Recent Progress in Understanding the Imaging and Metrology using the Helium Ion Microscope

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ABSTRACT

Nanotechnology is pushing imaging and measurement instrument technology to high levels of required performance. As this continues, new barriers confronting innovation in this field are encountered. Particle beam instrument resolution remains one of these barriers. A new tool for imaging and metrology for nanotechnology is the scanning Helium Ion Microscope (HIM). The HIM is a new approach to imaging and metrology for nanotechnology which may be able to push this barrier lower. As a new methodology, it is just beginning to show promise and the number of potentially advantageous applications for nanotechnology and nanometrology has yet to be fully exploited. This presentation will discuss some of the progress made at NIST in collaboration with the manufacturing community in understanding the imaging and metrology for this new technology.

Keywords: Helium ion, microscopy, HIM, scanning electron microscope, SEM, nanomanufacturing, nanometrology

1. INTRODUCTION

The scanning helium ion microscope (HIM) is a new tool for nanotechnology. As reported in earlier papers [1,2,3,4], the HIM is a new approach to imaging and metrology for nanotechnology which may be able to push the current resolution barrier lower. This instrument also promises the potential for new imaging modes, as well as, charge free imaging without the need for conductive coating. But, successful imaging and metrology with this instrument entails new ion beam/specimen interaction physics which must be fully understood. As a new methodology, HIM is beginning to show promise and the numerous potentially advantageous applications for nanotechnology have yet to be fully exploited. NIST was fortunate to receive the first commercial HIM [2] and this paper will discuss some of the progress made at NIST in improving its performance and to understand the science and capabilities of this new instrumentation. In addition, it is the NIST goal to understand potential differences between HIM and contemporary scanning electron microscope (SEM) instruments. Clearly, this is a difficult task since the SEM and the HIM appear similar in that both are scanned particle beam instruments but are quite different in their overall operational parameters.

2. MATERIALS and METHODS

The scanning helium ion microscopes used in this work were either a Zeiss Orion Plus installed in the NIST Advanced Measurement Laboratory or an engineering instrument installed at the Zeiss facility in Peabody, MA.

3. INSTRUMENT RESOLUTION

Imaging of the smallest details on a nanomaterial sample being viewed and then measuring it accurately is the goal of both the NIST Manufacturing Engineering Laboratory's Nanomanufacturing and Next-Generation Metrology Programs. Understanding the overall behavior and performance characteristics of particle beam instruments (SEM and HIM) is highly critical. For example, it is well known that at high accelerating voltages, particle beam instruments perform better than at low accelerating voltage. There are a number of reasons for this characteristic difference [5]. In the SEM, low

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landing energies are commonly employed when surface information is desired. But, the compromise is lower attainable resolution. For example, an SEM operating at 15 kV accelerating voltage will potentially have better than 1 nm performance, but that same SEM operating at 1.0 kV will perform at about 1.5 nm (or more). Newer aberration corrected SEM instruments have improved this situation and higher resolution low landing energy images now can be obtained in the subnanometer region. On the other hand, surface detail can be resolved with the HIM (discussed below) but, the current HIM instruments typically function only at high landing energies. So it must be understood that because of these and other fundamental issues direct comparisons between the performance of the HIM and SEM are rather difficult to do today even under high landing energy conditions.

Clearly, the ability to resolve fine detail with all particle beam microscopes has drastically improved over the past 20 years. The resolution achievable with the newest SEMs has been published by one manufacturer to be at or below 0.4 nm [6] and for the scanning helium ion microscope, 0.21 nm has been reported [7]. Even the best of the current instruments must perform at the highest level possible to resolve routinely the finest structures of nanomaterials the four contemporary scanning electron and ion microscopes installed within the Precision Engineering Division of NIST function at or better than the manufacturers specifications thanks to the generous commitment of the instrument manufacturers to high resolution performance. [8] Performance metrics are currently being researched to standardize the performance measurement of these instruments and recently, the development of a new standard for the evaluation of image sharpness has begun through the International Organization for Standardization (ISO). Currently, a working committee of ISO is reviewing a draft documentary standard of software programs for the evaluation of image sharpness in the SEM and HIM.

