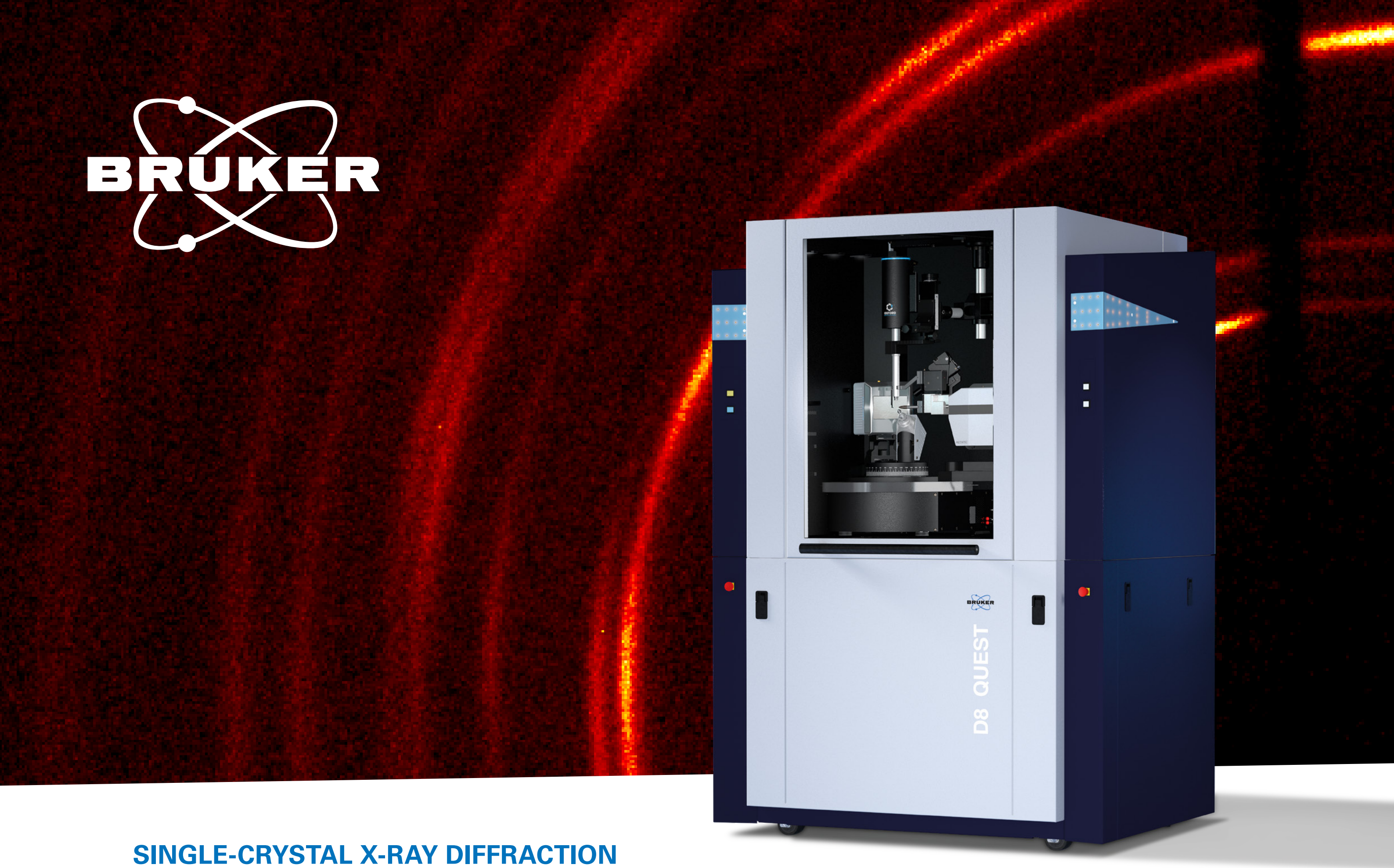

SINGLE-CRYSTAL X-RAY DIFFRACTION

Beyond SC-XRD: Texture Measurements

Running a texture measurement on the D8 QUEST

Introduction

Single-crystal X-ray diffraction (SC-XRD) is highly optimized for precise structure determination. To handle today's typically tiny samples, the instruments, such as the D8 QUEST or the D8 VENTURE feature a precise multi-axes goniometer, a high intensity X-ray source, and large active area 2D-detectors allowing an efficient and accurate detection of the diffracted signal. All instruments are equipped with a video microscope for sample alignment. Although dedicated to single-crystal experiments, the D8 QUEST and D8 VENTURE are versatile and can be successfully utilized for many other X-ray diffraction experiments.

