

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

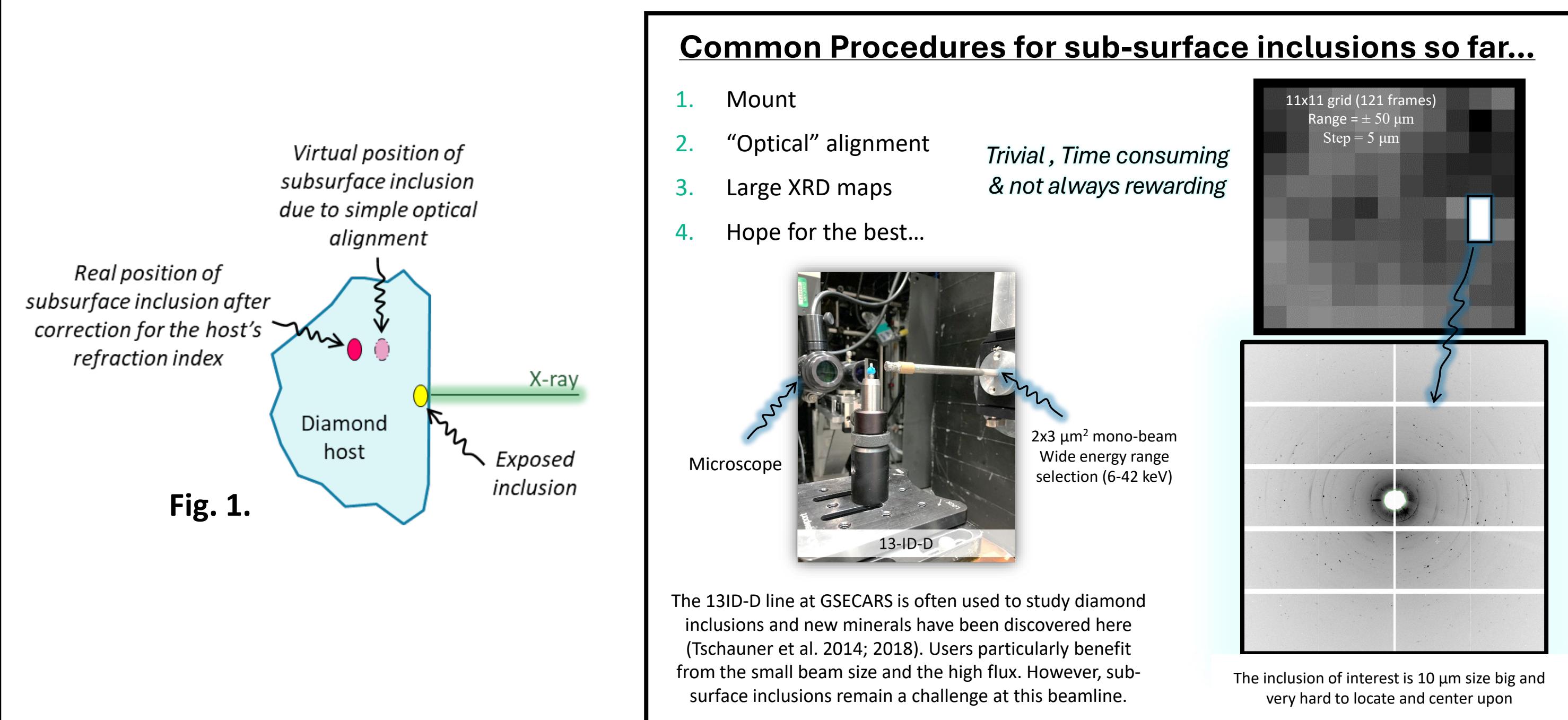
