers) of charged particle beams in a material can be estimated as:

$$R\,(\mathrm{nm})\cdot\rho \;= A\cdot E^{n} \qquad (1)$$

where E is the beam energy (in keV) and ρ is the density of the sample (gm/cc). The symbol A represents a constant, which for electrons, H+, and He+ ions, is 78 nm; whereas it is 16 nm for Ne+, 10 nm for Ar+, and 8 nm for Ga+. The exponent "n" depends on the choice of particles in the beam: for electrons, n is 1.67, and for all the ions considered here n can be taken as 0.75. As seen in Figure 3, the penetration (that is, the product of the beam range and sample density) of H+ and He+ ions in a material is typically from 20 to 100× less than for electrons of the same energy, and so it is in nanometers, rather than micrometers, in scale. Changing from a light ion (H+) to a heavy ion (Ga+) decreases the depth of penetration by about another order of magnitude, giving the user the ability to rapidly change the depth from which the image information will be recovered over a wide range without the need to change any other parameters of the instrument.

The Interaction Volume

The penetration is only one of the properties of the beam that changes with the choice of ion. Figure 4 shows how the shape and size of the interaction volume in a given material (silicon) varies with the choice of ion at an energy of 40 keV. For H+ and He+ the shape of the interaction volume is quite similar to that found for electrons although on a significantly smaller size scale. However, for successively heavier ions, the interaction volume shrinks in size and changes in form. The volume becomes almost spherical in shape rather than forward peaked; it also becomes less clearly defined because of the generation of a loose tangle of trajectories that is visible in the simulation. This is due to the effect known as "straggle" in which heavy ions may travel very large distances before finally giving up all their residual energy and coming to rest. Thus, while the use of heavier ions does reduce the irradiated volume by a factor of typically 500 to 1000× or more as compared to lighter ions, the definition of the beam-affected region is significantly degraded and extended.

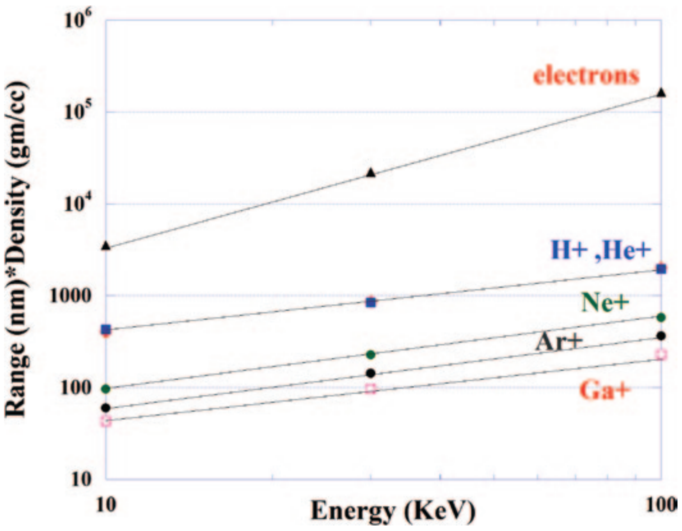

Figure 3: Penetration (range*density) computed from IONiSE for different ions as a function of energy.

