Investigations of the antiferromagnetic phase of YBCO nano-particles

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Abstract

Undoped bulk $YBa_2Cu_3O_{6+x}$ (YBCO) is antiferromagnetically ordered with a Néel temperature of approximately 410K. Doped bulk YBCO becomes a high T_C superconductor at doping levels $x > 0.35$. In our studies we investigate nano-sized particles of YBCO and thereby use the particle size as a new control parameter to gain additional insight into the physics of YBCO, where there central question is whether the magnetic phase and the superconducting phase are competing or coexisting phases.

We have investigated the antiferromagnetic phase diagram in YBCO nanoparticles with mean diameter of 40 nm. We present a mapping of the antiferromagnetically ordered phase as a function of oxygen doping level. The neutron powder diffraction investigations have been performed using the 7-blades triple axis instrument RITA-2 to focus on a narrow q-range and to suppress background, since the magnetic signal is very weak. The 7-blades data have after measurements been combined into a single data set. The hitherto obtained neutron diffraction data indicate that the Néel temperature as function of doping might be lower than that of bulk YBCO [1].

Sample preparation

The YBCO nanoparticles were prepared by a citrate gel modification of the sol-gel technique as described in [2,5], and subsequently reduced in a N atmosphere to zero oxygen doping. The mean size of the YBCO nanoparticles has been determined by Rietveld refinement of x-ray diffraction to approximately 40 nm in diameter and to be about 3-10 nm thick.

The particles have been stripped of water and oxygenated in a gas volumetric system.

The doping is set by regulating the equilibrium pressure and temperature according to the phase diagram [3,4], and allowing the sample to absorb the required amount of oxygen. Afterwards, the sample is sealed and cooled during a few hours. We have so far prepared four samples (samples 1 to 4, see Tab. 1) which both originally are parts of the same two powder batches. Each sample contained approximately 2 g of material.

Sample	Doping	Batch
1	0.20 ± 0.025	1
2	0.30 ± 0.025	1
3	0.35 ± 0.025	2
4	0.25 ± 0.025	2

Tab. 1: *Overview of the investigated nano-YBCO samples*

The instrument

The samples have been investigated using the triple axis spectrometer RITA-II at the SINQ neutron source, Paul Scherrer Institute in Villigen, Switzerland.

The RITA-II spectrometer has 7 analyzer blades and a position sensitive detector(PSD), and can therefore measure 7 data points simultaneously (see. fig. 2. (right)).

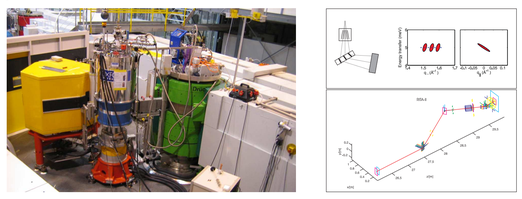

Fig. 1: **Left:** *Image of the RITA-II spectrometer. From the left, the yellow detector tank containing the analyzer assembly and the PSD. The sample is located further right, inside the blue cryo magnet. The monochromator is located inside the green tank. The grey box to the right of the monochromator tank contains the neutron guide.* **Top-right:** *A schematic of the RITA-II analyzer setup. Also shown is a resolution function for a multi blade mode (3 blades).* **Bottom-right:** *A schematic of RITA-II.*

Different arrangements of the analyzer blades, assembly and PSD, give rise to a variety of different user modes. The mode that we have used is the **monochromatic imaging mode** where neutrons are reflected onto separate regions on the PSD. In this mode, the setting of the blades is such that they all scatter neutrons of the same energy. As can be seen from fig. 1 the analyzer blades collect different scattering angles. Hence different q's are detected on different regions on the PSD. By defining software windows (counting bins) appropriately, the RITA-II acts as 7 triple axis spectrometers working in parallel. The data from the individual windows can later be combined, which henceforth can be analyzed in a standard fashion. Examples of combined spectra are presented in fig. 4.