Attaining high resolution depends directly on a number of factors such as: 1) the physics of the particle beam; 2) the electromagnetic focusing ability of the electron or ion-optical column; 3) the size and shape of the best focused beam; 4) the conical angle of the beam; and 5) overall signal-to-noise ratio. Attainable resolution is also directly related to the sample being analyzed since the size and shape of the information volume, the region of the excited volume where the collected (generally) secondary electron (SE) signal originates. Therefore, the apparent sharpness of the image obtained will be a function of the material being analyzed and not just the instrument performance. Moreover, the amount of noise present in the system plays an important role as well, because it limits the amount of information recoverable from the image generated by the microscope. In most cases, much of this is transparent to the user and, in almost all practical instances; the focusing ability of the optical column is not the limiting factor because other contributory factors (e.g., operator ability, environmental effects, sample contamination, electro-magnetic fields, sample stage and beam drift, and vibration) also at work to reduce the achievable instrument resolution.

4. The Primary Electron and Ion Beams and Excited Volumes

The scanning electron microscope and the helium ion microscope both share a number of additional fundamental characteristics these include depth-of-field, signal, and excited volume. But, upon study, the nature of the characteristics manifests themselves quite differently.

4. 1 Depth-of-Field

Figure 1 shows a diagrammatic representation of the beam relative to a nano-structure. In this diagram, a 3 nm difference in the beam diameter at the top and at the bottom of the 100 nm structure is shown. A more parallel beam results in a smaller difference hence, better depth-of-field. Under normal operating conditions, the HIM - where the source of ions begins at single atom [1,2,3,4] - ***can*** result in a narrower convergence angle than the SEM (depending upon the instrument operating conditions). Therefore, the overall depth-of-field can be larger than the SEM even at higher magnifications [3, 9]. The difference in the depth-of-field is illustrated in the micrograph of Figure 2 where the large tin spheres in the foreground clearly appear sharp and in focus just the ones in the background. In certain cases, using the most optimum conditions and instrument settings, the depth-of-field of the HIM can be 5 times greater than the SEM.

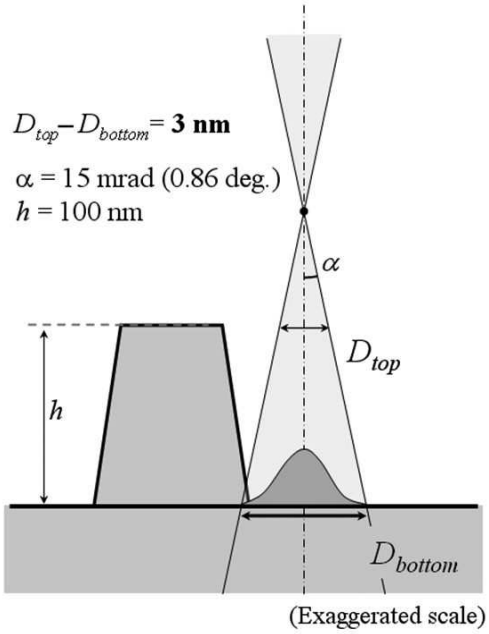

Figure 1. Idealized diagram of an electron or ion beam with a double-conical shape and Gaussian electron or ion intensity distribution across the beam. Note that the scales depicted have been exaggerated. (Courtesy of M. Tanaka of Hitachi High Technologies).

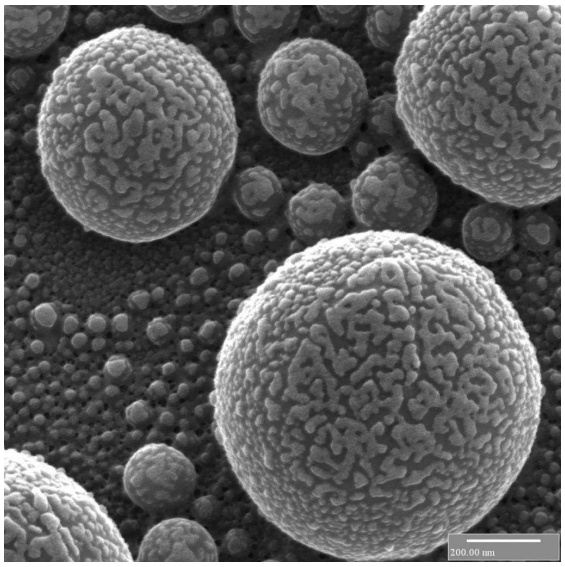

Figure 2. Helium ion microscope image of gold coated tin spheres showing the large depth of field possible with this instrument. The field of view is 1.5 µm.

