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Imaging of Materials through Aberration Corrected STEM

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Aberration correction has caused something of a revolution in electron microscopy. In Scanning Transmission Electron Microscopy (STEM), the enhanced sensitivity and resolution have enabled the detection of single atoms [1] and the resolving of sub-Ångstrom spacings [2] on an almost routine basis. Additionally, there have been a number of unexpected benefits from aberration correction. Firstly, because the Rayleigh diffraction limit is given by: $0.61\lambda/\theta$, in order to take advantage of the reduced aberrations, the objective aperture θ must be made larger. Thus aberration correction not only allows a smaller probe size, but also admits the possibility of increased current inside that smaller probe. Secondly, this increased aperture size allows a new technique for 3-dimensional imaging [3, 4], similar to optical confocal microscopy. For a diffraction limited system, in the absence of aberrations, the depth of field is commonly defined [5] as: λ/θ^2. This is the 80% of maximum on axis criteria, so in practice the depth resolution is a little worse than this. Even in the presence of aberrations, we would expect this result to be approximately true, because the aperture is normally chosen so that it is just smaller than the angle at which the aberrations become significant.

Three-dimensional imaging is important because all real samples are three-dimensional, yet most microscope images are two-dimensional representations of a sample and it is not always possible to reconstruct a three-dimensional model from these images. In addition, the limited depth of focus will become even more relevant with improvements in corrector design, because it is already comparable to specimen thickness. Thus for many samples, the variations in height are far larger than the depth of field in a C_S-corrected system. So this is not just a way to extract new information; it will become essential to consider the reduced depth of focus in order to interpret images from any non-flat sample. Similarly, for the case of single atoms buried in an amorphous substrate, it has been shown [4] that the single atoms are not necessarily visible unless the beam is focused within the sample. Thus, even where three-dimensional information is not explicitly required, it may be essential to use this technique in order to extract the correct distribution of dopants or impurities. In aligned crystals, the electrons are attracted to the columns of atoms, which is sometimes known as channeling, so there the behavior is more complicated [6]. This also has implications for tomography. If the depth of focus is too small, then the images at different tilts are not simple projections and the approximations used to derive the tomographic reconstruction are no longer valid, in which case tomography will necessarily involve some form of deconvolution.

By reciprocity, the bright field (BF) STEM image is equivalent to the BF TEM image. The main reason that the STEM is not more widely used for BF imaging is that the efficiency is very low. Only a small fraction of the electrons that are incident upon the sample are admitted by the collector aperture and used to form the image. This is because the damping envelope for bright field imaging is [7]: $E(k)=\exp(-\pi^2 q_0{}^2|\mathrm{grad}(\chi(k)|^2)$, where q_0 is the source size (Gaussian halfwidth) in TEM and the collector aperture size in STEM. The advantage of aberration correction is clear: Correcting the aberration function $\chi(k)$ results in a decreased $|\mathrm{grad}(\chi(k)|$, thus the collection angle q_0 can be increased. Further calculations suggest that this will allow an increase of approximately a factor of

10 in the collection angle that can be used, resulting in an increase in the efficiency of approximately 100 times! This exciting result suggests that BF imaging will become more widely used in the STEM in future. The aberration corrected BF image can be recorded at the same time as the aberration corrected Z-contrast image (although the optimal defocus for both images is not the same). This will greatly enhance the usefulness of this imaging mode, by combining the sensitivity to light atoms of phase contrast imaging and the ease of interpretation of Z-contrast images.

Catalysts are one of the most exciting classes of modern materials; almost every manufacturing process uses catalysts at some stage. Z-contrast STEM provides a unique tool to analyze catalysts, which often consist of heavy metal nanoparticles on a lighter support. Results showing the characterization of supported metal nanocatalysts prepared by a new technique [8] will be presented. Our technique allows the production of gold nanoparticles on both silica (fig. 1) and alumina (fig. 2) with almost identical size distributions, with a mean ~ 2 nm diameter, which was not possible with other techniques. This example will demonstrate how characterization of materials with aberration corrected STEM can help fuel the design of new materials and catalysts.

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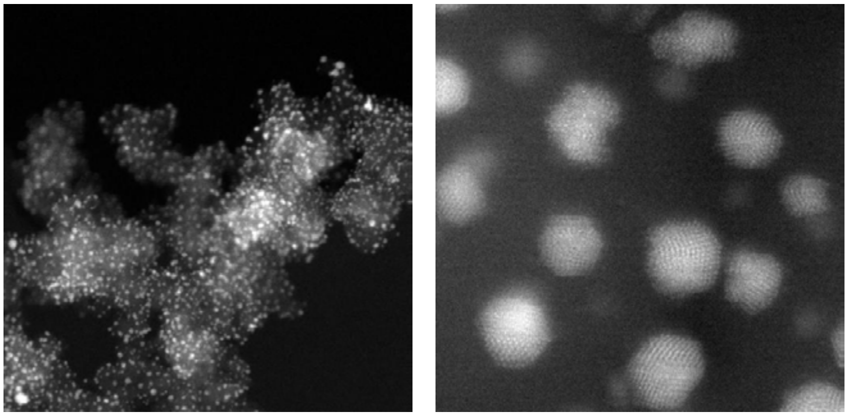

Fig. 1. Z-contrast images of gold nanoparticles on silica. At low magnification (left) the uniform distribution of nanoparticles is visible. At higher magnificiation (right) the structure of the nanoparticles can be resolved.

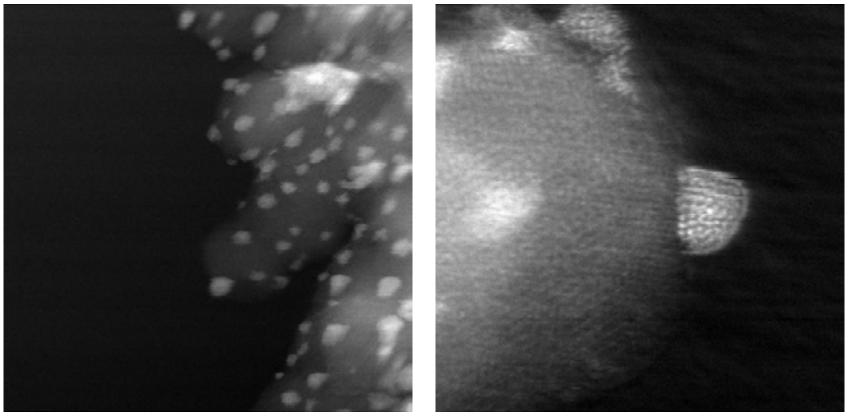

Fig. 2. Au nanoparticles on alumina. At low magnification (left) the uniform distribution and size range of nanoparticles is visible. At higher magnificiation (right) the structure of the nanoparticles can be resolved.