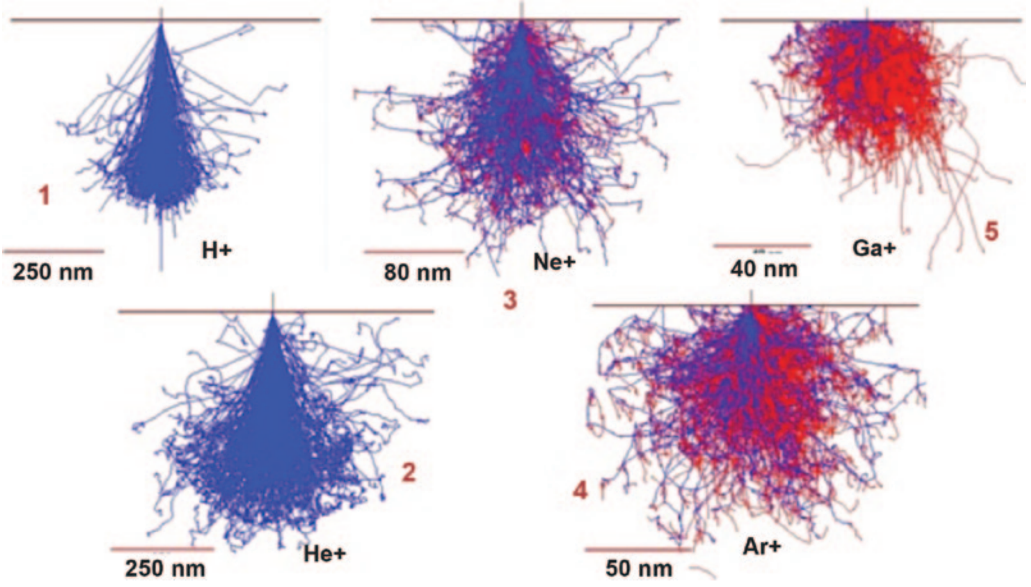

Figure 4: Interaction volumes of H+, He+, Ne+, Ar+, and Ga+ beams in silicon at 40 keV. Tracks shown in blue are generated by incident ions; tracks in red were made by recoiling target atoms.

The Generation of Secondary Electrons by Ions

The most important mode of image generation in ion microscopy is the secondary electron (iSE) signal, which is closely related to the familiar electron-generated (eSE) secondary signal but with some significant differences that are discussed below. To predict iSE yields, the IONiSE program [3] uses tabulated stopping power data for the chosen ion and sample to determine the energy ΔE available for iSE production along the beam trajectory. The rate, N(iSE), at which secondary electrons are generated at some point in the trajectory is then:

$$N(\mathrm{iSE}) = -(1/\varepsilon)\cdot(\mathrm{d}E/\mathrm{d}s) \qquad (2)$$

where (dE/ds) is the instantaneous stopping power of the ion in the target material and ε is a constant for a given material [4, 5]. The fraction $p(z)$ of these iSEs that escape the sample from a depth z beneath the surface is then [6]:

$$p(z) = A \cdot \exp[-z/\lambda] \tag{3}$$

so along each step of the Monte Carlo the incremental iSE emission is:

$$\delta(\text{iSE}) = N(\text{iSE}) \cdot p(z) \tag{4}$$

This approach is simple to implement, and detailed comparisons with both more elaborate models [7] and experimental yield data confirm that the accuracy obtained is more than good enough for any current application.

The problem in applying this technique is that values for the ε and λ parameters must be provided for each element. This can be done by fitting the model to experimental iSE yield curves [8] in the literature. For helium beams, fits for the two parameters have been determined for a total of 37 elements. For future use, however, the goal must be to provide ε and λ parameters to cover other beams of interest, such as H^+, Ne^+, Ar^+, and Ga^+. For a few elements, for example, C, Al, Mo, and W, some iSE yield data have been published for one or more of these additional ion beams of interest, and helpfully the ε and λ parameters derived in such cases have been found to be close to those for the original He+ beam. For these initial studies, therefore, the assumption has been made that the values of ε and λ depend only on the target material and not on the nature of ion employed.

The first requirement is to know how the yield of secondary electrons generated by an ion beam compares to that produced by a beam of electrons. As shown in Figures 5 and 6, respectively, the iSE yields from a low atomic number material (silicon) and a high atomic number material (platinum) vary widely as a function of beam energy and composition. Compared to the electron-beam-generated secondary electrons (eSE), iSE values show some very significant differences.

