

# Exploring diamond inclusions via combined Microtomography and single-crystal X-ray Diffraction at GSECARS

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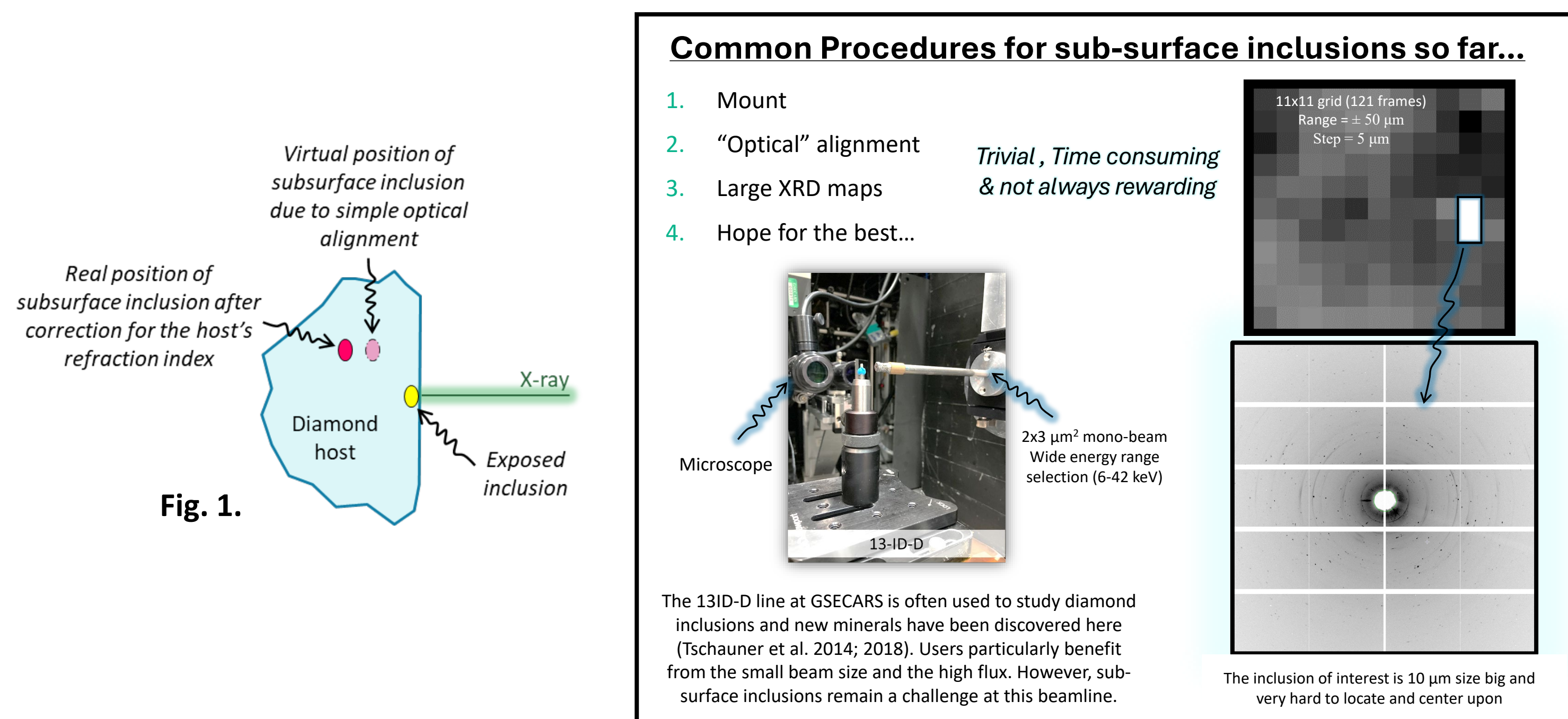


