

X-RAY DIFFRACTION

High Resolution X-Ray Diffraction from PZT Capacitors with Micrometer Beamsizes

Application Note 626

Introduction

The functionality of microelectronics devices is highly dependent on their crystal structure. High-Resolution X-Ray Diffraction (HRXRD) is a non-destructive analytical technique that enables the investigation of crystalline structure with sub-nanometer accuracy—even under non-ambient or in-operando conditions. The investigation of these micrometer-sized devices via HRXRD using laboratory X-ray diffractometers is especially challenging, as an X-ray beam smaller than the structure of interest is required.

Lead zirconate titanate (PZT) capacitors play a vital role in enhancing the functionality and performance of various electronic devices and systems. Due to their ability to switch between electrical and mechanical energy, they are used for applications requiring precision, control and efficient energy conversion.

Typical examples include micro-electromechanical systems (MEMS) devices, piezoelectric transformers, actuators and motors, vibration sensors, frequency generators or timers, to name a few. Controlling the structure of the different layers of these devices is of critical importance to realize proper functionality.

In this application note, 50 μm large PZT capacitors have been investigated. These small devices require beam sizes of less than 20 μm while achieving good intensity levels to allow for reasonable measurement times.

Instrument configuration

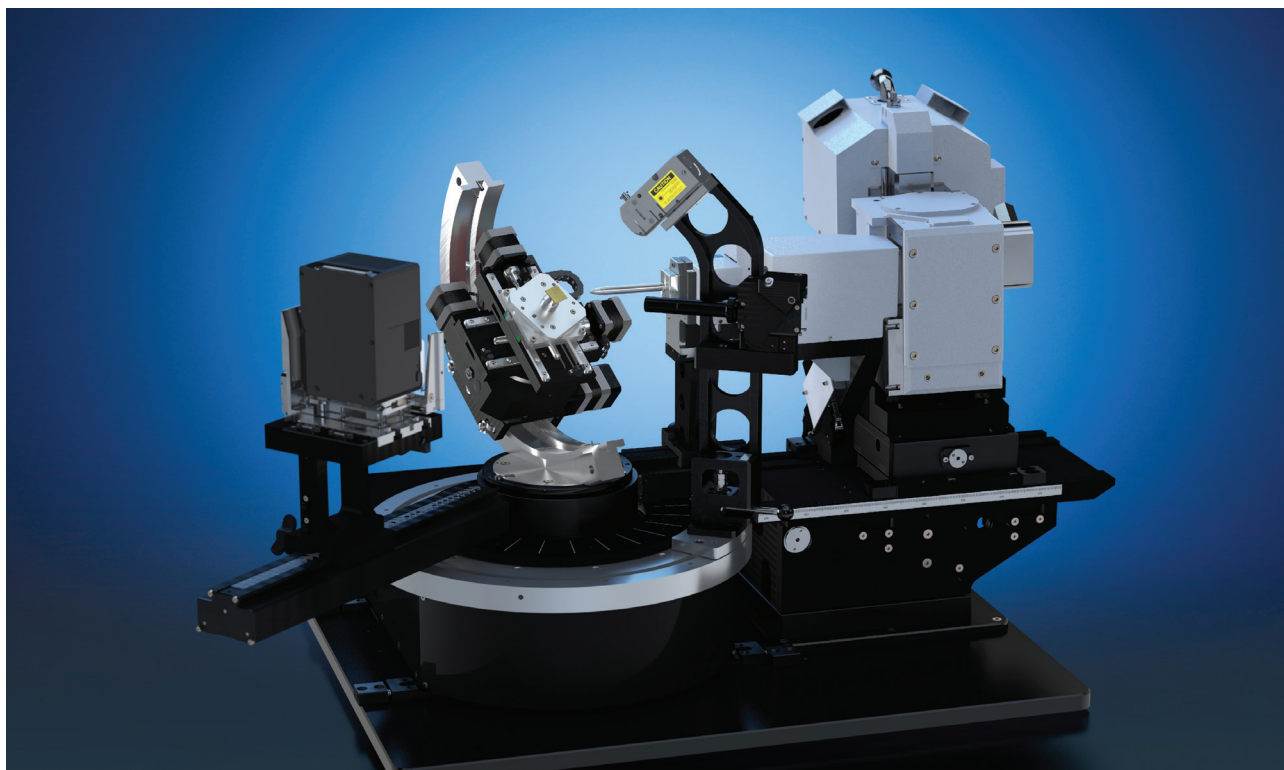


Fig. 1 D8 DISCOVER configuration with HB-TXS

The system consists of a 2.5 kW High-Brilliance Turbo X-ray Source (HB-TXS) with a Cu anode operating at 50 kV and 50 mA. The X-rays emitted from the focal spot of less than 100 μm size are reflected by a Montel optics. These optics produce a Cu- $K\alpha$ beam that is highly parallel in the equatorial direction, while being axially focused down to about 180 μm at the centre of the goniometer. A 2xGe(022a) monochromator can be employed to obtain a high-intensity pure Cu- $K\alpha_1$ beam with an equatorial divergence of better than 0.013°. The beam size at the sample position can be controlled by using so-called UBC collimators¹⁾. These collimators are mounted with μm precision via a magnetic fixture and allow flexible sizing of the full 2 mm x 180 μm beam down to 20 μm .

