SINGLE-CRYSTAL X-RAY DIFFRACTION

The World Beyond Conventional SC-XRD

Advanced applications for your Single-Crystal diffraction solution

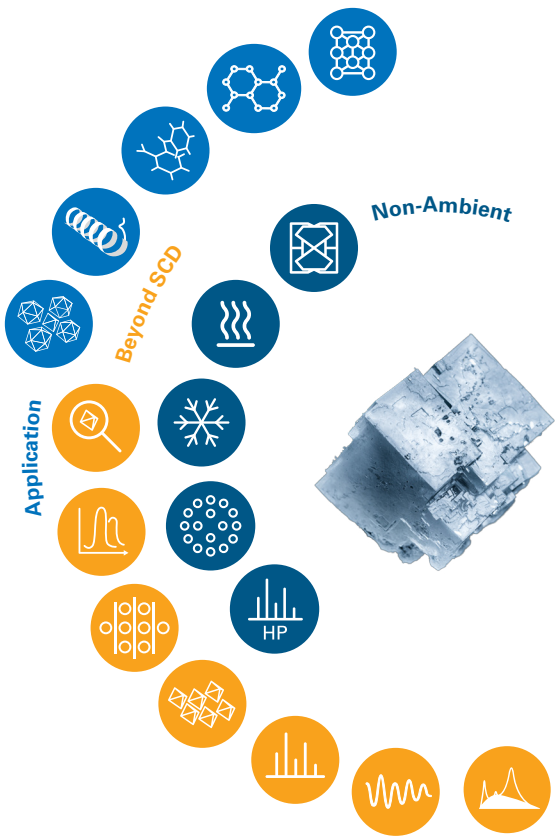
Introduction

Single-crystal X-ray diffraction (SC-XRD) is a precise analytical technique used for *de novo* structure determination, providing highly detailed insights into the arrangement of atoms, down to detecting fractions of electrons.

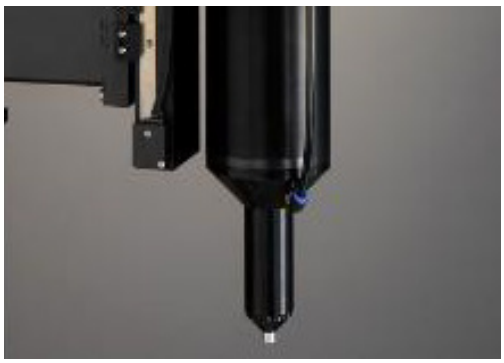
Our equipment is carefully optimized to deliver maximum brightness, handle small samples, and accurately measure the intensity of diffracted X-rays. A typical setup includes one or more high-brightness microfocus X-ray sources, a precise multi-axis goniometer, and a large-active-area, photon-counting detector. The setup is complemented by user-friendly software with thoroughly tested and powerful capabilities.

While being optimized for SC-XRD experiments, Bruker's instrumentation delivers excellent results in a variety of experiments beyond conventional single-crystal studies.

This Application Note provides an overview of these applications. Inspired by the creativity of our users, these innovative experiments highlight the versatility of SC-XRD. Even experienced scientists are impressed by the outstanding quality achieved in these experiments.

