Beamline G3 at DESY: Materials X-ray Imaging

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Why beamline G3?

- The **majority** of typical diffraction experiments determine microstructures probing larger parts of the specimen and thus **averaging** over the sample.

- But the microstructure **might vary** over the sample especially if it is of inhomogeneous composition or has been inhomogeneously processed (e.g. soldering, welding, brazing).

Why beamline G3?

Beamline G3 enables:

- **lateral resolution** of the microstructure in **direct space,**
- **visual evaluation** of *distribution* of crystal phases, *size* of crystallites, *texture* or *strain*, *structure discontinuities* like surfaces, interfaces, phase or grain boundaries.

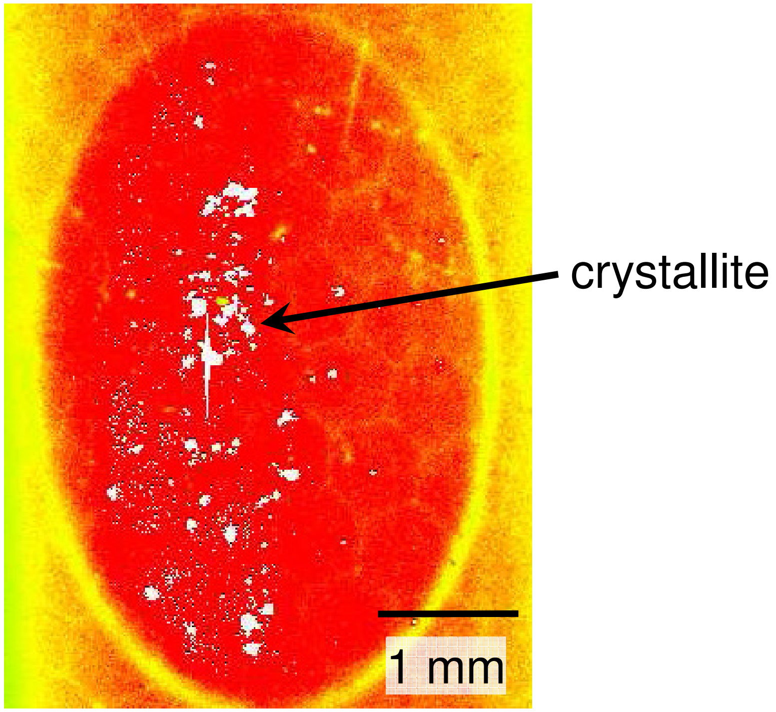


Fig. 1. Crystallites of β-Sn(312) on surface of solder joint

DORIS III at DESY

- **DORIS III**: storage ring for charged particles - positrons or electrons at an energy of 4.45 GeV, typically operates in 5 bunch mode.
- **Beam** current between 90-140 mA.
- **Synchrotron radiation** is produced in the bendings of the ring, where the insertion devices are located (wigglers, undulators, and bending magnets).
- **The spectrum** of DORIS III ranges from infrared radiation to hard X-rays, and is extreme intense particularly in the X-ray range.
- **31 beamlines**.
- **Circumference** of 289 meters.

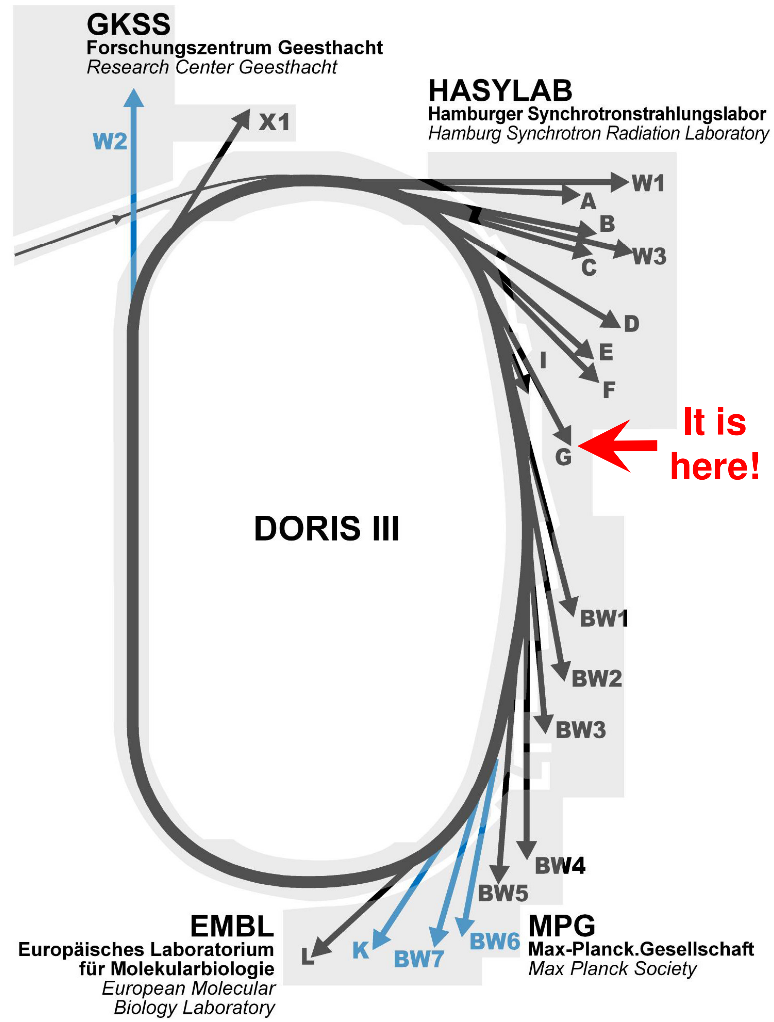


Fig. 2. DORIS III
[http://hasylab.desy.de]

Basic Parameters of Beamline G3

- **Monochromator:** fixed-exit double-crystal Ge (111), also further crystals available Ge (311), Si (111), Si (511), Si (400).
- **Wavelength range:** 1 Å < λ < 2.2 Å with mirror, down to 0.5 Å without mirror (standard Ge (111) crystal).
- **Beam dimensions** at monochromator: ~4x16 mm^2.
- **Flux at the sample**: ~10^8 sec^{-1} (depending on monochromator crystal and chosen energy range).

Beamline G3: Experimental Station

- Monochromator, slit system, air pressure shutter, scintillation counter, gold mirror.

