Backscattered Electron (BSE) Imaging in the Scanning Electron Microscope (SEM) - Measurement of Surface Layer Mass-Thickness

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ABSTRACT

Sometimes, the sample to be examined in the SEM will consist of a compositionally non-uniform substrate that is covered by an approximately uniform surface layer. With a low enough incident beam energy, only the surface layer can be seen in the SEM image. The underlying structure can be seen in the secondary electron (SE) image if the range of the incident electrons is greater than twice the thickness of the surface film. In the backscattered electron (BSE) image the threshold energy is higher because the BSE detector is insensitive to slow electrons. The information depth in the BSE image was investigated experimentally as a function of incident energy and BSE detector position using test specimens in which an Al layer of thickness either 210 or 1,100 nm was deposited onto an aluminised Si wafer covered by a pattern of gold lines. It was estimated that a lower limit to the surface mass-thickness that can be measured using a solid-state BSE detector is $-10 \mu g/cm^2$ ($=40$ nm of Al) for the BSE method, as compared with $-0.25 \mu g/cm^2$ ($=1$ nm of Al) for the low-loss electron method. There would seem to be no reason why measurements by the BSE method could not be carried out automatically in a computer-controlled SEM equipped with image analysis and using the standard BSE detector systems, to measure the mass-thickness of a surface layer.

Keywords: Backscattered electron image, Electron penetration, Image contrast in scanning electron microscopy, Information depth, Mass-thickness of surface layer, Multi-layered sample, Secondary electron image, Angular distribution of backscattered electrons.

1. INTRODUCTION

The idea which runs through this paper is the way in which both the secondary electron (SE) image and the backscattered electron (BSE) image of a solid specimen in the scanning electron microscope (SEM) are affected by BSE from the deeper layers. The samples consist of an approximately uniform surface layer over a substrate in which the atomic number $Z$ is non-uniform. A question of some interest is how to measure the mass-thickness of such a surface layer in terms of the beam energy at which the underlying structure becomes visible in the recorded image. A second question is whether such measurements would be feasible on a routine basis with an automated SEM.

In the SE image, the collected current arises jointly from SE that are excited as the primary electrons enter the specimen, by SE excited at the surface of the specimen by re-emerging BSE, by SE that are excited by the BSE from the surrounding objects in the specimen chamber, and by BSE that enter the detector directly. It is the last three of these that are affected by the structure that lies below the surface. The practical importance of this can be seen from the following examples from SEM service work. Fig. 1 shows an oxidised silicon wafer on which there had been deposited 0.19 $\mu m$ of Cr, 0.13 $\mu m$ of Si, and 0.15 $\mu m$ of Au. This had been heated to 300°C in He so that intermixing and segregation effects have occurred between the Si and Au layers. It was required to discover whether a surface layer of any kind had been formed over these high-Z and low-Z segregated regions. SE images obtained in a Hitachi S-450-LB SEM at beam energies of 3, 4, and 10 keV are shown in Figs. 1(a)-(c). The detector was of the type described by Everhart and Thornley (1960). The differences between these images are very striking. With the lowest beam energy, only the surface topography can be seen. As the beam energy is increased, the surface topography fades away, and the image contrast caused by the underlying structure becomes dominant. This effect is caused by a surface layer (actually of SiO$_2$) which lies over the segregated regions. In addition, the dust particles which are so evident at 3 keV can hardly be seen at 10 keV. A similar sample had been fractured and examined in cross-section by Wells and Aliotta (1979), and a low density surface layer of thickness 100 nm had been found.

Examination of Figs. 1(a) and 1(b) shows that the underlying structure is just beginning to become visible as the incident beam energy is raised from 3 keV to 4 keV. It is there-
degraded by the increased electron penetration in the target and some surface structure of these regions (Fig. 2b). At 30 keV, the BSE from the buried pole-tips give rise to SE which are then collected, and this shows both the positions and Reimer (1973; their Fig. 2) suggests that a 10 keV electron beam is broadened by ~50 nm following penetration through 100 nm of SiO₂. This is consistent with the observed sharpness of the image of the underlying structure shown in Fig. 1(c).