A DECTRIS EIGER2 R 500K detector enabling measurements in 0D²⁾, 1D, and 2D modes was used on the diffracted beam side. Working in 0D mode provides strong control over the background by selecting the appropriate field of view. The 1D and 2D modes are used to measure Reciprocal Space Maps (RSMs) with control over the axial integration window. The secondary dovetail track of the D8 goniometer allows continuous positioning of the detector to perfectly adapt angular resolution and field of view to the application's requirements. With its integrated detector-distance detection, the EIGER2 detector remains precisely calibrated at all times.

A high-resolution optical microscope is fully integrated into the measurement software to facilitate easy identification of the region of interest and accurate positioning of the sample in the X-ray beam with micron accuracy.

¹⁾ Universal-Beam Concept (UBC)

²⁾ Dimensional (D)

Application examples

This unique instrumental setup perfectly matches the requirements of thin film analysis on micrometer sized objects. Its outstanding brilliance translates into excellent data quality for spot sizes down to 20 μm , providing an integrated flux of more than 10^9 cps. To highlight this unmatched performance, two application examples are presented.

Example 1

The first example demonstrates the high spatial resolution of the setup. A UBC collimator with a 20 μm pinhole is used to define the beam size at the sample surface. To achieve a beam size of 20 μm or less, the pinhole needs to be positioned at 5 mm distance to the sample. The sample* consists of various differently shaped SiGe pads with a diameter of 50 μm . To probe the different pads, the SiGe(004) reflections were aligned, and by mapping X and Y, the spatial shape of the pads was obtained.

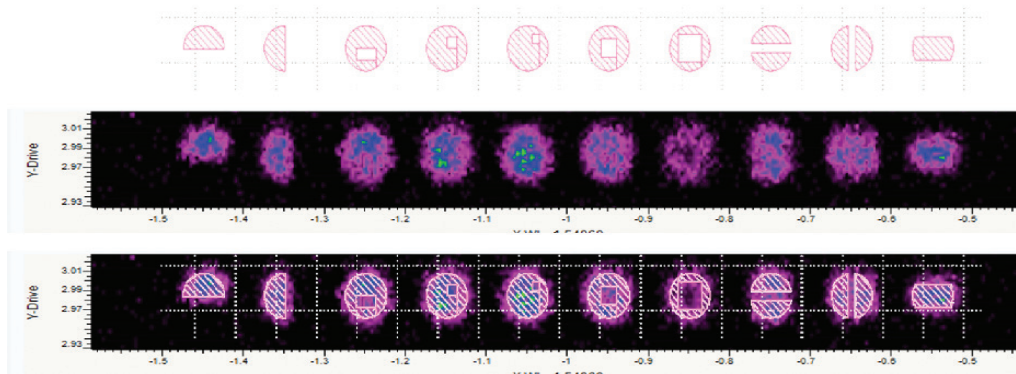


Fig. 2 Scheme of differently shaped SiGe pads with diameter 50 μm (top). An (X,Y) Mapping in diffraction condition of the (004) SiGe layer peak using a 20 μm beam (middle line). Overlay of scheme and measurement highlights the excellent spatial resolution of the micron beam.

