Optimization of secondary electron imaging of zeolite materials using in-lens techniques

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Zeolites have been used in many more industrial applications; the most recent one that has caused a big revolution in control of NOx reduction is as selective catalytic reduction (SCR) catalysts to control auto emissions. The special porous structure lets the zeolite material suitable for selecting the molecules of certain size. Unfortunately, this porous property coupled with a charge imbalance imposed by Al-Si oxide ratios makes the zeolite electron-beam sensitive for characterization of its structure. SEM is a primary instrument used to observe and analyze the zeolites in industry. Secondary electron microscopy provides a critical and essential way to analyze the topographic information of the material surface. The secondary electron images illustrate the surface morphology, topography, particle shape and size. Because of the sensitivity of the zeolite, the low accelerating voltage secondary electron microscopy is preferred in order to preserve its original structure and crystallinity. The adjustable accelerating voltage of the electron gun in SEM makes it possible to choose an optimal low voltage to image the zeolite. [1]

In this study, we used the Hitachi S-4800 FE SEM for imaging bare Beta zeolite and bare CHA zeolite samples to optimize imaging conditions using accelerating voltages at 1kV, 2 kV, 5 kV and 10 kV. The images are acquired at lower voltage first, and then adjusted to higher voltage. The images are acquired at the same area of the sample at the same magnification. Fig 1 shows the SE images of CHA zeolite under 1kV, 2 kV, 5 kV and 10 kV electron beam at 30 k mag. The image acquired at 2 kV gives the best resolution and surface topography. In the image acquired at 1 kV (Fig 1. (a)), a similar fine surface structure can be seen as shown in the 2 kV image. However, the image is less defined and surface lines are not visible. This could be caused by the energy spread of the electron beam at low voltage, especially more obvious at 1 kV in this study. This energy spread generates the chromatic errors at the focus and influences the resolution. In Fig 1 (c), the image acquired at 5 kV shows less surface morphological details compared to the one at 2 kV because the higher accelerating voltage makes the electron beam penetrate deeper into the zeolite bulk and generates more signals from the bulk. The larger number signals from the bulk would cover the secondary electron signals from the top surface and wash out the fine details. On the other hand, the depth of the field in 5 kV image is larger than in 2 kV as the electron probe is formed narrower. An image at 10 kV is shown in Fig 1 (d) as a comparison. It can be observed that the image at 10 kV is relatively flat and lacks surface detail while normally the higher accelerating voltage would generate an image with greater resolution. In Figure 2, the images acquired on bare Beta zeolite, which has less crystallographic habit surfaces, show a very similar trend to CHA zeolite. Image at 2 kV has a better resolution and more surface details than at 5 kV. [2, 3]

As we have discussed above, in this S-4800 FE SEM, the low accelerating voltage at 2 kV could be used as an optimal condition for imaging the zeolite particles and achieving the best resolution and surface fine structures. Moreover, the other factors like the working distance, condenser aperture size and beam current would also need to be considered but can be expected to be less so compared to the difference the accelerating voltage makes.

References:
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[2] D. Joy, Journal of Microscopy **147**, 51 (1987)
[3] D. Joy, Journal of Microscopy **140**, 283 (1985)

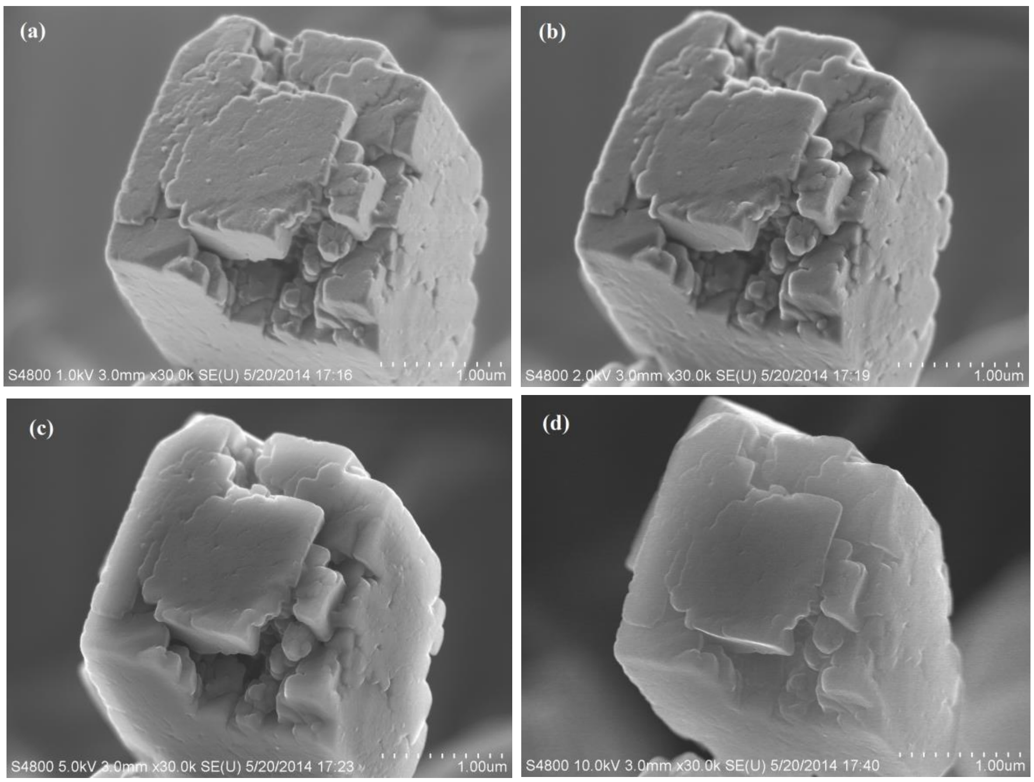

Figure.1. The Secondary Electron images of the bare CHA zeolite at (a) 1kV, (b) 2 kV, (c) 5kV, (d) 10kV.

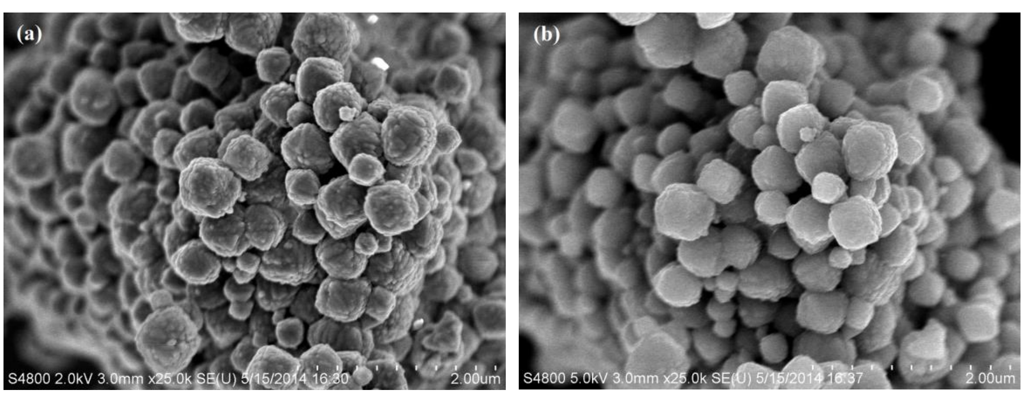

Figure.2. The Secondary Electron images of the bare Beta zeolite at (a) 2kV, (b) 5 kV.