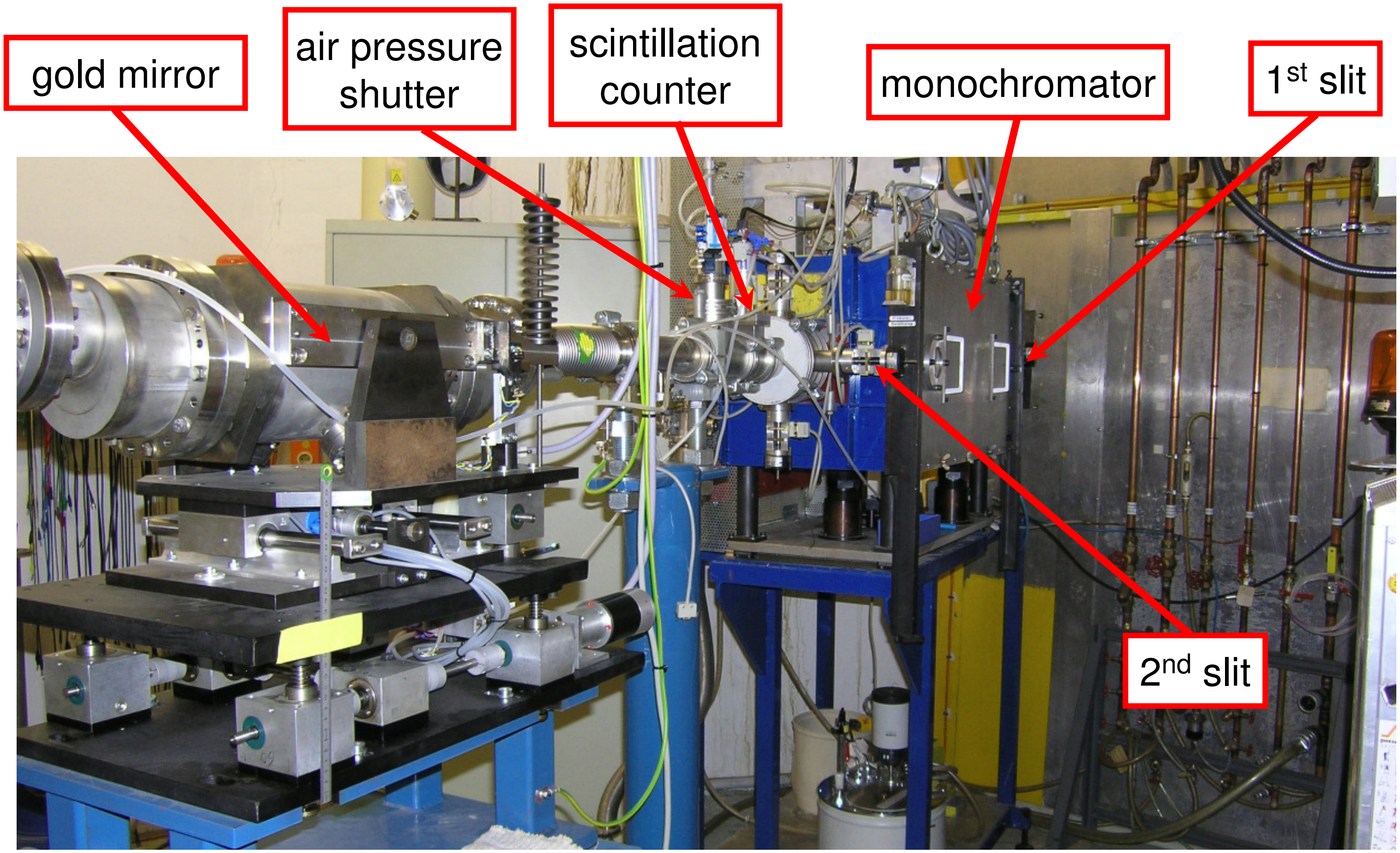


Fig. 3a. G3 experimental station
[Rothkirch]

Beamline G3: Experimental Station

- Beam tube, gold mirror, 2 detectors, 4 circle diffractometer.

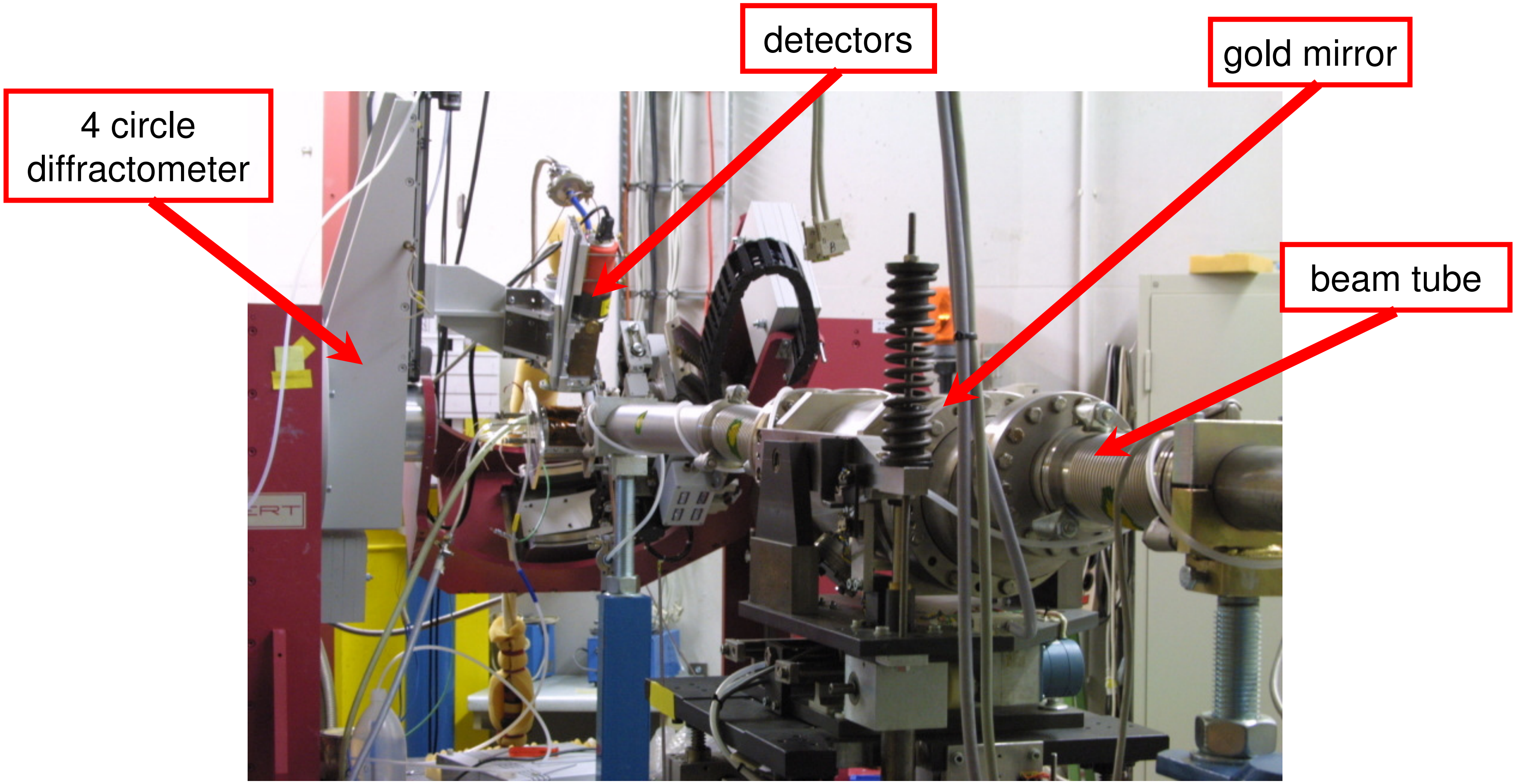


Fig. 3b. G3 experimental station
[http://hasylab.desy.de]

Beamline G3: Monochromator

- Fixed-exit **double-crystal** Ge (111), also further crystals available Ge (311), Si (111), Si (511), Si (400).
- All movements of the monochromator are driven by stepper motors.
- The monochromator is water cooled.
- The **tilted gold mirror** (7 mrad) is used for the suppression of higher harmonics.