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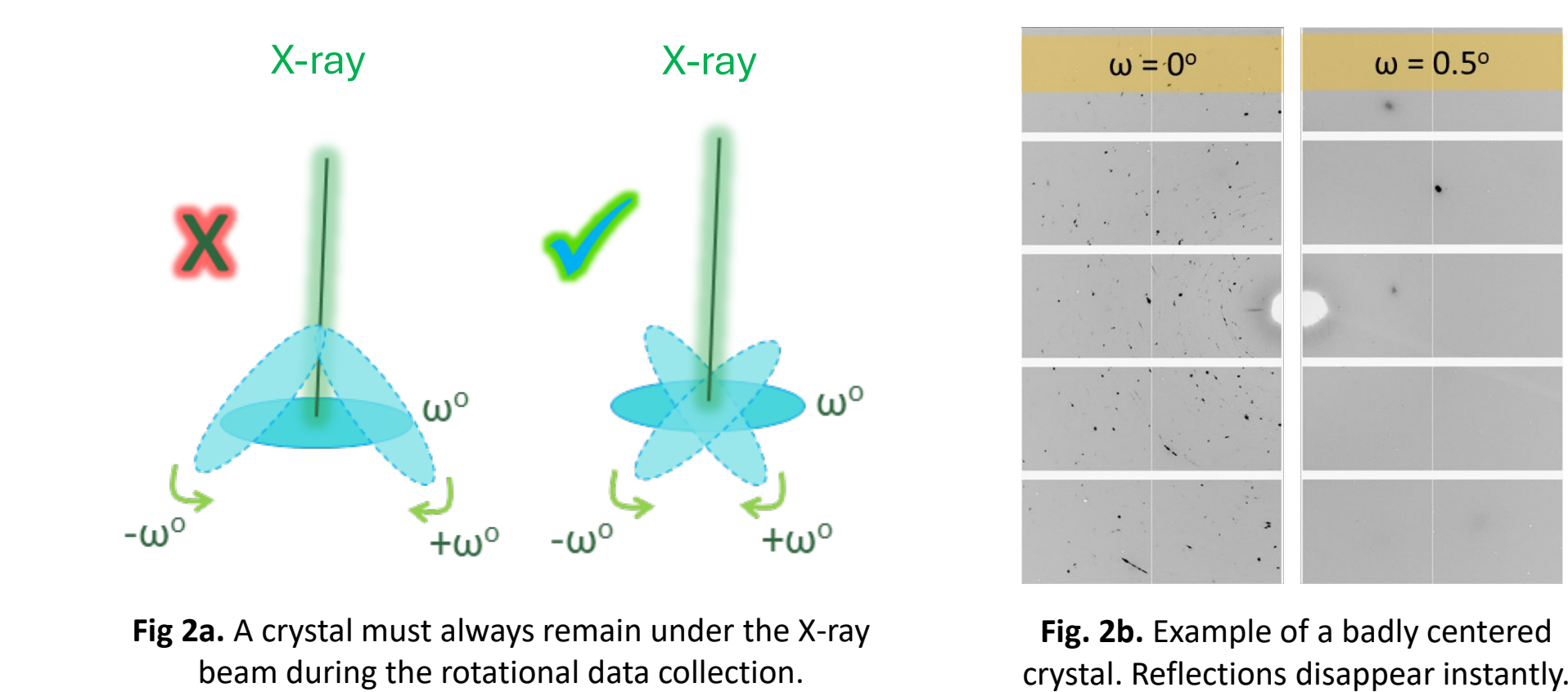
## Introduction to the problem

**Diamond inclusions** represent a direct window into the deepest layers of our planet and although rare specimens, the information they carry is invaluable in determining the mantle chemistry and understanding processes such as mantle convection and volatile cycling.

A **suite of techniques** may be used to study diamond inclusions and to identify the phases entrapped. However, many are considered destructive, often requiring exposure of the inclusion to the surface by diamond polishing. Such approaches pose a significant risk to the analysis and results interpretation due to possible alteration of the inclusions by exposure to the atmospheric air and pressure as well as loss of fluid phases.

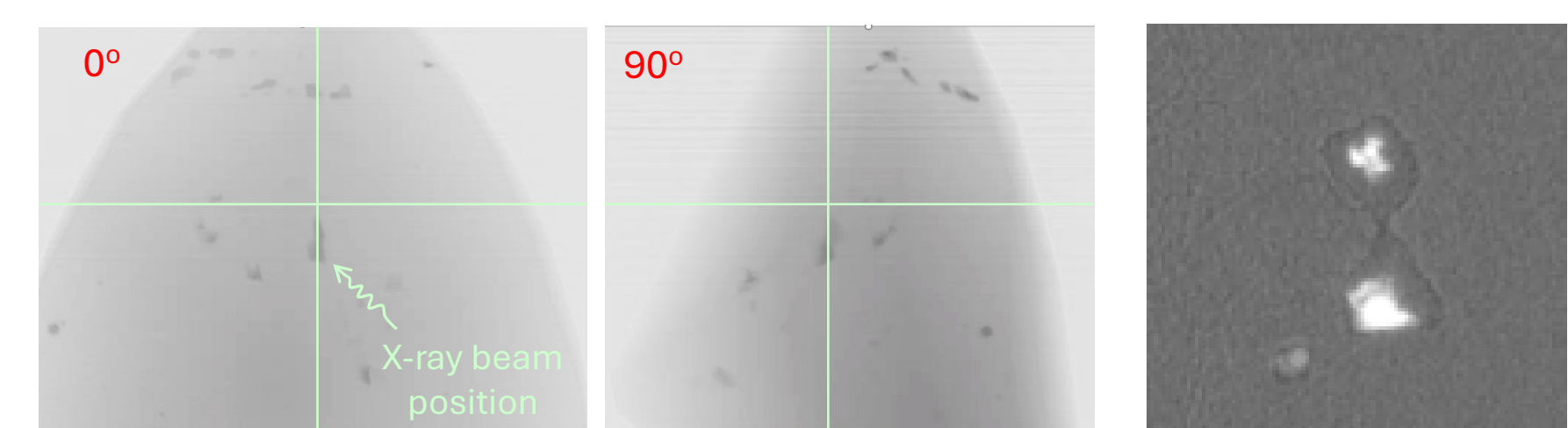


**Synchrotron single-crystal X-ray diffraction** is a powerful and unique tool in unravelling complex mineral assemblages by allowing accurate structure determination and phase identification. A critical requirement for a successful data collection is to bring the inclusion in the center of rotation (Fig. 2.). This is easily done for exposed inclusions using an optical approach (a.k.a. microscope camera). However, studying sub-surface inclusions presents a great challenge during alignment procedures. Due to the diamond refraction index, it is very hard to align optically on a non-exposed inclusion (Fig. 1.). A badly centered inclusion escapes the X-ray beam during collections (Fig. 2a.) which results in severely incomplete datasets (Fig. 2b.).