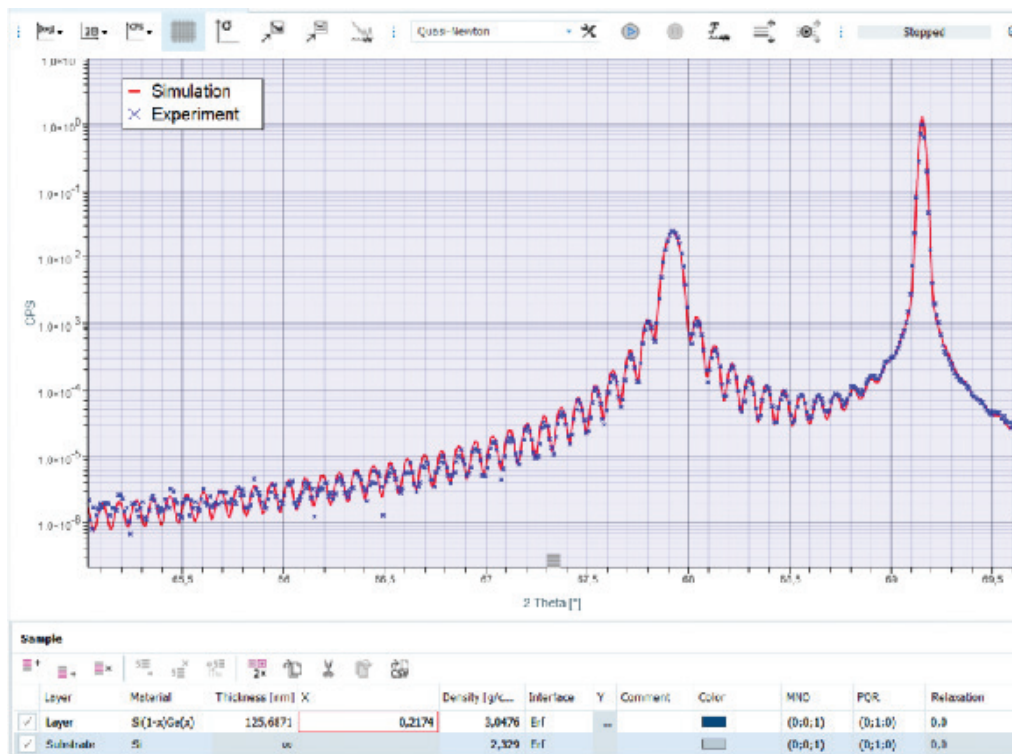


Fig. 3 2 θ / ω scan around the Si(004) reflection using the full 180 μm x 2 mm beam: In less than 5 minutes data of excellent quality can be obtained from a 2 x 2 mm² SiGe test structure. The measurement is perfectly explained using a single ~125 nm thick Si(1-x)Ge(x) with x=21.7% on top of a Si substrate.

The results are shown in Figure 2. The upper line depicts a sketch of the pads. The middle line shows the result of the spatial mapping. The differences between the different pads are clearly visible, and the agreement with the design of the pads (lower line) is very good. The capability to observe small deviations from a complete 50 μm circular pad highlights the excellent spatial resolution of this D8 DISCOVER configuration.

A second experiment was carried out using the full beam size. Using the full beam size, measurements of excellent data quality can be obtained within 5 minutes as demonstrated in the example shown in Figure 3.

Example 2

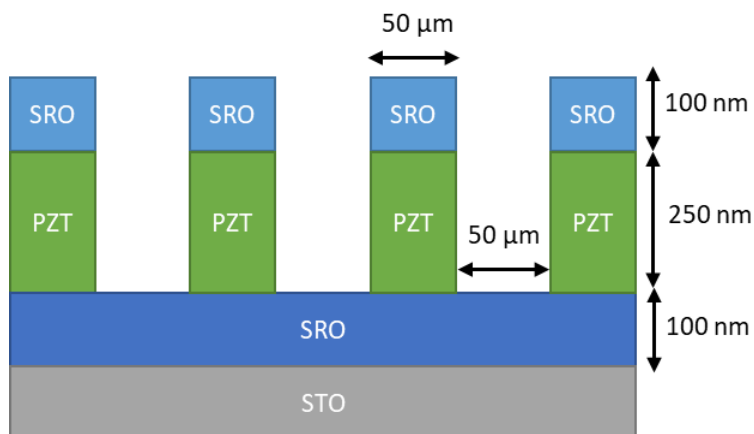


Fig. 4 Sketch of the PZT capacitor sample structure**.

The HB-TXS setup was used to investigate a structured multilayer sample** consisting of 100 nm of Strontium Ruthenium Oxide (SRO) deposited on a Strontium Titanate Oxide (STO) substrate, on top of which 50 μm sized pads consisting of a 250 nm Lead Zirconate Titanate (PZT) film with a 50 nm SRO top layer have been prepared. The SRO layers can serve as electrodes to apply an electric voltage and study the structural properties of the PZT film in-operando. A microscope view of the prepared structures is shown on the left picture of Figure 5. Two 100 μm pads and an array of 50 μm pads can be seen.