A second example is shown in Fig. 2. This is a thin-film recording head in which the pole-tip regions are covered by a thin surface layer. SE images obtained using a Cambridge S250 at energies of 5.1, 10 and 30 keV are shown in Fig. 2. At 10 keV, only the surface topography can be seen (Fig. 2a). At 10 keV, the BSE from the buried pole-tips give rise to SE which are then collected, and this shows both the positions and some surface structure of these regions (Fig. 2b). At 30 keV, the image is similar, except that the resolution is now degraded by the increased electron penetration in the target (Fig. 2c). When examining this sample, therefore, there is a critical energy at which the surface of the pole-tips can be seen most clearly.

Comparison pairs of SE images in which the underlying structure becomes visible as the beam energy is raised have also been published by Beaufreere (1974) and by Wolf (1974).

**ELECTRON BACKSCATTERING FROM MULTI-LAYER TARGETS**

Niedrig (1978 and 1982) has reviewed electron backscattering from thin-film and multi-layer targets. Only selected topics are considered here.

Electron backscattering from multi-layer targets (and with normal electron incidence) was investigated by Holliday and Sternglass (1955, 1957, 1959). Thin films of different materials and thicknesses were deposited onto substrates of substantially different Z. Electrons having an energy greater than 50 eV were collected over a large solid angle and were measured as a function of the incident energy. The range of electrons in the surface film for a particular beam energy was then defined as being twice the thickness of the film for which the backscattering coefficient was affected by the underlying substrate.

The values of electron range R cited below were taken from the best-fitting straight line to the curve published by Holliday and Sternglass (1959 their Fig. 5), and Kanter 1961 (his Fig. 5), for the energy range from ~1.5 to ~16 keV:

\[ R = 0.01 \times E^{1.42} \text{mg/cm}^2 \]  

or:

\[ R = 37 \times E^{1.40} \text{nm for Al} \]

where E is in keV. For energies greater than ~10 keV, the curve becomes steeper, and a more accurate relation was given by Everhart and Hoff (1971):

\[ R = 0.004 \times E^{1.35} \text{mg/cm}^2 \]  

or:

\[ R = 17 \times E^{1.75} \text{nm for Al} \]

where E is in keV. (The energy for which Eq. 1 and Eq. 3 give the same answer of \( R = 0.390 \text{mg/cm}^2 \) is E = 13.7 keV.)

**BSE IMAGE IN THE SEM**

In the SEM, the specimen can be mounted either at right angles or at an oblique angle to the incident electron beam. BSE have been detected in the SEM in the following ways:


2. With a grounded scintillator or phosphor screen which subtends a large solid angle at the specimen surface (Smith 1956; Coslett and Duncumb 1957; Wells 1957, 1970 and 1979; Everhart, Wells and Oatley 1959; Blaschke 1970; Schur, Blaschke and Pfieferkorn 1973 and 1974; Robinson 1974), or with a small solid angle (Everhart and Thornley 1960).
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Fig. 1. Multi-layer solid sample, in which a low-density surface layer lies on top of high-Z and low-Z segregated regions (see text). SE images obtained with incident beam energies of: (a) 3 keV, (b) 4 keV, and (c) 10 keV, $\theta_i = 45^\circ$. Field of view measures 5 $\mu$m from left to right.

(3) With one or more solid-state detectors (Kimoto and Hashimoto 1966; Wolf and Everhart 1969a and b; Murata 1976a and b; Hohn, Niedrig and Stuth 1976; Wells (1978).

(4) By detecting the SE from the far side of a self-supported thin film (Walker and Booker 1976).

The energy sensitivity with a scintillator or solid-state BSE detector is typically proportional to the excess energy over a threshold, which can be $\approx 2$ keV for a properly prepared scintillator (Everhart and Thornley 1960), or $\approx 3$ keV for the solid-state diode (Wells 1978).

