The Primary Energy Dependence of Backscattered Electron Images Up to 100 keV

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Abstract

The backscattered electron coefficient is known to be primarily dependent on the atomic number of the sample. If the atomic number increases, the backscattered electron coefficient increases, which results in a higher intensity in the backscattered electron image. The dependence of the primary electron energy is somewhat more complicated. Using photographic material (with composition AgBr-AgI), it is seen that the contrast in the backscattered electron image increases with the primary electron energy. Using three independent methods, based on image analysis techniques, it is shown that the difference between the backscattered electron coefficient of AgBr and AgI increases with the primary electron energy in the range from 40 to 100 keV.

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Introduction

The backscattered electron coefficient is dependent on the atomic number, the mass number, the thickness and the density of the sample, the primary electron energy, the tilt angle and the backscatter angle (Reimer, 1985). Of these parameters, the atomic number, the mass number and the density of the sample are closely related. Since, for these samples, the thickness can be considered constant, and the acquisition parameters tilt angle and backscatter angle are constant throughout the measurements, the backscattered electron coefficient is primarily dependent on the atomic number and the primary electron energy. The photographic microcrystals used in this study are Ag(Br,I) mixed crystals. They are of the tabular type with a thickness of typically about 150 nm and a diameter of 1 to 5 μm (Maskasky, 1987). Using backscattered electrons, one can visualize the distribution of the iodide over the crystal (Gao et al., 1989). Depending on the preparation of the photographic emulsion, the iodide can be distributed over one or several bands in the crystal. In these experiments the iodide is distributed over one band in the crystal. Since iodide has the highest backscattered electron coefficient, bright areas indicate high iodide content (figure 1). Energy dispersive X-ray analysis shows in an unambiguous way that the bright band in figure 1 has a high iodide content, and is not an edge effect.

Instrumentation and Sample Preparation

For the study of the backscattered electron images at different primary energies, the scanning transmission electron microscope JEOL 1200EX, equipped with a Wolf-Everhart backscattered electron detector, was used. Operating the microscope at different energies (40, 60, 80 and 100 keV) involves adjusting of the alignment at every energy. To make sure that the alignment had no influence on the results the measurements at different energies were carried out in a randomly chosen order. The backscattered electron images were transferred to a KONTRON IBAS image processing system, where image analysis was carried out. An off-line PC-AT computer, equipped with a DATA TRANSLATION DT2861 frame grabber board, was used in the calculation of the image information content. Since the photographic silverhalide microcrystals are
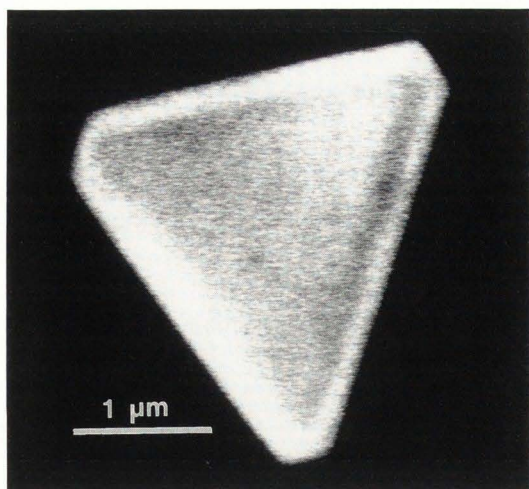
Fig. 1. Backscattered electron image of a tabular silverhalide microcrystal acquired at 100 keV, showing the iodide distribution. Since iodide has the highest backscattered electron coefficient, bright areas indicate high iodide content. Typical dimensions are a diameter of 1 to 5 μm and a thickness of 0.1 to 0.2 μm.

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sensitive to irradiation, a liquid nitrogen cooling stage was used to decrease the compositional changes and deformation of the sample. Carbon-coated Cu-grids were found to be a good supporting material for the crystals.

Measurements of the Backscattered Electron Coefficient at Low Energy

When comparing backscattered electron images of the microcrystals acquired at different primary electron energies, it is found that the images acquired at higher energies show more contrast. In the literature one finds that most of the work with backscattered electrons is being done at low electron energies up to 50 keV. Comparing the primary electron energy dependence expressions for the backscattered electron coefficient according to Love and Scott or Hunger and Küchler or with some experimental results (August and Wernisch1989), one observes the tendency that for materials with low atomic numbers (lower than 47) the value of the backscattered electron coefficient decreases with increasing primary electron energy, whereas materials with high atomic numbers show the contrary behaviour (Reed, 1975). Specifically for the AgBr-AgI crystals (with atomic numbers 41.88 and 50.18 respectively) (Danguy and Quivy, 1956), it can be expected that the difference in backscattered electron coefficient of AgI and AgBr is proportional to the primary electron energy. The difference in backscattered electron coefficient for the two constituents will be higher at 100 keV than at 10 keV. When the atomic numbers are all lower or all higher than 47, there will be no significant increase in backscattered electron contrast at higher energies.