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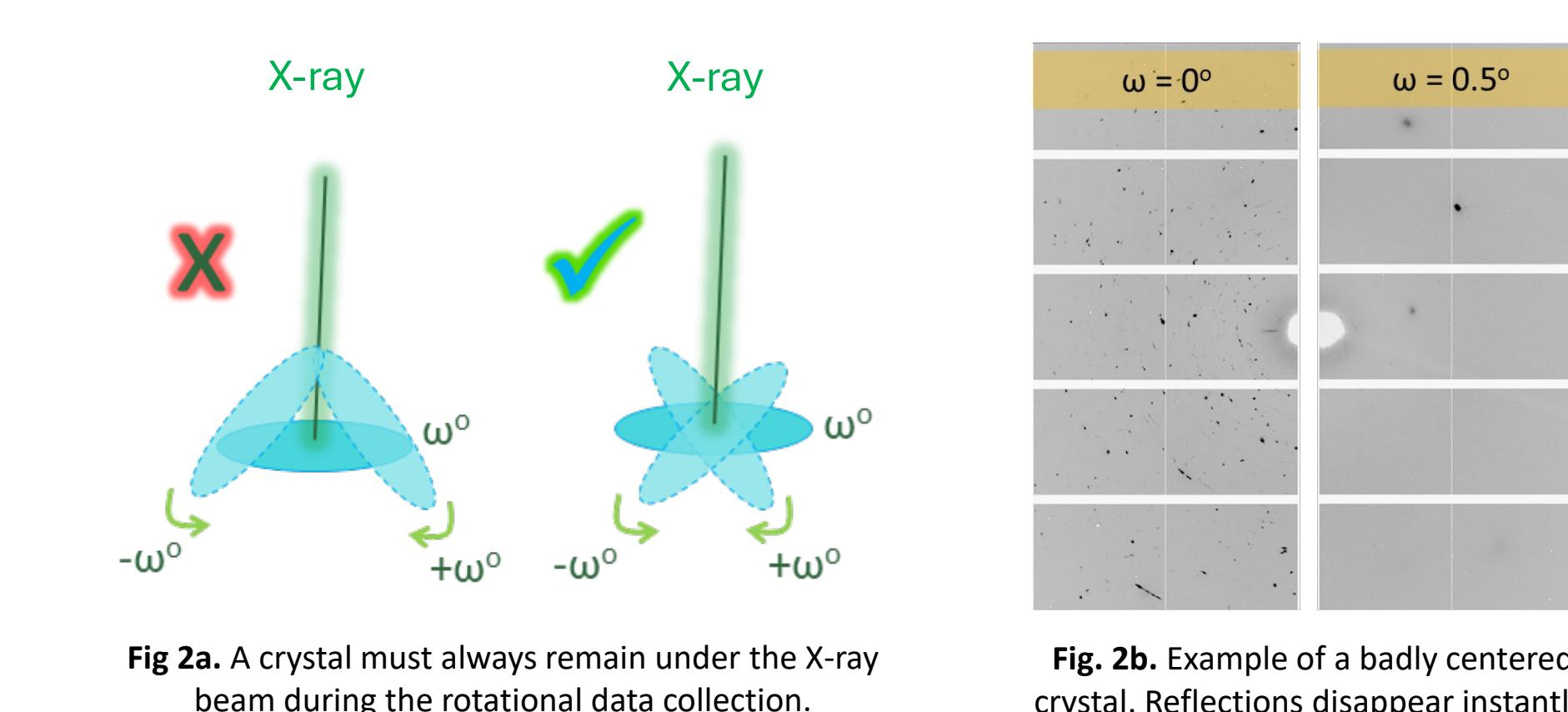
## Introduction to the problem