The BSE image can be seriously affected by the collector position, both as regards the image contrasts and the information depth. An example taken from Wells (1970) is shown in Fig. 3. The sample was an Al-Zn eutectic alloy which had been heat-treated to give segregated regions, mechanically polished, and then covered with a 50 nm layer of Al. The scintillator BSE detector could be changed by means of a turret mechanism without breaking the vacuum (Wells and Bremer 1970). In the images shown, which were obtained at 10 keV incident beam energy, only the topography can be seen with the lower takeoff angle, while only the underlying structure can be seen when the takeoff angle is raised. With the low takeoff angle, the underlying structure gradually
becomes visible as the beam energy is raised from 10 to 15 keV. This is a case when the information depth is affected by both the incident electron energy and by the detector position.

Extensive studies of the escape depth in the BSE image have been published by Murata (1971 through 1976). A self-supported thin copper film was placed across a hold in a copper target, and the image contrast was measured as a function of the film thickness, the incident beam energy and the detector position. The experimental results were compared with Monte Carlo calculations. Hohn, Niedrig and Stuth (1976) measured BSE from a self-supported film target using a moveable solid-state detector. Hohn, Kindt, Niedrig and Stuth (1976) measured BSE from multi-layer targets, and discussed the information depth. Seiler (1976) measured the information depth in the SE image for a Cu film over a substrate consisting of Ag, Al, Au and Fe. (His measurements, which were made for a signal-to-background ratio of 0.01 in the recorded SE image, can be expected to be slightly smaller than measurements made by extrapolation of the curves obtained here.)

The corresponding results for the information depth in the low-loss image are as follows (Wells 1971). The sample shown in Fig. 3 was cleaned, and then recoated with an Al layer of thickness 11 nm. With 15 keV primary energy, the underlying structure became visible in the image formed by BSE with less than 800 eV energy loss. Since a low-loss image with a loss of 100 eV or less is perfectly practical, this shows that Al surface layers of thicknesses down to ~1 nm should be measurable by this method. (These low-loss results were obtained using a retarding-field energy filter together with a scintillator-photomultiplier electron detector.)

The BSE detector system that was used in the present work consists of a pair of solar cells that can be moved round the specimen to vary the takeoff angle (Fig. 4). These were type 55CL (Optical Coating Lab. Inc., City of Industry, CA 91746). Each solar cell subtends a cone measuring 22° by 22° at the surface of the specimen, and provides a current gain proportional to the excess energy above ~1 keV (Fig. 5). (In this work, only one of the solar cells was used.)

Speaking in general terms, the effect of the collector takeoff angle $\theta_2$ (as defined in Fig. 4) on the BSE image can be summarised as follows:

(1) Topographic contrast can be minimised by the use of a high takeoff angle. Thus, with normal electron incidence, topographic contrast can be minimised by arranging the BSE detector symmetrically about the beam (see, for example McMullan 1953; his Fig. 4). With an inclined sample, a critical detector position exists in the neighborhood of $\theta_2 = 90°$ when topography in the form of surface waves (but not in the form of small holes) is minimised (Fathers et al. 1973 and 1974; Schur, Blaschke and Pfefferkorn 1974). This can be achieved, for a fixed detector position, by adjusting the tilt angle of the specimen until the collected BSE current is a maximum. For a magnetised sample, type-2 magnetic contrast is usually close to the optimum with the detector in this position (Fathers 1973 and 1974; Wells 1978).

(2) Topographic contrast from small features on an otherwise flat surface can be enhanced by the use of a takeoff angle $\theta_2$ less than ~20°.

(3) In general, the information depth can be reduced by using a small value of $\theta_2$.

(4) If both $\theta_1$ and $\theta_2$ are small enough, then contrast reversal can occur, so that the heavier material is less bright in the BSE image (Wells 1970 for precipitates in a Cu-Al alloy; Reimer, Popper and Brocker 1978 for more detailed studies, including the voltage-sensitive nature of this effect). This effect is discussed in connection with an Al and Au sample below.