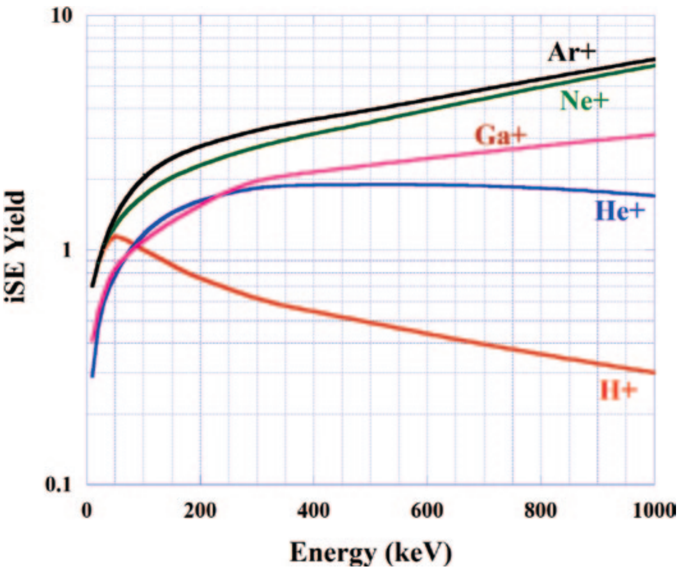

Figure 5: Computed iSE yields from silicon as a function of the incident beam energy for H+, He+, Ne+, Ar+, and Ga+.

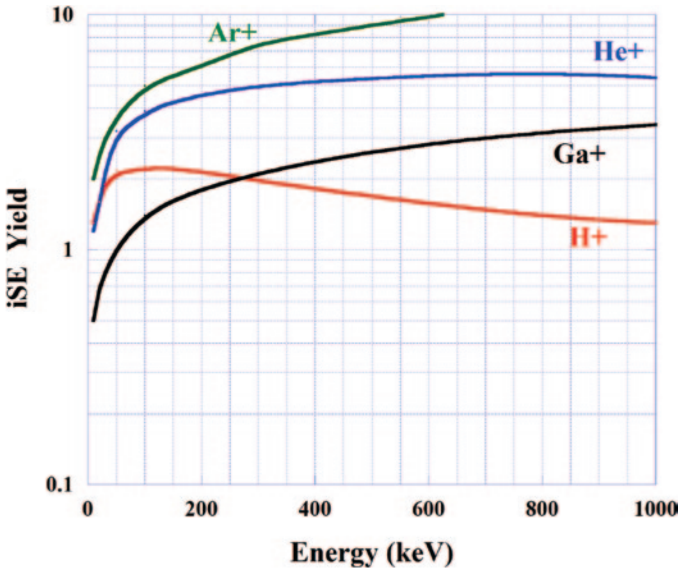

Figure 6: Computed iSE yields from platinum as a function of the incident beam energy for H+, He+, Ne+, Ar+, and Ga+.

Firstly, for all beam energies within the usual range (10 keV–100 keV), the ion iSE yield is almost always rising with energy, whereas for electrons it is always falling. This is because, as seen above, the yield of secondary electrons depends directly on the stopping power of the incident beam, and this in turn depends on the velocity of the incident particle. Stopping power for both ions and electrons reaches its maximum value for incident particle velocities of about $5 \cdot 10^8$ cm/sec, which corresponds to an energy of 250 eV for electrons, 50 keV for H+ ions, and 10 MeV for Ga+ ions. Secondly, as can be seen in Figures 5 and 6, the absolute yield of iSE is high compared to the corresponding electron case (where the yield is typically ≤ 1). This is because the incident ions only penetrate samples to a limited depth, and, as a consequence, a much higher fraction of the SE generation can diffuse back to the specimen surface and escape as compared to the electron case. Consequently, the iSE yield is typically an order of magnitude or more higher than for the corresponding electron beam, thus guaranteeing an enhanced signal-to-noise ratio even for a lower incident beam current. Furthermore, increasing the beam energy improves both the yield of iSE and the brightness of the ion source, giving even better performance. The optimum energy for a H+ (proton) beam SEM would be between 50 keV and 80 keV. For He+ ions this would rise to about 800 keV, and to many MeV for the heavier ions such as Ne+. Secondary electron images generated by ions from the same specimen, but originating from different ions, will be similar to each other but not identical in all details. For example, Monte Carlo simulations show that the topographic variation of iSE yield with the angle of incidence to the specimen surface decreases on moving from H+ to He+ to Ar+ and to Ga+, although all of these ion signals display better topographic contrast than the electron beam. The ability to choose the source ion therefore provides new options and opportunities.