Methods

The difference in contrast between images of silverhalide crystals acquired at 40 keV and 100 keV is easily discernible. It is rather difficult, however, to observe the differences in images acquired at the intermediate primary electron energies. Therefore, three objective methods were developed to measure the contrast differences in these images.

Grey Value Histogram Analysis

The backscattered electron images of the silverhalide microcrystals were transferred to the image processing system. After subtraction of the background intensities, caused by the carbon foil, a grey value histogram of the resulting image, containing only the crystal, was generated (figure 2). Using the histogram data of the four backscattered electron images of the same crystal, obtained at different primary electron energies, a scaling process was performed on the images in order to obtain the maximum contrast. Scaling of the image involves stretching of the old grey value range to the full range (0 to 255). Of these scaled images a new histogram was generated. In order to obtain a comparable scaling result for the four images, the scaling range limits were chosen so that the height of the resulting histograms at the extreme grey values are comparable. Since a large scale range indicates a large number of grey values in the original image, these images may contain more information than images with a smaller scale range. The maximum width attainable for the histogram of the processed image indicates the contrast of the image. The appearance of this contrast can be ascribed to the difference in backscattered electron coefficient in the constituents of the crystal.

This method offers an indication of the influence of the primary energy on the backscattered electron images (figure 3). When the primary energy increases, the scaling range increases accordingly. Therefore, there are more grey values in the images and the contrast increases. In the figure, two sets of measurements are presented, corresponding to measurements obtained from two crystals in a through-energy series. Acquisition parameters were kept constant throughout each measurement series. Since, however, the parameters can not be maintained identical between measurement series, different scale ranges are observed.

Measurement of Local Contrast on Both Sides of Phase Boundaries

This method directly measures the contrast in the images. It is however extremely user intensive as compared to the other methods.

Although the distribution of iodide in the crystal is visible in the backscattered electron images, the transition between the AgBr and AgI phases is not always clear. Moreover, due to topographic effects and noise in the backscattered electron image, it is possible even within one phase to find a large range of grey values. Moreover, the grey values of neighbouring pixels are to a certain degree correlated. So in order to measure the contrast at opposite sides of phase boundaries, it is necessary to average grey values of a number of neighbouring pixels. In this method, phase boundaries were interactively identified by drawing a line over them. Circular areas containing 129 pixels were positioned perpendicular across these phase boundaries (figure 4).
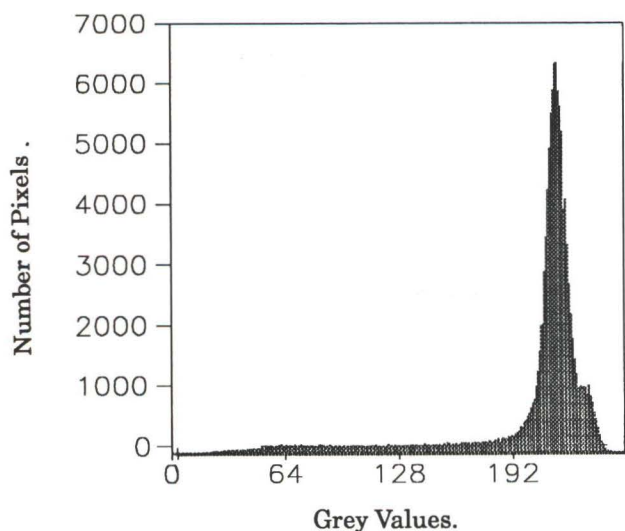
Fig. 2. The grey value histogram of the image of a microcrystal, after subtraction of the carbon foil contribution. The scale range is indicated. The grey values which lie outside the scale range are considered background.

