Mapping Grain Boundary Potentials in Ceramics by Nonlinear Inline Electron Holography and Impedance Spectroscopy

C.T. Koch[1], B. Rahmati[1], W. Sigle[1], M. Shirpour[2], R. Merkle[2], J. Maier[2], and P.A. van Aken[1]

1. Stuttgart Center for Electron Microscopy, Max Planck Institute for Metals Research, Heisenbergstraße 3, D-70569 Stuttgart, Germany
2. Max Planck Institute for Solid State Research, Heisenbergstraße 1, D-70569 Stuttgart, Germany

koch@mf.mpg.de
Keywords: inline holography, mean inner potential, focal series reconstruction, non-linear imaging theory, impedance spectroscopy

The electrostatic potential arising from charge bound at grain boundary cores in ceramics and the accumulation of space charge in their vicinity is in many cases made responsible for the ion blocking or conducting behavior of electroceramics. While interpretation of impedance spectra of nominally undoped and acceptor-doped $SrTiO_3$ ceramics and bicrystals implies that grain boundaries are positively charged and accompanied by a fairly wide region of negative space charge on both sides, a critical analysis of off-axis and inline electron holography data available in the literature yields very narrow potential profiles of the opposite sign.

Some of the advantages of inline holography over off-axis holography are: (a) very simple experimental setup (works in any FEG-TEM, and for small details even with a LaB_6 source), (b) possibility to record holograms far away from the specimen edge, (c) large fields of view because there is no need to oversample, and (d) specimen drift may easily be compensated, even during exposure. We will report on the application of a recently developed flux-preserving inline holography reconstruction algorithm [1] which allows the reconstruction of focal series recorded over a large focal range. Fig. 1 shows the (amplified) image distortions of the 15 images within a focal series of a near Σ13 grain boundary in $SrTiO_3$. These image distortions are a side effect of changing the objective lens current by a large amount (the defocus ranged from -16µm to +13µm) and have been determined and corrected for by the reconstruction algorithm. In addition to fitting and correcting relative image distortions the reconstruction algorithm also refines the relative defocus and translation of each image in the course of the reconstruction.

The reconstructed phase maps are shown in Figure 2. A double-tilt rotation holder has been used during the experiment. The grain boundary could therefore be aligned with the axis of the specimen holder. Rotating the holder by 18° therefore allowed a tilted projection of the grain boundary projection to be obtained (Fig. 2b). Using the local specimen thickness extracted from Fig. 2b the phase shift (Fig 2a) could be converted into a map of the mean inner potential (Fig. 3).

Inline holography results of various bicrystal geometries processed under varying oxygen partial pressures will be presented and will be correlated with grain boundary potential profiles fitted to impedance spectroscopy data as well as analytical TEM data.

1. C.T. Koch, "A flux-preserving inline electron holography reconstruction algorithm for illumination of partial spatial coherence", Ultramicroscopy 108, 141-150 (2008).
2. Financial support from the European Commission under contract nr. NMP3-CT-2005-013862 (INCEMS)

DOI: 10.3217/978-3-85125-062-6-537 , © Verlag der TU Graz 2009

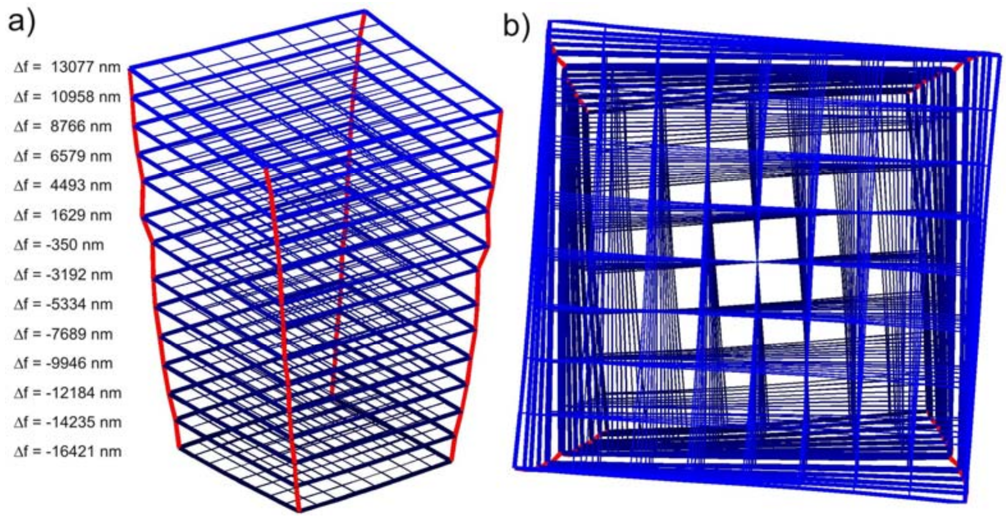

Figure 1. Defocus-induced image distortions enhanced 5 times. a) Relative image distortions (x5) that have been corrected for at each of the focal planes. b) Alternative (2D) representation (top view) of the plot shown in a).