Structure of YBCO

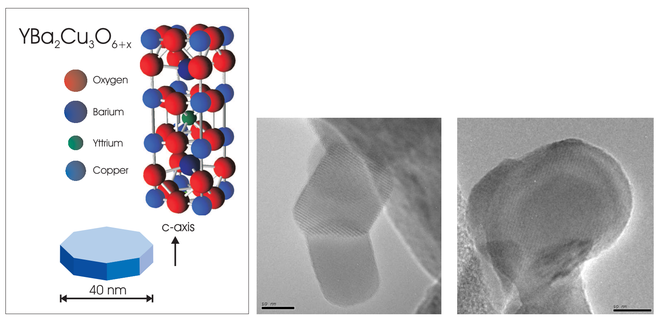

Fig. 2: **Left:** *A schematic of the YBCO unit cell, and a schematic of an YBCO nanoparticle.* **Center & Right:** *TEM image of an YBCO nanoparticle.* **Center:** *The bar is 10 nm.* **Right:** *The bar is 50 nm.*

AFM measurements

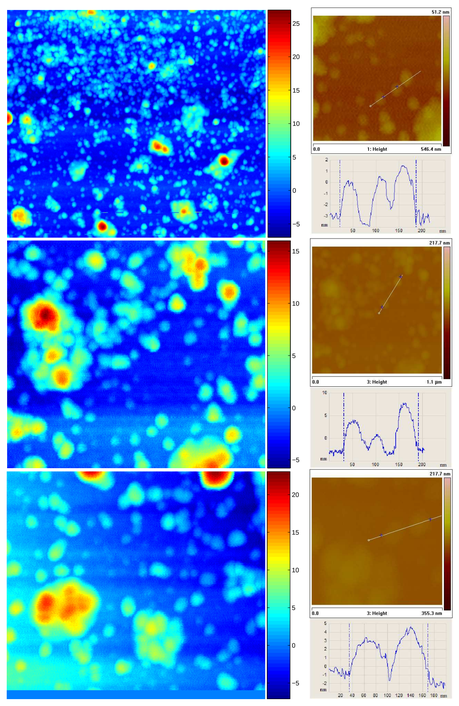

Fig. 3: *AFM images of YBCO nanoparticles on Si surface. The color scales are in nm. On the right a countour map of selected particles is available.* **1st from top:** *Image is 3.3x3.3 μm.* **2nd from top:** *Image is 1.1x1.1 μm.* **3rd from top:** *Image is 1.1x1.1 μm.*

