Add-on transmission attachments for the scanning electron microscope

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This article presents simulation and experimental results for add-on transmission attachments that have been developed for the scanning electron microscope (SEM). A primary beam energy of around 30 keV is used to irradiate thin test specimens (<100 nm). A transmission lens attachment enables a conventional field emission SEM to improve its image resolution by an order of magnitude, providing an image magnification of several million and a spatial resolution down to 0.3 nm. In addition, a compact energy electron loss spectrometer attachment has been developed that uses the SEM’s built-in secondary electron detector. © 2003 American Institute of Physics.

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I. INTRODUCTION

Scanning electron microscopes (SEMs) and scanning transmission electron microscopes (STEMs) are traditionally used for two quite distinct purposes. Both instruments focus an energetic primary beam of electrons onto a specimen and scan it in a raster-like fashion to produce a high-magnification image (>100,000) on a cathode ray tube (CRT) screen. SEMs typically provide surface topographical information on bulk specimens by detecting electrons that are scattered back from its surface. STEMs, on the other hand, detect electrons that travel through thin specimens, which yields bulk structural and compositional material information.

The image resolution of SEMs is often limited by the interaction volume that the primary beam makes with the specimen surface. At their top primary beam energy of around 30 keV, the image resolution does not improve much beyond 2–3 nm. Since transmitted electrons are not limited by the surface interaction volume effect, the image resolution of STEMs can be considerably better than that of SEMs. Crewe, Wall, and Welter reported an image resolution of 0.3 nm at a primary beam energy of 30 keV in the early development of STEMs. For thin specimens, where a transmitted beam can be detected, it is therefore desirable to operate SEMs in the transmission mode. More recent STEMs operate at higher primary beam energies, typically, in the 100–300 keV range, which enables them to approximately double their image resolution.

At present, there are some SEM attachments available that enable conventional SEMs to operate in the scanning transmission mode. However, they simply consist of specially made electron detectors that can be placed below the specimen. The spatial resolution of the transmission images obtained with these attachments is limited by the aberrations of the SEM objective lens. Most SEMs are not designed to provide a probe size below 1 nm.

This article shows that an add-on lens attachment can be made for conventional SEMs, which effectively enables them to provide STEM-like image resolution capability. The work reported here follows naturally from previous research that developed small permanent magnet lens/spectrometer attachments for SEMs. The 0.3 nm image resolution presented in this article may well be the highest spatial resolution to have been recorded by a conventional SEM.

STEMs usually divide their detection of transmitted electrons into two separate modes: the bright-field image, formed from inelastically scattered electrons, and the dark-field image, which is formed from elastically scattered electrons. In most STEM systems, the spectrum of transmitted electrons is usually acquired by the use of an electron energy loss spectrometer (EELS) placed below the specimen. The combination of a STEM with an EELS system greatly enhances its ability to provide quantitative material analysis. This article demonstrates the feasibility of using a small EELS attachment fitted on to the SEM specimen stage, which can be used with or without add-on transmission lens attachments. Providing EELS in combination with a SEM is not new, Crewe, Isaacson, and Johnson attached an energy spectrometer on to a field emission SEM in the early 1970s. However, the EELS attachment presented here is miniaturized so that it can either be fitted directly on to the specimen stage of conventional SEMs, comparable in size to their

Fig. 1. Transmission lens attachment in a SEM.

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specimen holders, or it can be fixed to a miniature STEM lens unit, so that a single compact attachment to a conventional SEM can enable it to simultaneously provide STEM and EELS information.

It should be noted that the SEM transmission mode in general needs thinner samples than those normally used for dedicated STEMs or transmission electron microscopes (TEMs). This is because the top primary beam energy in SEMs is around 3–5 times smaller than that routinely used in STEMs or TEMs. However, useful transmission images can be obtained with SEMs on standard TEM samples, as demonstrated by the experimental results presented in this article.

II. ADD-ON TRANSMISSION LENS ATTACHMENT

One possible way of incorporating a transmission lens attachment into a conventional SEM is shown in Fig. 1. Here, the backscattered detector is fitted onto the specimen stage and is used to collect transmitted electrons. The position of the central hole in the backscattered detector, measuring several millimeters in diameter, can be adjusted relative to the transmitted beam so that either wide-angle electrons are collected (dark field) or electrons traveling close to the optical axis are collected (bright field). The add-on lens attachment is fitted on to the backscattered detector holder. The specimen is placed within the add-on transmission lens unit prior to mounting it onto the backscattered holder.

The field distribution inside the add-on lens transmission attachment provides most of the focusing action applied to the primary beam. The specimen height within the transmission lens unit is adjusted so that the normal SEM objective lens is used only for fine focusing purposes. The SEM continues to collect the secondary electron image signal. Indeed, this capability proves to be extremely useful in aligning the transmission lens axis with that of the SEM optic axis. A secondary emission image of the lens top plate bore is first obtained via the normal SEM focusing and detection system. Specimen stage movement is then used to move the add-on transmission lens in the X and Y directions, aligning it to the electron beam axis.