4.2 Signal

The signal composing the image from the SEM or the HIM is a complex product of: 1) the interaction of the electron or ion probe with the sample; 2) the composition of the sample; 3) sample chamber geometry and chamber material; 4) electron detector employed; 5) and the electro-magnetic field present around the sample (either from the instrument itself or from sample biasing and/or charging). Clearly, the electron detector used is also a component. The three-dimensional

electron or ion beam and the sample define this 3-D information volume. This is the region from which the detected signals originate. Therefore, although imaging in the SEM or HIM appears straightforward, it is in reality a rather complex affair (as discussed further below). Additionally, for accurate dimensional measurements at the nanometer scale, sophisticated modeling methods that account for all the physical processes must be used to measure accurately the shape and size of the sample structures of interest [9, 10, 11]. To aid in this analysis, Ramachandra et al., [12] have developed the IONiSE Monte Carlo model to predict the topographic yield variation of helium generated SE as a function of sample composition. With this model, the important details of helium ion SE imaging can be compared with comparable electron-generated SE imaging. Such work is an important and primary step in the understanding of the imaging and metrology of the HIM.

4.3 Excited Volume

The current understanding is that the volume excited by the electron beam in the SEM and the volume excited by the HIM are quite different [1]. This volume is where the important beam-specimen interactions take place and it has been documented experimentally for the SEM [13, 14] but has not been done, yet for the HIM. In fact, this can be done much easier today because of the sample preparation capabilities of the ion beam instruments. Within this volume, the SE escape depth is the region from which the secondary electrons have enough energy to leave the sample surface to be potentially collected. The escape depth can be a few nm to up to more than 10 nm, depending on the sample material. For metals it is thinner, for insulators it is generally larger. The shape and size, the depth reached by electrons and ions and the secondary electron generation efficiency all strongly depend on the landing energy of the electrons or ions and the sample material irradiated. The landing energy of electrons in an SEM is variable; however, adjustable ion acceleration in the HIM is a capability still being implemented.

The size and shape of the excited and information volumes along with the secondary electron generation efficiency and location are important for another reason. The amount of surface-related information collected is directly dependent on these factors. Those secondary electrons carrying information about the finest details of the sample are generated by the primary electrons or ions at the point where the beam hits the sample. These are the so-called type SE1 electrons. The secondary electrons that were created by energetic electrons or ions backscattered within the sample are designated as SE2. Because of the location of their generation, the SE2 do not carry information about finest sample details; in fact, it is thought that they can emerge from a much larger area around the point of impact of the primary beam. The size of this area depends on the primary excitation and the material composing the sample under examination and can extend more than a micrometer in diameter [15]. Electrons are also generated by the backscattered electrons or ions that leave the sample and hit some other material within the sample chamber. These secondary electrons are called SE3. Again, these do not carry information about finest sample details. The well-focused beam always generates SE1, but the relative amounts of SE1 and SE2 and SE3 electrons have a profound effect on the appearance and the amount of fine details resolved in the secondary electron image. Peters [16] measured the individual contributions of the components of the SE signal, in the SEM, from gold crystals and found that, depending upon the sample viewed, the total SE image the contribution of the SE2 is approximately 30% and the contribution to the image of the SE3 electrons is approximately 60% as compared with approximately 10% of the image contributed by the SE1 derived signal. This ratio of SE2/SE1 generated by electrons [12, 17, 18, 19] significantly reduces the contrast and resolvability of small features. Clearly, this depends on landing energy and SE and backscattered electron or ion yields.