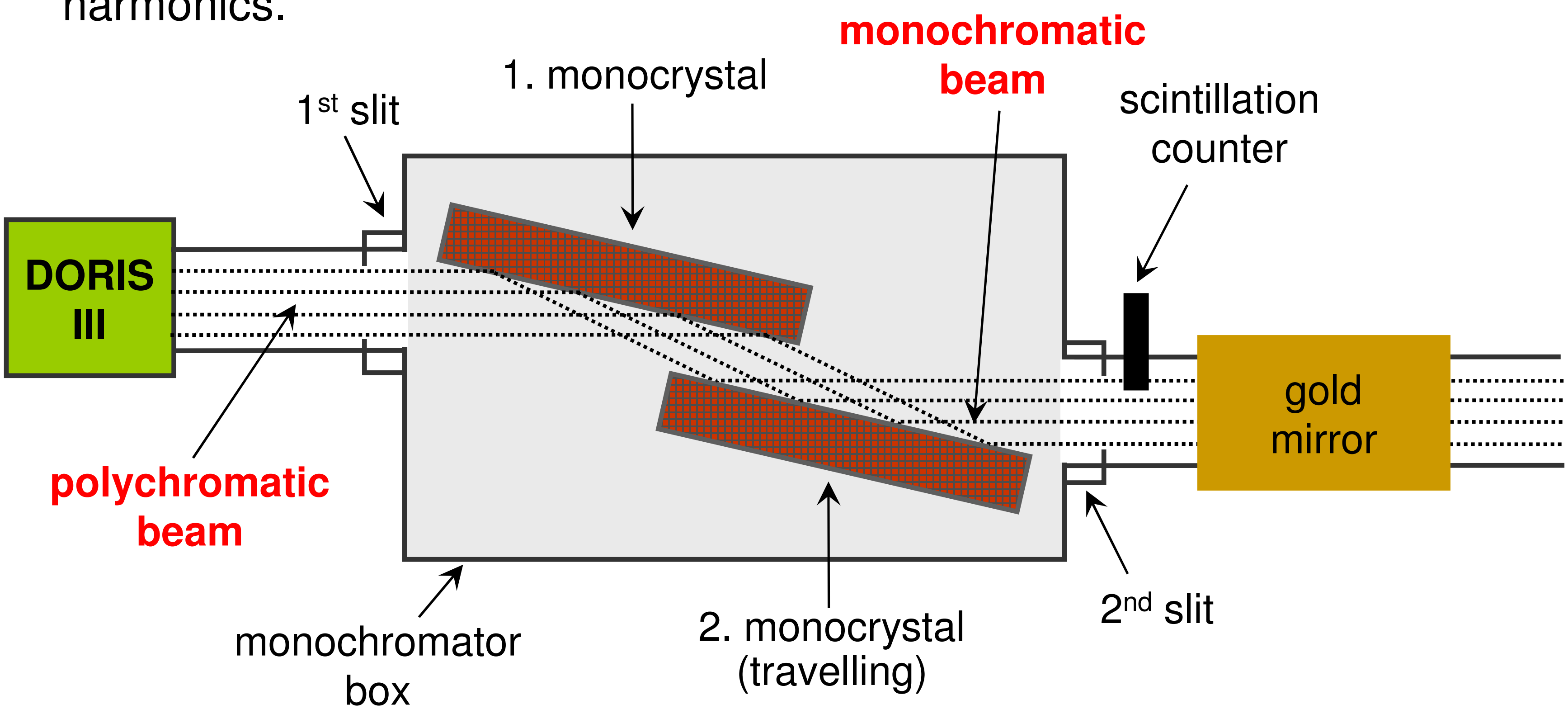


Fig. 4. G3 monochromator

Beamline G3: 4 Circle Diffractometer

- **Allows rotation** in 4 angles: 2θ, ω, φ , χ (sample tilt: 0°< χ< 90°).
- **Sample table** enables translations along x, y (±100 mm) axes and z axis (±10 mm).
- **A sphere** of 66 mm radius around the center of the diffractometer is free of mechanical components.
- **The detector arm** is equipped with a traverse allowing a radial translation of the detectors of up to 380 mm.
- The diffractometer holds **CCD detector with the MCP** and **point scintillation detector** with **Soller collimator.**

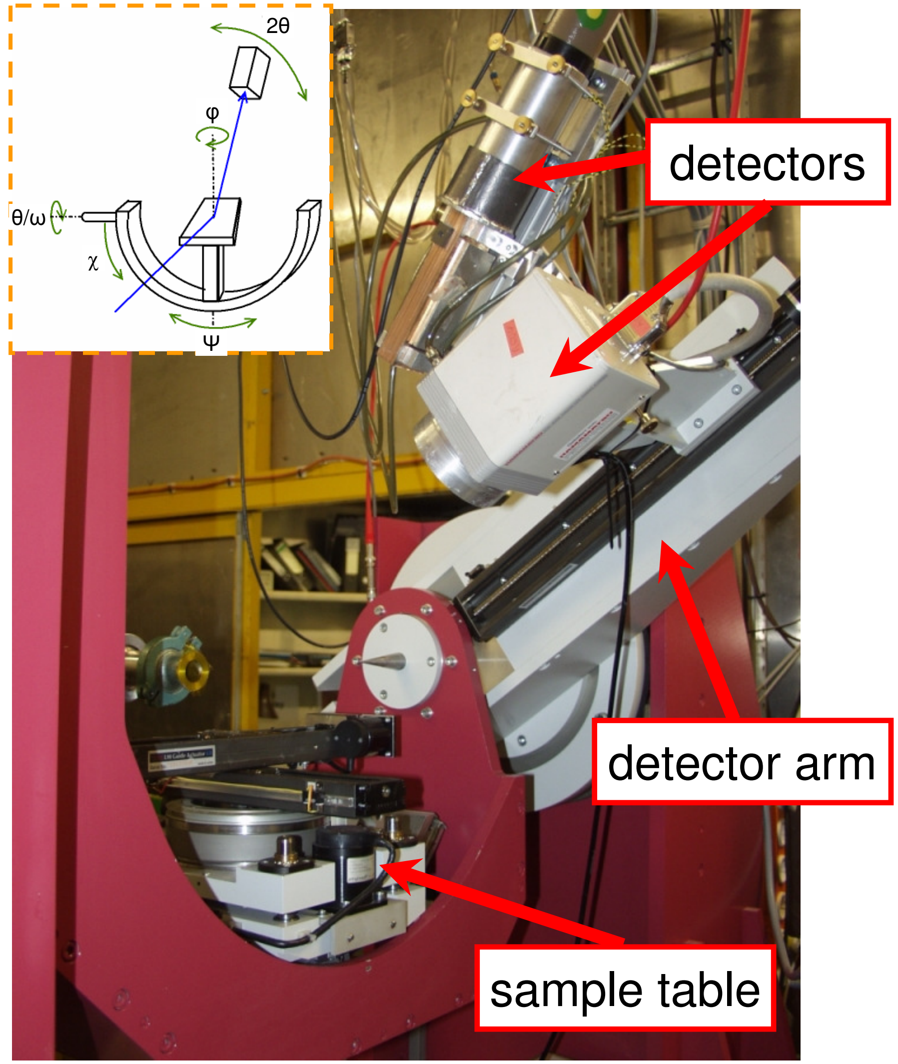


Fig. 5. 4 circle diffratometer and detectors
[http://hasylab.desy.de]

