# Measuring the Size and Distribution of NiO Particles in individual SBA-15 Pores by Electron Tomography.

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## Introduction

The development of new catalyst characterization procedures is one important factor for the design and fabrication of more efficient catalysts. In particular, size, location, distribution, and accessibility of the active phase within its support are of major interest in heterogeneous catalysis. In this respect, electron tomography (ET) has shown to be a versatile tool because of its unique imaging capabilities that provide detailed three-dimensional (3D) information on the nanometer scale [1]. Studies combining ET with posteriori image processing prove that a transition from qualitative to quantitative assessments is possible [2,3]. Here we present for the first time results on visualization and quantification of the NiO particle distribution in individual pores of an SBA-15 support material.

## Materials and Methods

The sample of 24 wt% NiO in SBA-15 was synthesized by incipient wetness impregnation of SBA-15 followed by a drying and a calcination treatment. A detailed description of the procedure is given in an accompanying abstract [4]. ET was performed in bright-field TEM mode using a Tecnai 20 electron microscope. The images were recorded with Xplore3D software (FEI) over a tilt range of at least -70 to + 70 degrees at 1 degree increments. Alignment and reconstruction by filtered backprojection was performed in IMOD [5]. Image segmentation was carried out in Amira 3.1 (Mercury Computer Systems, Inc.) by manually tracing the NiO particle contours throughout the volume of the examined pores. Finally the volume and the center-of-mass were calculated for each NiO particle. To complement and verify nanoscale observations $N_2$ physisorption and XRD were applied.

## Results and Discussion

In Figure 1A a numerical cross-section through a reconstruction of the NiO/SBA-15 is displayed. As reported earlier for another metal oxide / SBA-15 material [2], not all pores of the support were occupied by NiO crystals. In return, filled pores seemed to be heavily occupied. In this particular structure only 43 % of the pores were filled. A statistical analysis of 299 NiO volumes shows that mainly NiO crystals below 50 $nm^3$ were present. For comparison with XRD, the size of each NiO crystal was estimated by its equivalent diameter, i.e. the diameter of a sphere having the same volume. Although the crystals did not appear to be spherical a close match between the volume weighted distribution maximum at 4 nm to the diameter obtained by XRD was found. The observation that particles at around 4 nm diameter were present was also supported by $N_2$- physisorption data (not shown) and the inferred absence of mesopore blocking.

The distribution of particles along a 134 nm pore is shown in Figure 1B and C. In general a homogeneous distribution of the number of NiO particles was observed. This could be confirmed by a statistical analysis of the 3D distances between neighboring NiO particles. The nearest, second, and third nearest neighbors were separated by 4.6±0.9 nm, 5.9±1.8 nm, and 7.1±2.9 nm (mean±std), respectively.

In Figure 1D the calculated NiO loading for 10 consecutive pore segments is displayed. It is apparent that a local loading much higher than the applied 24 wt% was found and that variations along the pore existed. This observation is in line with the large percentage of unfilled pores that arguably compensate for the high local loading. This suggests that a high loading with good dispersion is achievable if the percentage of filled pores can be increased.

## Significance

ET in combination with image segmentation was successfully applied to study the size, 3D location, distribution, and local loading of NiO crystals inside SBA-15. To our knowledge, this is the first application of ET to measure these properties on the level of individual mesopores.

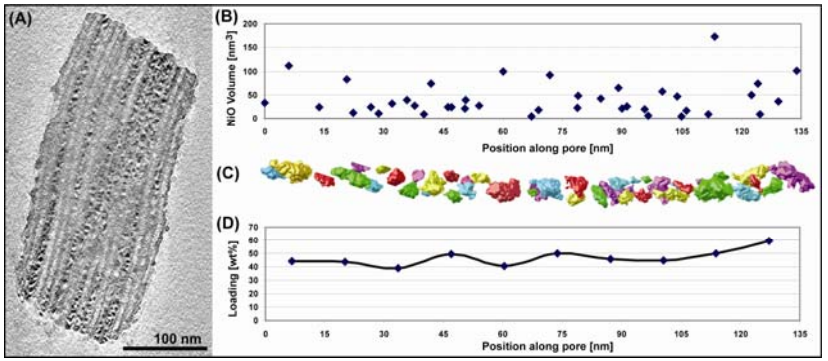


**Figure 1.** 0.54 nm thick numerical cross-section through NiO/SBA-15 (A), properties of 134 nm long SBA-15 pore occupied by NiO crystals: NiO particle volume vs. position along the pore (B), surface view of the NiO particles (C), and local loading of NiO calculated for 10 successive pore segments (D).

## References

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