This makes Bruker's SC-XRD solutions a valuable tool extending beyond structural analysis. Although a D8 VENTURE, offering a larger working space, would be equally well able to perform this analysis, in this Application Note we use our D8 QUEST to investigate the texture of a piece of aluminum metal.

Texture measurement (Figure 1) is a sophisticated, but established, technique used in material science to analyze the crystallographic orientation distribution within polycrystalline materials. This method provides detailed insights into the preferred orientation, or texture, of grains in a material, which directly affects its mechanical, thermal, and electrical properties.

X-ray texture measurements are essential in metallurgy, ceramics, semiconductors and geology. The data obtained from these measurements are key in understanding and predicting material behavior under various conditions. This enables researchers developing advanced materials with tailored properties and controlling or optimizing processing techniques in production.

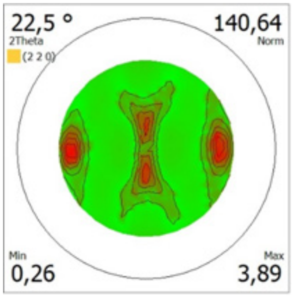

Figure 1
Example of a pole figure of processed aluminum.

Sample Preparation and Experiment

A plate of aluminum (diameter: 10mm, thickness: 2mm) was fixed on a flat holder, fitted on a standard XYZ goniometer head and mounted on a FIXED CHI (χ=54.85°) goniometer in a D8 QUEST (Figure 2).

The goniometer's ϕ-axis was positioned in line with the optical axis of the instrument's video microscope. The aluminum sample was roughly centered in the goniometer head's xy-plane, while the sample surface was accurately positioned at the goniometer center by adjusting the z-direction of the goniometer head, bringing the surface in focus to the video camera.

The sample was then moved to ω = 5° to minimize shadowing. A full 360° ϕ -scan was collected on the D8 QUEST equipped with an IµS DIAMOND II with Ag radiation and a PHOTON III 14 detector positioned at 2θ = 25° (Figure 3).

The best balance between resolution and γ-coverage was achieved using a detector distance of Dx = 100mm, a frame width of 5° per frame and an exposure time of 5 sec per frame leading to 72 images in total. All data was imported and analyzed using the Bruker texture software package DIFFRAC.TEXTURE.[1]

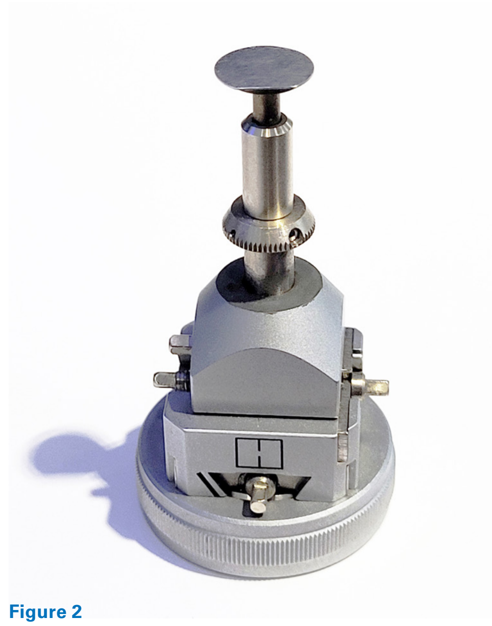

Figure 2
Aluminum sample on flat holder mounted on a standard XYZ goniometer head.

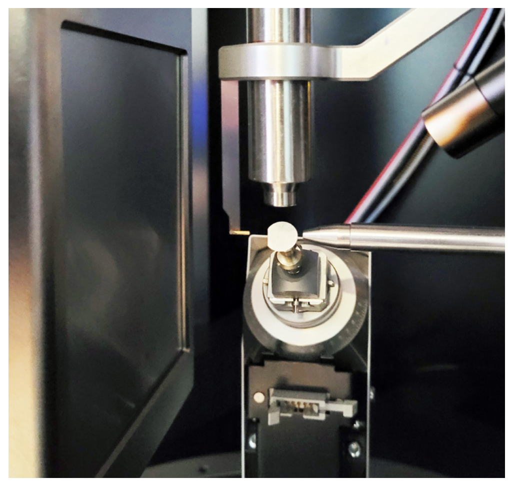

Figure 3
Sample mounted on the FIXED CHI goniometer of the D8 QUEST in measurement position.