In the HIM, secondary electrons are produced at (or very near, within the escape depth of the SEs) the point of initial interaction with the sample and thus, are equivalent to SE1 electrons of the SEM. The initial SEs produce images with strong and familiar topographic contrast, and generally appear very similar to the secondary electron images obtained from an SEM, upon first inspection. IONiSE modeling [12] predicts that the helium-ion-generated SE2/SE1 ratio should be lower than that for electron irradiation especially at the higher landing energies. Hence, the contrast and the surface details are enhanced. Contrary to the SEM interactions, the ion beam passes much more deeply into the sample matrix and very few SE2 or SE3 type electrons dominant in the SEM imaging are generated. The flood of SE2 and SE3 electrons resulting from the backscatter of electrons in the SEM can essentially “wash-out” the surface detail potentially resolved by the electron beam. This does not occur to the same extent in the HIM, resulting in the enhanced surface

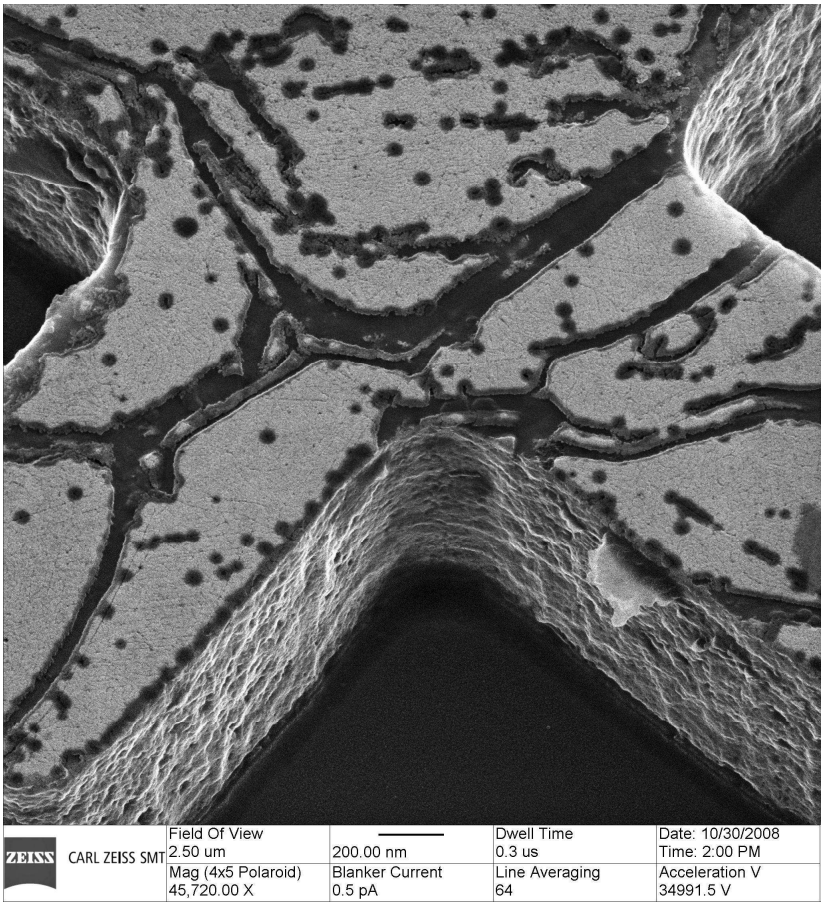

Figure 3. 35 keV landing energy HIM SE image of a contaminated Al marker showing excellent surface details and depth of field. The field of view is 2.5 μm (Courtesy of Carl Zeiss SMT).

detail as shown in Figure 3. Thus, in a material infinitely thicker than the secondary electron escape depth, clear surface detail may be resolved.

