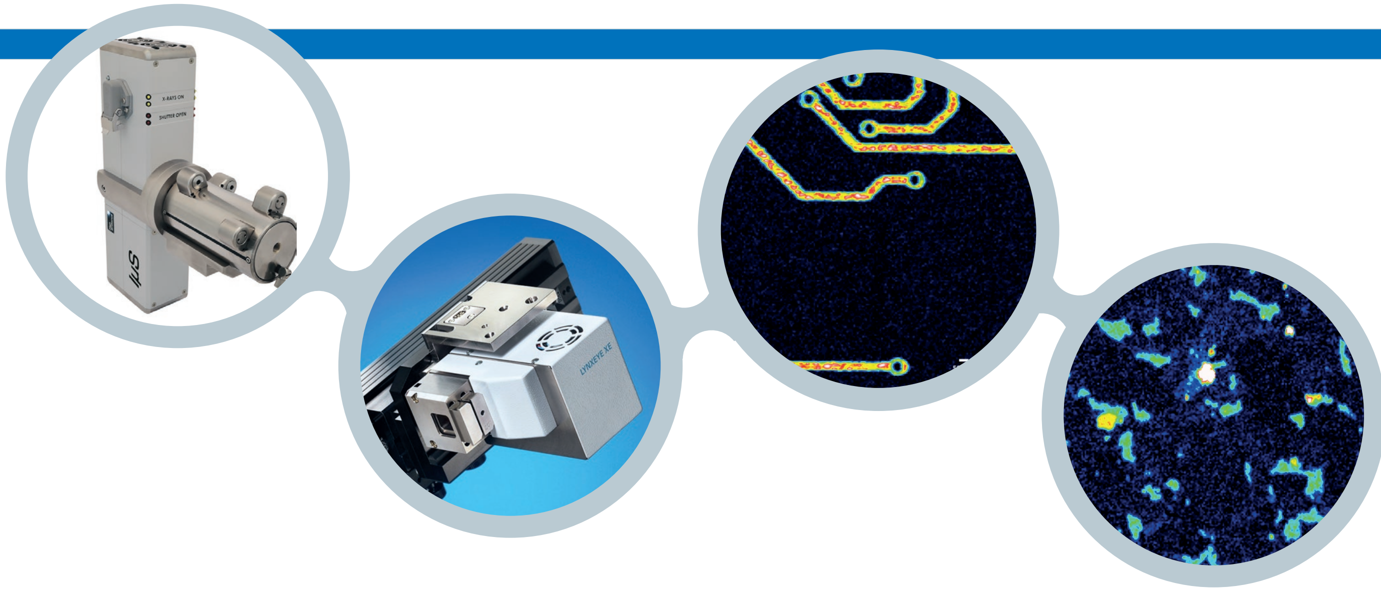

Application Note XRD 606

D8 DISCOVER with IµS

Application: µXRD Phase mapping

Introduction

The D8 DISCOVER combined with the IµS microfocus x-ray source is an innovative diffraction (XRD) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in a micro-diffraction (µXRD) configuration to analyze the crystalline phase distribution and properties of materials with probe sizes less than 50 µm.

As a second example, µXRD phase mapping will be presented on several materials including a slice of granite and copper interconnect circuits. In addition, orientation distribution function (ODF) analysis is demonstrated on a circuit with 500 µm line width.