To determine the structure of the pads, a 20 μm beam was used to align the PZT (103+) reflection. With an incident angle of $\sim 54^\circ$, the footprint of the X-ray beam is $\sim 25 \mu\text{m}$, which is small enough to precisely measure on or between the pads. To illustrate the excellent spatial resolution, an (X,Y)-mapping was performed and the result is shown on the right side of Figure 5.

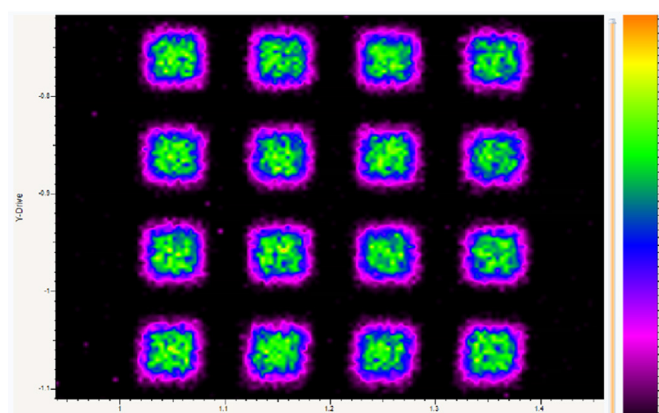
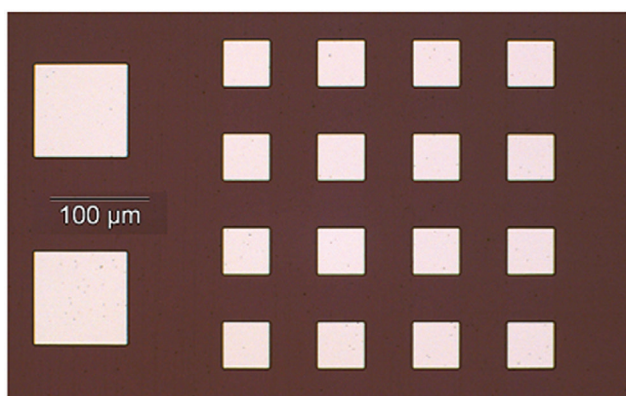


Fig. 5 Left side: Microscope view on the patterned test structure** showing 100 μm and 50 μm pads.

Right side: (X,Y) mapping of the array of 50 μm pads: The PZT (103+) reflection was used for probing the pads using a 20 μm sized beam.

Data analysis and results

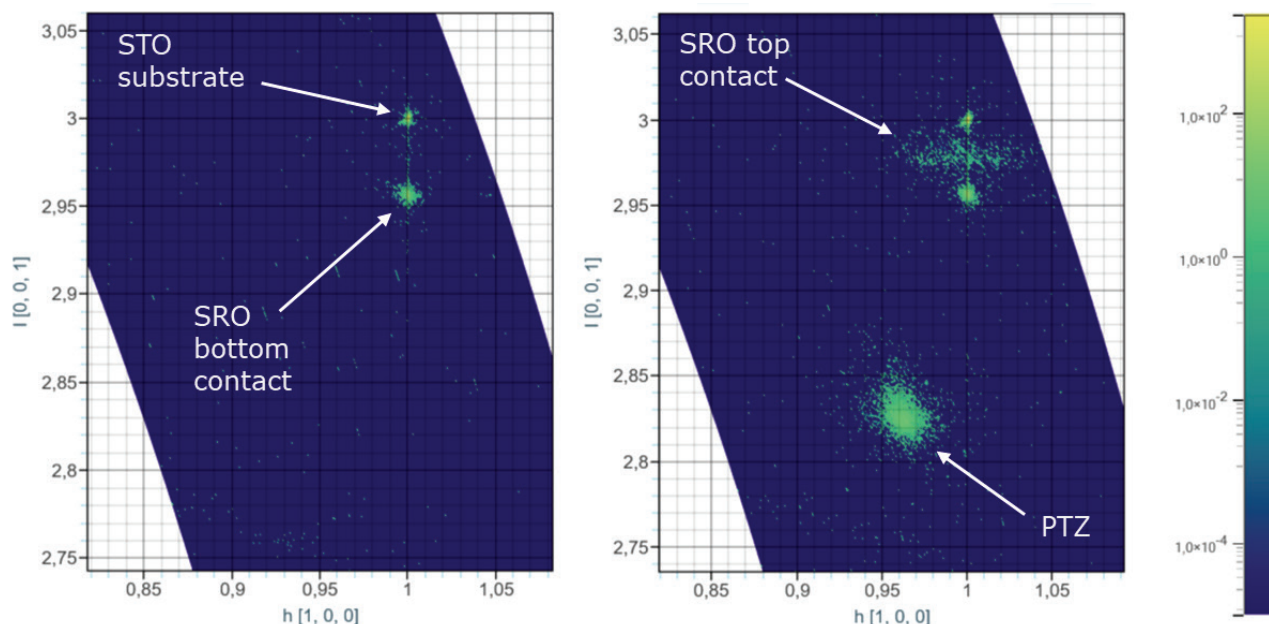


Fig. 6 Reciprocal space mapping at the STO (103+) reflection between (left) and on (right) a 50 μm pad.