**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.

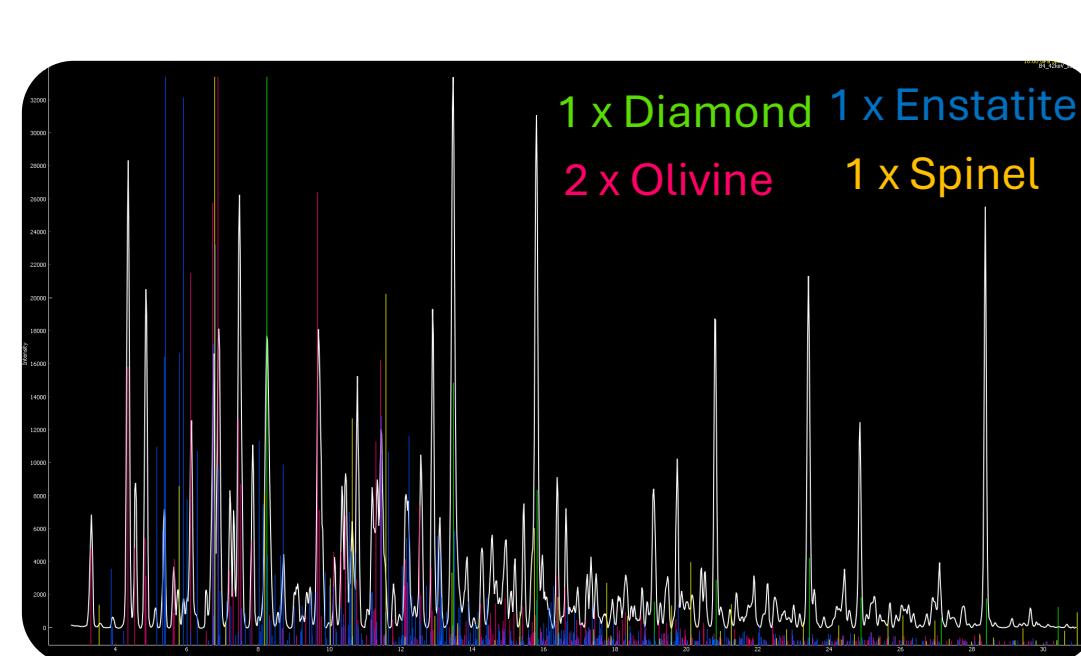


**Synchrotron single-crystal X-ray diffraction** is a powerful and unique tool in unravelling complex mineral assemblages by allowing accurate structure determination and phase identification. A critical requirement for a successful data collection is to bring the inclusion in the center of rotation (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).

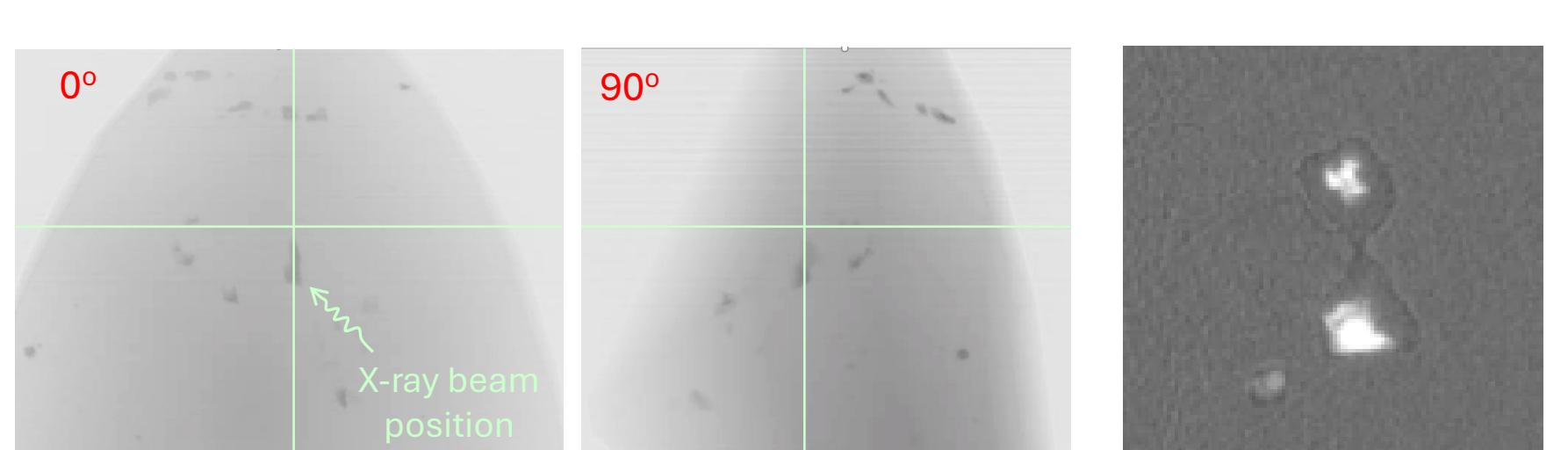


## Example 1 :

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.