Figure 2(a) shows a schematic diagram cross section taken through the center of the transmission lens attachment. Various permanent magnet lens designs were considered, but this lens design was found to be the most effective at im-

![Image](image_url)
mersing the specimen in a high magnetic field strength while at the same time minimizing the lens height. Two strong NbFeB permanent ring magnets, magnetized in the vertical direction, are arranged so that their respective coercive forces (900 000 A/m) are pointing in opposing directions. The outer casing of the transmission lens and an iron middle plate form a magnetic circuit that has two air gaps. The specimen is placed into its own detachable holder, which is lowered into the top air gap. The specimen position typically lies in the middle of the air gap. Adjustment of the specimen height is important for optimizing the image resolution, so for this reason, the specimen height within the lens can be altered by rotating the specimen holder. The fact that the specimen holder forms an important part of the magnetic circuit and at the same time is detachable does not pose any significant problems. After the specimen is changed, the transmission lens axis is recentered via normal specimen stage movement while monitoring the secondary emission image of the lens top plate bore, and since fine focusing is achieved through using the SEM objective lens, small changes in the transmission lens field strength will not affect the quality of the final image.

Figure 2(b) shows simulated flux lines for the flux inside the transmission lens unit. This flux distribution was calculated by finite element programs, KEOS, written by Khursheed. A prototype transmission lens attachment was designed, made, and tested. The simulated and experimentally measured axial field distributions for this lens are shown in Fig. 3. The average variation between experiment and simulation lies well below 5%. The peak field strength of 1.5 T in the upper air gap is significantly higher than the peak field strength found in most SEM objective lenses. In this type of immersion lens, increasing the peak field strength has the effect of lowering lens aberrations. The negative part of the axial field distribution produced by the lower air gap will tend to have a focusing effect on the transmitted beam. This is not expected to change the image resolution, but it does give rise to the possibility of moving between dark- and bright-field images by varying the backscattered detector’s height.

Using another KEOS program, on-axis aberrations were calculated for the transmission lens. Simulation predicts that a parallel incoming 30 keV primary beam will have a focal length of 0.956 mm, a spherical aberration coefficient \( C_s \) of 0.627 mm, and a chromatic aberration coefficient \( C_c \) of 0.696 mm. Using the Barth and Kruit root-sum formula for combining aberrations, and assuming that there is an energy spread of 0.4 eV in the primary beam, typical for a thermal field emission gun, the aberration limited probe diameter at the specimen surface is predicted to be 0.325 nm. These simulation results, therefore, predict that the add-on transmission lens will have a resolution capability similar to dedicated STEM systems.

The add-on lens attachment was fitted into a Philips XL30 field emission SEM. At first, an Agar Scientific Lacey Carbon Film S166-3 specimen was used. This specimen consists of a thin film of carbon (~100 nm) that has holes in it for a variety of different TEM applications.

Figures 4(a) and 4(b) show dark-field and secondary electron images collected by using the transmission lens at-
A beam voltage of 30 kV was used with an image magnification of 42 700. The demagnification action of the add-on transmission lens was calculated by first-order optics, and also measured on specimen features of known size. It was possible to move between dark- and bright-field images by varying the beam voltage. The dark-field image shows improvement in edge definition over the secondary image. This improvement in resolution is expected, since the transmitted mode of operation overcomes the surface interaction volume limit. On the other hand, the secondary electron image clearly shows more surface detail than the dark-field image. The two images should, therefore, be used together since they provide complementary information.

Figures 5(a) and 5(b) show similar results to Figs. 4(a) and 4(b), but were obtained at an image magnification of 200 000. The better resolution of the dark-field image compared with the secondary electron image is much more apparent at this higher magnification.

Figures 6(a) and 6(b) show how the add-on transmission lens performance compares with the transmission mode of the Philips XL30 SEM using its normal optics (own objective lens). A Philips “STEM detector” is placed below the specimen and a dark-field image of the Lacey Carbon specimen is obtained at an image magnification of 100 000, as shown in Fig. 6(a). Figure 6(b) shows a dark-field image taken at the same magnification with the transmission lens attachment. The dark-field image taken with the transmission add-on lens attachment appears to have higher edge definition, and superior resolution.