**EXPERIMENTAL RESULTS**

Experiments were made to determine the information depth in the BSE image as a function of the incident beam
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Fig. 3. Al-Zn eutectic alloy heat treated to give segregated regions, mechanically polished, and covered with 50 nm of Al. (a) Scintillator BSE detector with low take-off angle. (b) BSE detector with high takeoff angle. (c, d) BSE images obtained with detectors shown above. [$\theta_t = 45^\circ$. Field of view measures 15 \(\mu\)m from left to right. From Wells 1970.]

Fig. 4. Movement of solid-state BSE detectors around the sample (from Wells 1978).

Fig. 5. Energy sensitivity of solid-state BSE detector (from Wells 1978).
Fig. 6. Sample used in present work. Definitions of $\theta_1$ and $\theta_2$. See text for definitions of $\eta_A$ and $\eta_B$.

Fig. 7. Angular distribution of BSE from: "Al," "Al-on-Au," and "Au" (as defined in text). [Incident beam energy = 10 keV.]
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The signal was measured with a second digital voltmeter as the beam was scanned at (effectively) television speed in a reduced-area raster. This raster was moved back and forth across the edge of the underlying gold layer. For each experimental setting, the average was taken of twelve values of \( \eta_B/\eta_A \) as measured in this way. This ratio was plotted as a function of incident beam energy for different values of the takeoff angle (Figs. 9 and 10).

Similar (but considerably less extensive) measurements of the ratio between the corresponding secondary emission coefficients \( \delta_B \) and \( \delta_A \) were plotted as a function of the beam energy, and gave an intercept energy of \( \sim 5 \) keV. This is in substantial agreement with measurements from an Al film of very nearly 210 nm thickness over an Au substrate made by Holliday and Sternglass (1957; their Fig. 4). (The advantage of the BSE method lies in the possibility of eliminating topographic contrast.)

**Discussion**

It is clear that the ratio \( \eta_B/\eta_A \) will be unity for a low enough incident beam energy, and that it will increase in some manner if the energy exceeds a "threshold" value which must now be defined in some way. Holliday and Sternglass (1957 and 1959) plotted the absolute values of the unweighted BSE coefficient \( \eta \), and this is probably best as far as studying the process is concerned. Easier options are, however, to measure either the ratio \( \eta_B/\eta_A \) or the image contrast \( C_{BA} \) defined as:

\[
C_{BA} = \frac{\eta_B - \eta_A}{\eta_B + \eta_A}
\]

The two quantities \( \eta_B/\eta_A \) and \( C_{BA} \) are plotted as a function of incident electron energy in Fig. 9. If \( \eta_B/\eta_A < -1.2 \), then there is very little to choose between the values that are obtained. But if \( \eta_B/\eta_A > -1.2 \), then the curve for \( \eta_B/\eta_A \) is found to be essentially straight over a greater range of incident energy. This makes it easier to determine the point at which the extrapolated curve meets the zero-contrast axis. In these studies, the curves obtained by plotting \( \eta_B/\eta_A \) were used.

Fig. 10 shows the dependence of the ratio \( \eta_B/\eta_A \) on the incident electron energy of the electron and X-ray energies for comparison in Table 1. The curves with the line \( \eta_B/\eta_A = 1 \) do not change too greatly as \( \eta_B/\eta_A \) increases. However, energy intercept of these curves with the larger values of the ratio \( \eta_B/\eta_A \).

Numerical data in Table 1 for 210 nm of Al, and in Table 2 for 1100 nm of Al on the specimen shown in Fig. 6. The rows in these tables are as follows:

**Path length AB + BC.** Everhart (1960) described an electron backscattering model for normal electron incidence in which the incident electrons are assumed to travel in straight lines except for a single wide-angle scattering event. This was modified for oblique incidence by Shimizu and Shinoda (1963). The path length AB + BC as defined in Fig. 11 is the shortest distance that a BSE must travel if it is to reach the surface according to this model. The electron ranges as calculated from the energy intercepts are normalised relative to AB + BC in rows 9-11 in Tables 1 and 2.

