

innovative coating technologies gmbh

Texture measurements on thin films using an X-ray microfocus source

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Introduction

The Incoatec Microfocus Source I μ S is a 30 W air-cooled sealed tube (Fig. 1). It is combined with 2D beam shaping multilayer optics. Specs of often used optics are given in Table 1. The $I\mu$ S in detail:



The source

- high brilliance
- $< 50 \,\mu\text{m}$ spot size
- 30 W and air-cooled
- Cu, Mo, Cr and Ag radiation available
- convenience of sealed-tube

Useful accessories • detectors for beam characterization

- collimators
- pumps + vacuum gauges

 $I\mu$ S System with optics housing and 19" wide generator

The optics

- new Montel type optics named Quazar
- 2-dim beam shaping
- collimating (LD), focussing (SCD) or collimating + focusing (hybrid)
- patented housing for high stability and easy alignment
- motorized alignment (optional)



Fig. 2: Frame of the sample with the 2θ -range from 22° to 49° with 600 s exposure time. During exposure the sample was rotated in ω , so the Bragg-conditions for the YBCO spots are fulfilled. The integration of the frame is shown below.





To measure a pole figure of the (1 1 0) reflex of the BaHfO₃, with the program MULTEX (Bruker AXS) a measurement scheme of 126 frames was planed, covering the χ -range up to 65°. The frames were recorded with 20 s exposure time each resulting in a total measurement time of about 50 minutes including all movements. Pole figures calculated with MULTEX are shown in Figure 3.





Figure 1: IµS System integrated in a mar desktop beamline (left) and in a Bruker AXS GADDS system (right).

	type	length (mm)	divergence (deg)	beam size (mm)
E 23 (Mo)	focusing	150	0.28	0.12
E 31 (Cu)	focusing	150	0.69	0.23
E 32 (Cu)	collimating	60	0.06	0.65
HY (Cu)	hybride	60	0.24 x 0.06	0.65 x 0.18

 Table 1: Different types of standard Quazar optics (others on request)

The I μ S can be used for different applications, stress and texture measurements, (GI)SAXS, phase identification, or single crystal diffraction to name but a few.

The IµS with copper anode and collimating optics was integrated in a Bruker D8 GADDS-system (Figure 1, right) with eulerian cradle and VÅNTEC2000-detector. This setup was used for pole figure measurements on nano particles.

The similarity of the orientation is also visible in the frame shown in Figure 4.





Fig. 3: Pole figure of the BaHfO₃-(1 1 0) reflex (left) and the YBCO (0 0 4) reflex (right). A strong orientation of the crystals is clearly visible.



Fig. 4: Frame recorded during the pole measurement at $2\theta = 35^{\circ}$,

The Sample

The superconductor $YBa_2Cu_3O_7$ (YBCO) was deposited on a strontium titanate substrate (SrTiO₃) using chemical solution deposition. An Hf-compound was added to the chemical precursor in order to form BaHfO₃ nanoparticle with a size of 10 to 20 nm inside the YBCO matrix acting as pinning centers for an improvement of the critical current density J_c in magnetic fields. The main goal of the measurement was to clarify the orientation distribution of the BaHfO₃ nanoparticles with respect to the epitaxially grown YBCO matrix.

In Fig. 2 a frame recorded with the GADDS system is shown. The (0 0 I) spots of the single crystalline SrTiO₃ and of the epitaxially grown YBCO are clearly visible together with (1 1 0) and (0 0 2) Scherrer rings from BaHfO₃. Additionally, a small (0 0 2) peak was found at $2\theta \approx 30^{\circ}$ arising from an epitaxial BaHfO₃ component.

 $\omega = 18.7^{\circ}, \chi = 54.3^{\circ}, \text{ exposure time 20 seconds. The Scherer-ring of the}$ $BaHfO_3$ (1 1 0) peak is visible together with the (0 0 4) and (0 0 3) spots of YBCO.

Conclusion The measurements indicate, that the majority of the nanoparticles are randomly incorporated in the YBCO matrix. Nevertheless, it was possible to measure a (1 1 0) pole figure of the epitaxially grown BaHfO₃ fraction (Fig. 3), which has a fourfold-symmetry similar to the YBCO layer. With a modern set-up it is possible to perform a complete pole figure measurement in less than one hour.

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