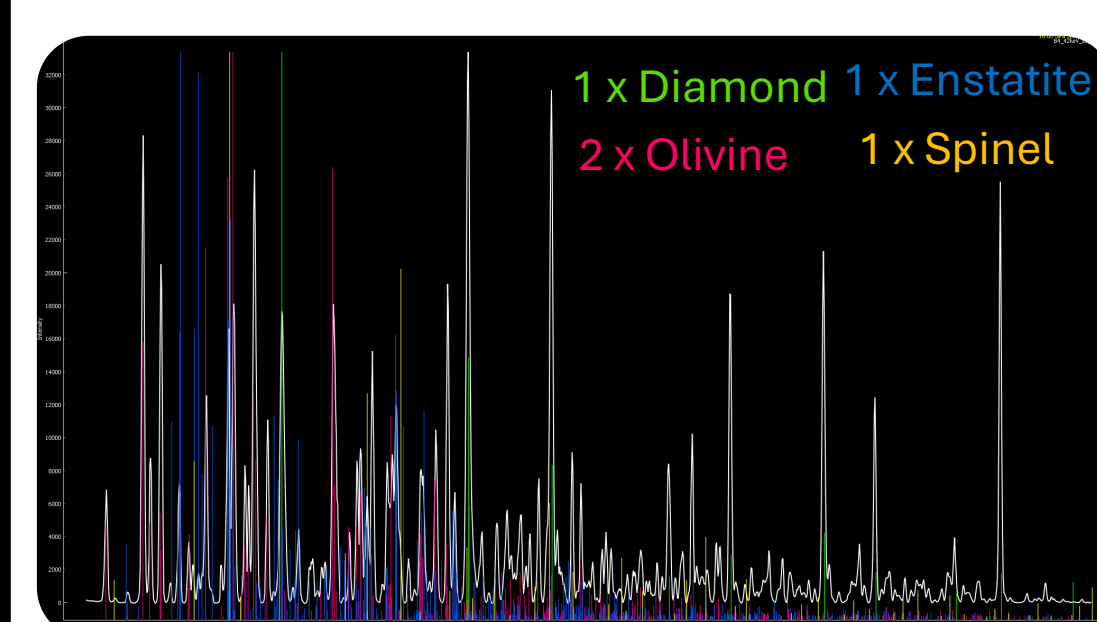
## Example 1 :

A sublithospheric diamond contains several sub-surfaced inclusions. The user wants to characterize one of them before they decide if it is worth risking to expose it in the surface for further analysis.



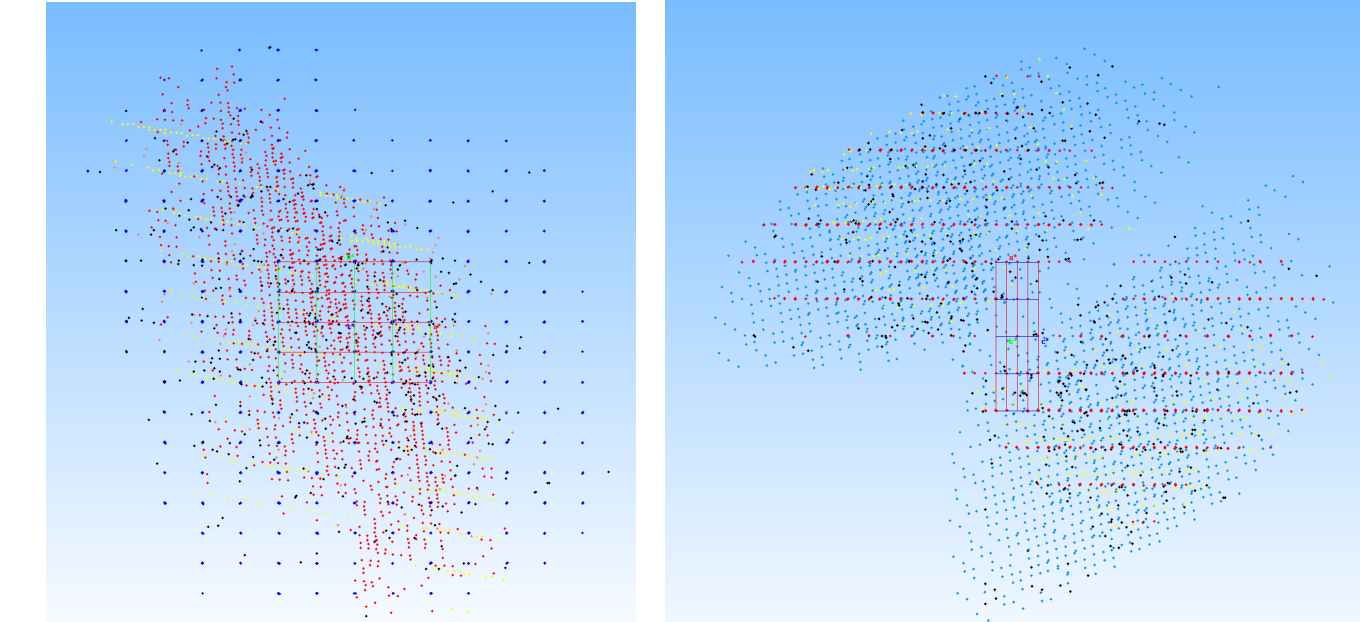
The inclusion of interest is clearly visible using X-ray imaging. The cross represents the X-ray beam position. We correct the XYZ position of the inclusion so that it remains under the X-ray during rotation.