To investigate the matter further, the Agar Scientific S132/S133 TEM test specimen was used. This specimen has fine gold particles deposited on a thin film of carbon. The gold particles typically range from 2 to 9 nm. To resolve the smallest feature sizes on this specimen, an image magnification of greater than one million is required. It was not possible to resolve much detail on this specimen using the objective lens of the Philips XL30 SEM in its normal mode of operation. However, with the aid of the transmission lens attachment, this high resolution TEM test sample could be successfully imaged. Figure 7(a) shows a bright-field image of the gold on carbon specimen; the gold particles appear black, while the amorphous carbon layer appears bright. To obtain the bright-field image, it was necessary to lower the primary beam voltage down to 27 kV. The width of this image represents a distance of around 300 nm on the specimen. Figure 7(b) clearly contains fine detail, and it was possible to directly measure the smallest distinguishable feature sizes to be around 0.3 nm. To provide further confirmation of this result, a line scan was performed, a part of which is shown in Fig. 7(b). Here, the rising edge of a gold particle is examined. The 25%–75% change in signal height occurs over a distance of around 0.3 nm. This result clearly demonstrates the ability of the add-on transmission lens to improve a conventional SEM’s image resolution by an order of magnitude. The line signal over the amorphous carbon regions in this sample was noisy, and for this reason only points that lie just beyond the walls of the gold particle are included in Fig. 7(b). However, the signal over the gold particles and their edges were, in general, well defined, and this was also ap-

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**FIG. 7.** Transmission mode inspection of a gold-on-carbon TEM test specimen. (a) Bright-field image taken at an image magnification of one million. (b) Line scan across a gold particle.

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**FIG. 8.** EELS attachment in the SEM.
parent in the corresponding transmitted image. The authors are not sure why this should be the case. Surface contamination may be one possible cause, since this experiment was not performed under ultra-high-vacuum conditions.

**III. ADD-ON EELS ATTACHMENT**

Figure 8 illustrates the principle of the add-on EELS attachment. In this case, the EELS attachment is mounted directly onto the specimen stage and the normal SEM objective lens is used to focus the primary beam on to the specimen. A ring electrode located at the top of the unit is biased positively \((V_r = +100 \text{ V})\) so that it attracts secondary electrons. A transverse magnetic sector field causes the transmitted beam to undergo a deflection of approximately \(90^\circ\). A field strength of 0.1 T is typically required to bend a 30 keV beam around a radius of 5.8 mm. The transmitted beam then traverses a small slit opening, and strikes the specimen chamber wall below the scintillator detector. The tertiary electrons generated from this collision are then collected by the secondary electron detector. The signal on the secondary electron detector will, therefore, represent the transmitted image.

The add-on EELS attachment uses a standard magnetic sector prism energy spectrometer. A schematic drawing of the plan and sideviews of a prototype EELS attachment is shown in Fig. 9(a). Simulation using the KEOS programs for the field distribution predict that a manageable solenoid current can generate the required field strength of around 0.1 T between the sector plates. A diagram of the simulated flux lines for the EELS attachment is shown in Fig. 9(b). To simplify the simulation work, the rectangular-shaped box and sector plates were approximated to be cylindrical in shape.
This approximation makes it possible to use a KEOS two-dimensional finite element program to calculate the field strength between sector plates for a given coil current and display the flux lines. A total current of less than 1 A was predicted to be sufficient, and this was confirmed by experiment. No adverse heating effects were observed. Trajectory paths of a 30 keV beam leaving the specimen were also simulated, as shown in Fig. 9(c). These simulation results predict that a 30 keV transmitted beam comes to a focus several millimeters beyond the slit. Although not fully optimal, this design is sufficient to produce bright- and dark-field transmitted images.

Figures 10(a)–10(c) show bright-field, dark-field, and secondary electron images of the Lacey Carbon TEM test specimen obtained from the EELS attachment being fitted on to the Philips XL30 field emission SEM operating at an energy of 30 keV. An image magnification of 10,000 is used. The results not only show the transmission images to have better resolution than the secondary electron image, but show that the dark-field image can be used to provide a contour map of the specimen local mass thickness. Bright areas of the carbon film in Fig. 10(b) indicate a certain level of energy loss in the beam, related to a certain specimen mass thickness, while darker areas in the carbon indicate areas of greater energy loss, that is, areas of greater specimen mass thickness. Measurement of mass thickness is, of course, a well-known contrast mechanism performed by dedicated
STEM instruments. Both varying the primary beam energy and magnetic sector coil current caused the mass-thickness contour map to change in predictable ways. As the energy range of the detected transmitted beam was selectively scanned from the collection of elastic electrons to high loss electrons by the EELS attachment, the bright areas in the transmission image moved away from the holes in the specimen to regions between the holes. Figures 11(a)–11(c) show this for the magnetic sector coil current changing from 758, 741, and 711 mA. These images were taken on another part of the same specimen. In this preliminary design, a 1 mA change in the sector coils causes an approximate energy dispersion of around 20 eV. Figure 11(b) clearly indicates an intermediate energy loss condition, where the brightness distribution is related to variations in specimen mass thickness. These results serve to demonstrate that the EELS attachment is able to selectively map different parts of the transmitted beam energy spectrum. The EELS design needs to be further optimized before it can acquire ionization edge information.

Combining the compact STEM and EELS units presented in this article is relatively straightforward. A prototype STEM/EELS unit has already been constructed that has a height of less than 45 mm and weighs less than 840 g, and can comfortably be directly mounted on the specimen stages of most conventional SEMs. Future work will present results from this combined STEM/EELS compact add-on SEM attachment.