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An Anomalous Contrast in Scanning Electron Microscopy of Insulators: The Pseudo Mirror Effect.

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Summary : In a scanning electron microscope (SEM), electron-beam irradiation of insulators may induce a strong electric field, due to the trapping of charges within the specimen interaction volume. On one hand, this field modifies the trajectories of the beam of electrons subsequently entering the specimen, resulting in reduced penetration depth into the bulk specimen. On the other hand, it leads to the acceleration in the vacuum of the emitted secondary electrons and also to a strong distortion of their angular distribution. Among others, the consequences concern an anomalous contrast in the secondary electron image. This contrast is due to the so-called pseudo-mirror effect. The aim of this work is first to report the observation of this anomalous contrast and then to give an explanation of this effect and finally to discuss the factors affecting it. Practical consequences such as contrast interpretations will be highlighted.

Key words : Scanning electron microscopy; Charging; Secondary electrons; Anomalous contrast; X-Ray maps.

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Introduction

When certain insulating materials are observed in a scanning electron microscope without prior metallic coating, charging artefacts occur. The built-up electric field in the vacuum resulting from the trapped electrons in the specimen leads to an image distortion and to secondary electrons (SE) contrast dependence on the local sample charging. One of the most spectacular observations related to the negative charging is the well known mirror effect (Clarke *et al* 1970, Vallayer *et al* 1999, Wintle 1999). The mirror image is the result of a two-step process. In the first one, the sample is subjected to electron irradiation with an energy E_0, higher than its second cross-over energy E_{II}. The negative charge increases until the number of electrons emitted (secondary electrons: SE and backscattered electrons: BSE) equals the number of primary electrons and the surface potential reaches its negative saturation value V_S such that $-eV_S = E_0 - E_{II}$ (Reimer 1985). In this equilibrium situation the landing energy of primary electrons is reduced and the primary beam is in the worst case partially deflected, but still strikes the sample surface. In the second step, the negative trapped charge in the sample acts as an electrostatic mirror if the sample is irradiated by an electron beam with an energy sufficiently reduced, such that $E_0 < -eV_S$. As a consequence, a mirror image corresponding to a distorted view of the SEM chamber is obtained. Here, a curious contrast similar to that given by the mirror effect, although the primary beam energy is not lowered (in comparison to the second step), has been observed, but the image contains two contrast contributions corresponding not only to a distorted view of the SEM chamber but also to a view of the sample scan area. Unlike the mirror image the present effect shows less resolved details to such a point (especially for flat samples) that they are often assigned to a local change of SE emission. The origin of this anomalous contrast due to the so called “pseudo-mirror effect“ can not be explained by the total reflection of the incoming electron beam on the equipotential of interest.

In this paper an explanation for the formation of this contrast is proposed. This explanation is based on i) the distortion of the angular distribution of secondary electrons, which is more elongated in the direction of the external electric field and ii) also on the modification of their spectral distribution that is shifted to higher energies (the results of measurements of this distribution that have been carried out using a special spectrometer (Rau and Robinson 1996) are published elsewhere (Jbara *et al.*)). These considerations are supported by experimental results showing secondary electron images as well as characteristic X-ray maps, paying particular attention to the problems related to misinterpretation of recorded images.

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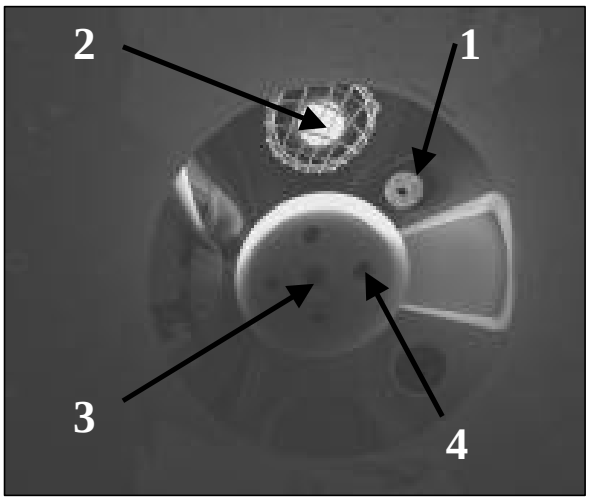
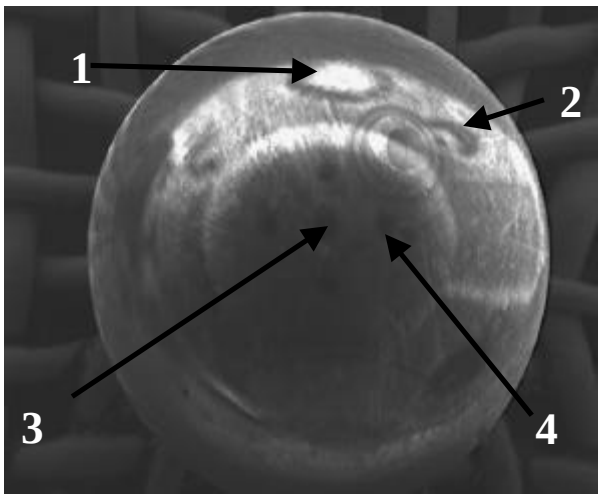
Experimental results and discussion