The microstructure of the inclusion from the 3D reconstructed images.

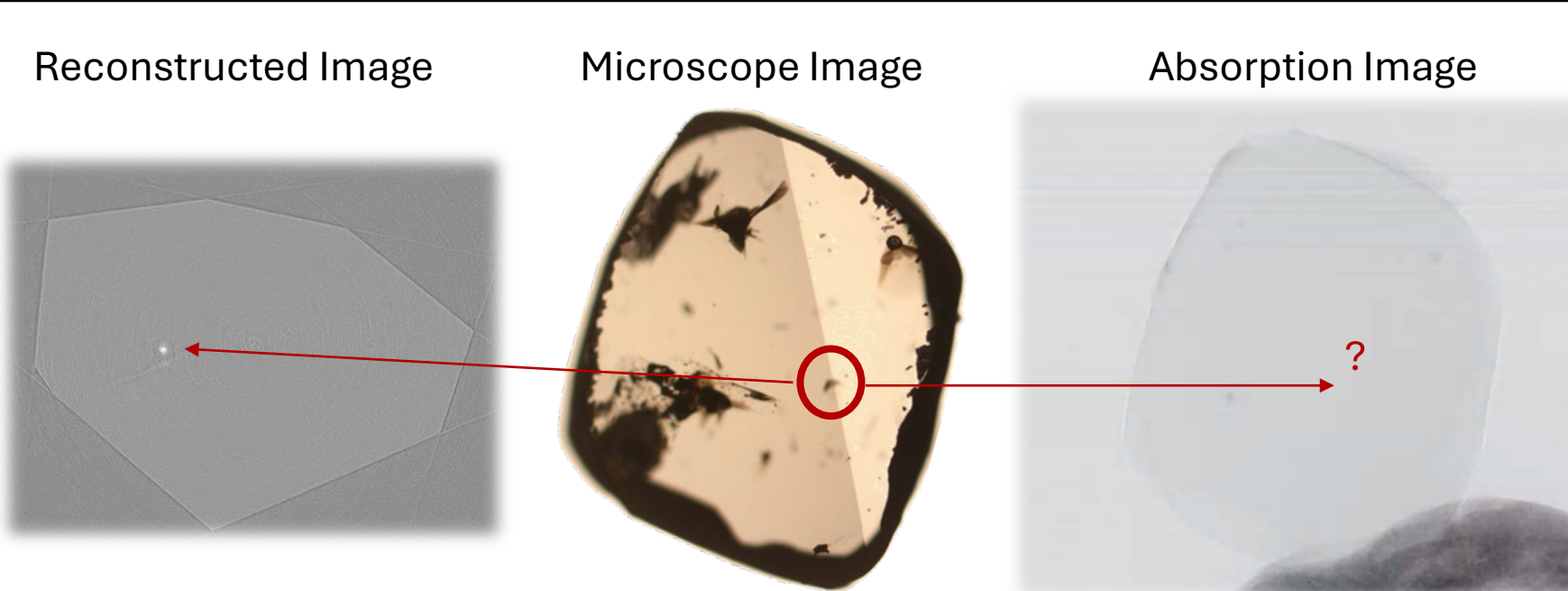


Conventional powder diffraction approach is not sufficient to deconvolute these complex XRD patterns.

A single-crystal XRD approach on this multiphase/multigrain inclusion allows us to identify all the present phases and in the case of new mineral, solve its structure.



