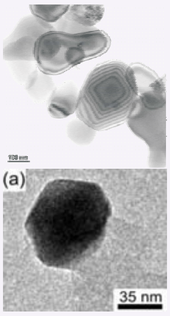
High Angle Centered Dark Field Electron Tomography: A First Example

O.G. Abrosimov,[a] A.L. Chuvilin,[b] and U. Kaiser[b]

[a]Laboratory of Structural Methods of Investigations, Boreskov Institute of Catalysis, Novosibirsk 630090, Russia
[b]Materialwissenschaftliche Elektronenmikroskopie, University of Ulm, Ulm D-89069, Germany

Motivation

Electron tomography provides information about three-dimentional substance structure, which is very important in nanomaterial research.



The suitability of conventional TEM for electron tomography in materials science is limited by the so-called projection requirement.

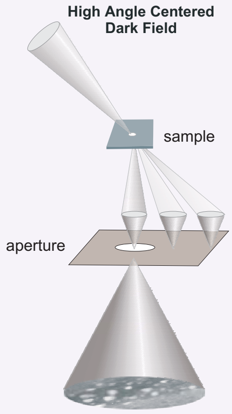
This limitation is overcome by STEM HAADF [1], EFTEM [2], and ADF TEM with Cs-corrector [3]. Recently [4], a new technique called High Angle Centered Dark Field (HACDF) was introduced, which achieves this goal without expensive STEM HAADF, FEG or Cs-corrected instruments, but instead within a conventional TEM. The resolution of this technique is about 1 nm.

In this work we investigate the use of the HACDF technique for three-dimensional reconstruction of the shape of nanosized catalytic particle.

The High Angle Centered Dark Field Technique

Technique description

In the HACDF imaging mode a conventional aperture, strongly centered on the optic axis, is used for the separation of high-angle dispersed electrons, high-angle being achieved with inclination of beam itself



Advantages

☑ In contrast to ADF TEM the absence of spherical aberrations allows to maintain resolution

☑ The resolution of HACDF technique achieved amounts to 1 nm

☑ HACDF technique, like HAADF STEM, is sensitive to atomic number (Z-contrast)

☑ An important feature of this technique is that convenient TEM instrument is enough for it, so one can be able to use the method without any need for expensive STEM HAADF, EFTEM instruments, or ADF TEM with Cs - corrector.

Electron Tomography: Equipment

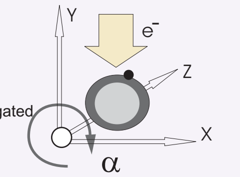
The resolution of the Electron Tomography is non isotropic

d_z = the original images's resolution

$d_x = \frac{2\pi D}{N}$, where D is a size of an area being investigated and N is a number of images in a tilt series

$d_y = d_x e_{xy}$

$e_{xy} = \sqrt{\frac{\alpha + \sin\alpha \cdot \cos\alpha}{\alpha - \sin\alpha \cdot \cos\alpha}}$, where α is a maximum tilt angle allowed

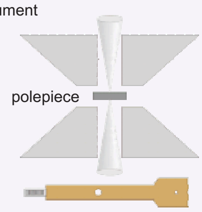


acquiring projections in high angle range $\pm 70^{o} - \pm 80^{o}$

For the standard JEOL specimen holder for the JEOL JEM2010 instrument

$\alpha \approx 40^{o}$ $e_{xy} \approx 2.1$

The loss of spatial resolution along Y axis is ~100%

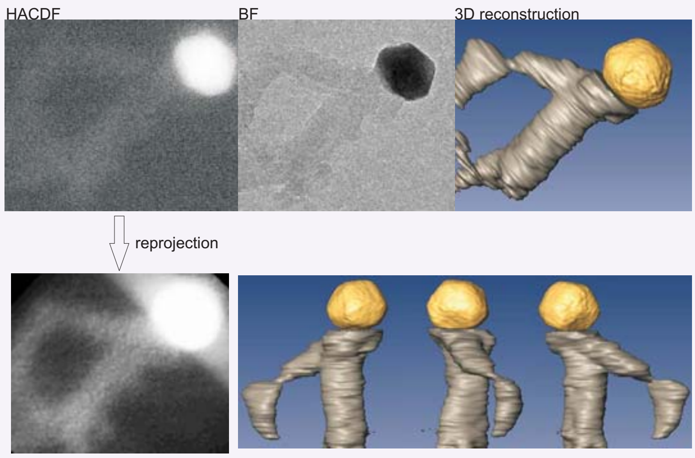


In order to improve the spatial resolution, special specimen holder and grids were developed, that allows

$\alpha \approx 80^{o}$ $e_{xy} \approx 1.13$

The loss of spatial resolution along Y axis in this case is ~13%, that is sufficient

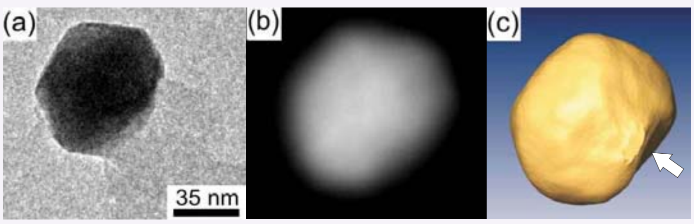
HACDF Tomography: Signal to noise ratio



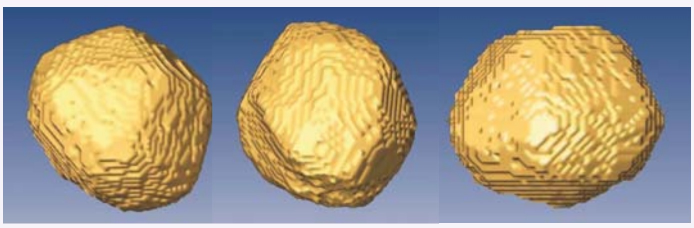
The signal to noise ratio (SNR) of the image of the carbon filament was about 3.5. Surprisingly, it was enough to successfully reconstruct the filament. The SNR of the reprojected carbon filament image was higher - about 5.6. This effect was already mentioned in other works.

Not only the shape of the metallic particle, but also the shape of the carbon filament can be reconstructed. In this case the metallic particle serves as a marker for alignment. However, intensity differences make difficulties for visualization. The empty space between the particle and the filament as seen in 3D models originates from these difficulties.

Results: NiCu particle's habitus



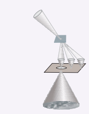
(a) The bright-field image of Cu-Ni nanoparticle with carbon filament attached. The particle is faceted well.
(b) The HACDF image of the same particle. The faceting is preserved, diffraction contrast is absent.
(c) The surface model of the reconstructed nanoparticle. In the right-bottom corner a carbon filament-corroded hole can be seen.



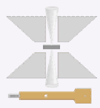
A special visualization mode reveals the crystal faces more.

The HACDF tomography technique can be used to determine the habitus of one separated nanoparticle without the use of fiducial markers.

Conclusions



The HACDF-tomography technique can be successfully used in materials science. (See also our poster MS5 N98.)



In order to improve the spatial resolution, a special specimen holder and grids were developed, that extends the available tilts range to 80 degrees.

Even for low signal to noise ratio images of nanostructures with low Z-number the HACDF-tomography technique could be successfully applied.

The shape of a Cu-Ni nanoparticle guiding catalytic filamentous carbon formation was reconstructed using the technique of HACDF-tomography. The different crystal faces are clearly seen on this reconstruction.

Aknowledgements

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