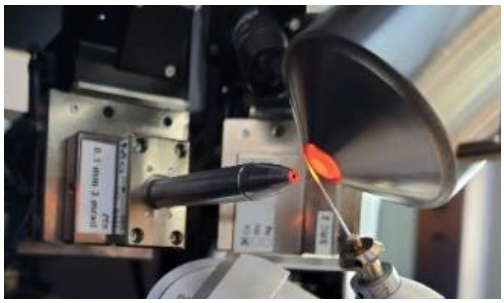


Low-Temperature Experiments



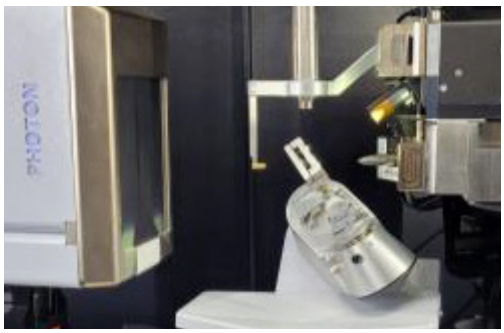
Low-temperature experiments[1] down to 100K are now routine in single-crystal X-ray diffraction (SC-XRD). Bruker's instrumentation guarantees seamless integration of modern low-temperature devices allowing for convenient remote control, enhancing sample stability and data quality. For experiments at even lower temperatures, the Oxford N-Helix can be added to the D8 VENTURE instrument. Our vibration-free mount guarantees best data quality down to 28K. Modern low temperature experiments greatly benefit from the automated masking of reflections affected by cryo nozzle shading and the failsafe collision prevention provided by our software.

High-Temperature Experiments



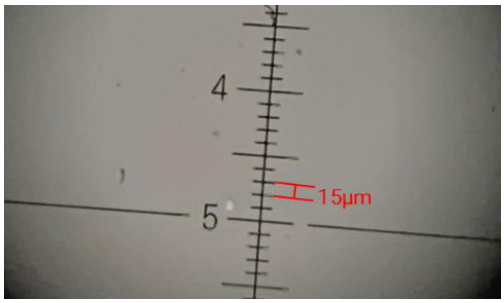
The fields of material science, geology, and mineralogy can greatly benefit from determining the structure of materials at high temperatures. Today's low-temperature devices can be used to access a moderate high-temperature range up to 500K, while dedicated equipment is available for higher temperatures. When combined with a D8 VENTURE, modern heater cans reach sample temperatures of up to 1300K. The user-friendly design of the mounting ensures convenient operation combined with operator and instrument safety.

High-Pressure SC-XRD[2]



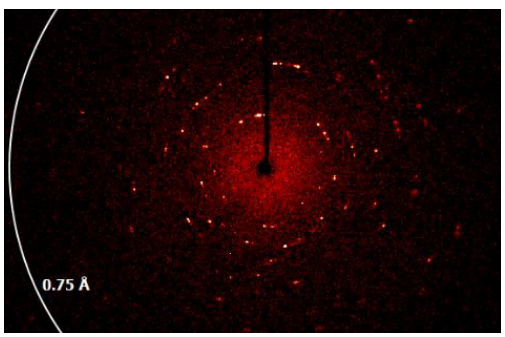
Metastable phases, such as those found in geology, can be easily studied under high pressure using Diamond Anvil Cells (DAC). The standard goniometers of both the D8 QUEST and the D8 VENTURE can easily accommodate all commonly used cells. In addition to being easy to use, our most advanced IμS X-ray sources ensure the best possible data quality for Mo- and/or Ag-radiation. Even the smallest samples remain reliably centered on our accurate and robust goniometer, while our PHOTON detectors offer unmatched efficiency for the intense radiation needed for these experiments.

Micro-Crystal SC-XRD (MC SC-XRD)



Even though the minimum crystal size that can be analyzed using single-crystal X-ray diffraction (SC-XRD) has significantly decreased over the past few decades, crystals smaller than 20 µm are generally considered the practical minimum limit for modern SC-XRD equipment. However, with Bruker's high brightness sources such as the IμS DIAMOND II using Cu radiation and METALJET MC using Ga radiation, crystallographers can now routinely study samples smaller than even 10 µm.