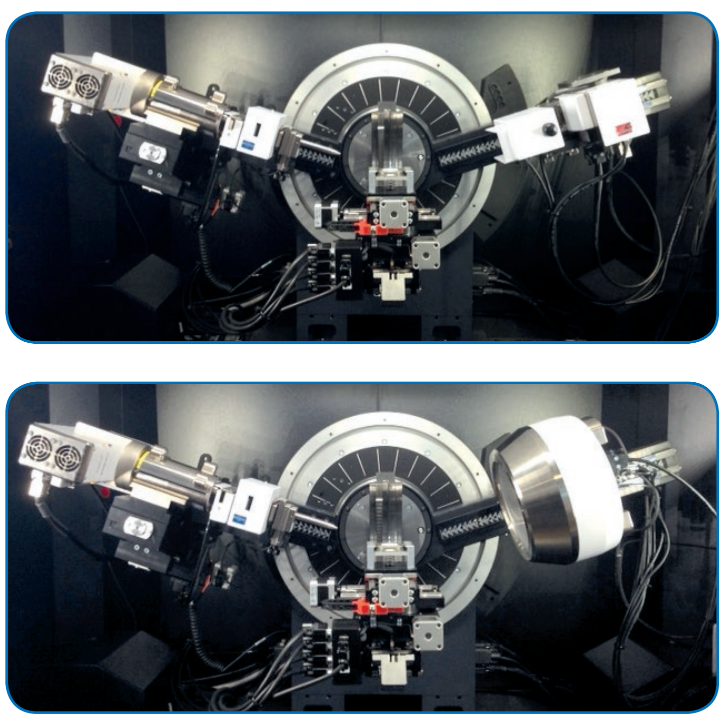

Figure 1. D8 DISCOVER with IµS configured for µXRD, from left to right: IµS, Montel, collimator, centric cradle, 1D and 2D detector respectively. (Top) The LYNXEYE XE is used to produce orientation dependent maps. (Bottom) The VÅNTEC-500 is used for pole figure measurements for ODF analysis.

µXRD

The configuration used for µXRD is shown in figure 1 and detailed in table 1. The IµS and integrated MONTEL optic produces a very brilliant spot type primary beam which is ideal for this application. The final beam size can be controlled by using collimators and is typically selected to 20 µm and 100 µm. The data in this report are achieved with a dedicated MONTEL optic, which focuses the beam on the sample position to provide higher intensity for the smallest spot size. The laser-video microscope is used to achieve simple and precise (< 5µm) contact-free positioning of the sample in the x-ray beam. For mapping experiments where a single peak is used to indicate the presence of a phase, the LYNXEYE XE detector was used. It maximizes sensitivity by combining speed and extremely low background due to its unique energy discrimination capability. The detector is positioned on a peak of interest and then the scan is performed as a series of X translation scans sequenced in the Y direction. Typical scans settings are 0.05 mm step, 1 second per step, giving a total scan time of 7 min per mm^2. The sample can be oscillated in Phi (rotation) and Psi (tilt) during measurement to reduce preferred orientation and/or poor particle statistics so that peak intensity is more representative of phase concentration. Once an area of interest is identified, further analysis can be performed, such as residual stress or texture measurements. A large 2D detector such as the VÅNTEC-500 can be used to speed up data collection in case many measurements at different sample orientations would be required.

Source	IµS Microfocus (Cu)
Optics	MONTEL
Collimator	20 µm - 1.0 mm, several intermediate sizes
Stage	Centric Eulerian Cradle (CEC)
Sample Positioning	Laser Video Microscope
Detector	LYNXEYE XE or VÅNTEC-500

Table 1. Typical µXRD Phase mapping instrument setup for the D8 DISCOVER with IµS.

Measurement of Biotite in Granite

A piece of granite, shown in Figure 2a, roughly 20 mm x 20 mm x 5 mm was used for this experiment. Figure 2b shows a phase map performed on the granite section at 34 degrees, corresponding to the (131) reflection of biotite. This map consists of a series of X scans with 0.1 mm step and 0.5 second per step count time covering a range of 20 mm. This X scan is then repeated across 20 mm of Y, resulting in a total scan time of 11 hours. There is good correlation between the black areas of the granite in the optical image and the biotite in the µXRD phase map.