Data

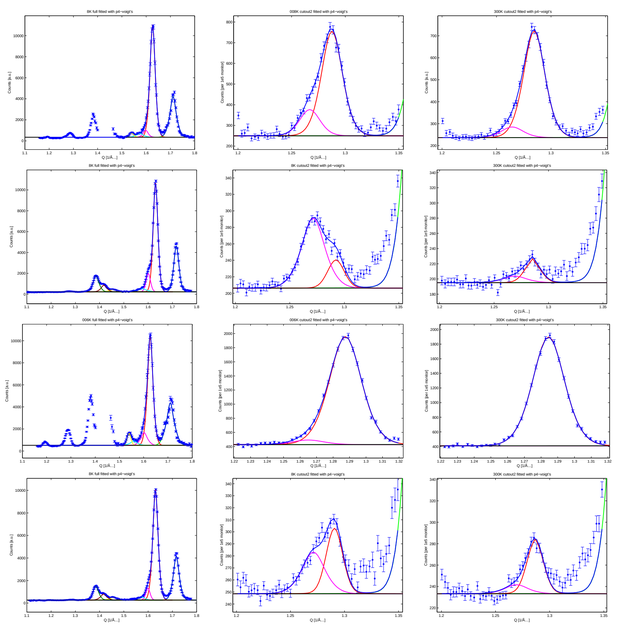

Fig. 3: *Neutron spectra of nano YBCO. Doping are: 0.20, 0.25, 0.30, 0.35.* **1st from top:** **Left:** *A combined fitted spectrum of sample 1 (x=0.20) at T=8 K* **Center:** *A zoom on the combined fitted spectrum of sample 1 (x=0.25) at at T=8 K, on the $(\frac{1}{2}\frac{1}{2}1)$ (marked) antiferromagnetic reflection in YBCO at $q = 1.27$ Å^{-1}.* **Right:** *A zoom on the combined fitted spectrum of sample 1 (x=0.25) at at T=300 K.* **2nd from top:** *Sample 4 (x=0.25).* **Left & center:** *T=8 K.* **Right:** *T=300 K.* **3rd from top:** *Sample 2 (x=0.30).* **Left & center:** *T=6 K.* **Right:** *T=300 K.* **4th from top:** *Sample 3 (x=0.35).* **Left & center:** *T=8 K.* **Right:** *T=300 K.*

In our experiments we have investigated the $(\frac{1}{2}\frac{1}{2}1)$ antiferromagnetic reflection in the YBCO nanopowder at different T (8-450 K) in zero field by powder diffraction. In addition to the YBCO reflections the spectra contain structural reflections from the non-magnetic impurity $BaCuO_2$. Namely the phase $(3\,2\,1)$ reflection which is located at q=1.286 Å^{-1}, may be disturbing since it is located adjacent to the $(\frac{1}{2}\frac{1}{2}1)$ magnetic reflection of YBCO.

Fitting Strategy

In stead of describing the fitting procedure in detail, we provide the most important factors in the fitting work.

- All fitting performed on a cutout of the full spectrum.
- Line shape used is two pseudo-voigt's for each peak fitted, one for intrument broadening and one for signal.
- All fit parameters free at lowest temperature.
- Only amplitude of peaks and background allowed to vary freely at all other temperatures.
- Width of peaks limited/constant while fitting.
- Position of magnetic peak found from the position of the structural peaks (100) and (101).

Magnetic order

A preliminary conclusion of the data (especially the x=0.25 and x=0.35 dopings) indicate that the Néel temperature of nano YBCO might be lower than that of bulk YBCO.

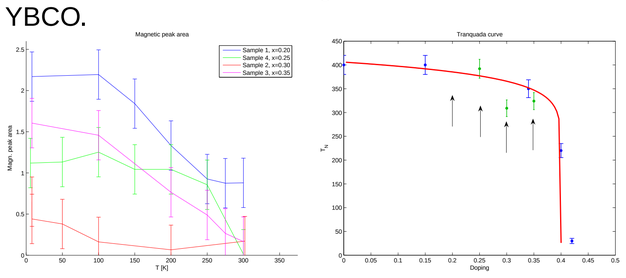

Fig. 5:
Left: *The area of the magnetic peak as a function of doping and temperature. The error bars are estimated.* **Right:** *Data from [1] combined with our data. Our points are green and marked with arrows.*

New data - super- on semiconductor

In one of our recent experiments we have used PLD to deposit YBCO nano-islands on semiconductor substrates to investigate the formation of islands. The formation of nano-islands of superconductor on semiconductor, is interesting seen from the point of view of the electronics community. As substrates we have used Si and $SrTiO_3$:Nb 0.5% wafers.

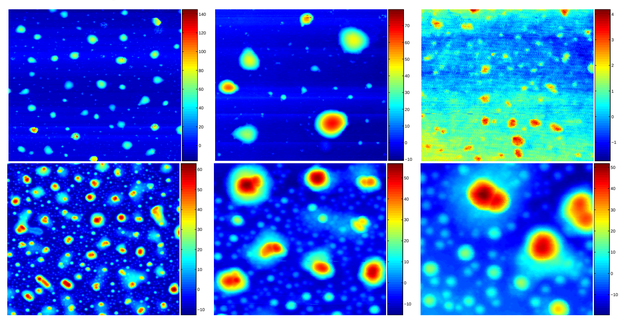

Fig. 6: *AFM images of PLD-deposited YBCO on semiconductor substrate.* **Top row:** *YBCO on Si: 20x20 μm, 6.6x6.6 μm, 746x746 nm.* **Bottom row:** *YBCO on $SrTiO_3$:Nb: 3.3x3.3 μm, 1.15x1.15 μm, 723x723 nm.*

Contact

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