Fig. 4. Illustration of the local contrast measurement. The phase boundaries are indicated, together with the probe areas, used in calculating the inter-phase contrast.
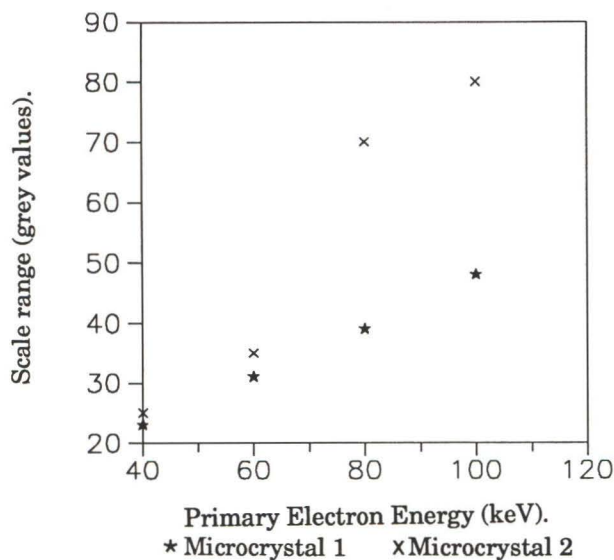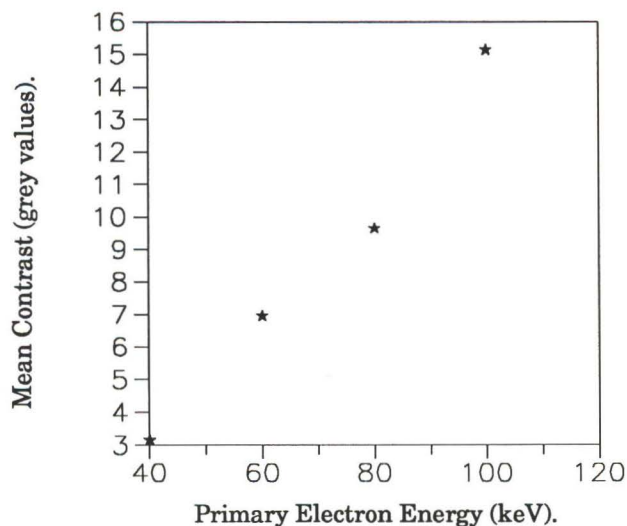

* Microcrystal 1   × Microcrystal 2

Fig. 3. Results of the grey value histogram analysis. The dependence of the scale range on primary electron energy is shown for several crystals. The scale range increases with the primary electron energy.
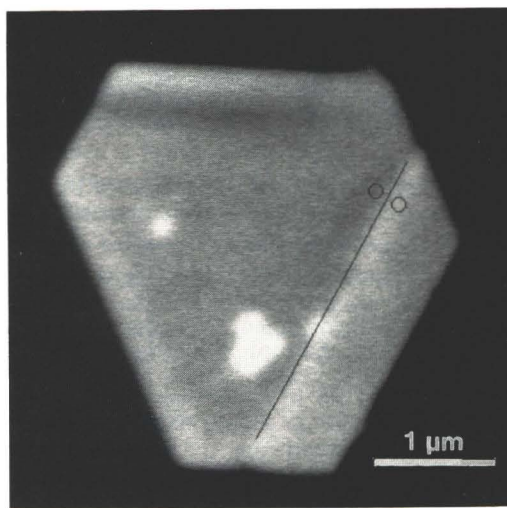

Fig. 5. Results of the local contrast measurement method. The mean inter-phase contrast of one crystal as a function of primary electron energy is shown. Each point in the graph is based on fifteen local contrast measurements.