Although the HIM is very surface-sensitive in its imaging, sub-surface contributions to the HIM image are also possible. In very thin materials, low in atomic number or those that are flocculent, significant contributions to the image can be made from underlying structures. The SEM or HIM beam enters the first layer of the sample and generates signal then passes through (potentially generating signal, as well) striking another fiber or portion of the sample also generating signal. The sum of both interactions is collected by the secondary electron detector resulting in an image with a "ghost-like" appearance. One interpretation of this image is that the ion beam penetrated through the initial thin fiber providing signal from both the upper and lower fibers of the sample. Other possible mechanisms for generating sub-surface signal may be from electron channeling effects. Further research into these mechanisms and the underlying physics needs to be done in order to understand fully these observations. For now, adjusting instrument operating conditions helps to minimize these contributions.

5. HELIUM ION BEAM MILLING AND SAMPLE DAMAGE

Depending upon the beam current, the flux of particles within the excited volume of either the SEM or the HIM can be substantial and can lead to significant sample degradation [20]. Moreover, much of the energy of the primary electrons or ions stays within the sample, which can cause adverse effects such as sample melting, swelling or other dimensional changes. This is not to say that the electron and ion are equivalent in their effects. An electron stopping in a sample does

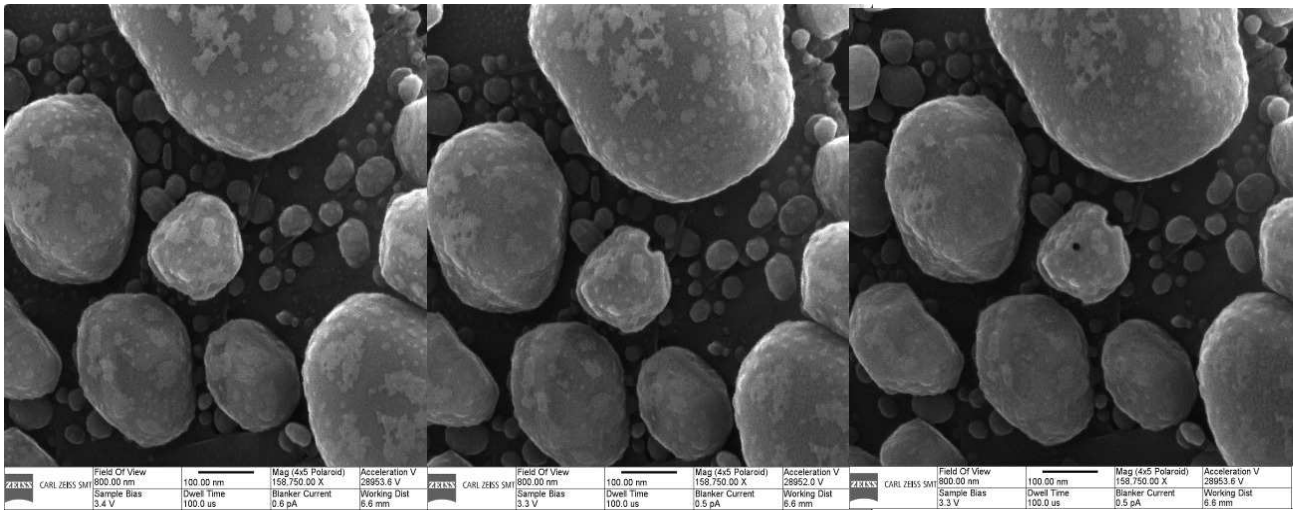

Figure 4. HIM SE images of He ion beam modification of nanostructures of a Pt-decorated gold-on-carbon sample. First image with no modification (left). After nano-milling at 2 o'clock position (center) and after drilling a 12 nm diameter hole (right). 800 nm field-of-view images.

not occupy a significant volume; however, the larger helium ions remaining in the sample could result in a swelling of the structures. However, this is highly sample-dependent; multiple imaging of robust structures shows no swelling or detrimental modifications to the structure under observation. This effect needs to be studied in greater detail. Unwanted sample modification is an issue which is a function of the material being viewed and the operating conditions of the instrument (beam current, accelerating voltage, etc.). It is up to the operator to find the optimal imaging and measurement conditions to obtain the best and repeatable results. For the scanning electron microscope many years of experience has provided some of the answers to sample damage for the SEM, but the HIM is still a new development and much has yet to be learned.