Polycrystalline sample SC-XRD[3]

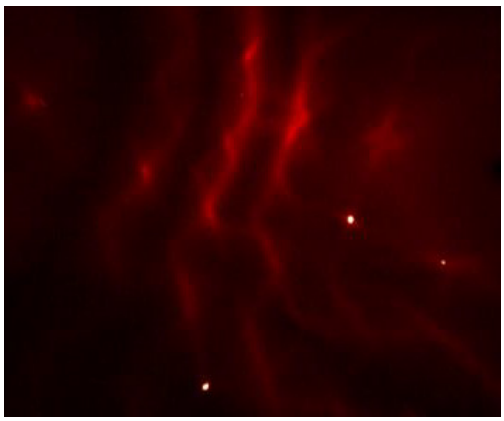


It is common to encounter polycrystalline agglomerates in naturally occurring minerals and samples resulting from industrial processes. These agglomerates often make it difficult to isolate single crystals. The small, highly brilliant X-ray beam resulting from our IμS sources eases the collection of high quality data from all contributing grains. In combination with DAFi[3] and the sophisticated twin handling in APEX, individual grain contribution identification, data integration and structure determination from polycrystalline samples can be accomplished as efficiently as never before.

Diffuse scattering, 3D-ΔPDF

X-ray diffraction is able to detect the periodicity of a sample, although no sample is ever perfectly periodic. Disorder in the sample leads to very weak diffuse scattering signals in the diffraction image. To extract useful information from diffuse scattering, extremely sensitive detectors are required to reliably detect these weak signals alongside the much stronger Bragg-reflections.

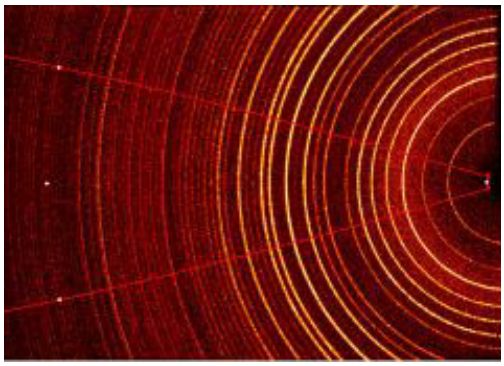
Additionally, a large active-area detector, such as that provided by the PHOTON series, is essential for efficiently mapping the complete diffraction space. Evaluation relies on 3D-ΔPDF[4] analysis applying a holistic approach to deliver detailed insight into the local structure of the compound under investigation.



Micro Powder Diffraction (µPXRD)

The small, intense X-ray beams paired with large 2D area detectors create optimal conditions for quick PXRD measurements on small sample quantities. While dedicated PXRD systems are unmatched, Bruker's SC-XRD equipment, with an angular resolution as low as 0.11° FWHM, serves as a robust tool for µPXRD on minimal sample quantities to rapidly identify phases, check phase purity, or assess crystallinity.

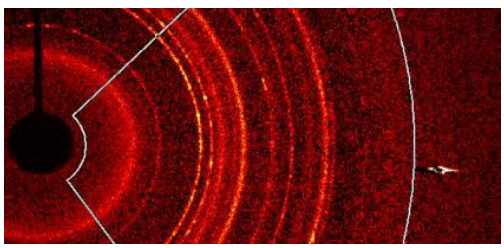
The unique combination of APEX[5] and DIFFRAC.EVA[6] facilitate easy and efficient processing of the data from SC-XRD's large 2D detectors.



The availability of multiple wavelength and potential integration with previously described non-ambient equipment offers a wide range of experimental flexibility which will be briefly described.

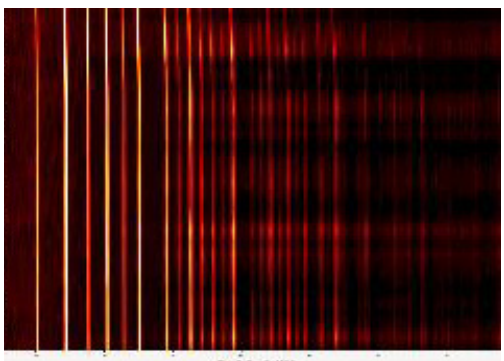
High-Pressure Micro Powder Diffraction (HP-µPXRD)

The concept of µPXRD can be combined with high-pressure DAC cells as described above. The D8 QUEST and D8 VENTURE provide all the experimental flexibility and tools to conveniently execute µPXRD in a DAC cell. In fact, HP-µPXRD benefits from the small, brilliant beam which provides significantly better signal to noise without any parasitic contribution from the cell itself.