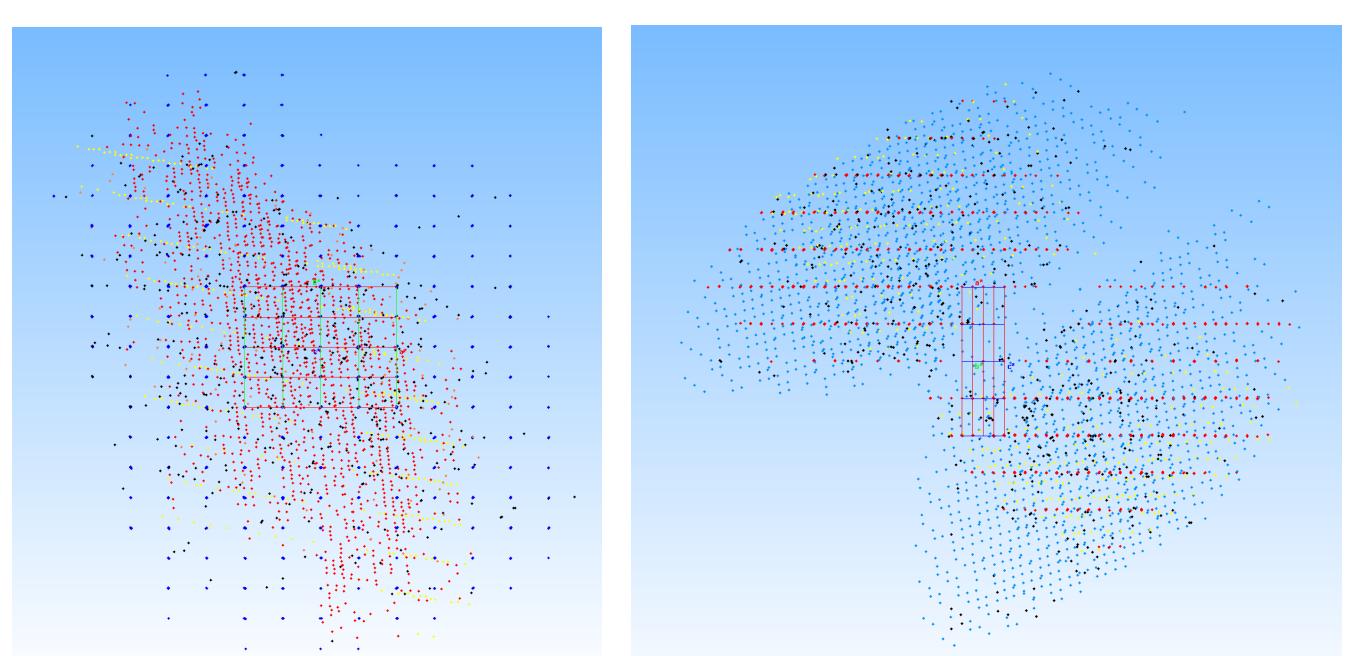


Conventional powder diffraction approach is not sufficient to deconvolute these complex XRD patterns.



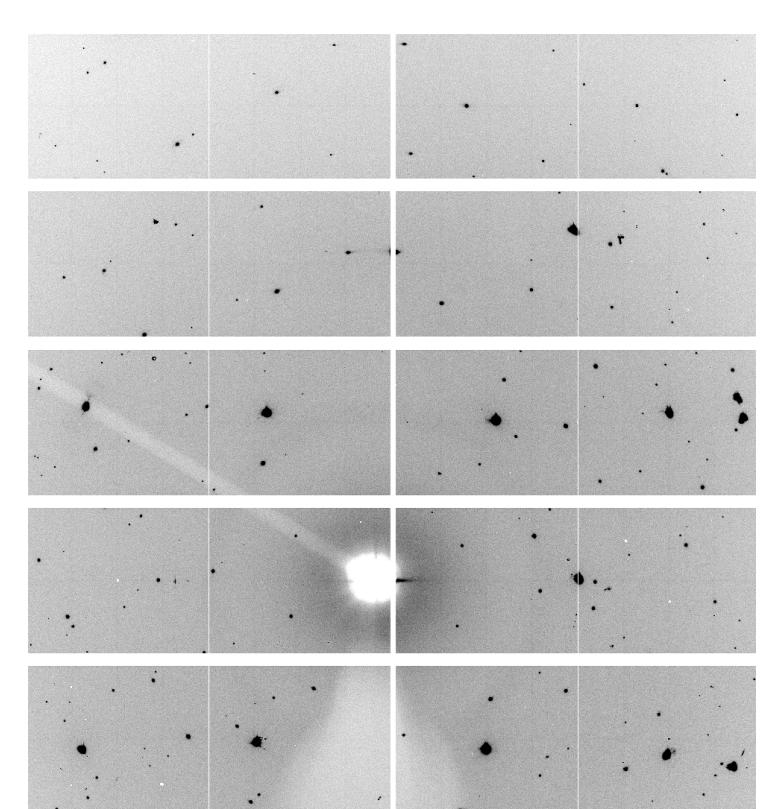
The microstructure of the inclusion from the 3D reconstructed images.