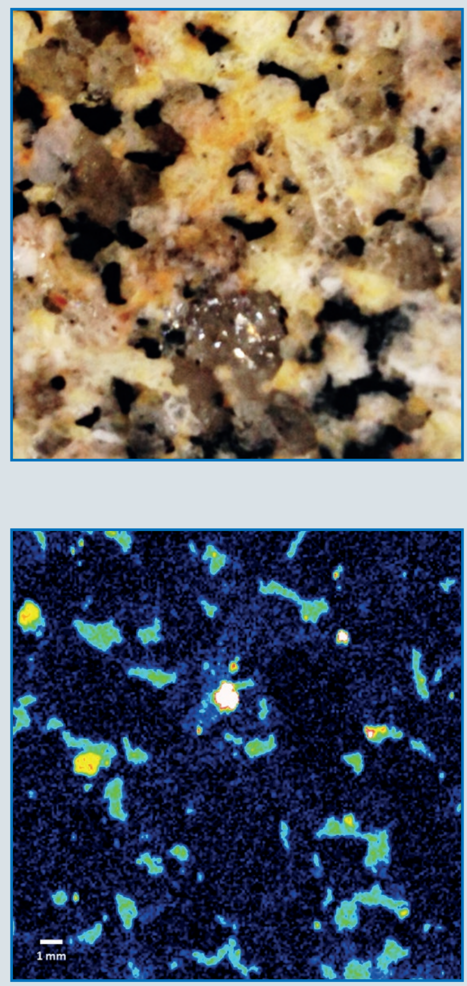

Figure 2. Optical image and µXRD phase mapping of biotite in granite with the D8 DISCOVER with IµS and LYNXEYE XE.

Measurement of 50µm Copper Traces on a PCB

The interconnection circuits on printed circuit boards are essential in connecting devices in electronics. The quality of the interconnection circuit has a dramatic effect on device performance, as grain boundaries are known to increase resistance, degrading signals and increasing power consumption. As the size of these interconnection circuits shrinks, it can become increasingly difficult to maintain the desired preferred orientation minimizing the above mentioned effects. Figure 3 shows the integration of a single 2D frame (shown in the inset) collected from a circuit board containing 50 micron copper interconnects. The primary phase identified in the scan corresponds to copper, with the (111), (200) and (220) peaks shown. Figure 4a shows an image of the circuit that is being analyzed. Figure 4b displays a micro x-ray fluorescence (µXRF) map of the circuit collected with the M4 TORNADO. Set to Copper sensitivity a very uniform elemental composition continuity of the interconnection wires is detected. Figures 4c-e show the (111), (200) and (220) µXRD phase maps of the sample. Bright areas indicate a grain of that orientation normal to the surface of the printed circuit board. Even though the wires show good elemental continuity and seem uniform in the µXRF analysis, structural analysis through µXRD reveals a large number of grain boundaries which may be problematic. In addition to the primary phase of Cu observed in figure 3, a secondary phase, $BaSO_4$ was found to be present. Figure 5a-b shows the elemental mapping of Ba and S done with the M4 TORNADO. Although the elements can be visualized, there is no information of the crystalline phases that are present. Figure 5c presents a phase map of (121) $BaSO_4$, clearly showing that the Ba and S observed in the elemental mapping is present in the $BaSO_4$ phase.

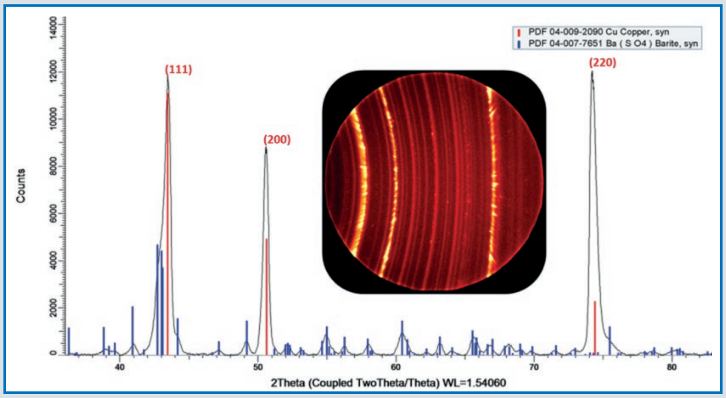

Figure 3. XRD Measurement of a printed circuit board collected with IµS and LYNXEYE XE while oscillating in X and Y over the whole surface.

