WHAT ARE THE OPTIMAL CONDITIONS FOR THE DETECTION OF A SMALL PORE BY HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY?

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Introduction

Pores are an important microstructural feature that can degrade material properties in various ways: pores can degrade the mechanical properties or degrade the optical properties by scattering. Alternatively, pores can be used to prevent conduction or engineer the dielectric properties of materials. The detection and characterization of pores is therefore important. However, at the nanometer length-scale the detection and characterization of pores can be challenging.

The goals of this work is to evaluate the optimal working conditions of aberration corrected high resolution transmission electron microscope (HRTEM) for detection of nanometric-sized closed pores.

Methodology

In order to simulate aberration corrected HRTEM images of yttria-stabilized cubic zirconia (YSZ), a YSZ crystal with a thickness of 11.7nm was made using Crystal Maker software. Using the software, a surface roughness of ~2Å was generated.

MATLAB was used to create a closed spherical pore in the YSZ crystal, by removing clusters of atoms with varying radii (4-15Å).

A simulated defocus series for each pore diameter was calculated using EMS (electron microscopy simulations) software [1] based on the multi-slice method [2] at 300 keV and $C_s = 3\mu m$.

Results and Discussion

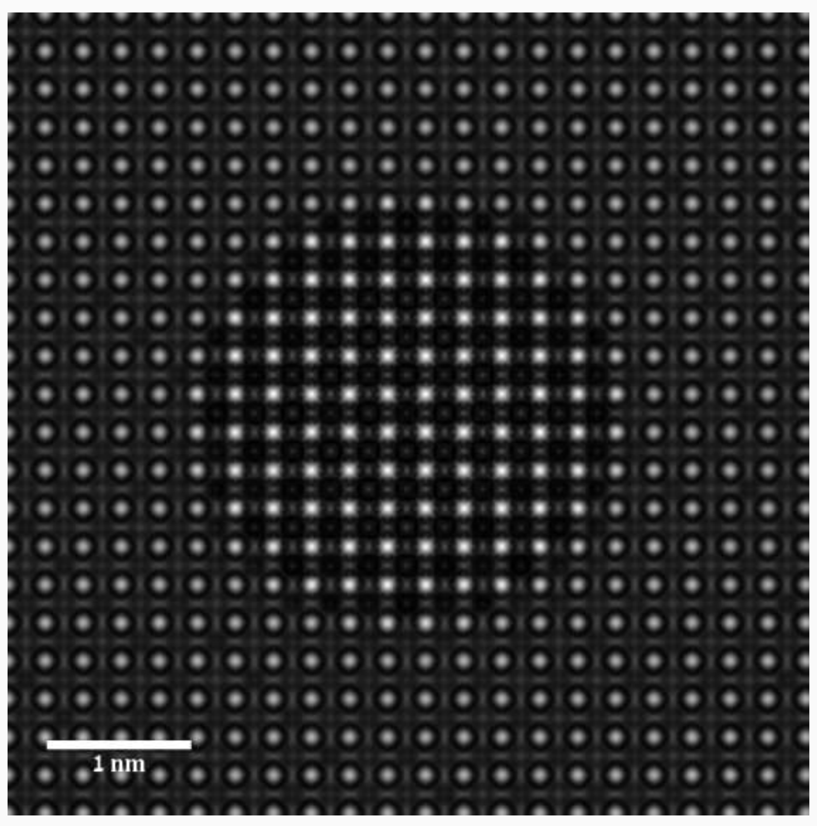

Figure 1: Simulated image of a YSZ [100] crystal with a spherical pore ($r = 15$Å) at an overfocus of 3nm.

Simulated images of YSZ with nanometric sized pores (for example figure 1) shows that the inclusion of a pore changes the image intensity. The change in intensity in the region of the pore varies with defocus of the objective lens and pore diameter.

A comparative analysis of three different metrics was conducted for a unit cell in the column of the pore and far away from the pore (i.e. bulk): Normalized Euclidian Distance (NED) [3], Cross Correlation Coefficient (XCC) and the normalized intensity ratio (R) (figure 2).

At smaller pore dimensions and across all defocus values the intensity ratio and the XCC are close to 1, while the NED is close to 0 which means that the detection of a nanometric pore at that size is harder as the changes to the intensity caused by the pore are very small. For the most part the detection of a pore with $d < 12$Å is the most difficult across all 3 parameters.

For larger pores (d>18Å) the detection of the pore using NED (figure 2b) is preferred across most defocus values, while using XCC (figure 2c) as a metric is preferred at lower defocus values and R (figure 2a) for higher overfocus values.

The cross-correlation coefficient (figure 2c) and the NED (figure 2b) at lower defocus values provides more sensitivity for detection than the normalized intensity ratio R (figure 2a) for larger pores ($d > 18$Å).

Regardless of pore detection, pore size measurements would also depend on contrast delocalization [2]. Delocalization results in image details being displaced from their true locations in the image plane. The delocalization effect is demonstrated in figure 3 for an open pore in YSZ.