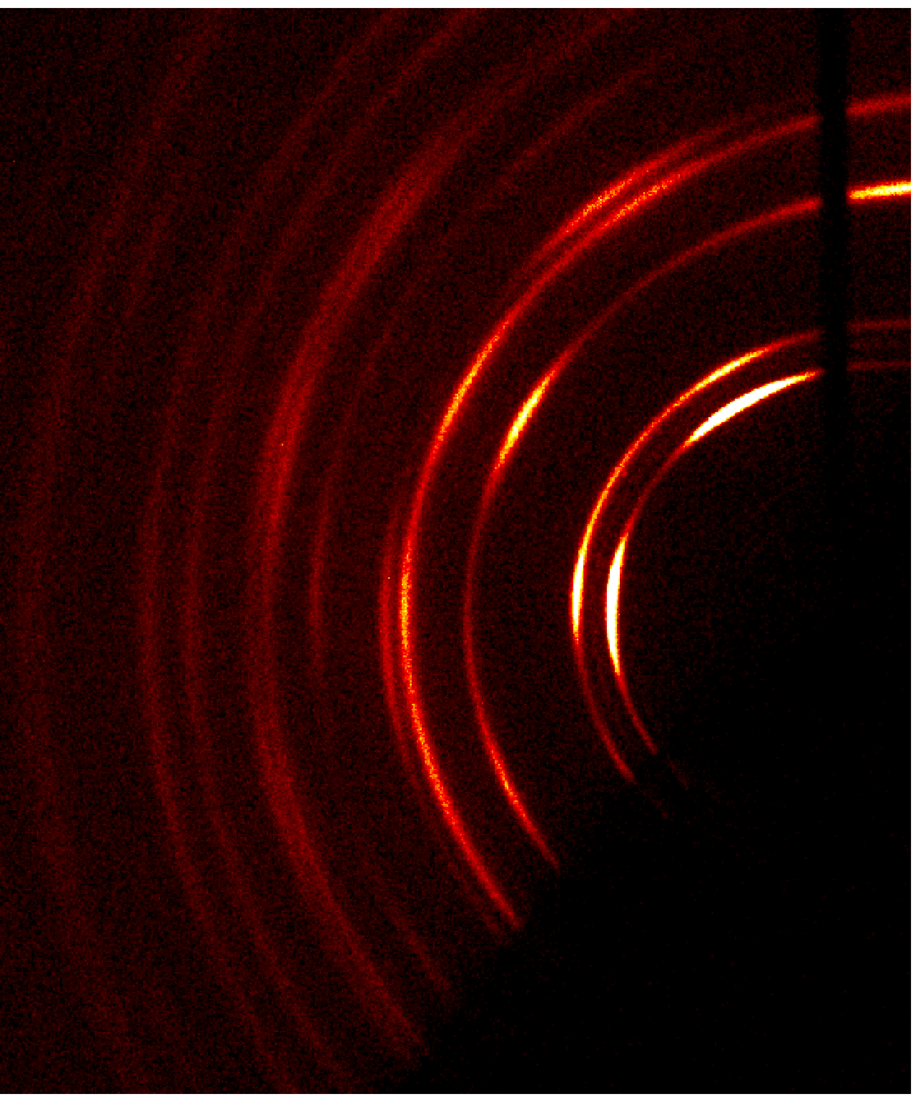

Figure 4
Diffraction image of the textured aluminum sample.

Results

The collected frames (Figure 4) exhibit excellent quality with well-defined diffraction rings, low background and a sharp contrast to zero-noise shaded areas. The presence of extensive texture effects in the sample is clearly visible from the intensity modulation along the diffraction rings. As determined from the d-values, the inner three rings correspond to the (111), (200), and (220) reflections (Table1). Further, higher order reflections are recorded right in the same image and can be analysed, if required for model fitting or consistency verification.

The complete set of 72 frames was processed with the DIFFRAC.TEXTURE software package. Based on this data the pole figures of the reflections (111), (200) and (220) were generated as shown in Figure 5. It should be noted that the PHOTON III 14 detector enabled collection of an impressively large γ-range by just one single ϕ-scan. Using a KAPPA goniometer would further enhance the γ-range.