Gallium ion microscopes are well known for their ability to perform ion beam milling of samples for a vast array of purposes including transmission electron microscope (TEM) sample preparation. The helium ion microscope can also be used for focused ion beam mediated material removal by adjusting the instrument parameters and increasing the beam current and dwell time. Because of the inherently small source size, the HIM has demonstrated very fine material removal. Figure 4 is a series of images depicting the high precision material removal from gold on carbon sample using the HIM. This illustrates the effects of irradiation of approximately 3 pA beam current for approximately 60 seconds at 28 keV landing energy. Figure 4 (left) is the initial image taken with conditions conducive to imaging, whereas in Figure 4 (middle) the instrument conditions were changed to a very small scanned region (very high magnification image setting) for material removal. The small square region at the 2 o'clock location has been removed through irradiation with the helium ion beam. In Figure 4 (right), a small 10 nm hole was precisely milled near the center of the gold island in point irradiation mode thus, showing how precisely the beam can be positioned and material removed.

6. SEMICONDUCTOR IMAGING AND METROLOGY

The potential of achieving higher resolution and greater surface sensitivity has prompted a great deal of interest in the HIM for semiconductor metrology applications. The SEM is currently the tool of choice for semiconductor production. The many samples in the SEM are prone to charging and charge reduction is commonly achieved by lowering the accelerating voltage down into the 1 kV range to achieve a charge balance [20]. The immediate result has, historically been, a significant reduction in resolution due to beam broadening. Hence, edge definition is also broadened. Modeling has been able to de-convolute edge information from the images, but requires an accurate model to be used in conjunction with the instrumentation obtaining the data. Aberration corrected SEMs working at low accelerating voltage may, in the near future, improve upon this situation and this avenue is currently being explored, as well.

Semiconductor metrology with the HIM is different in that current models of the HIM only operate at high accelerating voltages. Therefore, no direct comparisons of the HIM and the SEM at low landing energy is possible. But, the edge sharpness would be expected to be much higher than that of the low accelerating voltage SEM. Additional work to understand the proper conditions for semiconductor metrology with the HIM is ongoing.

7. CHARGE REDUCTION

Current HIM instruments, as stated earlier, operate routinely at high accelerating voltages. Some semiconductor samples being viewed can build up a positive charge on the surface. Unlike the SEM, where the negative charge build-up can be quite high – high enough to deflect the electron beam in some instances, the positive charging in the HIM can be eliminated or at least minimized by employing an electron flood gun. Operating conditions can be established to facilitate viewing materials such as photoresist with the flood gun. HIM imaging of patterned photoresist structures on a silicon substrate has been successfully done in this manner. Typically, this type of sample would require use of a low voltage SEM due to the possibility of negative charge build-up, but with the HIM, these samples can be imaged without the disturbing sample charging by employing the electron flood gun.

The electron flood gun is also useful in imaging and metrology of chromium on quartz photomasks. Photomasks are very difficult to image in the SEM due to charge build-up in the quartz. Postek et al. (2003) successfully used variable pressure SEM to dissipate the charge build-up for the imaging and metrology of chromium photomasks [21]. However, as shown in Figure 5, the HIM can also be successfully employed in the imaging and metrology of these samples. The positive charge build-up is removed by electron flood gun enabling high resolution imaging of the chromium photomasks (Figure 5). Optimization of the electron flood gun is currently being undertaken to determine the proper conditions for common semiconductor materials and the effects of this tool upon measurements made while it is being operated.