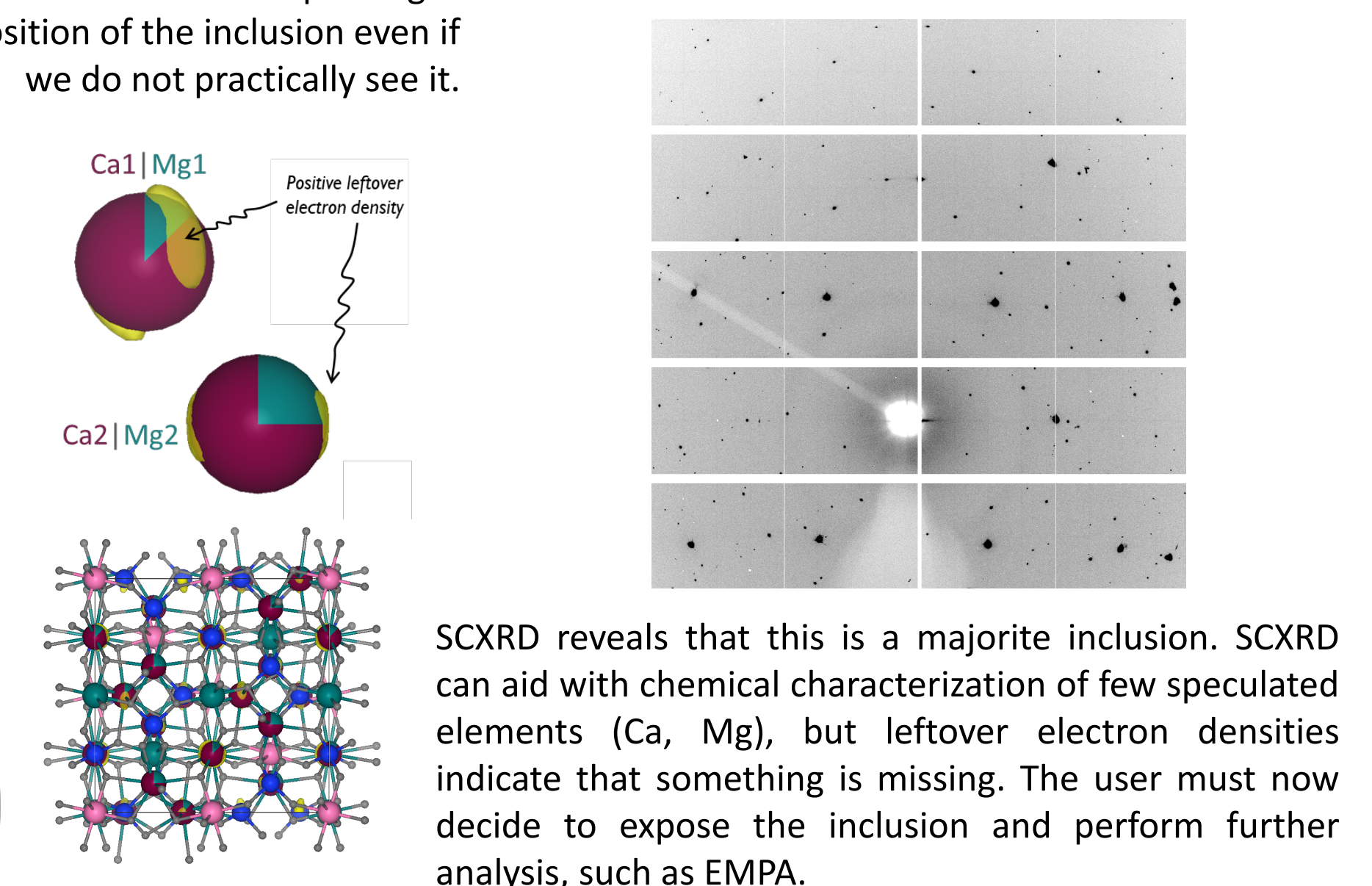
Software like CrysAlisPRO have excellent tools in untangling and reducing such datasets. Here we found reflections from 5 grains : diamond, enstatite, olivine and magnetite.



Using the reconstructed images, we can drive the sample stages to the XYZ position of the inclusion even if we do not practically see it.

## Example 2 :

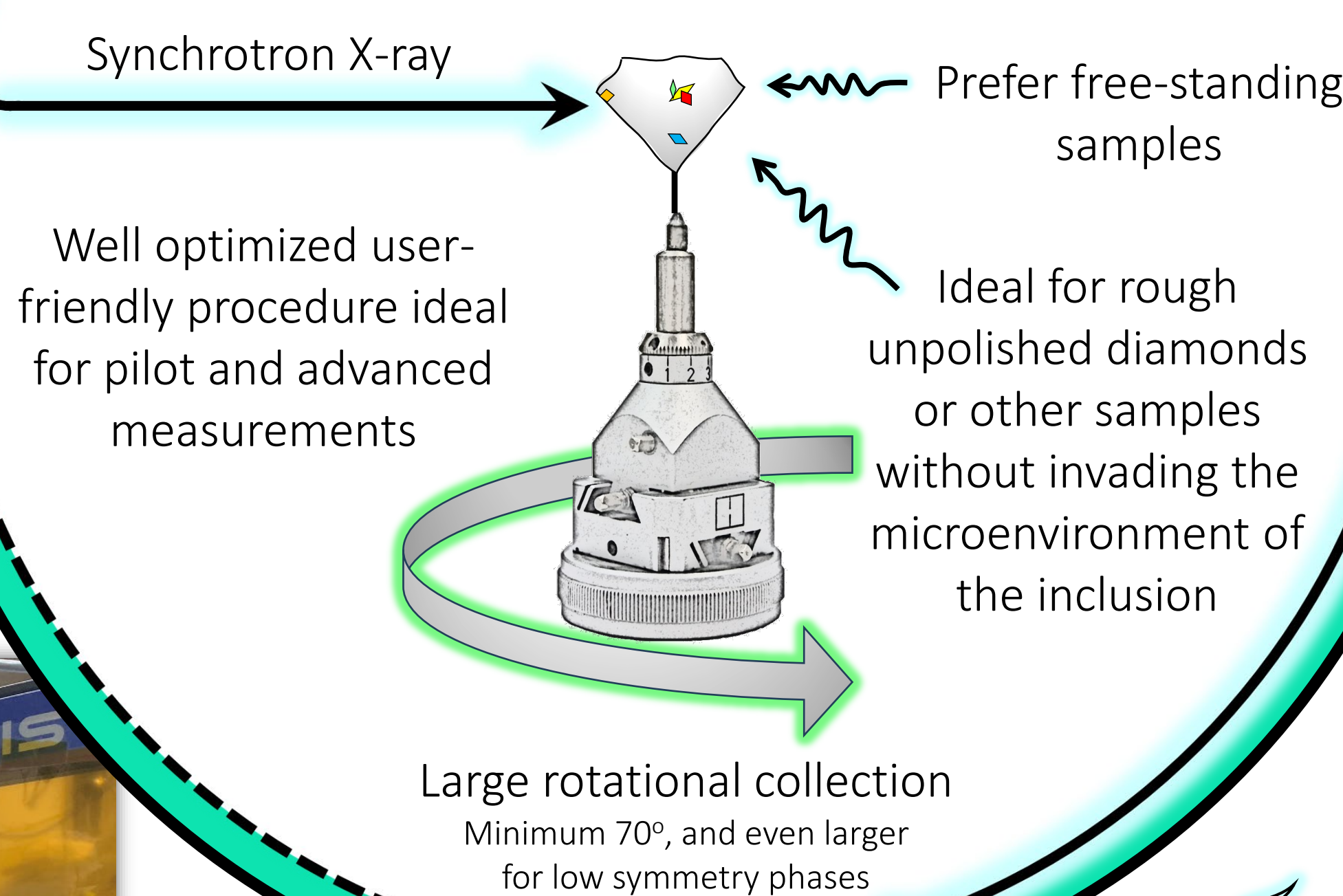
A user can see a subsurface inclusion under the microscope, but they can't see it using X-ray radiography, due to its low Z-elements content and poor absorption contrast.



