HAADF-STEM tomography of carbonitrides in steel

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Keywords: Carbonitrides, Electron Tomography, HAADF-STEM

The image observed in TEM is two-dimensional (2D), and might be treated as "a shadow" occurring in the investigated samples of spatial (three-dimensional 3D) elements. Electron tomography (ET) is an excellent method for investigation of a particle morphology (size and shape), especially for complicated shape. It is a method for generating 3D images on the basis of multiple 2D projection images of a 3D object, obtained over a wide range of viewing directions. High resolution of the details in the final reconstructed tomogram can only be achieved using very accurate alignment of the tilt series images. The classical reconstruction technique of weighted back-projection (WBP) is limited by both noise and the loss frequency (high and low) information. These effects are better dealt with by iterative techniques such as ART and SIRT; both are incorporated in Inspect3D software.

Conventional electron tomography utilises BF contrast, however for crystalline metallic specimens, BF images can be unsuitable due to non-monotonic contrast, such as that generated by diffraction or Fresnel fringes. Therefore an alternative imaging must be used. One suitable mechanism is Z-contrast imaging, generated by STEM-HAADF [1]. By using a STEM detector with a large inner radius (a HAADF detector), electrons are collected which are not Bragg-scattered. As such HAADF images show little or no diffraction effects, and their intensity is approximately proportional to Z^2. This imaging technique is ideal for tomographic reconstruction as it generates a strong contrast that has a fully monotonic relationship with specimen thickness [2].

In the present work, a shape of the carbonitrides occurring in high strength low-alloyed steel was analysed. The shape of small particles is here essential, because it has influence on physical properties of the material. Thin foils are little useful for this investigation. The BF image exhibits occurrence of defects, as dislocations and changing of extinction contours (**Figure 1**). The DF imaging may eliminate from the image other defects and extinction contours, but the images of small particles are observed only at small range of inclination. During inclining of sample, the small particles are invisible, simply disappear. The sample was preparing as a double sided extraction replica. In our investigations tilt series were acquired semi automatically at 200 kV on an FEI Tecnai G^2 microscope using FEI Xplore 3D software. Digital images of the interesting microstructures were recorded on a 1K Gatan 794 slow scan CCD camera as they were tilted from –74 deg to +74 deg at intervals of 2 deg (**Figure 2**). The tomograms were generated using Xplore 3D software. All tilted images were aligned to a common tilt axis using cross-correlation and the volume was reconstructed using weighted back projection method. The 3D visualization of reconstructed space was performed using Amira 4.3 software (**Figure 3**). In spite of the exact shift correction of the object in a relation to the rotation axis, the effect of visualization is not ideal. The improvement of the reconstruction image quality can be obtained by using the iterative methods (ART, SIRT). The further studies are in progress.

DOI: 10.3217/978-3-85125-062-6-041,

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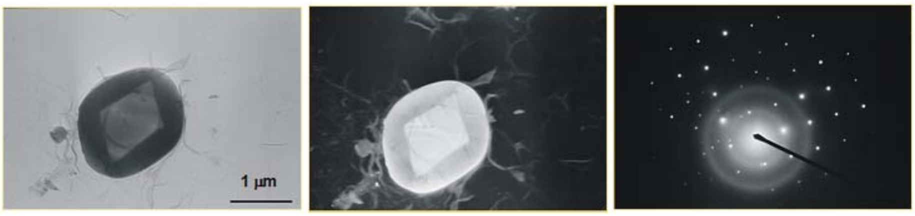
3. The financial support from the European Union under the Framework 6 program, contract for an Integrated Infrastructure Initiative, reference 026019 ESTEEM, is kindly acknowledged.
Helpful discussions with Prof. Paul A. Midgley are gratefully acknowledged.



BF **DF** **Diffraction**

Figure 1. TEM, 2D Visualization of Ti(C, N).

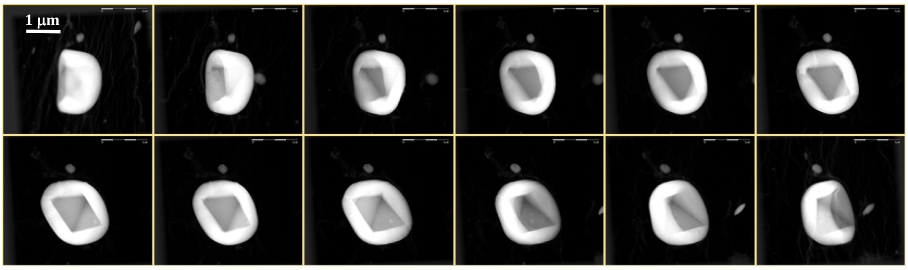


Figure 2. Acquired experimental images after axis alignment. HAADF-STEM.

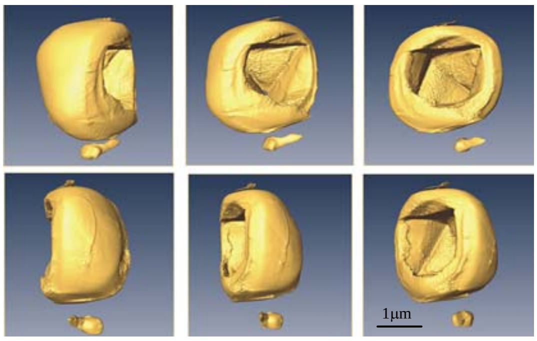


Figure 3. 3D Visualization of the carbonitride particle after reconstruction and 3D rendering.

G. Kothleitner, M. Leisch (Eds.): MC2009, Vol. 1: Instrumentation and Methodology,
DOI: 10.3217/978-3-85125-062-6-041, © Verlag der TU Graz 2009