Beamline G3: Detectors

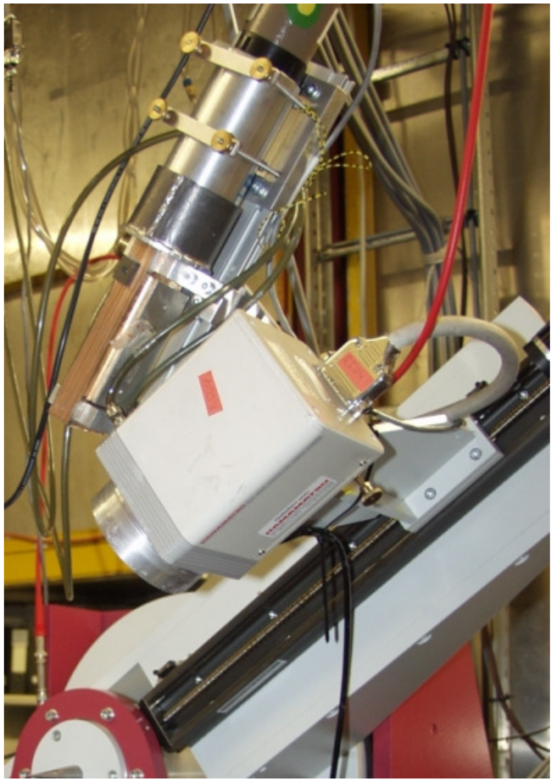


Fig. 5. G3 detectors
[http://hasylab.desy.de]

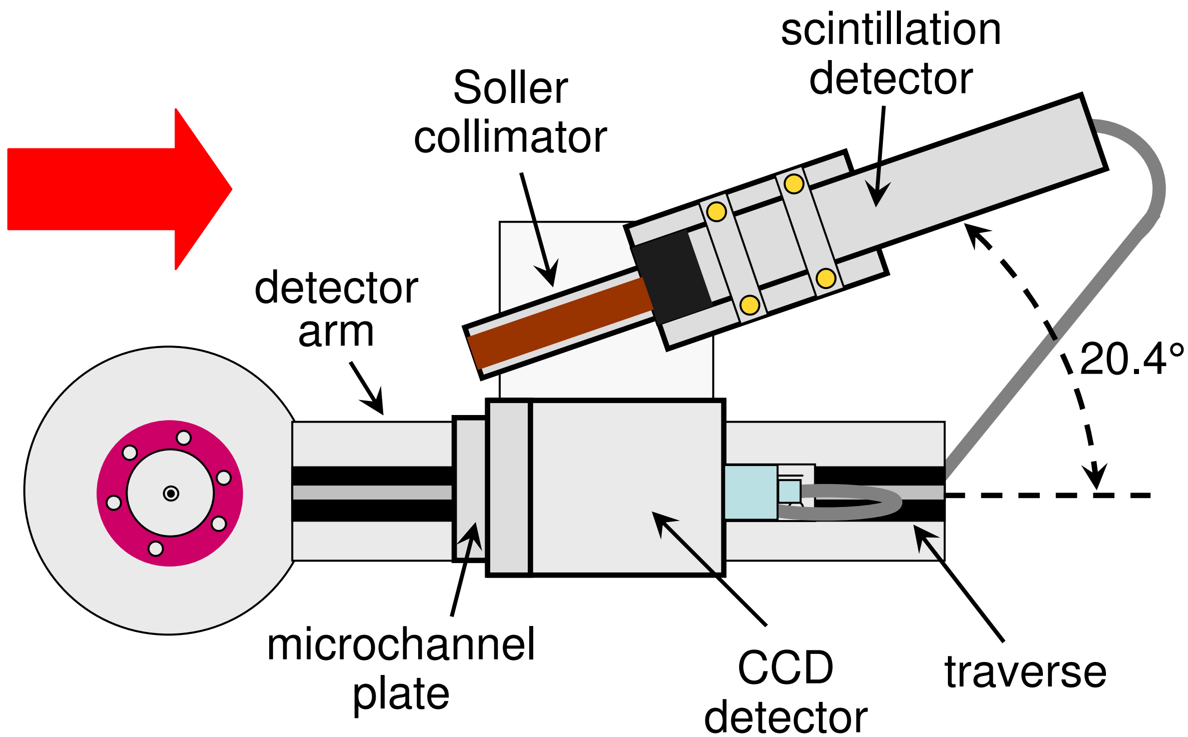


Fig. 6. Scheme of the detectors position (side view)

- On the platform for the CCD/MCP detector, inclined by about 20.4º, a scintillation counter with Soller collimator in front is mounted.

Beamline G3: Detectors

CCD camera

- Hamamatsu 4880,
- 1024 x 1024 pixels (13 µm),
- Peltier-cooled CCD chip (-65ºC),
- the MCP is in contact
  with capton foil of the CCD camera.

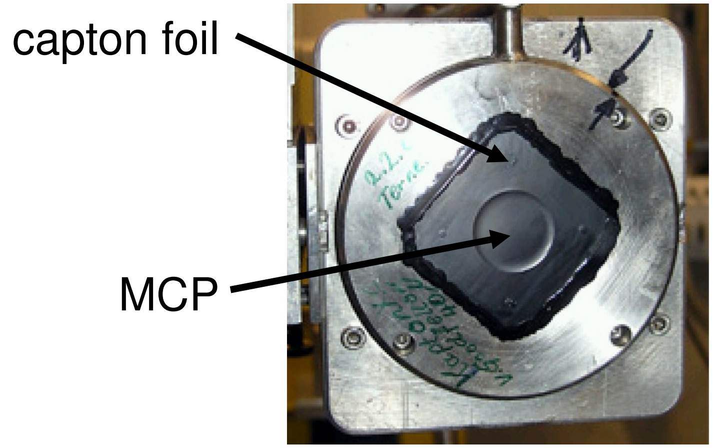


Fig. 7. CCD camera - front view
[Rothkirch]

Point scintillation detector

- Ortec LABR-1X1,
- lanthanum bromide ($LaBr_3(Ce)$)
  detector.