Choosing Ions for the Problem of Charging

The ability to select the incident ion and its energy can be helpful in managing the problem of charging in the HIM. As in the SEM, specimens that are insulators or of low conductivity can charge-up under the beam causing problems ranging from image instability and exaggerated bright and dark contrast to distortions and random changes in the field of view. These problems will be more serious in the HIM because most iSE yields are greater than unity, so more negative charge is removed. In addition, only a small fraction of the incident ions are backscattered from the sample, which implies that the remainder must come to rest and deposit their positive charge into the depths of the specimen. Consequently, the surface potential of a target, in this case silicon, always rises with increasing ion energy (Figure 7), as compared to the electron case where the surface potential initially rises, then falls, goes through zero at the "E2" energy, and afterwards stays negative. Poorly conducting or insulating samples exposed to ion beams will always acquire a net positive charge, which will partially suppress the iSE yield, degrade the signal-to-noise ratio, distort the image field of view, and could cause image instability. For the observation of non-conductors, therefore, H+ or He+ ions would be the best choices as they minimize the growth of positive charge.

Charging can be further reduced, and often even eliminated, using either a localized jet of gas or a low-energy electron flood gun. The flood gun can maintain the sample surface potential at zero for any incident beam energy, does not degrade resolution, and, unlike the electron "E2" charge balance method, works even when the surface has significant topographic detail. Alternatively, a localized low-pressure gas jet can also stabilize charging with minimal impact on resolution. However, the surface potential will not in general be zero, so the landing energy of the beam and parameters such as the field of view for a given "magnification" setting may vary slightly from one region of a sample to another.

Backscattered Imaging with Ions—Why Sometimes Less May Be Better than More

Incident ions, like electrons, suffer backscattering from a specimen with a probability that increases with the atomic number Z of the target material; this provides a "backscatter image" in which the image brightness increases approximately as about Z^2 to give a "Z contrast" image related to specimen surface chemistry. The yield of the backscattered ion signal is very dependent on the choice of incident ion and its energy, as demonstrated in Figure 8. H+ and He+ ions are backscattered from a target with a probability that is 5 to 10 times higher than that for heavier ions (for example, Ag+) of similar energy; however, in every case the backscatter yield falls rapidly with an increase in the incident ion energy, becoming of little use for energies above about 100 keV. Consequently, although backscattered ions can provide some information on sample chemistry, in practice this method is limited to low-energy beams of H+ or He+.

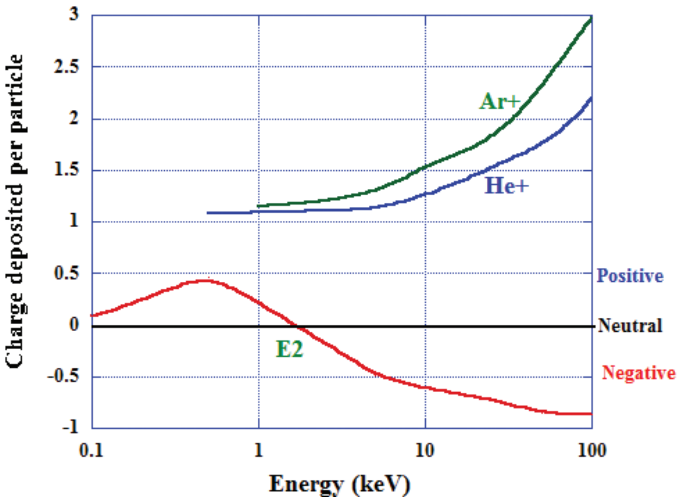

Figure 7: Variation of surface potential build-up on silicon as a function of beam energy for electron, helium, and argon beams.