Observation of the effect

The results presented in this paper are carried out using a SEM (Philips 505) equipped with a standard Thornley-Everhart SE detector and with a SiLi energy dispersive spectrometer (EDS). The sample is 99.9% pure Al_2O_3 sapphire sphere of 1.5 mm diameter, negatively charged in the fast scanning mode (TV mode) by an 20 keV electron beam. When the surface potential reaches its saturation value, which corresponds to the stabilisation state of the image distortion, the acceleration voltage was reduced to 10 kV and then the sample was imaged. The recorded image, figure 1a, is a result of the mirror effect described above. The sample surface is charged so negatively (the surface potential is higher than 10 kV) that the resulting electric field is higher enough to repels the primary electrons. Consequently, the SEM inner-shell was scanned rather than the sample surface.



(a)



(b)

FIG. 1 (a) Mirror image of the scanning electron microscope (SEM) chamber obtained at a 10 kV primary electron accelerating voltage. The charging was performed at 20 kV. Details of the SEM inner-shell are clearly distinguished : (1) collimator of the SiLi detector, (2) secondary electron detector (grid and scintillator), (3) output electron gun aperture, (4) screw holes. (b) Pseudo-mirror image recorded at 20 kV primary beam accelerating voltage. Horizontal width = 1.6 mm.

The recorded micrograph presented in figure 1b, obtained at a 20 kV accelerating voltage, shows clearly a contrast similar to that given by the mirror effect, although the primary beam energy is not lowered. Some remarkable elements of the SEM chamber (the SiLi detector (1), the secondary electron detector (2), output electrons gun aperture (3) and also the surrounding screw holes (4) etc.) can be distinguished clearly . One may also note that the details in this micrograph are less resolved than in the mirror image (fig.1a).

In order to obtain the maximum spatial extent of the SEM chamber image and to see clearly the difference between the generated contrast in the two micrographs, a sample of spherical shape was chosen. Nevertheless the anomalous contrast is also obtained using a flat specimen of Al_2O_3 insulator as shown in the middle of figure 2 where, dark spots, independent of the irradiated area of the homogenous sample (the spots keep the same position when the sample is translated), clearly appear. These spots are attributed to the distorted view of the output electrons gun aperture (1) and also to the surrounding screw holes (2).

Formation of the pseudo mirror image.

The presence of negative charge in the irradiated insulator generates an electric field in the vacuum (between the negatively charged sample surface and the grounded chamber walls) which products several effects (internal and external).

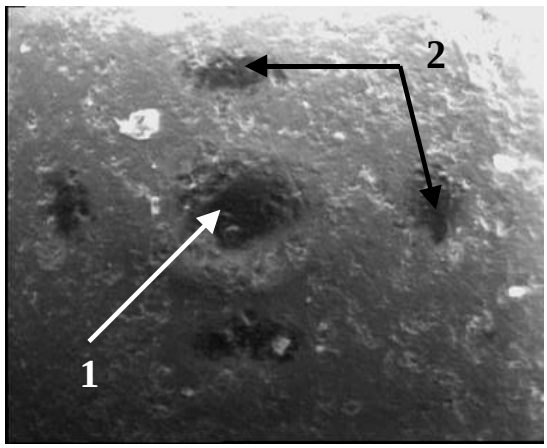


FIG. 2 Secondary electron image of an homogeneous flat Al_2O_3 sample. The dark spot in the middle corresponds to the distorted view of the last output aperture of the SEM gun (1). The screw holes are also observed (2). “horizontal width = 0.5 mm”.