Fig. 8. Scintillation detector
[www.ortec-online.com]

Beamline G3: Microchannel plate

- The microchannel plate (MCP) consists of millions of very-thin, conductive **glass capillaries** fused together and sliced into a thin plate.
- MCP is a **collimator array** between the sample and a position-sensitive detector (CCD camera).
- Each channel of the MCP acts as a collimator tube pointing to a certain location of the sample, channels arranged in **hexagonal lattice**.
- Diameter of channels is 10 μm, 12.5 μm between their centers.

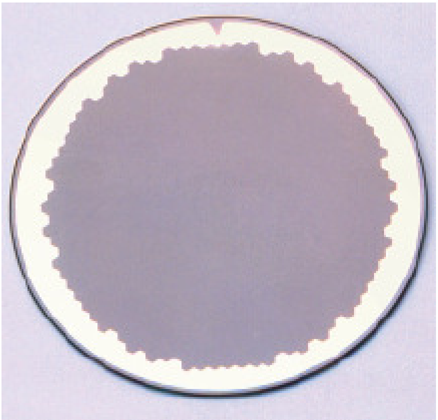


Fig. 9. Microchannel plate (front view) [www.hamamatsu.com]

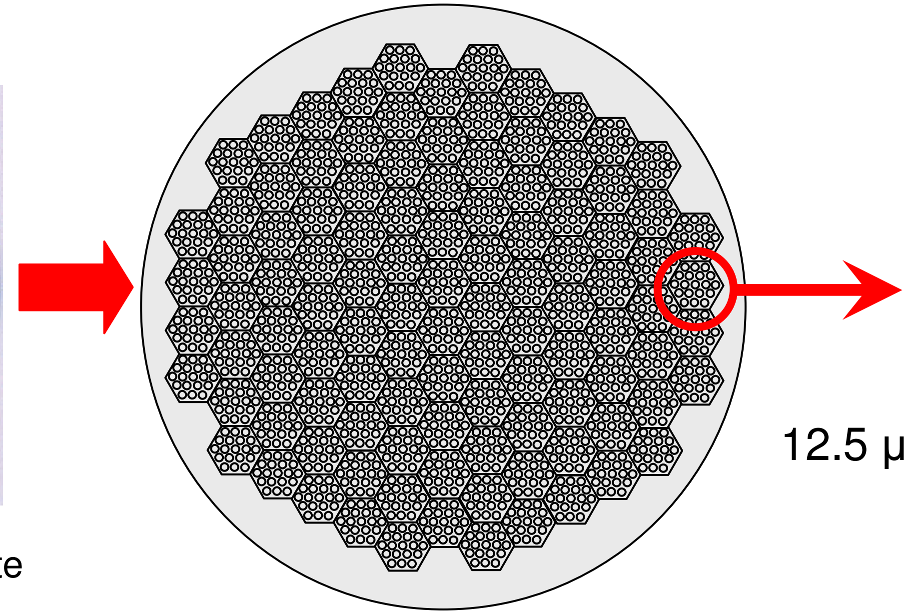


Fig. 10. Scheme of the microchannel plate (front view)

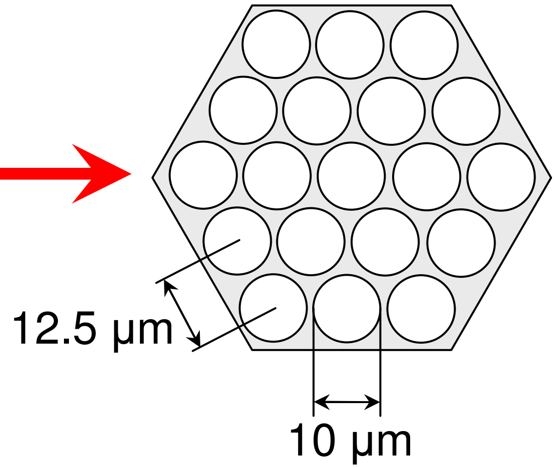


Fig. 11. Detail of hexagonal lattice (front view)

Beamline G3: Microchannel plate

- The thickness of the plate is 4 mm resulting in an **angular acceptance** of 2.5 mrad full width and, considering the circular shape of the channels, a FWHM of 1 mrad.
- **Typical sample to detector distance** is 12 mm resulting in a spatial resolution of 13 µm which is also the size of each of 1024 x 1024 pixels of the CCD detector.

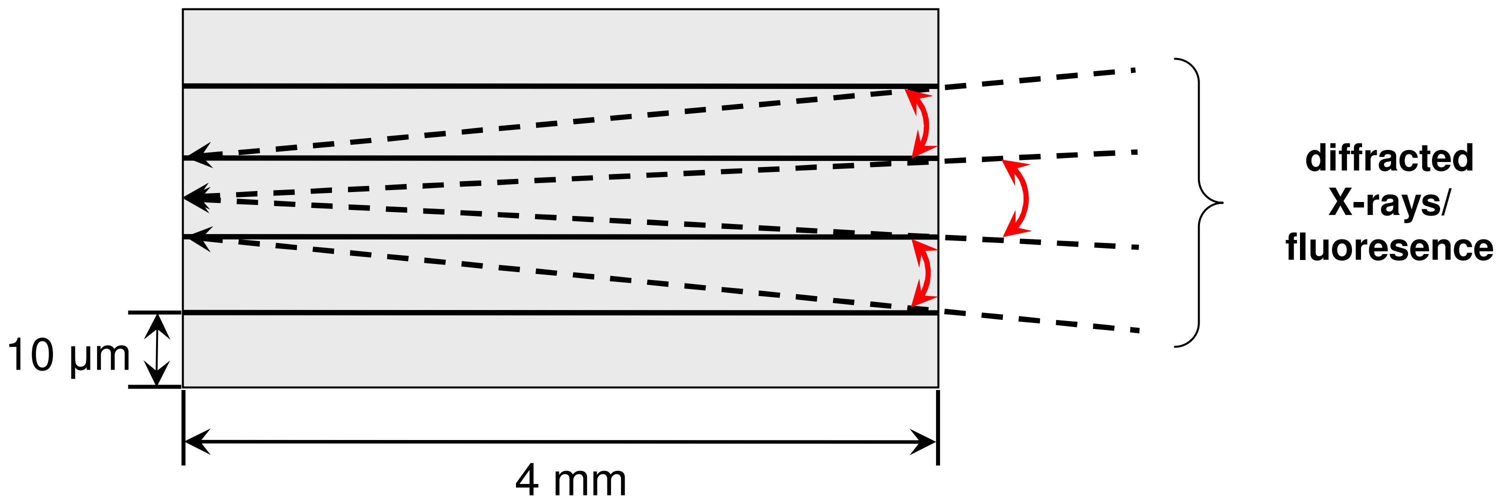


Fig. 12. Detail of hexagonal lattice
(side view)

Beamline G3: Principle of Diffraction Imaging

- Principle of diffraction imaging lies in application of the **MCP**.
- The MCP prevents detection of **crossfire** from diffracted X-rays.
- **Spatial distribution of crystal phases, ...**