**Threshold energies.** The three energies \( E_{1,0} \), \( E_{1,1} \), and \( E_{1,2} \) shown in rows 3-5 are defined as the intercepts of the best-fitting straight lines in Figs. 9 and 10 with \( \eta_B/\eta_A = 1.0, 1.1 \) and 1.2.

**Electron range.** The three ranges \( R_{1,0} \), \( R_{1,1} \), and \( R_{1,2} \) shown in rows 6-8 were calculated from Eq. 1 or Eq. 3 to correspond with \( E_{1,0} \), \( E_{1,1} \), and \( E_{1,2} \). These are expressed as a multiple of the appropriate value of AB + BC in rows 9-11. For \( \theta_2 \) greater than 30°, the ratio \( R_{1,0}/AB + BC \) is within 10% of unity, indicating that \( R_{1,0} \) is a useful estimate of the film thickness in that case.

A correction should be made to \( R_{1,0} \) by subtracting the range \( R_{1,0} \) corresponding to the energy threshold \( E_{1,0} \) of the BSE detector. Thus, if \( E_{1,0} = 3 \) keV, then (from Eq. 1), \( R_{1,0} = 0.047 \) mg/cm² (= 170 nm for Al).

**Information depth.** Intuitively, it would appear from Fig. 3 that the information depth should be significantly increased if the takeoff angle is raised. The comparatively minor variations in \( E_{1,0} \) with \( \theta_2 \) shown in Figs. 9 and 10 and in Tables 1 and 2 may therefore come as a surprise. However, the variations in \( E_{1,1} \) and \( E_{1,2} \) with \( \theta_2 \) are considerably greater, which shows that the depth to give a specified contribution to the image contrast varies more rapidly with the takeoff angle than does the thickness as estimated from \( E_{1,0} \).

**Conclusions**

The main conclusion from this work is that the BSE method for measuring the mass-thickness of a surface layer on a solid target is ready to be more widely applied (using the standard BSE detector systems) by means of automated techniques.

A lower limit to the surface mass-thickness that can be measured is imposed by the need to have the incident beam energy greater than the energy threshold \( E_{1,0} \) of the BSE detector by an amount that is large enough to give a measurable signal. Since the sensitivity of a solid-state detector increases gradually from zero above \( E_{1,0} \), it might be expected that the BSE detector will become adequately sensitive for BSE having an energy of several times \( E_{1,0} \). As a first approximation, it might therefore be expected that the smallest measurable film thickness will be of the same order of magnitude as the range \( R_{1,0} \). Thus, if a solid-state BSE detector can be found with a threshold energy of 1 keV, then a lower thickness limit of \( \sim 10 \mu g/cm² \) (= 40 nm of Al) might be expected. The possibility of using a converter-type BSE detector with its improved low energy response must also be considered. In the low-loss image, the energy threshold of the detector is precisely defined by a retarding-field energy filter, and thicknesses down to 0.25 \mu g/cm² (= 1 nm of Al) can be measured by adjusting this cut-off energy relative to the incident beam energy.
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Fig. 8. Sometimes the compositional contrast reverses between Au and Al (see text). Here, $\theta_1 = 30^\circ$ in all images. At 6 keV the contrast reverses: (a) $\theta_2 = 40^\circ$, (b) $\theta_2 = 50^\circ$, (c) $\theta_2 = 110^\circ$. At 10 keV it does not (d as in a, e as in b, f as in c). Field of view measures 180 $\mu$m from left to right.

Fig. 9. Ratios $\eta_B/\eta_A$, and $\frac{\eta_B - \eta_A}{\eta_B + \eta_A}$ as functions of incident electron energy for $\theta_1 = 45^\circ$, $\theta_2 = 110^\circ$ and $D = 210$ nm.

Fig. 10. Ratio $\eta_B/\eta_A$ as a function of incident electron energy (with $\theta_1 = 45^\circ$) for $\theta_2 = 110^\circ$, $45^\circ$, $33^\circ$, and $9^\circ$: (a) $D = 210$ nm, and (b) $D = 1100$ nm.