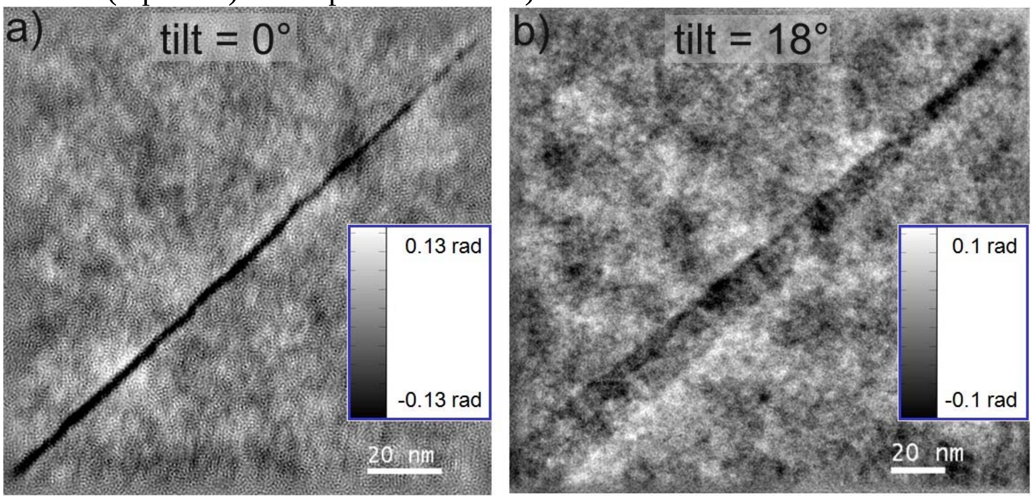

Figure 2. Phase reconstructed from a focal series of Fresnel contrast images of a near Σ13 grain boundary in $SrTiO_3$. a) Phase map of the grain boundary viewed ‘edge-on’. b) Phase map of the grain boundary tilted by 18°. This second projection was used to normalize the projected potential w.r.t. specimen thickness.

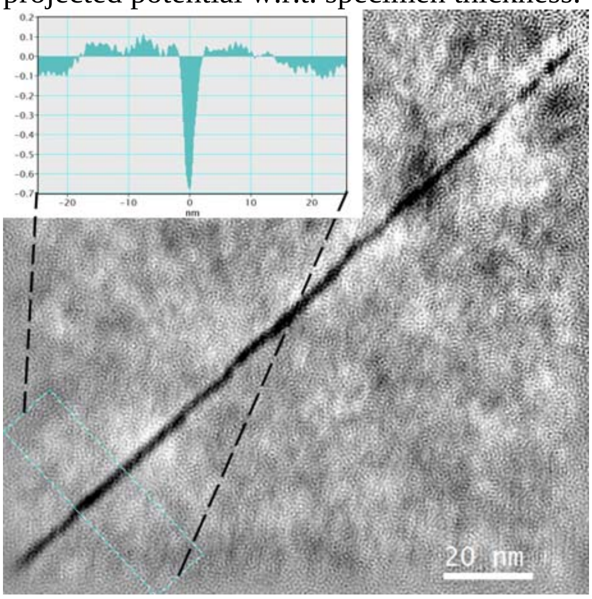

Figure 3. Thickness-normalized mean inner potential reconstructed from the phase map shown in Fig. 2a. The inset shows a line scan across the grain boundary. The slow variation in the mean inner potential hints at the positive potential of the grain boundary. The sharp dark grain boundary core is caused by local changes in stoichiometry.

W. Grogger, F. Hofer, P. Pölt (Eds.): MC2009, Vol. 3: Materials Science,
DOI: 10.3217/978-3-85125-062-6-537 , © Verlag der TU Graz 2009