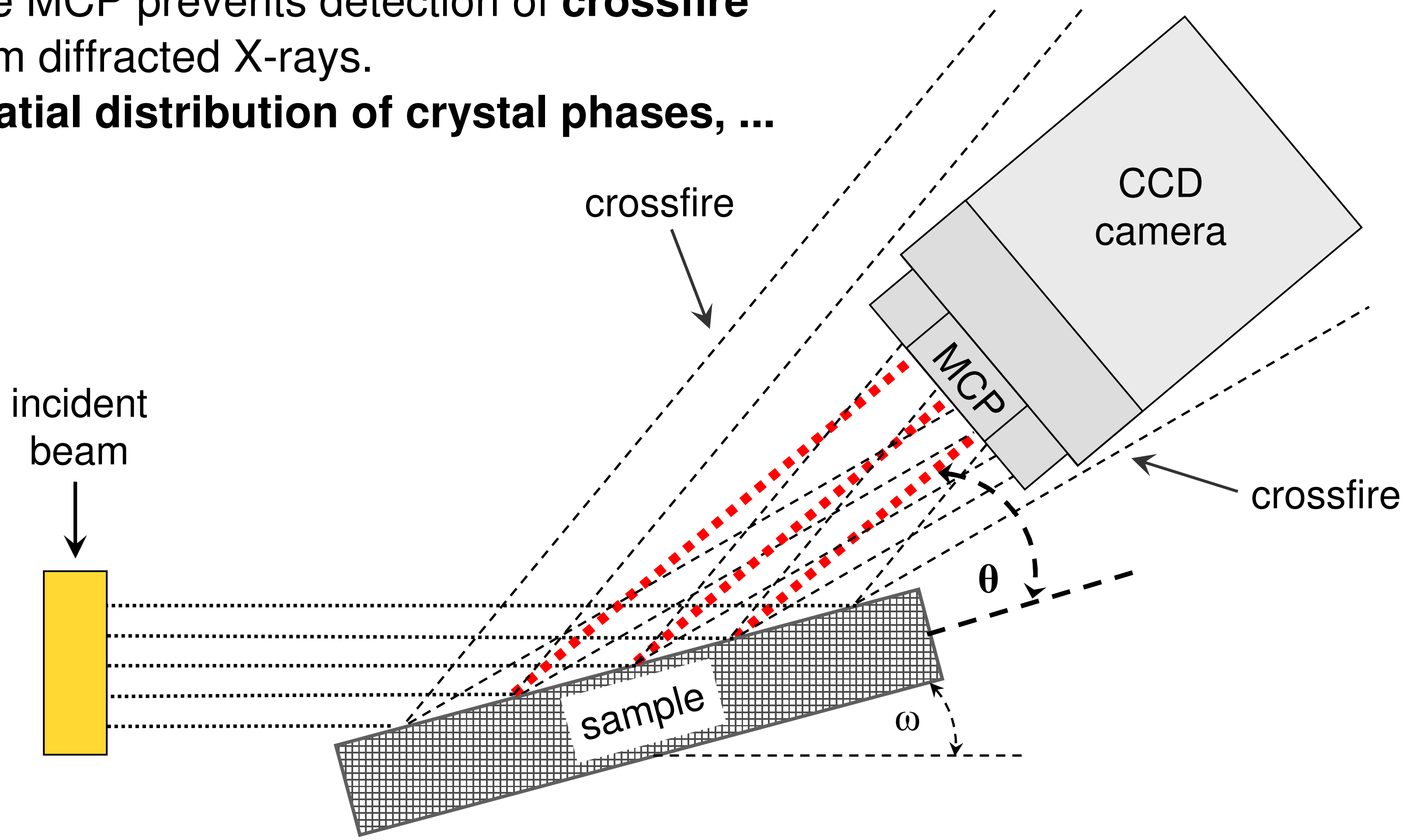


Fig. 13. Principle of diffraction imaging

Beamline G3: Principle of Fluorescence Imaging

- Variation of applied beam wavelength by crystal monochromator.
- The MCP prevents detection of "**crossfire**" from fluorescent X-rays.
- **Spatial distribution of chemical elements.**