Fig. 11. The "path length" in Table 1 is defined as $AB + BC$. 
Table 1. Numerical data with $D = 210$ nm.

<table>
<thead>
<tr>
<th>$\theta_2$</th>
<th>$\theta_1 = 45^\circ$</th>
<th>$\theta_1 = 90^\circ$</th>
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<tr>
<td>in nm:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\theta_2$</td>
<td>110°</td>
<td>45°</td>
</tr>
<tr>
<td>AB + BC</td>
<td>520</td>
<td>594</td>
</tr>
<tr>
<td>in keV:</td>
<td></td>
<td></td>
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<tr>
<td>$E_{1,0}$</td>
<td>6.92</td>
<td>7.63</td>
</tr>
<tr>
<td>$E_{1,1}$</td>
<td>7.5</td>
<td>9.0</td>
</tr>
<tr>
<td>$E_{1,2}$</td>
<td>8.2</td>
<td>10.1</td>
</tr>
<tr>
<td>in nm:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$R_{1,0}$</td>
<td>550</td>
<td>636</td>
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<td>$R_{1,1}$</td>
<td>621</td>
<td>802</td>
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<tr>
<td>$R_{1,2}$</td>
<td>704</td>
<td>942</td>
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<tr>
<td>$\frac{R_{1,0}}{AB + BC}$</td>
<td>1.06</td>
<td>1.07</td>
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<td>$\frac{R_{1,1}}{AB + BC}$</td>
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<tr>
<td>$\frac{R_{1,2}}{AB + BC}$</td>
<td>1.35</td>
<td>1.59</td>
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Table 2. Numerical data with $D = 1,100$ nm.

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<th>$\theta_1 = 90^\circ$</th>
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<tbody>
<tr>
<td>in nm:</td>
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<tr>
<td>$\theta_2$</td>
<td>110°</td>
<td>45°</td>
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<tr>
<td>AB + BC</td>
<td>2730</td>
<td>3110</td>
</tr>
<tr>
<td>in keV:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$E_{1,0}$</td>
<td>19.6</td>
<td>20.1</td>
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<tr>
<td>$E_{1,1}$</td>
<td>20.0</td>
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<td>$E_{1,2}$</td>
<td>20.8</td>
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<tr>
<td>in nm:</td>
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<td></td>
</tr>
<tr>
<td>$R_{1,0}$</td>
<td>2740</td>
<td>2860</td>
</tr>
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<td>$R_{1,1}$</td>
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<td>$R_{1,2}$</td>
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<td>1.21</td>
</tr>
<tr>
<td>$\frac{R_{1,2}}{AB + BC}$</td>
<td>1.11</td>
<td>1.54</td>
</tr>
</tbody>
</table>
The measurements described in this paper were very time-consuming, involving the repeated readjustment of instrumental parameters, and a repetitive calculation, averaging and plotting of the ratios. Also, it is not easy to make measurements from an image having irregular areas of different contrast as is the case in Figs. 1 or 3. In a properly automated SEM having an image processing facility, the different regions could be identified from the peaks in the histogram of the gray levels present in the image, after which the curves could be plotted and evaluated automatically. This would therefore add another method for quantitative measurement to existing techniques.

ACKNOWLEDGMENTS

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Wells OC. (1970). New contrast mechanism for scanning electron microscope. Appl. Phys. Lett., 16, 151-153. (In Fig. 1, the words “plural scattering” should be replaced by “low takeoff angle” and “multiple scattering” by “high takeoff angle”.)


Wells OC. (1979). Effects of collector takeoff angle and energy filtering on the BSE image in the SEM. Scanning, 2, 199-216.

Wells OC and Aliotta CF. (1979). Studies of contamination buildup in the SEM using the BSE imaging technique. Scanning, 2, 257-259. (The correct sequence of the Cr, Si and Au layers is as stated in this present paper.)