Firstly the landing energy of the incident electrons is reduced leading to an enhancement of the SE yield. Secondly the electric field in the vacuum may interfere with the collection of SE. eventually, adding to these effects, the pseudo-mirror effect is simply due to the high sensitivity of the emitted SE trajectories to the built-up electric field. This field mainly affects the emitted secondary electrons in two ways:

(1) These electrons coming out the near surface region of the sample and which have energies in the range 0-50 eV are accelerated to an energy -eVs corresponding to the surface potential, as presented in figure 3a. The figure shows the measured spectral distribution of all emitted electrons from the charged Al_2O_3 sample at 20 kV primary electrons accelerating voltage

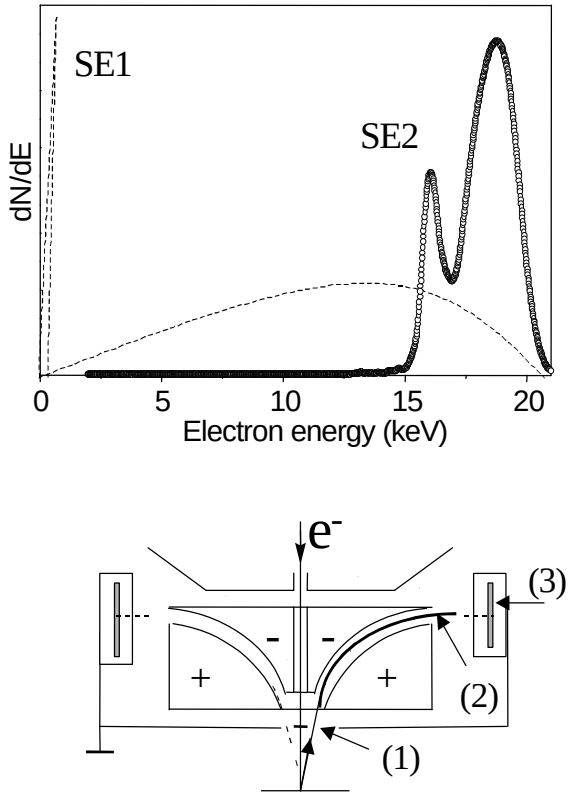


FIG. 3 (a): Comparison of energy distributions of emitted electrons. Dashed line: free from charging electron-irradiated Al_2O_3 (schematic shape). Solid line: charged electron irradiated Al_2O_3 (measured distribution at 20 kV primary electron accelerating voltage).(b): Schematic illustration of the toroidal electrostatic spectrometer. A fraction of the emitted electrons coming out in hollow cone at the angle 20° ± 1° with respect to the normal impinges at the input annular diaphragm (1). These electrons travel along the radius r_0 in the space between two toroidal electrodes, with the lower electrode at the potential +V and the upper one at –V. Only the electrons with a definite energy can come out of the analyser and through the output annular gap (2) can reach the detector (3). The detector consists of a ring made of 20 connected Si crystals with shallow p-n junctions. These semiconductors exhibit a high quantum output, which made it possible to detect electrons with energies ranging from 2 to 40 keV.

and also the schematic shape of free from charging sample distribution (dashed line). The measured electron spectrum (circles) proves indeed the acceleration of SE electrons. In fact a shift of the secondary electrons peak from its initial value, free of charging (SE1: some eV), to (SE2: 16 keV) at the entrance of the grounded apertures of the electron energy analyser, is observed. The last value corresponds to the surface potential of charged sample. The energy analyser used is a highly compact toroidal electrostatic spectrometer, specially adapted for SEM (Rau and Robinson 1996). A brief description of this spectrometer is given schematically in figure 3b.

(2) The SE angular distribution is highly distorted and points at the ceiling of the SEM chamber. This statement was corroborated using numerical simulations of the SE trajectories, taking into account the built-up electric field in the vacuum. Indeed, this field tends to bend their trajectories from the straight line, defined by their take-off emission angle, to parabolic trajectories. Consequently the secondary electrons tend to be focussed and form a spot of diameter D at the grounded SEM inner-shell. Calculations of the emitted SE trajectories has been used in order to deduce the evolution of D as function of the surface potential. In these calculations, a charge flat sample was considered as an infinite charged plane set at the negative potential Vs. The objective lens was considered as an infinite grounded conductor plane placed at d (work distance) above the sample surface. The electric field is then uniform and is $F = V_s / d$. Considering simple electrostatic and mechanics law arguments, the SE electrons trajectories in this field are parabolic and symmetric regarding the z axis as shown in the inset of figure 4. The results of trajectories calculations as a function of Vs potential are shown in figure 4.