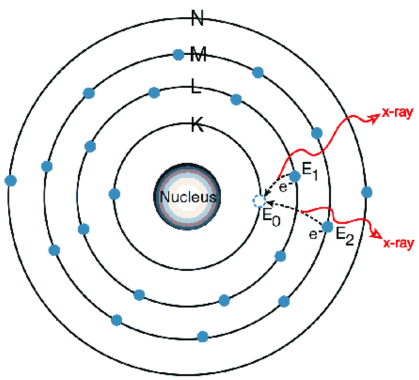


Fig. 14. Effect of fluorescence

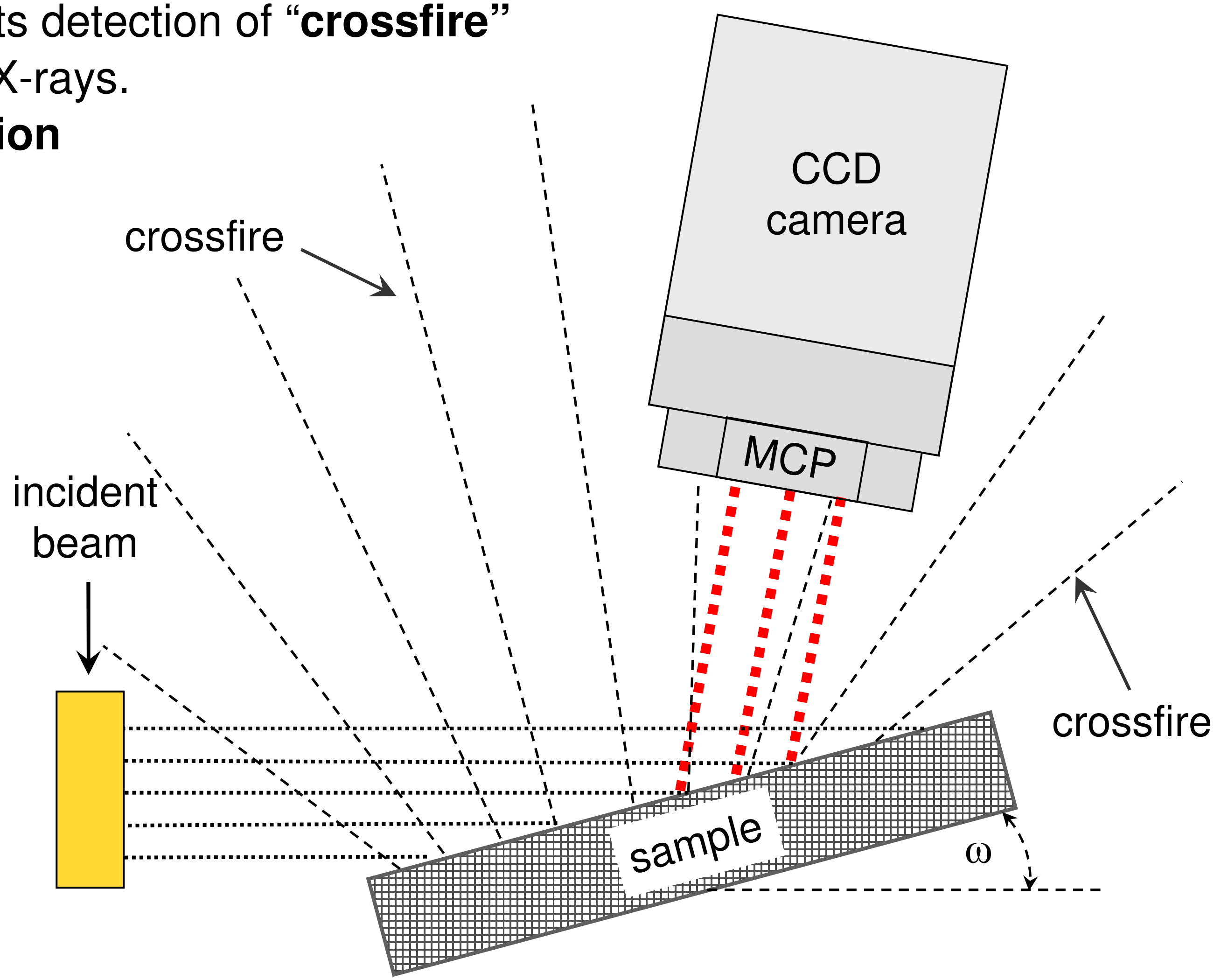


Fig. 15. Principle of fluorescence imaging

Beamline G3: Parallel polycapillaries

- Length of 300 mm, diameter of 30 μm, arranged in a hexagonal array of 8 mm distance between opposing sides.
- **Lower angular acceptance -** 30 μm/ 300 mm.
- **Small angle scattering -** CCD does not move into the primary beam.
- **High energy imaging** - total reflection at the capillary walls is much less pronounced at high X-ray energies.

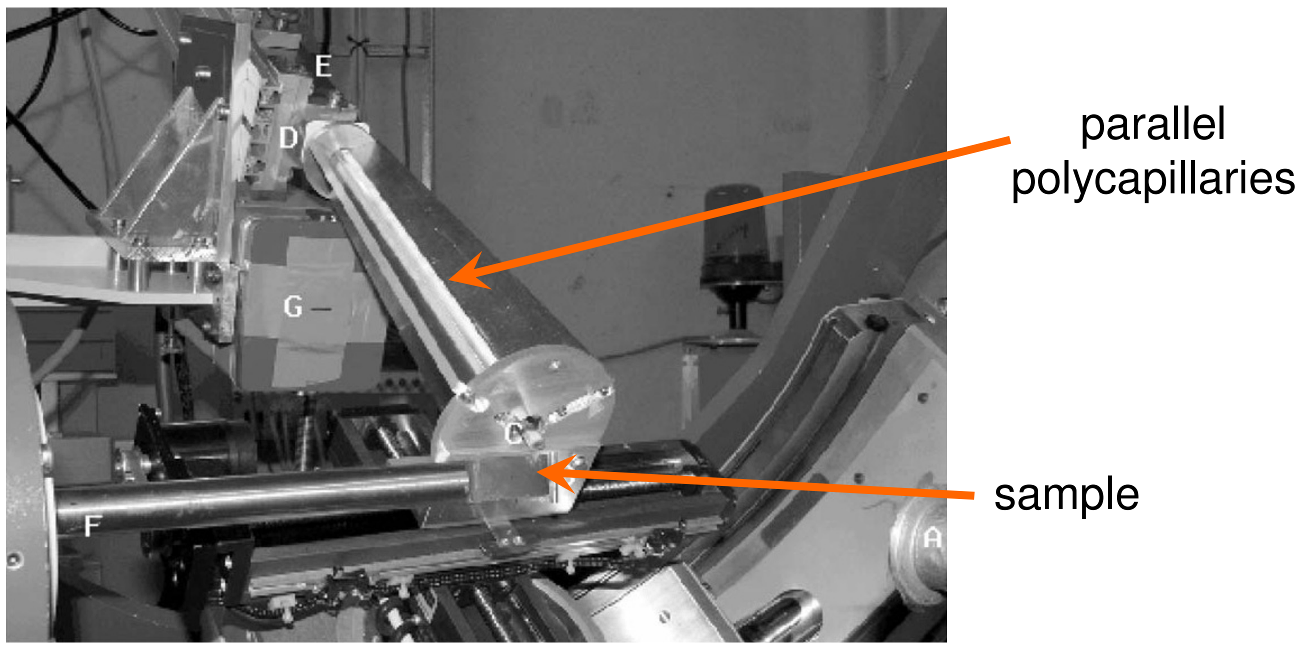


Fig. 16. Parallel polycapillaries at G3 diffractometer [Wroblewski, Bjeoumikhov]

Beamline G3: Solid-Liquid Phase Transitions

- Includes application of **additional monocrystal Ge (111).**
- The monocrystal enables measurements of **liquid samples**.
- Liquid samples can not be tilted.

