SINGLE-CRYSTAL X-RAY DIFFRACTION

Deep Learning Solutions for Smart Crystal Alignment

Unlock Smart Crystal Alignment with AI–Fast, Accurate and Reliable

Introduction

A well aligned sample, leveraging the small sphere of confusion of today's goniometers, is critical for collecting the best possible data in single crystal X-ray diffraction (SC-XRD) experiments. Video camera-based methods often struggle with variations in image quality caused by different sample mounts and crystal shapes, making it difficult to achieve optimal alignment.

On the other hand, experienced crystallographers typically outperform traditional sample recognition algorithms, highlighting the limitations of these conventional approaches. Accurate crystal alignment can be a significant barrier of entry into the field of SC-XRD. Wouldn't it be nice to streamline the process as much as possible and have a solution that automatically ensures perfect sample alignment?

Recently refined deep learning ("artificial intelligence", AI) technology has proven to be a powerful tool to address this problem. Here we report on how integrating AI into our X-ray diffractometers makes crystal alignment the most accurate and efficient (Figure 1).

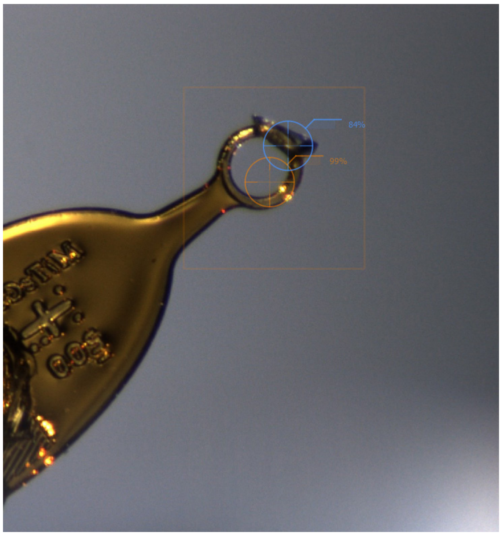

Figure 1
Visual feedback during deep learning assisted crystal centering.

How does it work?

New Deep Learning techniques are impacting many aspects of modern research [1]. One area where deep neural networks have proven powerful is image processing and interpretation.[2]

We built on top of these methods developing a novel technique to streamline the process of aligning crystal samples to the center of a single crystal X-ray diffractometer. A multi-staged neural network delivers the backbone for the real time analysis of the live camera feed. The network emulates typical steps in the decision process of an expert user:

- Evaluate lighting conditions
- Find the relevant region of interest
- Check for icing and other pathologies
- Identify the loop/mount center
- Identify the position of the sample.

For all steps real time feedback is provided to the user and can be utilized as a guiding tool, or as a sample alignment automaton (Figure 2).

The intense training on numerous samples with highly varied conditions created a reliable model. For a typical crystal of 50 µm in size, AI centering identifies the crystal with an accuracy of 98%. The crystal is accurately moved inside the beam path of a 75 µm wide beam in 99.5% of cases (Figure 3).

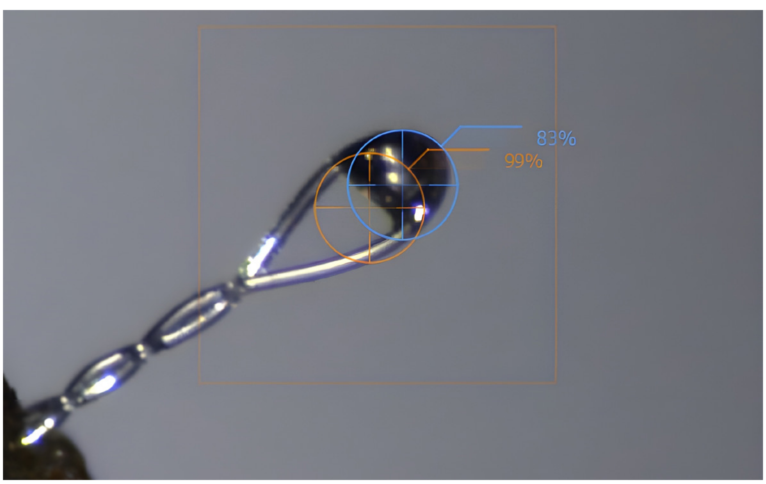

Figure 2
Real life feedback after the algorithm identified a region of interest, searched for the loop center and the crystal within this region. The confidence score is calculated and displayed.

