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Particle Size Analysis of Supported Platinum Catalysts by TEM

Particle size analysis on catalysts has been carried out for many years as the most direct way to predict the effective surface area available for catalytic activity. There are many techniques available for characterising the particle size in the range between 100 μm and 10 nm. However, only X-ray diffraction (XRD) and transmission electron microscopy (TEM) can conveniently provide information below 10 nm.

These two techniques differ significantly in their approach. XRD analysis provides information on crystallite size rather than particle size (particles could be formed of several crystallite grains). Furthermore, XRD provides an average particle size from a volume average across the whole sample, rather than specific particles. TEM, on the other hand, provides particle size analysis from individual particles observed in a transmission electron micrograph. The technique gives localised size information from the areas of sample where the images are obtained. The counting is carried out one by one manually, or on a large scale by digital particle size analysis. The results are number-averaged rather than volume-averaged. The analysis is based on 'thresholding' the intensities from each pixel of an image, and exploiting the differences in intensity between particles and the background.

Digital processing of particle images within a smooth background is fairly straightforward, provided there is a marked intensity difference between the particle and the background. However, for supported particles where the background intensity varies due to the uneven thickness and surface features of the support, it is very complicated. The human eye does a better job in this situation, because it looks at a particle within its immediate surrounding rather than

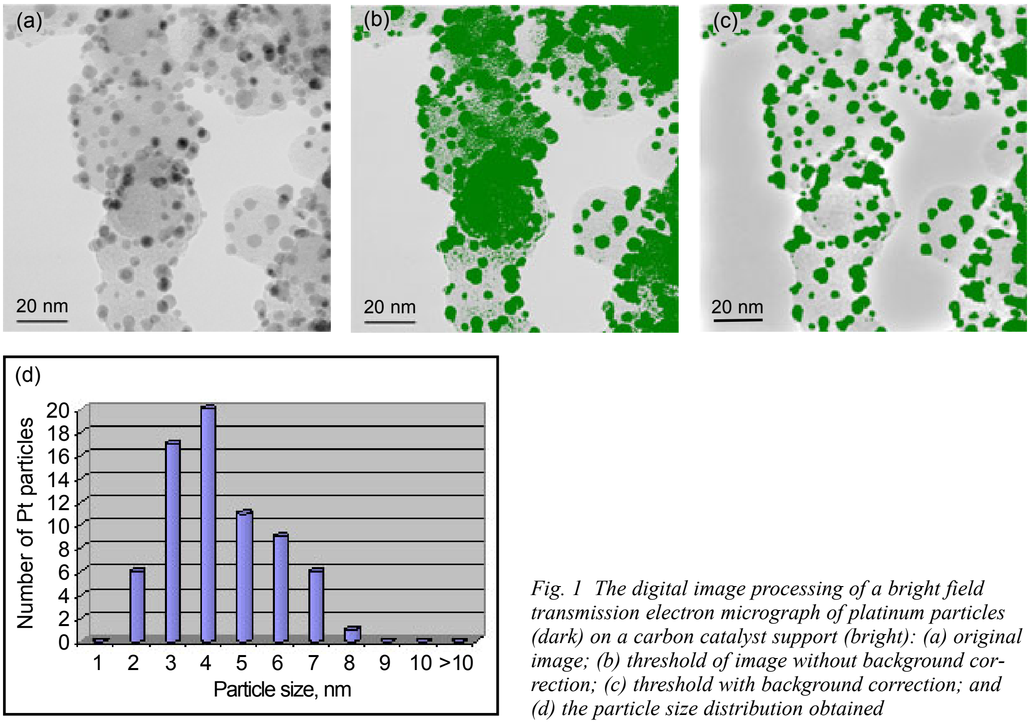

Fig. 1 The digital image processing of a bright field transmission electron micrograph of platinum particles (dark) on a carbon catalyst support (bright): (a) original image; (b) threshold of image without background correction; (c) threshold with background correction; and (d) the particle size distribution obtained

considering it as part of the whole intensity scale of the image. Digital analysis relies on absolute intensities, and if the intensity of a thick part of a support were the same as the intensity within a particle, both would be recognised as particle and/or support. The only way to resolve this conflict is to pre-process the digital image, to convert the localised intensity differences between particles and the support to absolute intensity differences which are global to the image (see Figure 1(b)).

There are a number of approaches for digitally reducing the background levels and boosting the global intensity differences between particle and support. The method used here is based on dividing the image into equivalent areas and thresholding these areas locally, in very much the same way as the human eye. Figure 1 shows the steps used in image analysis, with a view to obtaining particle size distributions using TEM. In Figure 1(a), the difference in intensity between the particles and support is due mostly to the difference in atomic number between platinum and carbon. Figure 1(c) shows that thresholding with background correction improves the detection of individual particles.

The next stage is to deselect some of these particles, especially ones that do not truly represent a full particle due to overlapping or being at the edge of the image. This is followed by calculating the statistical variables, such as the area, major and minor axes, and aspect ratio of the particle. These variables can then be plotted to give a chart showing the distribution of size *vs.* number of platinum particles, such as that shown in Figure 1(d).

The process relies on the assumption of a homogeneous distribution of particle sizes, which can be checked by moving to different parts of the sample. It is also important to remember that the particle sizes are calculated from projected volumes; although this assumption holds very well for most catalyst nanoparticles, if the particles are far from spherical (for example, rods or disks), then this assumption would not hold. In this case, a more laborious tomography routine would need to be developed.

D. OZKAYA

The Author

Dogan Ozkaya works as a Principal Analyst, responsible for electron microscopy in the Analytical Department at the Johnson Matthey Technology Centre, Sonning Common, U.K. He holds a Ph.D. in Materials Science and Metallurgy from the University of Cambridge. He carried out postdoctoral research related to electron microscopy in several university departments, including the Cavendish Laboratory, University of Cambridge, and the Materials Department, University of Oxford, before joining Johnson Matthey in 2003. He has been involved in research on platinum group metal catalysts for 12 years.