The difference between the mean grey value of the areas situated in the AgI phase and the areas situated in the AgBr phase was defined as the inter-phase contrast. Thus the mean grey value of the two phases can be obtained. Several regions of the images were measured, yielding the mean inter-phase contrast of the crystal. The result of this method, as applied to the images acquired at the various primary electron energies, gives an idea of the inter-phase contrast as a function of the primary electron energy (figure 5). The maximum difference in grey value within one crystal increases with the primary electron energy. This result is a direct indication for the dependence of the backscattered electron coefficient on the primary electron energy. One can conclude that the effect of the primary electron energy on the backscattered electron coefficient varies significantly for different atomic numbers.
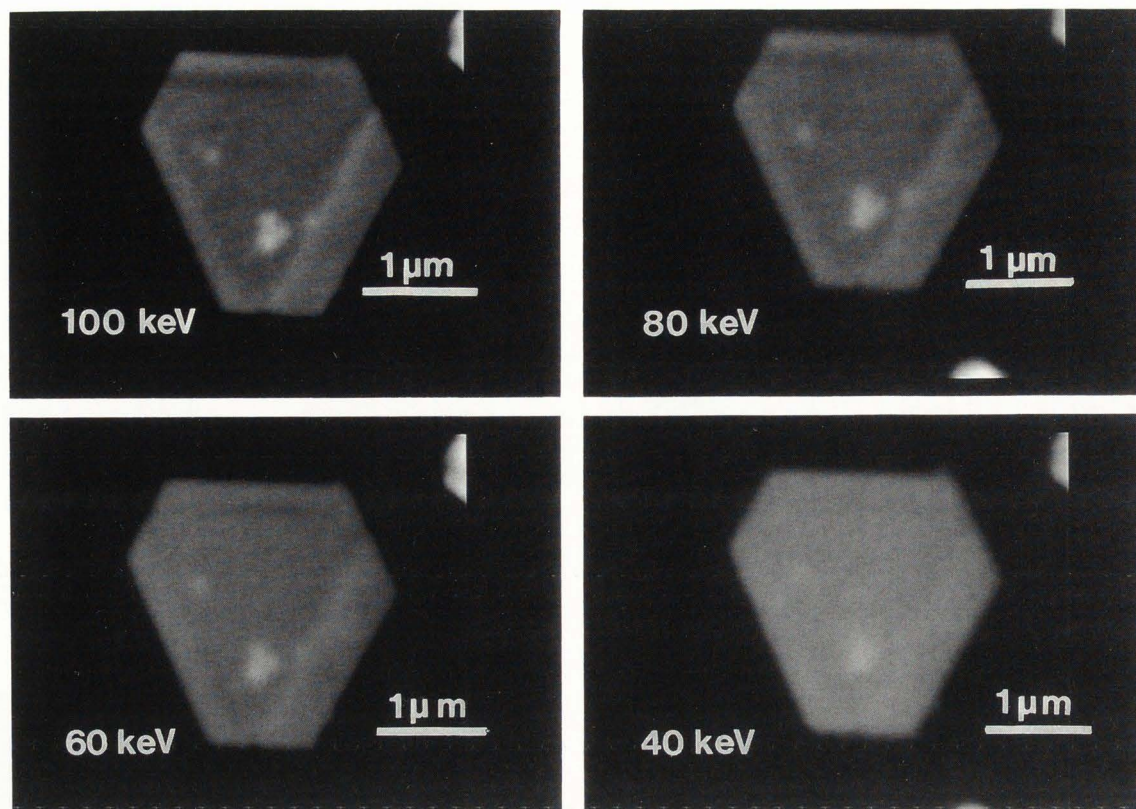
Fig. 6. Backscattered electron images of microcrystals at the various primary electron energies 100, 80, 60 and 40 keV. The information content is respectively 9.68, 8.79, 8.56 and 8.01 bits/pixel.

Calculation of the Information Content

The methods discussed are in principle not completely unbiased since they still require the operator to make manual adjustments during the analysis. Therefore, another, more objective method, was developed. This method uses the information content of the image as a parameter in determining the difference between the backscattered electron coefficients for different energies (Shannon and Weaver, 1949). The method was developed on an off-line PC-AT computer, equipped with a DATA TRANSLATION DT2861 frame grabber board. It is important to calculate the information content in the crystal only, to make sure that no background effects and contrast of image against background will influence the result. Also topographic information influences the result. The topographic information is for the most part filtered out by using a semiconductor Wolf-Everhart detector. Nevertheless, when small crystals have grown on top of the analysed crystal, topographic contrast becomes an important interference. This is one of the reasons why the information content is calculated only in a selected area of 128x128 pixels, without any topographic effects or edge effects of the crystal against the background. The selection of a subimage is also necessary to accommodate the limitations in storage and processing speed, posed by the AT computer, while still maintaining a representative part of the image for the calculation. Calculation of the information content of the pixels in the image is based on a two parameter image model, in which it is assumed that the mean and the gradient of the image are the most important carriers of the information contained in this type of images (Nys et al., 1991). Using the occurrence of all the possible combinations of mean and gradient of the 3x3 environment of each pixel, one can calculate the information content in the subimage (figure 6). The same part of the crystal was used as a subimage for the four pictures at the four different primary electron energies. It is clear that the information content increases with the primary electron energy (figure 7). More information indicates a higher contrast in the image.