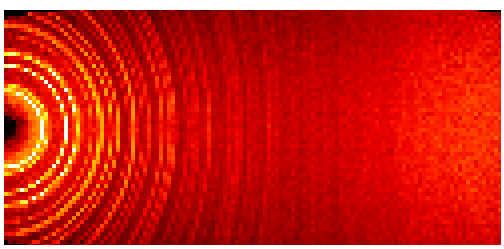
Variable Temperature SC-XRD and µPXRD (VT-SC-XRD/µPXRD)

To investigate phase transitions and material performance requires a flexible setup with access to a wide temperature range. Our SC-XRD systems can accommodate both low- and high-temperature devices to cover a total temperature range from 80K to 1300K. This makes our SC-XRD solutions equipped for variable temperature SC-XRD also ideal for µPXRD. Serial measurements of single crystals or powder over a range of temperatures can be easily set up within the APEX software suite enabling convenient investigation of the effects of temperature on the sample.

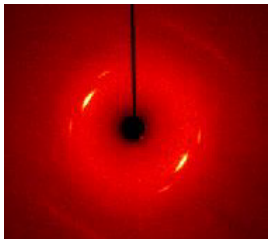


Micro PDF (µPDF)

The PDF[7] technique analyzes the local structure of both crystalline and non-crystalline materials. In 3D-ΔPDF, it is important to accurately determine even weak signals at very high resolution for the experiment to be successful. The availability of hard radiation (Mo, Ag, In) and large, highly sensitive detectors provides a significant advantage for collecting PDF data.

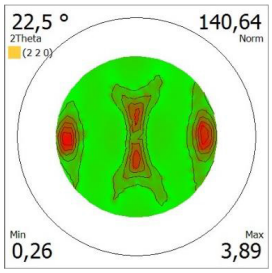


WAXS Analysis on polymers



Wide-angle scattering[8, 9] is a powerful tool for analyzing structural properties such as crystallinity, crystal sizes, and conformation. The advanced SC-XRD setup offers the flexibility to conduct WAXS measurements. In this example, the degree and orientation of crystallinity in a post-processed polymer are determined. Similarly, stretch and durability investigations can be carried out on a D8 QUEST or D8 VENTURE.

Texture Analysis



X-ray texture measurements[10] analyze the crystallographic orientation distribution within polycrystalline materials. The large detector of the D8 QUEST and D8 VENTURE provides a large γ-range. Therefore, typically only one φ-scan is needed to record a high-quality pole figure. The availability of two different wavelengths enables effective probing of materials with different thicknesses using a single instrument.

Conclusion

Single crystal X-ray instrumentation is optimized to provide best data for high quality structure determination. Modern instruments are also capable of conducting various other X-ray experiments. We have demonstrated that the D8 QUEST and D8 VENTURE, both can generate high-quality data beyond the standard SC-XRD approach. This overview is not exhaustive, and we believe that innovative thinking will lead to many more applications. Bruker has a strong tradition of supporting creativity by incorporating new technologies in hardware and software.

References

[1] A. Goeta, J. Howard, *Chem. Soc. Rev.*, **2004**, 33, 490.
[2] A. Katrusiak, *Acta Cryst. A*, **2008**, 64, 135–148.
[3] Aslandukov et al., *J. Appl. Crystallogr.*, **2022**, 55, 1383.
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[6] *DIFFRAC.EVA*, v7.1, Bruker AXS SE.
[7] D. Keen, *Crystallogr. Rev.*, **2020**, 26.
[8] Van der Heijden et al., *Macromol.*, **2004**, 37, 5327-5336.
[9] Grünig et al., *Macromol.*, **2021**, 54, 4816 – 4826.
[10] *International Tables for Crystallography*, **2019**. Vol. H, Ch. 2.5.4.2, 136-140.

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