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.



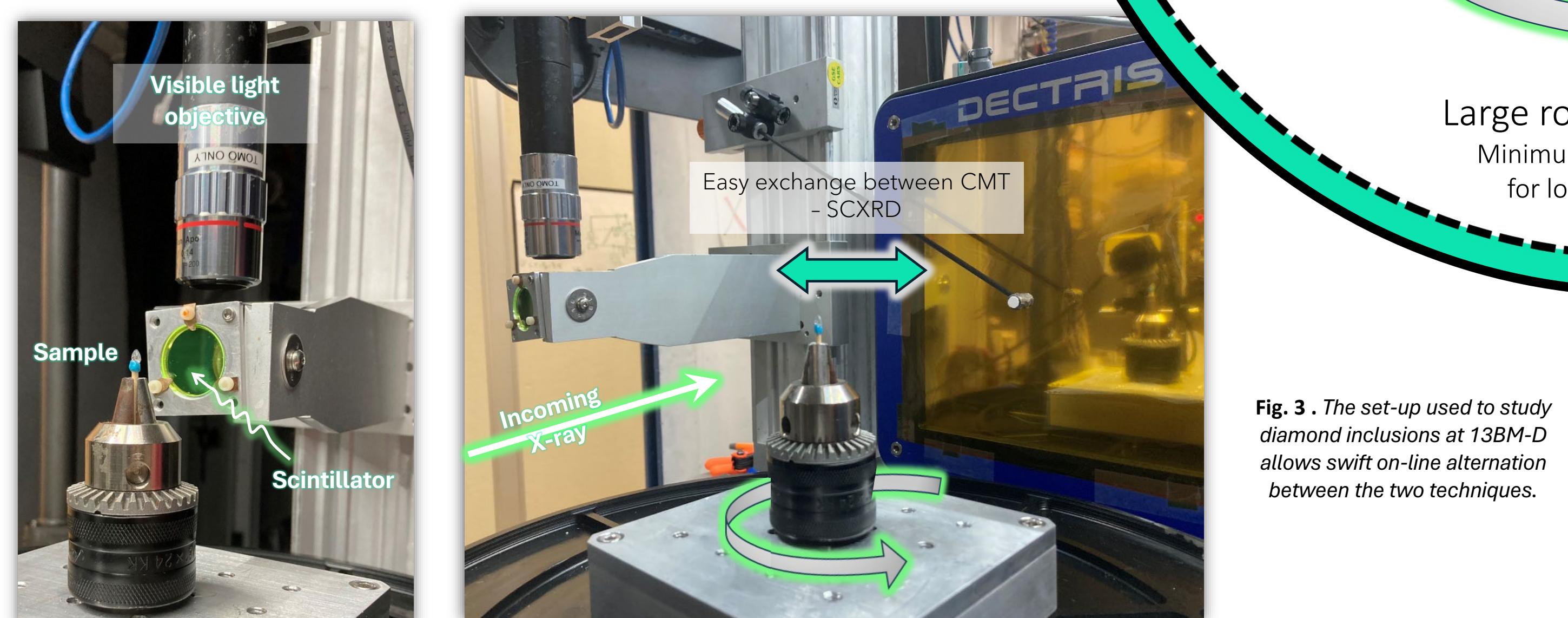
## Example 2 :

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.