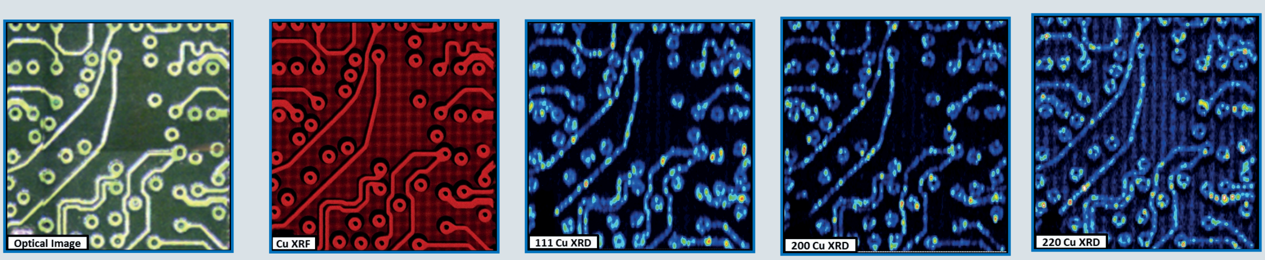

Figure 4. (a) Optical image of the printed circuit board sample. (b) µXRF elemental map of Cu collected with the M4 TORNADO. (c-e)) µXRD phase map of (111), (200) and (220) Cu collected with IµS and LYNXEYE XE.

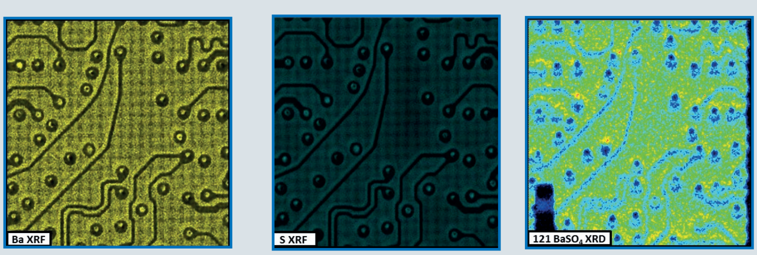

Figure 5. (a-b) µXRF elemental map of Ba and S collected with the M4 TORNADO.
(c) µXRD phase map of (121) $BaSO_4$ collected with IµS and LYNXEYE XE.

µXRD Measurement of Texture

As a consequence of the µXRD phase mapping, there may be regions of interest that justify more detailed analysis. These regions can be selected easily for further investigations based on the µXRD phase map. For example, the macroscopic properties of a material can be strongly dependent on the crystallographic orientation, often referred to as the texture, of the phases present. It may be of use to further quantify this texture with pole figures and the resulting orientation distribution function (ODF). A two dimensional detector, such as the VÅNTEC-500, with large coverage in 2θ and gamma offers a tremendous time savings in collecting pole figures. Figure 6 shows an optical image and a (111) Cu µXRD phase map of a printed circuit board containing copper traces with 500 µm line width. Although there is some difference of peak intensity, as indicated by red and yellow regions along the Cu trace, the (111) peak is always well represented indicating good out of plane alignment of the grain structure of the copper traces. However, further texture analysis may be required to determine the true three dimensional texture states. A texture data set was collected with 100 µm beam size and total collection time of 20 minutes. The resulting (111) and (200) pole figures are shown in Figure 7. A so-called fiber orientation can clearly be seen. DIFFRAC.MULTEX was used to create the pole figures and to analyze the texture using the component method, with the reconstructed pole figure also shown in figure 7. The ODF was calculated based on 3 fiber texture components and is shown in Figure 8.