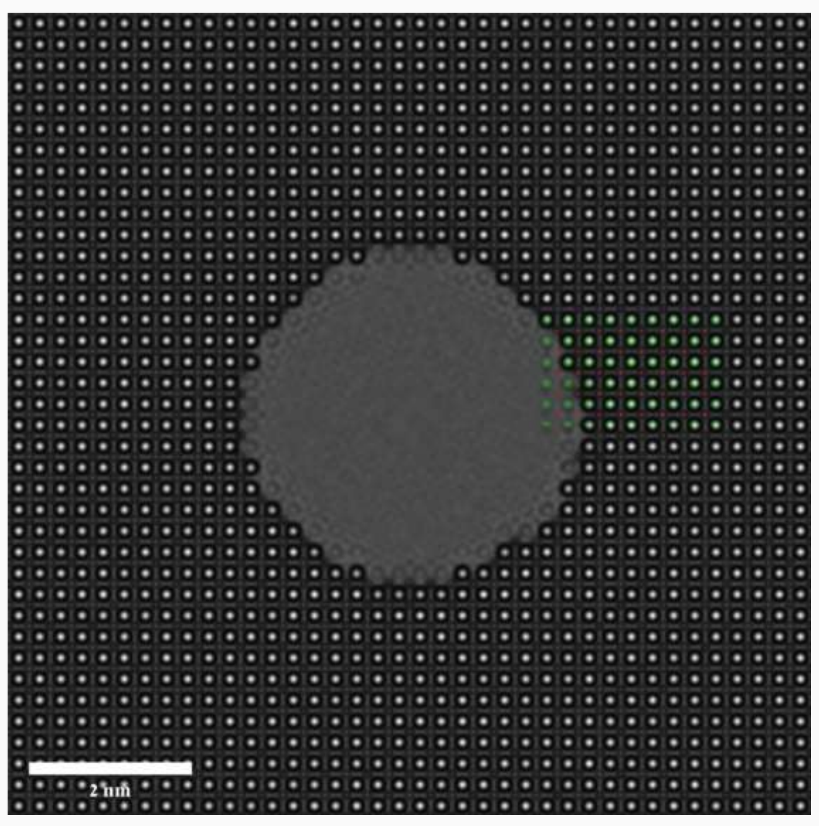

Figure 3: Delocalization effect in simulated YSZ [100] at an overfocus of 3nm.

The maximum delocalization distance is:

$$R(g) = \Delta f \cdot \lambda \cdot g + C_s \lambda^3 g^3$$

where Δf is the objective lens defocus, C_s the spherical aberration coefficient, λ the wavelength and $\boldsymbol{g}$ the reciprocal diffraction vector.

The maximum delocalization for the conditions here reach 8 Å. The maximum delocalization distance can reach the scale of the pore dimensions for smaller pores and therefore can effectively hinder detection and make pore size measurements from raw data impossible.

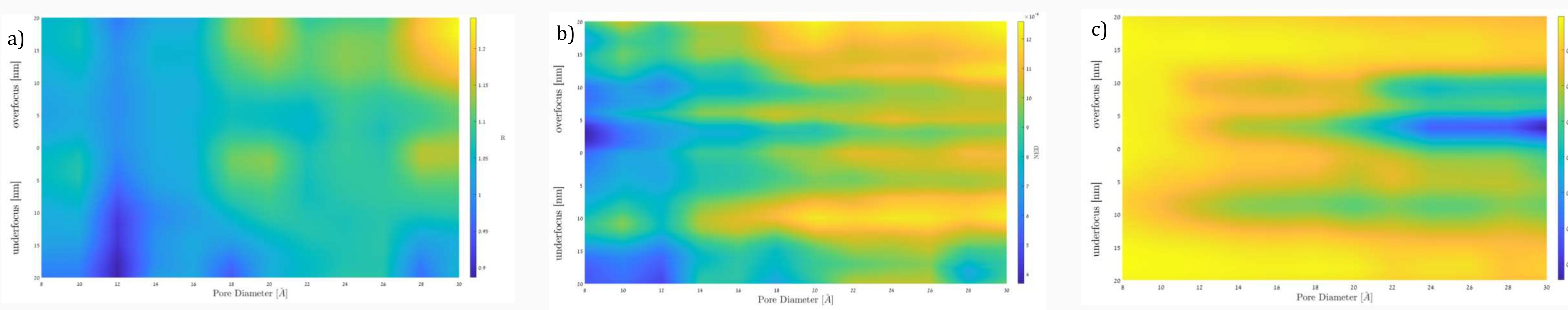

Figure 2: The 3 different metrics for a region in the sample containing a pore and a region in the same sample not containing a pore, as a function of pore diameter and objective lens defocus: a) normalized intensity ratio ($R = \frac{\Sigma I_p}{\Sigma I_b}$), b) NED, and c) XCC.

Summary & Conclusions

- Using aberration corrected HRTEM simulations we explored the influence of nanometric sized pores in a thin specimen on image contrast and evaluated the working conditions for pore detection with 3 different metrics.
- The contrast delocalization phenomenon can prevent pore detection and characterization.

References:

[1] Stadelmann, P.A. "Ems - a Software Package for Electron Diffraction Analysis and HREM Image Simulation in Materials Science." Ultramicroscopy, vol. 21, no. 2, 1987, pp. 131–145., doi:10.1016/0304-3991(87)90080-5.

[2] D. B. Williams, C. B. Carter. Transmission Electron Microscopy. New York: Springer, 2009.

[3] Di Gesu V, Starovoitov V. Distance-based functions for image comparison. Pattern Recogn Lett 1999;20:207– 14.