The sample shows pronounced texture effects with a pattern typical for roll processed aluminum. A detailed interpretation often depends on the subject area in which the material is examined.[2],[3] Comparable measurements on the D8 DISCOVER, a dedicated materials research instrument, shows the exact same features substantiating the quality of the texture data collected on the D8 QUEST. [2],[3]

The choice of wavelength determines to which depth the sample will be investigated. Cu will probe the topmost 30 µm and therefore is more surface sensitive, while the higher penetration depth of the herein used Ag radiation (200 µm) on the other hand allows for a deeper insight into the material.

For routine texture analysis a dedicated instrument like the D8 DISCOVER is equipped with an χ-cradle and a XYZ-sample holder. This provides additional experimental flexibility to accommodate large or bulky samples, as well as conducting multiple position analysis and texture mapping. However, the results of this study show that basic texture measurements on a small sample can also be performed on a D8 QUEST or D8 VENTURE in notable quality.

Table 1
Reflection HKL and corresponding 2θ values for Cu and Ag radiation.

Reflection HKL	Reflection 2θ [°]	
Radiation	**Cu**	**Ag**
(111)	38.4	14.0
(200)	44.5	15.9
(220)	65.1	22.5

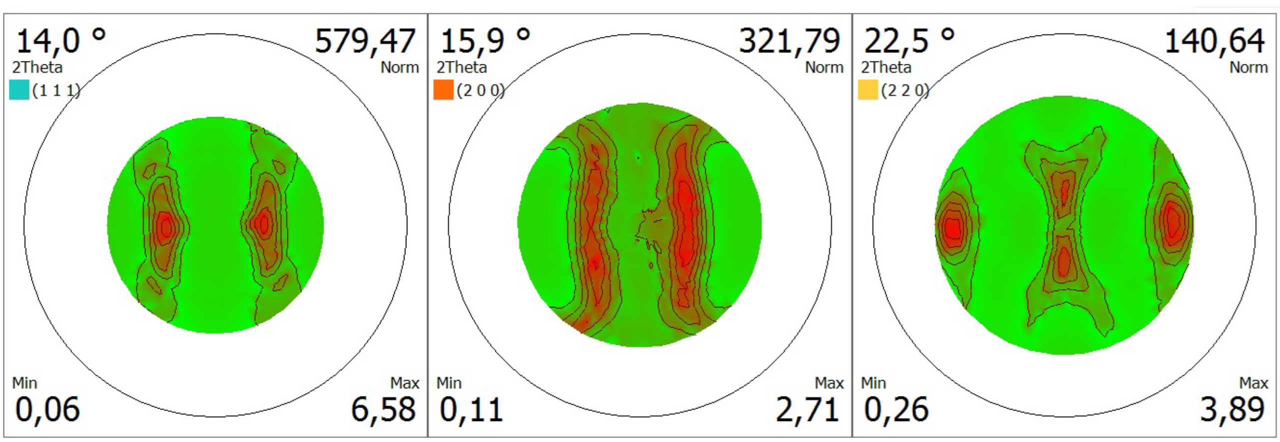

Figure 5
Pole figures of the reflections (111), (200), (220) based on data measured on a D8 QUEST with Ag radiation.

Conclusions

X-ray texture measurements analyze the crystallographic orientation distribution within polycrystalline materials. Routinely, these are conducted on multi configuration platform materials research instruments like the D8 DISCOVER providing the experimental flexibility to run all variations of samples and texture measurement.

Although SC-XRD instrumentation is highly specialized for structure determination we demonstrated in this work that high quality texture data can be obtained with these solutions. Benefits of the SC-XRD setup are the large detector, the brilliant X-ray beam and, optionally, the availability of two wavelengths. On the other hand, SC-XRD instrumentation is limited to small samples, precludes advanced texture measurements, and requires a little extra effort in sample preparation and experiment planning.

Routine texture measurements need to rely on dedicated instrumentation. The unmatched flexibility of the Bruker single crystal diffraction instrumentation nevertheless allows crystallographers to go beyond the core application. This enables SC-XRD operators to expand their portfolio to basic texture measurements if no access to dedicated materials research equipment is available.

References

1. DIFFRAC.TEXTURE V4.2.1

2. Application Note XRD 613, DOC-A88-EXS613, 2018, Bruker AXS

3. Application Report XRD 26, DOC-R88-EXS026, 2016 Bruker AXS

Authors

Dr. Kurt Erlacher,
Dr. Livia Chitu,
Dr. Hugues Guerault,
Dr. Tobias Stürzer

Bruker AXS

info.baxs@bruker.com

bruker.com