

Determining particle size of NiO nanoparticles

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Introduction

Bulk NiO is an antiferromagnetic material, but in 1956 Richardson and Milligan [1] reported that NiO exhibit anomalous effects such as ferromagnetic behavior when the particle size is less than 100 nm. Néel proposed [2] that the permanent moment of nanoscale particles is due to an incomplete magnetic compensation between moments from atoms A and B situated on different sublattices. In NiO each [111] plane has a magnetic moment in opposite direction to the neighboring [111] plane. If the number of atomic layers is even the spin of the layers cancel each other and the particle shows no magnetic moment. If the number of atomic layers is odd all layers except one cancel each other, and the magnetic moment of the particle is approximately equal to the magnetic moment of this last uncompensated atomic layer. For large particles the number of atoms and hence the number of layers is very big so no difference is observable, thus this effect is only seen in nanoparticles.

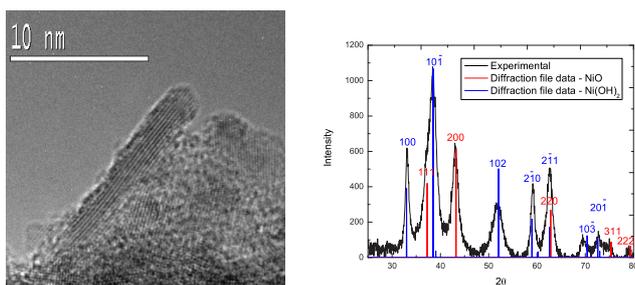


Figure 1: TEM micrograph and XRD spectrum of NiO.

Investigation

This effect has been studied at the Nanostructured Materials Group at DTU. Naturally the size and shape of the particles are of interest and therefore many samples have been examined by X-ray diffraction as the line width in the spectrum is dependent on average particle dimensions [3]. In this way we could very well determine the dimensions of the Ni(OH)₂ precursor particles which have a hexagonal close packed crystal structure, the particle dimensions were determined to be around 3x15x15 nm. However, NiO has a simple cubic structure and thus it is not possible to determine directional dimensions by X-ray diffraction. NiO is obtained from Ni(OH)₂ by thermal annealing so it is expected that the NiO particles inherit the shape of the precursor particles. In order to investigate this Transmission Electron Microscopy images were obtained with the JEOL 3000F TEM at Risø National Laboratory as seen in Figure 1. These images did reveal some information about the particle dimensions of our samples, but still some information was missing, thus the samples were investigated with STM and AFM at the newly opened Nanotek.

In order to study the NiO particles with STM and AFM a

small amount of material was suspended in ethanol. This was subsequently treated with ultrasound for about 20 minutes to ensure that all particles had separated and did not lie in big clusters. A small drop was then put on a HOPG crystal with a pipette. Theoretically STM can only be used on conducting materials and in this case the material to be probed is an oxide, but due to the small particle size some electrical conductance should remain.

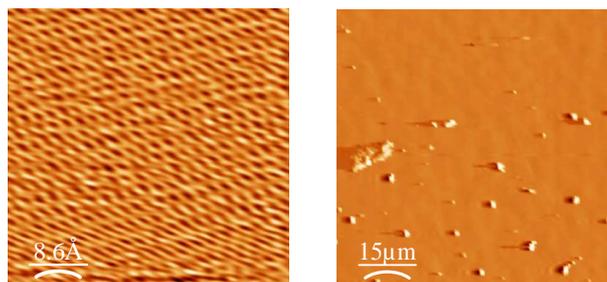


Figure 2: STM image of HOPG surface and AFM image of NiO particle clusters.

However sadly we were not able to see any particles with STM. Many areas of the sample were examined but nowhere did any NiO particles turn up. All we were able to get from STM was an image of the atomic ordering of the HOPG crystal itself as seen in Figure 2. In order to find out why we were unable to see NiO particles we turned to the AFM. An AFM image is also seen in Figure 2. It tells us why we are not able to see the NiO nanoparticles. They appear to be gathered in big clusters and not dispersed as single particles as hoped. Thus they are not observable with STM and AFM does not have the resolution needed for our investigation. A small problem encountered during the process was that the AFM tip would simply move the particles around on the surface. The failure to observe singly dispersed particles can very well rely in our sample preparation. As stated the sample was treated with ultrasound to separate the particles, perhaps not enough ultrasound treatment was given. When time allows it this theory will be tested in a new attempt to observe NiO nanoparticles with STM.

References

- [1] J. T. Richardson and W. O. Milligan, "Magnetic properties of colloidal nickelous oxide," *Phys. Rev.* **102**(5), 1289–1294 (1956).
- [2] L. Néel, *Low Temperature Physics*, Gordon and Beach, New York (1962).
- [3] L. Gerward, *X-ray analytical methods*, Technical University of Denmark, Department of Physics (2001).