Conclusion

The D8 DISCOVER with IµS was used to collect data from a variety of samples including granite and copper interconnects on printed circuit boards. The IµS combined with MONTEL optic creates a probe size of 20 to 100 µm, resulting in an ideal configuration for µXRD phase mapping and texture analysis. The LYNXEYE XE was used with the X and Y mapping stage of the Centric Eulerian Cradle to create the µXRD maps, while the VÅNTEC-500 was used for detailed analysis, texture in this example, of a specific sample region. The DIFFRAC.MEASUREMENT software suite with DAVINCI optical component recognition facilitates the change from LYNXEYE XE to VÅNTEC-500 in a matter of minutes, allowing the instrument to be optimized for the experiment.

Author

Jonathan Giencke, Bruker AXS Inc.

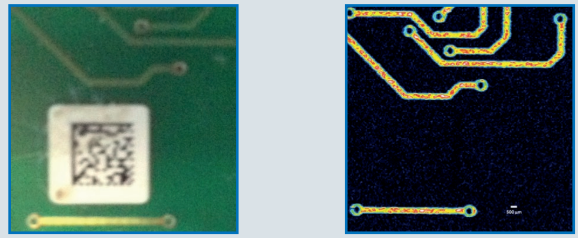

Figure 6. Optical image and µXRD Cu (111) phase mapping with the D8 DISCOVER with IµS and LYNXEYE XE.

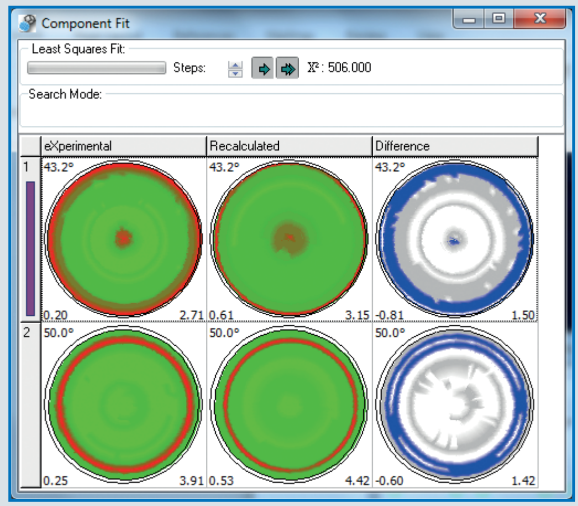

Figure 7. Measured (left) and simulated (right) pole figures using only a fiber texture components of a 500 µm copper interconnect. The pole figures are of the (111) (top) and (200) (bottom) reflections of copper.

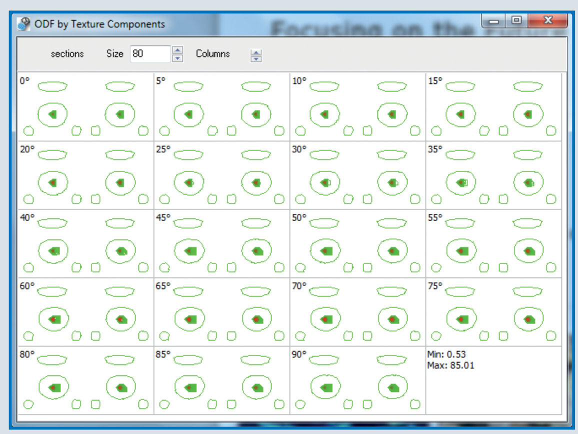

Figure 8. Orientation Distribution Function (ODF) of a 500 µm copper interconnect.

Bruker AXS GmbH

Karlsruhe · Germany
Phone +49 721 50997-0
Fax +49 721 50997-5654
info.baxs@bruker.com

www.bruker.com