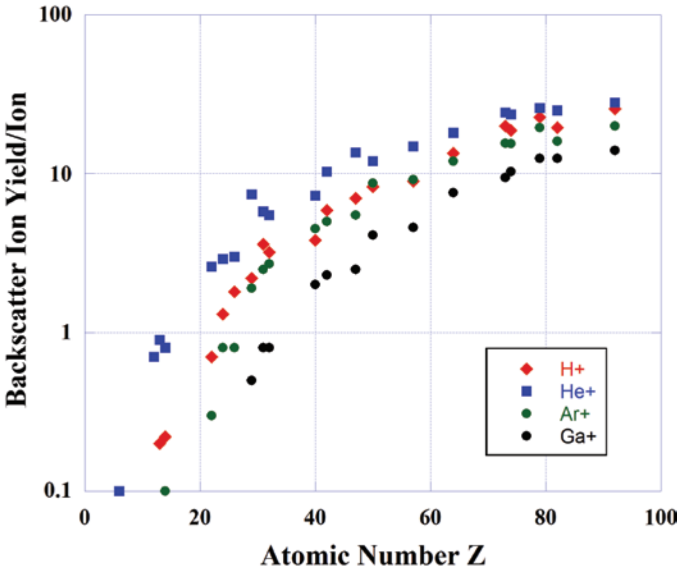

Figure 8: Computed backscattered ion yields as a function of ion type and energy for H+, He+, Ar+, and Ga+.

It can be noted, however, that the rapid decrease in ion backscatter yields with increasing beam energy might provide an effective means to optimize the iSE image. Both electron- and ion-generated secondary electron signals can be considered as being made up of two distinct components. The first, the SE1 signal, is produced where the incident beam impacts the specimen and gives an image rich in high spatial resolution surface detail. However, the second, the SE2 signal, is produced by backscattered electrons or ions as they leave the specimen and is of low resolution. Unfortunately, the SE2 component is typically 3 or 4 times larger than the SE1 component and so greatly dilutes the benefits of the SE1 signal, degrading both resolution and contrast. For ion beams, however, the iSE2 yield is much less significant, especially for heavy ions, and decreases with increasing energy, whereas the iSE1 yield is higher and rises with energy. As a result, the surface detail and the resolution of iSE images is much superior to those from an electron beam because of the absence of the unwanted SE2 contribution, especially for high beam energies and heavy ions.

Damage Control

Energetic ions can, and usually will, modify the materials that they are bombarding, and such damage can occur in

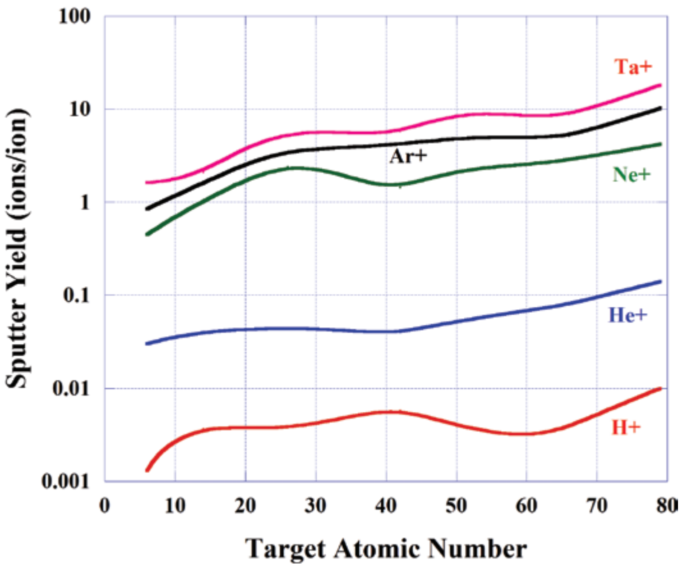

Figure 9: Sputter yield as a function of the incident ion beam type and the atomic number Z of the target material. Ion energy = 40 keV.

several different ways. For example, ions can be implanted into the sample resulting in the formation of gas bubbles, the incident ions can also break atomic bonds in organic and biological materials by the process of radiolysis, and ions can also directly "sputter" material from the specimen resulting in surface modification and possibly significant mass loss. The severity of such modifications depends on many factors. For example (see Figure 9) the rate at which the sample can be removed by sputtering varies with the atomic number of the specimen of interest and the choice of incident ion and its energy. At a fixed beam energy (here 40 keV), the rate at which material is "sputtered" away increases by orders of magnitude with an increase in the atomic number of the incident ion, although it does not depend much on the target material itself. However, for the lightest ions H+ and He+ the sputter yield falls rapidly with increasing energy (Figure 10). This behavior can be used to advantage when imaging beam-sensitive materials because increasing the beam energy reduces the rate of damage while simultaneously increasing the yield of secondary electrons (as noted earlier). The increase in iSE yield can then be used to advantage by reducing the beam current so as to maintain the original signal-to-noise ratio. As a result the damage rate is lowered by an order of magnitude while maintaining the quality of the image. Thus, as noted elsewhere, the ideal arrangement for an HIM is to have both light and heavy ion beams available so that all options are available for use when they are required for a given specimen.