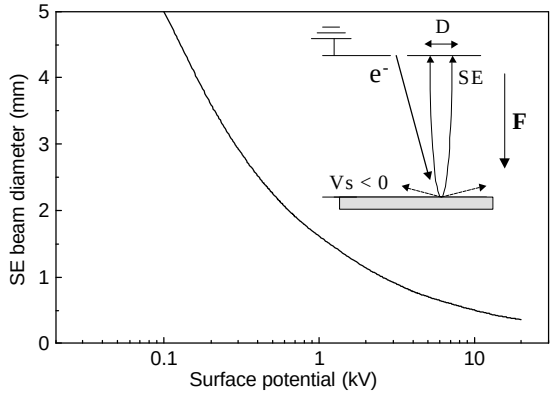


FIG. 4 Influence of the surface potential on the path of secondary electrons emitted from the charged insulator: results of simulations for uniformed charged infinite flat sample and a working distance of 10 mm.

The higher the charging magnitude, the smaller the SE spot diameter; and smaller than compared with some topographic details of the SEM chamber walls and detectors inside. Note that, SE trajectories calculations are shown here as qualitative results. Rigorous calculations must consider the finite dimension of the irradiated area and the electric field created by the positive biased SE detector as well as its spatial location.

Taking into account the above considerations (i and ii), the scanning of the SEM inner shell by the relatively high energy SE pseudo beam (2) (see fig.5) is in synchronism with the scanning of the surface sample by the primary electron beam (1).
As a result, the collected (SED) secondary electrons are a mixture of the electrons emitted from the sample (2') and of the electrons emitted from the chamber walls (3). The resulting image is a combination of the sample surface image and that of the more or less resolved pseudo-mirror.
In order to support the above explanation, additional experiments have been performed. The technique used was energy-dispersive X-ray (EDS) microanalysis and the specific measurements made were the characteristic X-ray maps of the sample and of the SEM chamber walls. In the first case, the OK line is used because sample is an oxide (Al_2O_3), while the CuK line is used in the second case because the support of the last output aperture of the electron column is made of brass. The EDS detector was disposed in manner that can detect emitted X-ray with a large solid angle as shown in figure 6d. The X-ray images simultaneously recorded at a 20 keV primary electrons accelerating voltage, are shown in figure 6 where also we present the secondary electron image, figure 6a, for comparison. Under this electron irradiation, the steady-state surface potential is about 16 kV (see spectrum of figure 3a) that is why the emitted secondary electrons impact the support of the output aperture (containing Cu), with enough energy to generate thereafter the CuK line. In figure 6b the OK X-ray map shows two areas, the dark one corresponds to a normal shadow due to the geometrical limitation of the X-ray solid angle detection, the second one corresponds to an homogeneous distribution of X-ray signal coming from the part of the irradiated sample (sphere) seen by the detector. This OK X-ray map highlights the fact that the primary beam was not mirror reflected. The Cu K X-ray map (fig.6c) generated from the electron gun shell clearly shows an inhomogeneous contrast similar to that of the secondary electron image without the presence of the typical shadow reflecting the sample geometry. Finally, this result confirms, on one hand that the secondary electrons are enough accelerated in the vacuum to generate CuK X-ray line as was underlined above and on the other hand, the angular distribution of the secondary electrons is enough distorted to produce a relatively focussed spot, able to produce an image.

Conclusion

When certain insulating materials are observed in SEM, the contrast results from the superposition of two components, one is due to the detected electrons emitted from the sample and the other is related to the electrons coming from the sample environment (electron gun shell, detectors…).
As it has been stressed in the section II, the pseudo mirror image is formed under the condition that the negative surface potential reaches an order of magnitude so great that the secondary electron are enough accelerated to be not completely attracted by the positive biased SE grid detector. Moreover, they must be sufficiently focussed to form a spot of diameter less than the SEM inner topographic details (see fig. 4).
The new developments of low voltage scanning electron microscope (LVSEM), allows the charging effects to be attenuated, by reducing the acceleration voltage to below 5 kV. In order to obtain the best operating conditions it is convenient to adjust the primary beam energy at a close value to E_{II} and to achieve a dynamic charge balance between the emitted electrons and the primary electrons. However the exact value of E_{II} was in the most cases not well known and, moreover, sensitive to the sample preparation and to the operating conditions (Cazaux 1999, Joy and Joy 1996, 1999) As a consequence, reducing the accelerating voltage reduces considerably the charging magnitude but does not guarantee the complete cancelling of this charge. Under low voltage electron irradiation the pseudo mirror effect is certainly less pronounced since the secondary electron spot is not so well focussed, but the resulting contrast is that of the sample reinforced by a constant SE signal coming from the SEM chamber walls. So it will be interesting to study the evolution and the consequences of anomalous contrast when the charging magnitude is reduced.