Two Reciprocal Space Maps (RSM) around the STO (103+) have been measured. One between the 50 μm PZT pads and the other one on one of the pads. The first one between the 50 μm PZT pads and the second one on one of the pads. Both measurements were performed using rapidRSM™ technology: The RSM is recorded by continuously taking 2Theta snapshots while rocking the sample. This technique reduces the measurement time down to ~ 40 min for each map. At a sample-to-detector distance of 290 mm, the EIGER2 R 500K covers $\sim 14.7^\circ$ in 2Theta while providing an angular resolution of $\sim 0.0145^\circ$. The measurements have been loaded into the thin-film analysis software DIFFRAC.LEPTOS X and converted into reciprocal lattice units. Both RSMs are depicted in Figure 6.

The reciprocal space map (RSM) measured between the pads shows the (103+) reflections from the STO substrate and the fully strained SRO bottom contact layer. The sharpness of both peaks indicates the excellent crystalline quality of both lattices.

The RSM taken on the pad clearly shows the additional peaks from the PZT layer and the top SRO electrode. Both peaks show a significant broadening compared to the peaks of the STO substrate and the lower SRO electrode indicating a higher crystalline mosaicity. This results in a higher dislocations density and therefore a (partial) relaxation of the films is expected. For the PZT film, the peak is located at (0.9584/2.8346).

This corresponds to lattice parameters of $a = 0.4133$ nm and $c = 0.4075$ nm which are in very good agreement with literature values of the lattice parameters of $\text{PbZr}_{1-x}\text{Ti}_x\text{O}_3$ powders with $x \approx 0.46$ as reported in [3].

For the SRO layers, the peak positions are (1/2.9643) and (0.9905/2.9866) for SRO_{bottom} and SRO_{top}, respectively. These positions match very well the fully strained and the fully relaxed peak positions of a cubic lattice with $a \approx 0.3934$ nm — assuming a simple cubic deformation model with Poisson's ratio $\nu = 1/3$. The lattice parameter agrees well with that of the pseudo-cubic representation of SRO with $a \approx 0.5567$ nm / $\sqrt{2} = 0.3936$ nm [1][2].

The RSMs taken from the 50 μm PZT capacitor have been analyzed and the results are in excellent agreement with other results presented in the literature. It should be emphasized that this kind of experiment can normally only be performed at synchrotron radiation facilities and not on laboratory X-ray systems.

Summary and Conclusion

The D8 DISCOVER with HB-TXS is a powerful XRD laboratory solution for thin-film analysis with spatial resolution down to the 20 μm range:

- When equipped with a UBC collimator, a beam size of $\sim 20 \mu\text{m}$ can be achieved, allowing for the clear separation of 50 μm test structures in a spatial (X, Y) mapping.
- When operated with the full beam dimensions of 2 mm x 180 μm , the Montel optics provide a total flux of more than 2×10^9 cps. This high intensity enables rapid measurements of standard thin film applications, such as grazing incidence diffraction, X-ray reflectometry, HRXRD, and texture analysis, in a very short time. This capability has been demonstrated with a measurement from a 2 x 2 mm² SiGe pad, requiring less than 5 minutes of data collection time.
- Reciprocal space maps (RSMs) have been measured on a PZT capacitor with a 50 μm pad size and between two capacitors. The RSMs allow for the determination of lattice parameters of different films with high accuracy, and the epitaxial quality can be extracted from the width of the peaks in the RSM.
- The D8 DISCOVER, equipped with HB-TXS and the EIGER2 R 500K detector, enables micron beam applications that were previously limited to synchrotron radiation facilities. This significantly extends the analytical capabilities of laboratories in both industry and academia.

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*) Sample courtesy of Bruker Nano Surfaces & Metrology, X-ray Business Unit

**) Sample courtesy of M. Nguyen and Y. Birkholzer, MESA + Institute for Nanotechnology, University of Twente

References

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