Identify the crystal and center – all fully automated

The incorporation of the deep learning routines into our D8 X-ray diffractometers has revolutionized our way of crystal alignment when operating the instrument. After the crystal is mounted on a pin the sample is first placed on the automated goniometer head (AGH). Once the enclosure doors are closed, the video stream from the internal camera is automatically fed into the deep learning algorithm.[3]

This algorithm, trained to recognize and analyze the crystal's position, processes the camera feed in real-time. The revolutionary step of the new process is the fully automated and autonomous adjustment of the sample position to the center of the goniometer, right into the center of the X-ray beam. In practice this means that the crystal is centered before the operarator will sit down at the computer to start the experiment.

AI centering removes the need for manual intervention and eliminates human error. At the same time the process is much faster than in the past. This advancement consistently achieves the optimal sample position and consequently ensures highest experimental reproducibility. The routine is not only incredibly fast, but it is also robust and versatile. As shown in Figure 3, AI centering can deal with all typical sample mounts as well as with a large variety of crystal shapes and sizes.

The algorithm is reliable in recognizing samples whether they are mounted inside or outside of the loop. It handles all kind of crystals regardless of color or appearance. The routine is impressively powerful with poor sample illumination conditions and changing sample background. It even identifies a small crystal fully covered by ice (Figure 3 f).

No matter the size, shape or color of your crystal, the improved centering defines a region of interest and finds even ice-covered crystals. The new AI centering engine is available in the current version of APEX and PROTEUM – for fully automated or manual centering. Your new tool for fast, reliable, and effective autonomous centering.

The facts

- **98% Accuracy** – Assuming an average crystal size of 50 µm, 98% of predictions are within 50 µm of the ground truth.
- **99.5% Reliability** – Assuming a beam size of 75 µm, 99.5% of predictions place the crystal within the beam path.
- Crystal and mount detection via Deep Learning is feasible and practical for automated and semi-automated workflows.

- Current consumer-grade computer hardware is more than capable of using the model to make predictions.

- The resulting model is lightweight enough to run on any modern desktop or laptop PC, providing seamless real-time feedback to users of Bruker's APEX and PROTEUM software suites.[4]

By integrating AI technology, we once more push the boundaries of precision and automation in crystallography. This innovation marks a significant step forward in the streamlining of structural analysis.