## Overview

**STEP 1: X-ray Computed Microtomography**  
Locate, Visualize & Align

**STEP 2: Single-crystal X-ray Diffraction**  
Identify phases & solve crystal structures



## How to solve the problem

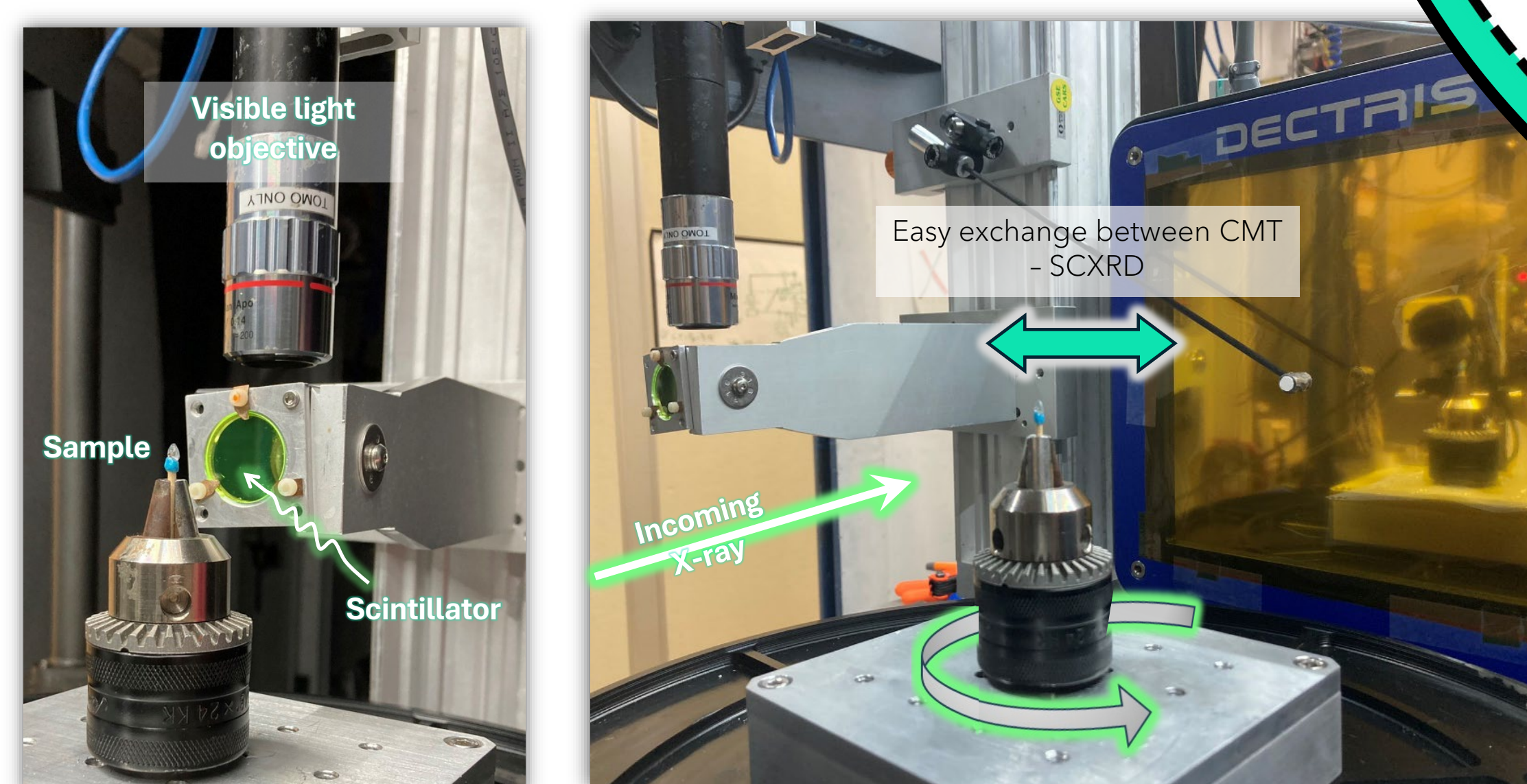
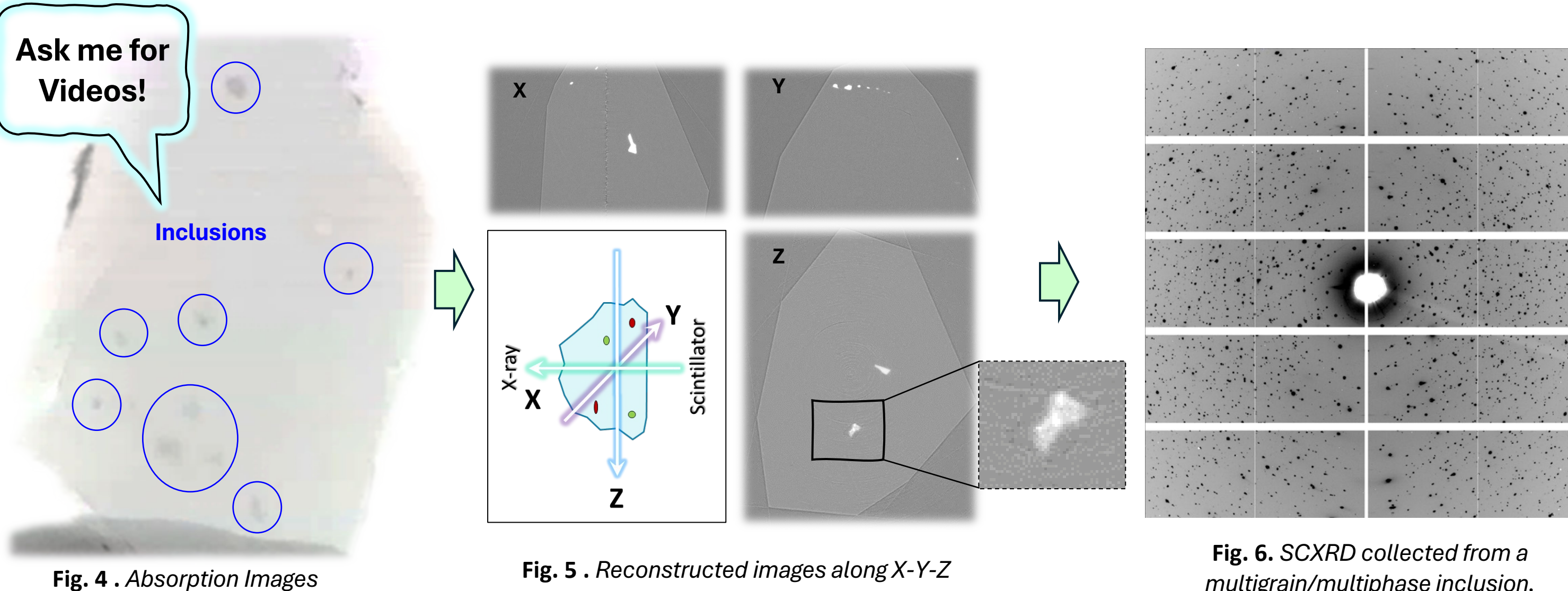