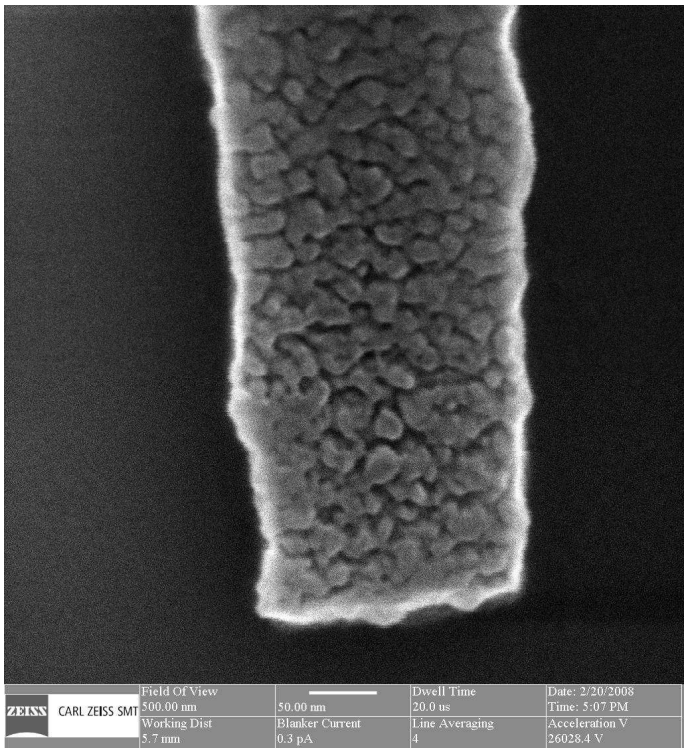

Figure 10. HIM micrograph of a chromium on quartz photomask 500 nm field-of-view.

8. HELIUM ION BEAM LITHOGRAPHY

Pushing lithography to the limits is important to semiconductor manufacturing. Electron beam lithography (EBL) has been used in lithographic patterning for many years. EBL suffers from many of the same issues as secondary electron imaging such as exposure from backscattered electrons, SE2s and other fast secondary electrons. Focused ion beam lithography with a gallium source has not been used extensively for resist patterning mainly because of the resolution constraints. Helium ion lithography with ~200 nm resolution was demonstrated over 20 years ago, but it suffered from a lack of ion column technology to make it a viable competition to EBL [22]. One advantage to using helium ions for lithography is the potential for higher resolution lithography than electrons [23]. The helium ion microscope has demonstrated that it is capable of generating extremely fine lines with extremely straight walls due to the deep penetration of the helium ions into the substrate and the lack of additional secondary signals which can degrade the lithographic fidelity. Dense array of approximately 15 nm diameter hydrogen silsesquioxane (HSQ) resist posts have been generated by helium ion lithography. HSQ is a high resolution electron beam resist and it permits high-resolution SEM inspection following patterning and development. The helium ion beam lithography technology is in its beginnings but it has already demonstrated an ability to fabricate 10 nm lines with a 20 nm pitch [24] and the direct fabrication of 7 nm structures have been reported [25]. There is still much to be done, but ion beam lithography shows promise and the sensitivity of the resist materials can be substantially higher, so higher –throughput direct lithography may be possible.

9. SUMMARY

In the interim between the original 2007, paper when the first commercial helium ion microscope was delivered to NIST was presented [2], a substantial evolution of the instrumentation has occurred. The performance of the instrument has markedly improved, as well as, the attainable resolution. Typically, the benchmark of performance for scanned beam instruments is the measure of resolution. But, it must be understood that where scanned particle beam instrumentation is concerned, resolution determination is more complicated than just laying a ruler on a micrograph and measuring the distance between two points. This concept is more involved than either the classical light microscope or even TEM. In this instance, the specimen plays a major part in the overall performance measure. Clearly, instrument performance is a strong issue and vast improvements in instrument design have strongly contributed to advances over the years in both SEMs and now the evolution of the HIM. Improved lens designs and illumination sources have been the main contributors to increased SEM instrument performance. But, instrument resolution relies not only on a high performing instrument design, and instrument operating conditions, but also on the material being viewed to demonstrate successfully the performance characteristics. Particle beam interactions and the nature and manner of the signal being collected are major contributors, as well. Hence, sample choice plays a significant role to demonstrate the performance of any instrument. It is also likely that the perceived instrument resolution for each sample will vary depending upon the materials composing that particular sample. For that reason, specialized samples have been developed for the demonstration of resolution capabilities. These samples have had to evolve as did the instruments. But, one must always keep in mind that one sample may work better than another for a particular set of operating conditions or instrument design so one must be continually vigilant that fair evaluations are made.

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