SCXRD reveals that this is a majorite inclusion. SCXRD can aid with chemical characterization of few speculated elements (Ca, Mg), but leftover electron densities indicate that something is missing. The user must now decide to expose the inclusion and perform further analysis, such as EMPA.

## How to solve the problem



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

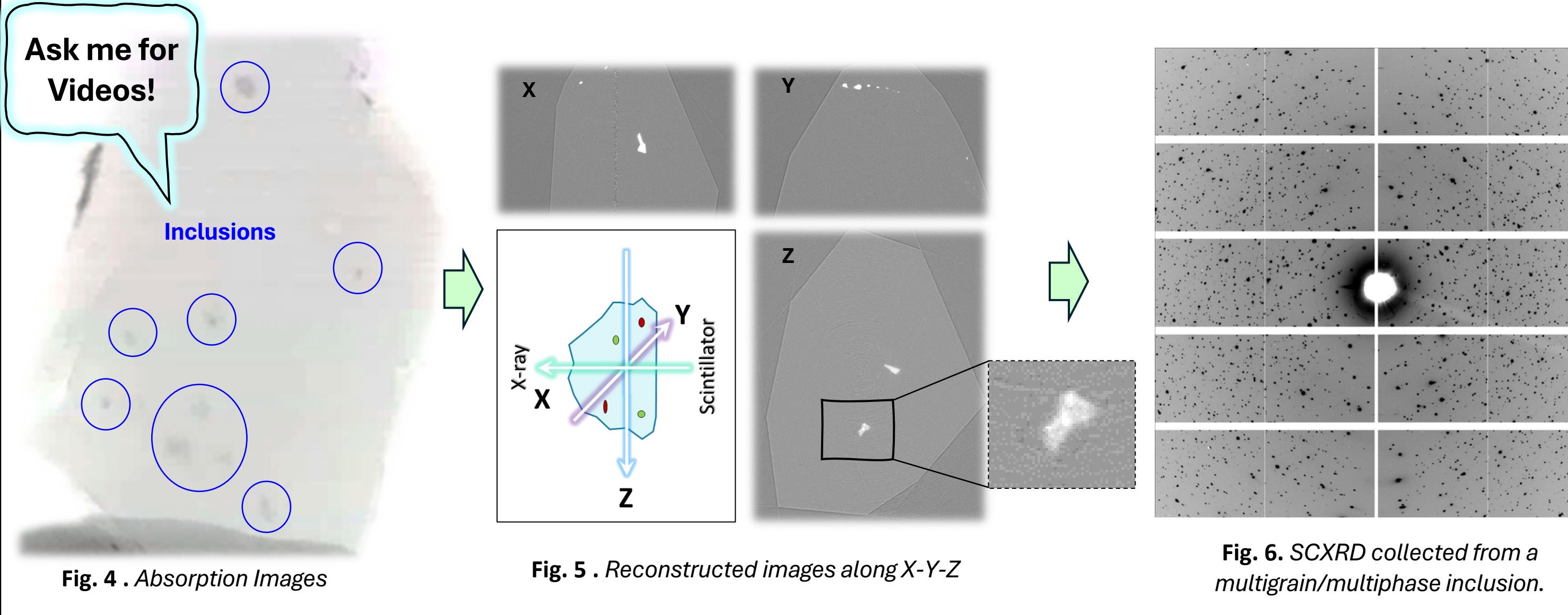


Fig. 4 . Absorption Images

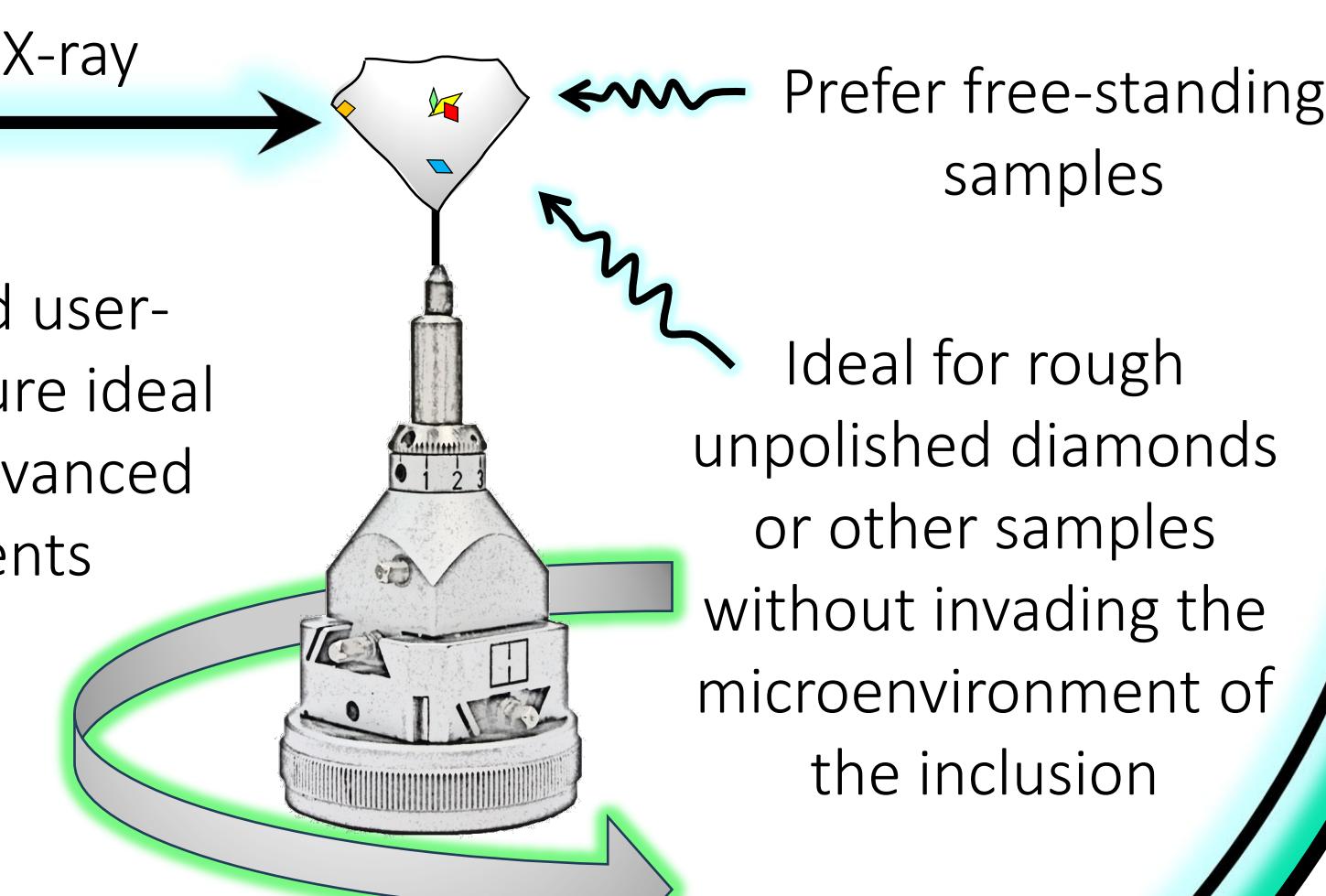
Fig. 5 . Reconstructed images along X-Y-Z