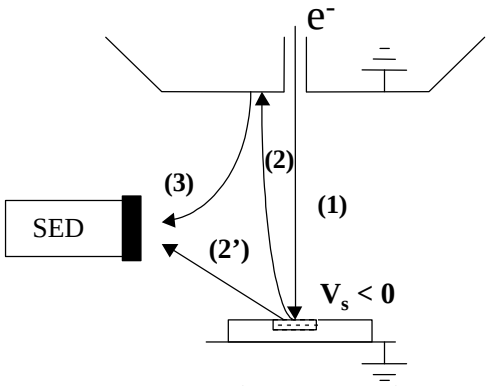


FIG. 5 Schematic drawing of the mechanism formation of the pseudo mirror image. The scanning of the SEM inner shell by the relatively high energy SE pseudo beam (2) is in synchronism with the scanning of the surface sample by the primary electron beam (1).

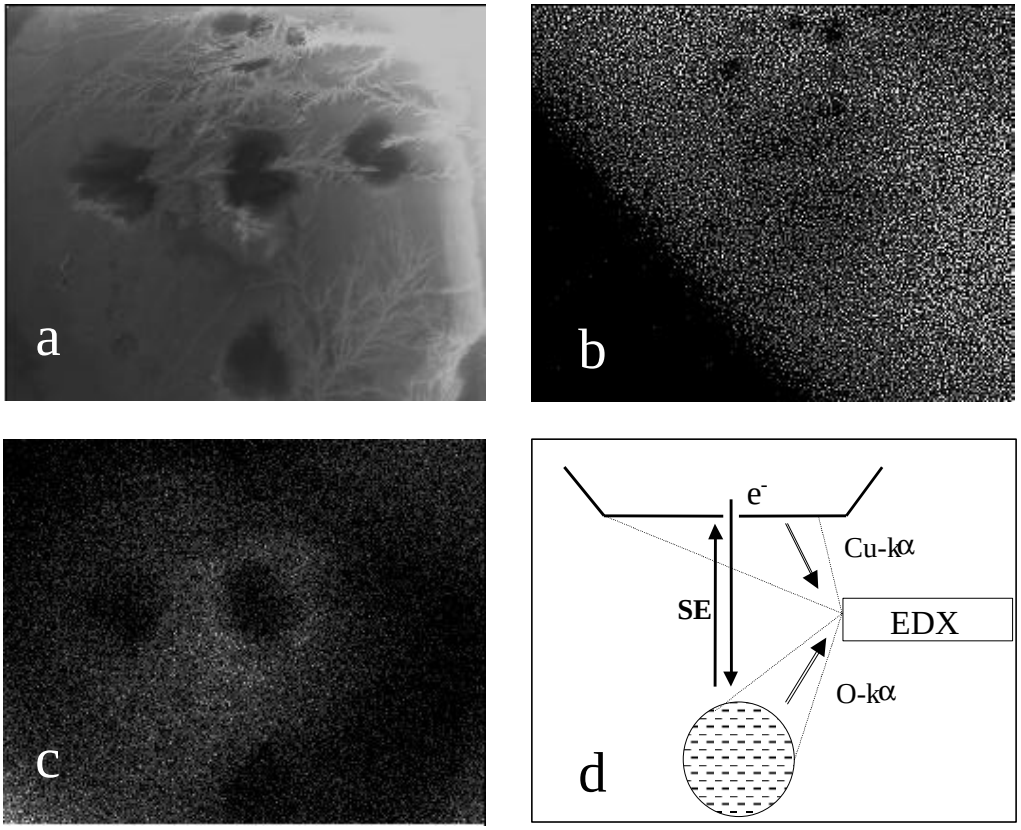


FIG. 6 Images simultaneously recorded at a primary beam energy of 20 keV for a Al_2O_3 sphere sample and the ceiling of the SEM chamber. (a) Secondary electron image of the upper part of Al_2O_3 sample. (b) and (c) X-ray maps using OK and CuK lines respectively. (d) Experimental set-up and detection geometry. Horizontal field width = 0.8 mm.

This study needs to take into account more experimental parameters such as the SE detector polarisation bias, the work distance, etc. (the work is still in progress). An other inevitable consequence of operating at low beam energies is the difficulty of performing chemical microanalysis as an adjunct to imaging. This is firstly because electron induced X-ray production demands that the beam energy exceeds the critical excitation energy for the elemental line of interest, and also because, as the energy is reduced a quite large area of the periodic table becomes inaccessible, either because no characteristic line can be fluoresced or because the available lines lies too low in energy to be efficiently detected.

From the electron probe microanalyses point of view, operating with sufficiently high primary energies, hence creating X-ray from the chamber walls by accelerated SE electrons, constitutes an impediment to elemental identification of electron-irradiated insulators especially if the sample and the chamber walls contain the same elements.

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