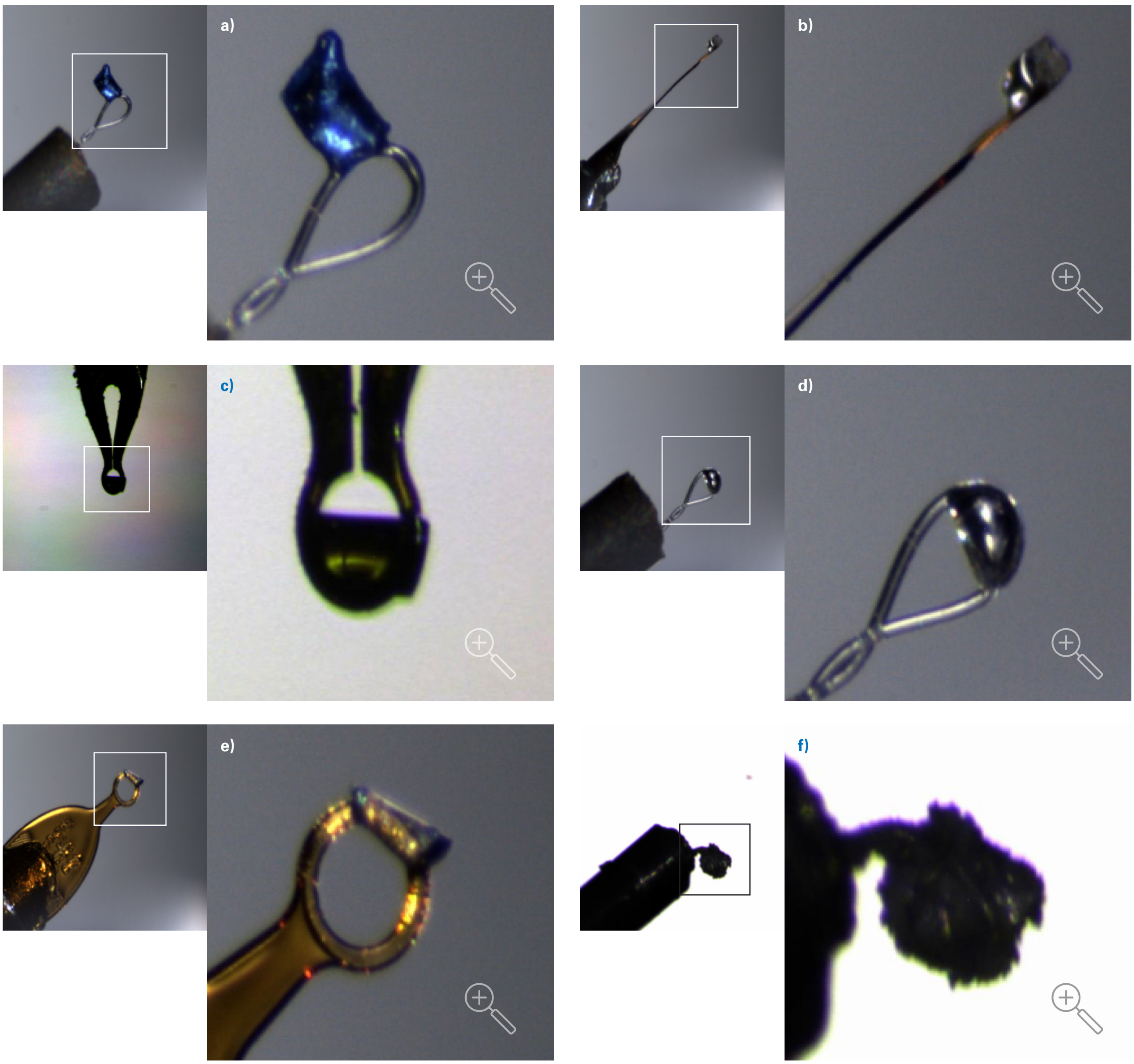

Figure 3
The AI based centering algorithm can successfully handle as variety of samples, such as:
a) large, blue colored crystal outside of a nylon loop
b) colorless crystal on a capton loop
c) dark crystal on a micromount
d) a colorless crystal in liquid drop on a nylon loop
e) colorless crystal aligned on the edge of a capton loop
f) a colorless crystal in a loop completely covered in ice.

Literature:

[1] Alzubaidi, L., Zhang, J., Humaidi, A.J. et al. (2021). *J Big Data*, **8**, 53.

[2] Ito S, Ueno G, Yamamoto M. (2019). *J Synchrotron Radiat*. **26** (Pt 4), 1361.

[3] Kaiming He, Xiangyu Zhang, Shaoqing Ren, Jian Sun (2015). Deep Residual Learning for Image Recognition. arXiv:1512.03385.

[4] Bruker AXS (2024). APEX: Version 2023.9, Bruker AXS., Karlsruhe, Germany.

Bruker AXS

info.baxs@bruker.com

bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/sc-xrd