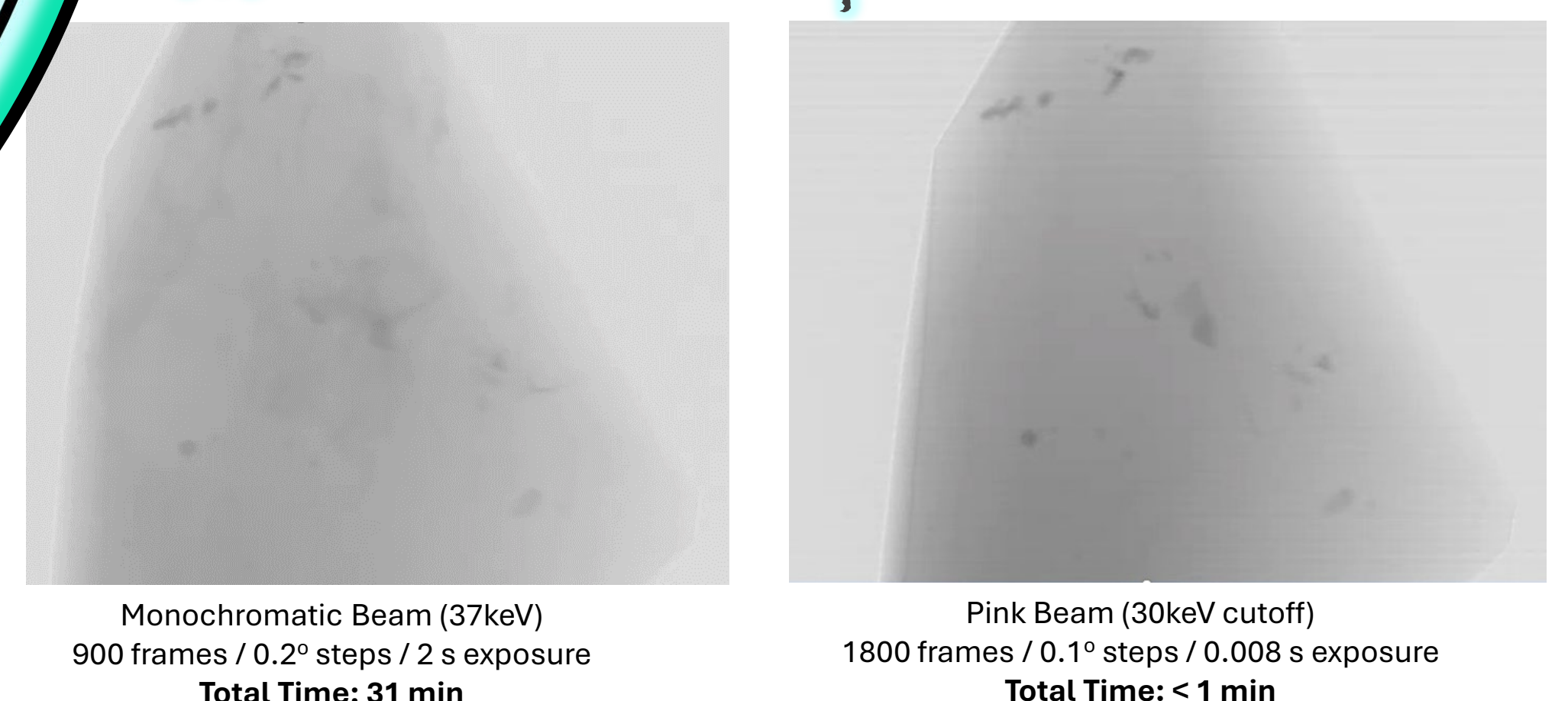
Fig. 3. The set-up used to study diamond inclusions at 13BM-D allows swift on-line alternation between the two techniques.

At the 13BM-D beamline at GSECARS we combined microtomography (CMT) and SCXRD to approach this problem:

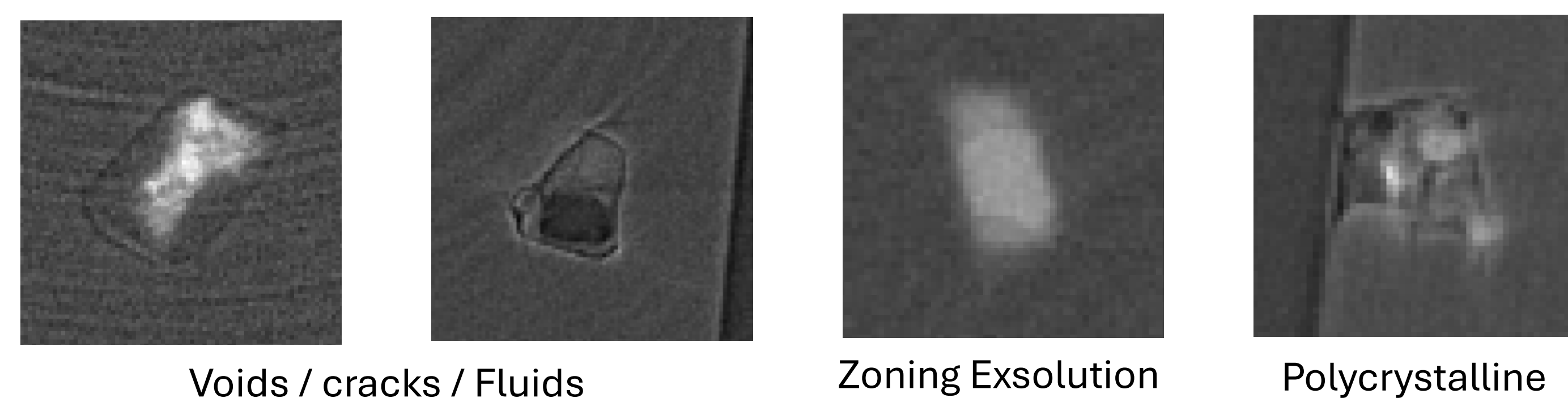
1. Scan diamonds with pink or monochromatic beam to produce a series of high contrast absorption images (Fig. 4.)
2. Filtered back-projection analysis produces their 3D reconstructed images (Fig. 5)
3. This allows the visualization, location and alignment of inclusions under the beam to collect SCXRD patterns (Fig. 6)
4. SCXRD data analysis allows accurate phase identification and structure solution and refinements



## Monochromatic or pink beam for CMT?



## Other Features



3D reconstructions can help reveal sample features such as density contrast, cracks, zoning, fluid rims etc. Such macroscopic characteristics are important in describing the history of the inclusion.

## Other Techniques

The **13BM-C** beamline hosts a six-circle diffractometer and is ideal for SCXRD studies.

A portable camera system allows X-ray radiography experiments on diamond inclusions.

Although not of the same spatial resolution as CMT at 13BM-D, the user here can still locate and align on strongly or semi-absorbing inclusions. There is the extra benefit of the multiple rotation axes which allows improved data completeness.