## Overview

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

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

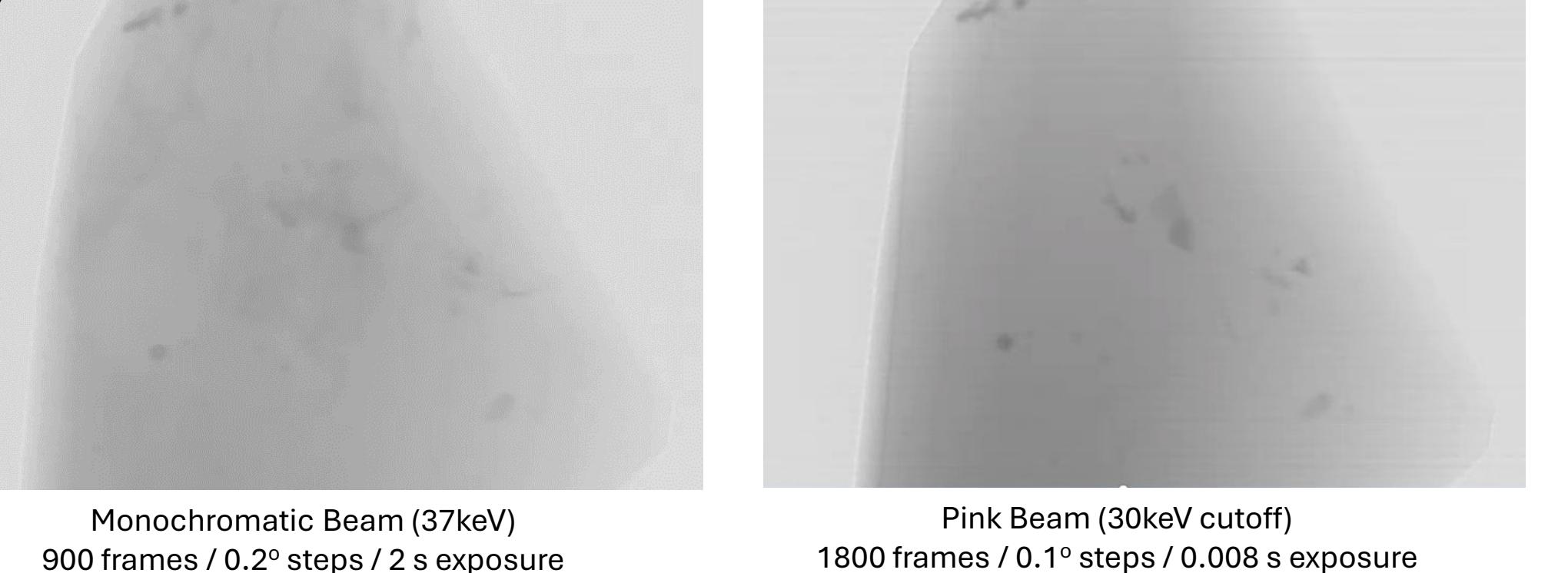
Synchrotron X-ray



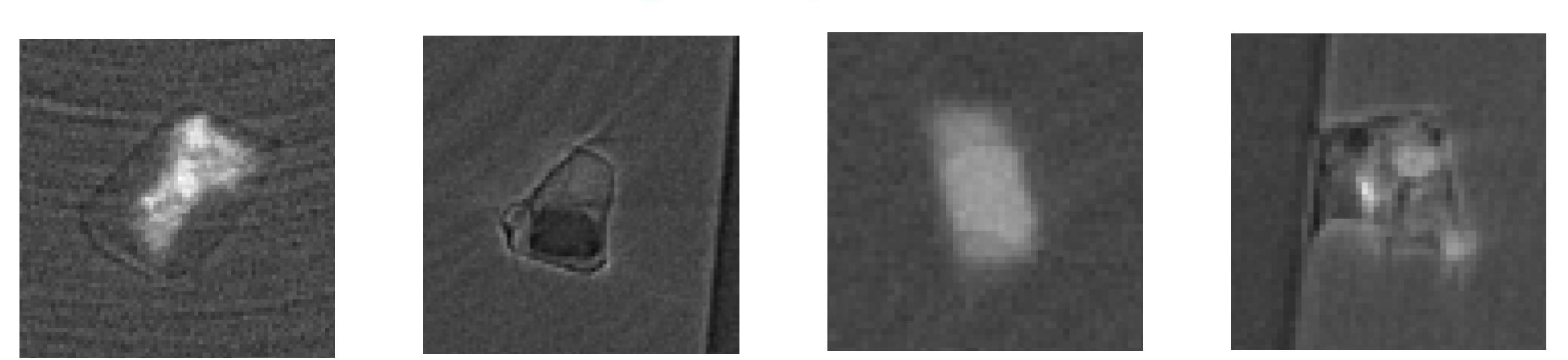
Large rotational collection  
Minimum 70°, and even larger for low symmetry phases

Ask me for  
Videos!

## 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.