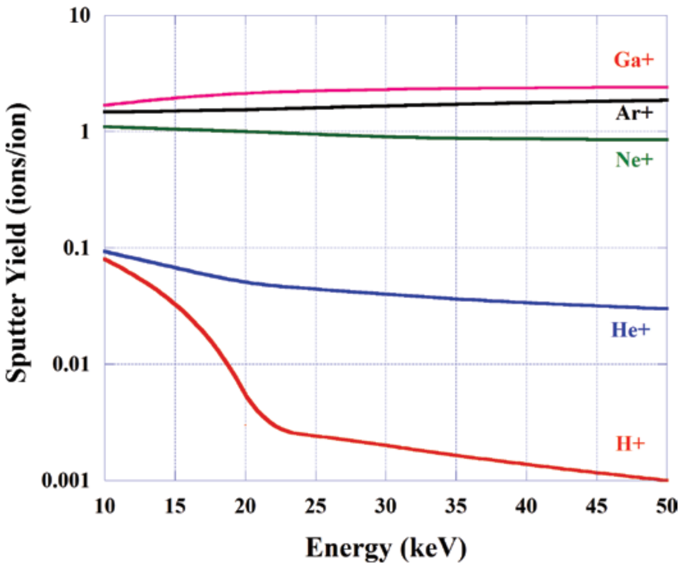

Figure 10: Variation of sputter yield from a silicon sample as a function of ion type and beam energy.

Microanalysis Possibilities

A significant future "application" of the options offered by the properties of different ion beams is likely to be in microanalysis. Microscopes that use ions, rather than electrons, do not generate X rays and so cannot perform a conventional elemental microanalysis. Although, as noted earlier, the yield of backscattered ions from a specimen gives some indication of the atomic numbers of the elements present, the reliability of the data is poor. Rutherford back scattered ion (RBI) spectroscopy, which measures the energy of the backscattered ions after a specific angular deviation, is more accurate in principle but is of little practical use for bulk samples because the ions lose much or all of their energy before they reach the surface. A potential alternative method for microanalysis is secondary ion mass spectrometry (SIMS), which measures the mass-to-charge ratio for atomic and molecular species sputtered from the sample by the incident ion beam, providing an unambiguous identification. In this mode a low-Z ion beam such as He+ would be used to scan the surface and identify a feature or area of interest but without generating damage. Then more massive ions, such as Ne+, could be used to sputter the top monolayers of the surface in the chosen area and eject the mass fragments into the SIMS system for analysis of their mass-to-charge ratios. This approach offers not just elemental analysis but "fingerprints" of complex materials and so could have applications in such fields as analysis of biological systems, polymers, semiconductors, and other complex nanoscale materials.

Summary

In summary, although the HIM shares many features in common with the SEM, it is more flexible and much more powerful. In particular, the ability of an HIM to generate a variety of different ion beams by simply changing the gas source allows the performance to be optimized for a variety of different applications and outcomes:

High-resolution imaging. Choose to use He+ or H+ ions and beam energies between 40 and 100 keV. This permits sub-nanometer iSE imaging resolution with low damage and a high signal-to-noise ratio.

Damage-free imaging. Use H+ or He+ beams and select an energy at or above 50 keV, which minimizes or eliminates sputter damage. The enhanced iSE yield at this energy allows the beam current to be reduced to maintain an acceptable signal-to-noise while causing minimum damage.

Sample preparation and sectioning. Use the heavy (Ga+) beam for surface machining, Ar+ beams for surface polishing, and then a He+ beam for the final imaging.

Secondary ion mass spectrometry. Use Ar+ or Ga+ to expose the region of interest, then employ He+ or Ne+ beams at medium energies to eject sputter fragments for secondary ion mass spectrometry.

Gas-induced nanofabrication. Use Ne+ or Ar+ beams at high energies interacting with a precursor gas for high speed, high spatial resolution, 3-D pattern deposition. Then choose H+ or He+ beams at a reduced energy for observation and, if required, final editing of the pattern.

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