Conclusion

Three different methods were shown to confirm that for the samples discussed here, the difference in backscattered electron intensities increases with primary electron energy. This results in a better contrast of the backscattered electron images at higher electron energies. However, more extensive testing of this phenomenon is necessary for other materials.
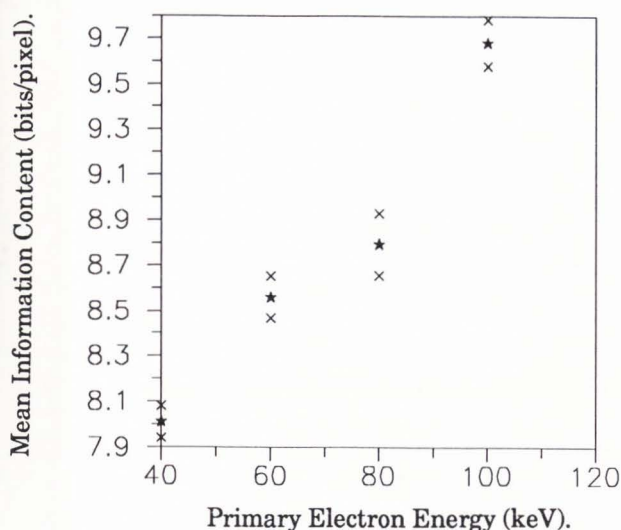
Acknowledgements

Fig. 7. Results of the information measurement method. The graph shows the mean pixel information as a function of primary electron energy. The error bars are based on the measurement of the pixel information at six locations in the crystal.

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Discussion with Reviewers

F. Hasselbach: What is the philosophy behind expressing the image information content in bits/pixel?

Authors: When one considers a noisy image the information content is low. Taking the mean and gradient images of this image, pixels with the same combination of mean and gradient values are abundant throughout the image. In analogy with the expression of numbers in a computer using bits, the information content in an image can be expressed in bits per pixel.

F. Hasselbach: Why do you not use the conventional contrast definition when measuring the local contrast on phase boundaries, i.e.,

$$C = (I_1 - I_2) / (I_1 + I_2) ?$$

Authors: When using the conventional definition, measurements at different energies give rise to different contrast values which cannot be compared since the mean intensities vary too much among images. When using absolute grey value differences, however, contrast values are comparable at different energies.

V.N.E. Robinson: There are other possible explanations for the observed image contrast. You have used the hypothesis that it is due to the increase in scattering by the higher atomic number at higher accelerating voltages. To verify that, you need to eliminate the other possibilities. These include:

a) The iodide and bromide constituents of the crystals are not uniform, and that there exists the possibility that the bromide is partially covering the iodide (or vice versa). Under these circumstances, at lower beam energy, the iodide would be masked by the bromide. As the beam energy was increased, the effect of the iodide would be greater and the contrast from the iodide would be seen to be greater with greater beam energy.

b) The crystals are thin and electrons are transmitted through them. At lower beam energies, this difference is a particular value. At the higher energies, a greater proportion of the electrons are transmitted through the lower atomic (mass) number bromide, than through the higher atomic and mass number iodide. With the stated size of your crystals being 0.1 to 0.2 μm, this effect would lead to observed increase in BSE contrast with increasing beam accelerating voltage.

Have you considered these possibilities? If so can you please comment on them, particularly the possibility that the contrast you are observing is due to the reduced scattering in the lower atomic number thin crystals than the higher atomic number ones, at increased beam accelerating voltages. In other words, this may be a beam penetration effect, not a change in differential BSE scattering with increased beam accelerating voltages, and as such, would not be present in the bulk material.

Authors: The tabular silver bromide-iodide crystals are

composed of a AgBr core and a AgBrI shell. They exhibit a characteristic two dimensional growth. It is however possible that there is a certain outgrow in the Z direction also. If so, the effect of that layer would indeed be greater at lower energy. The three dimensional structure of the crystals was examined with secondary ion mass spectrometry (SIMS). This showed that the iodide-enriched upper layer is thinner than 5 nm. The difference in contrast of the images at different primary electron energies could not be due to this very thin layer. Moreover, the backscattered electron detector measures the backscattered electrons in the complete energy range, eliminating the effect of measuring only the high-energy electrons.

In the scanning transmission images it is possible to see the phase transition between the AgBr and AgBrI phase due to the stacking faults which occur at these positions. This has nothing to do with the atomic number composition. It is however impossible to observe a contrast difference due to the atomic number difference of AgBr and AgBrI unless the core would consist of pure AgBr and the shell pure AgI. This type of crystal was not available for our experiments.