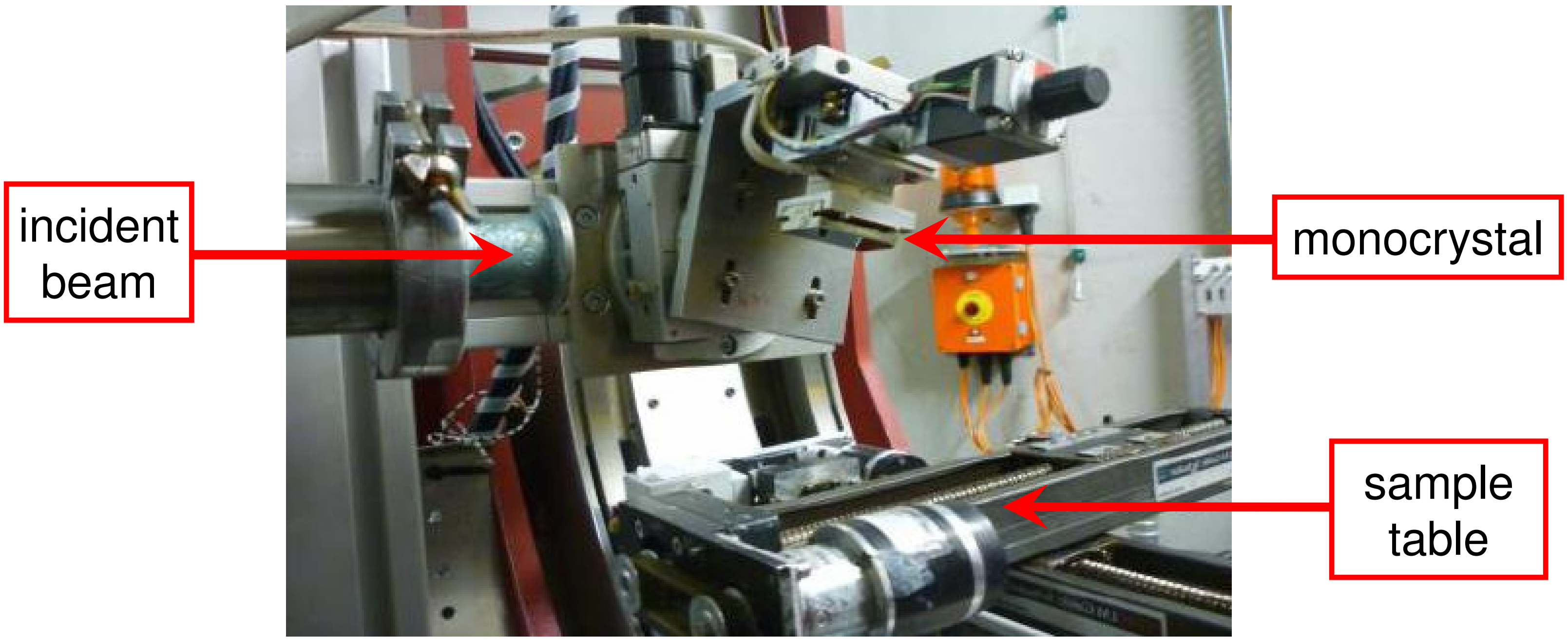


Fig. 17. Monocrystal mounted on 4 circle diffractometer
[Donges, Ovchinnikov]

Beamline G3: Solid-Liquid Phase Transitions

- Includes application of additional monocrystal Ge (111).
- **Tilt angle** of the monochromator depends on **applied wavelength**.

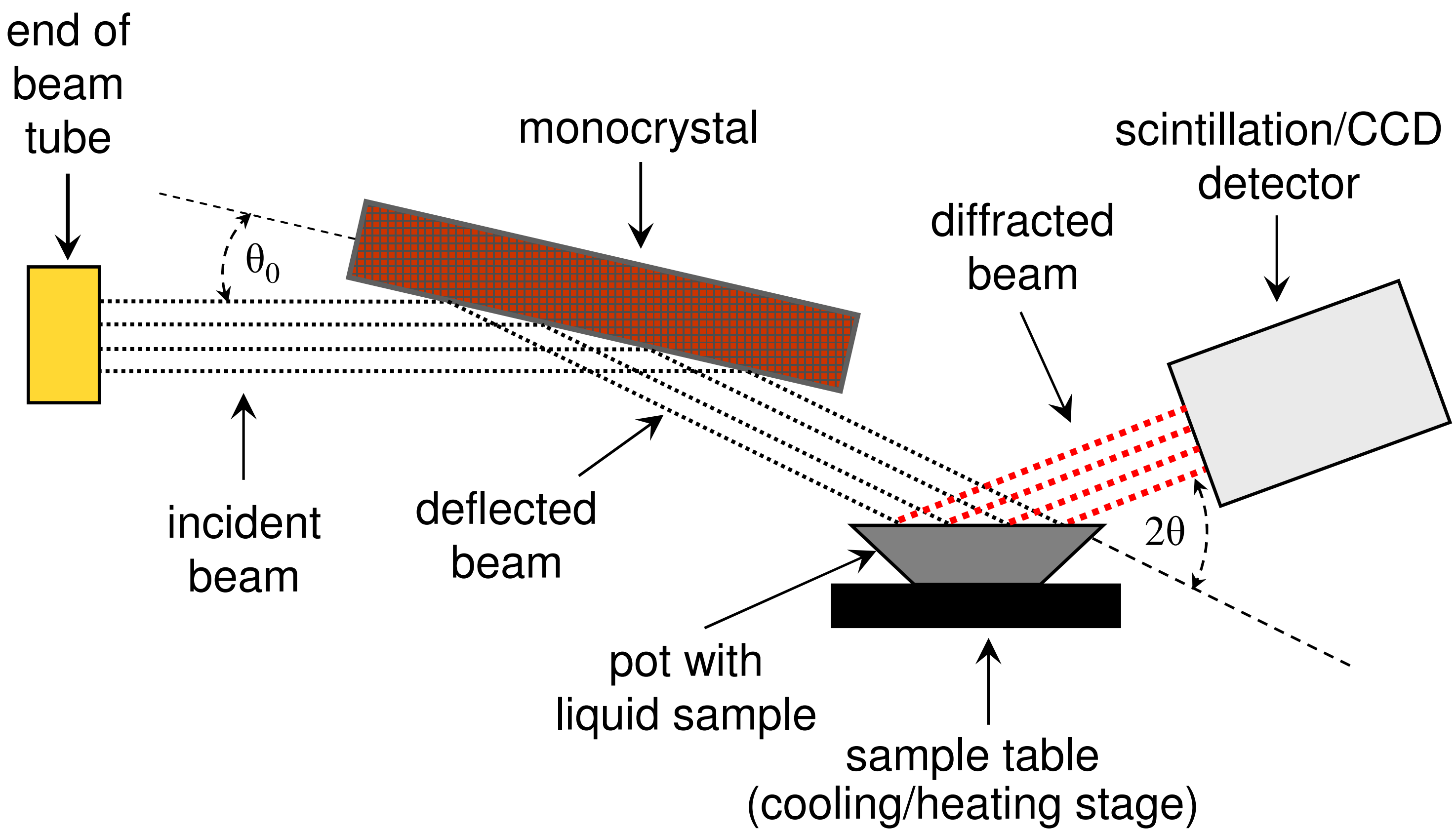
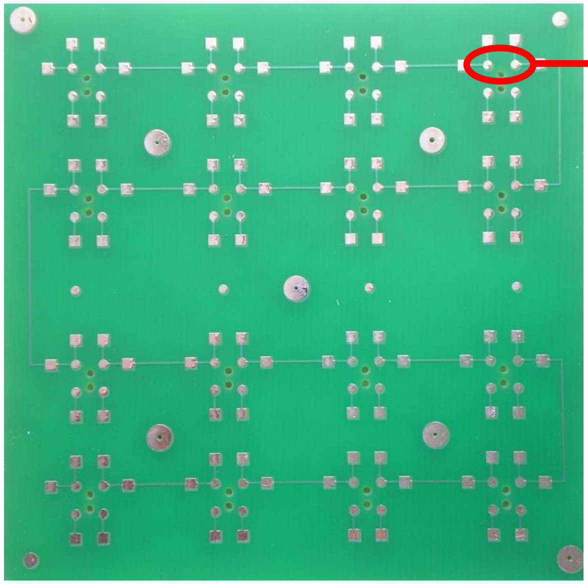


Fig. 18. Principle of liquid samples measurement

Beamline G3: Solder Alloy Measurement

- **Solder alloy:** 96.5Sn3Ag0.5Cu (wt. %).
- The alloy was soldered on copper (Cu) foil.
- **Goal of the measurement**: visualization of distribution and orientation of crystal phases of the solder (β-Sn, intermetallics – Ag_3Sn, Cu_6Sn_5) via **CCD camera**.
- Samples - **2 solder spots**.



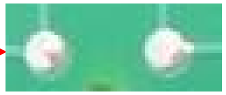


Fig. 19. PCB pattern

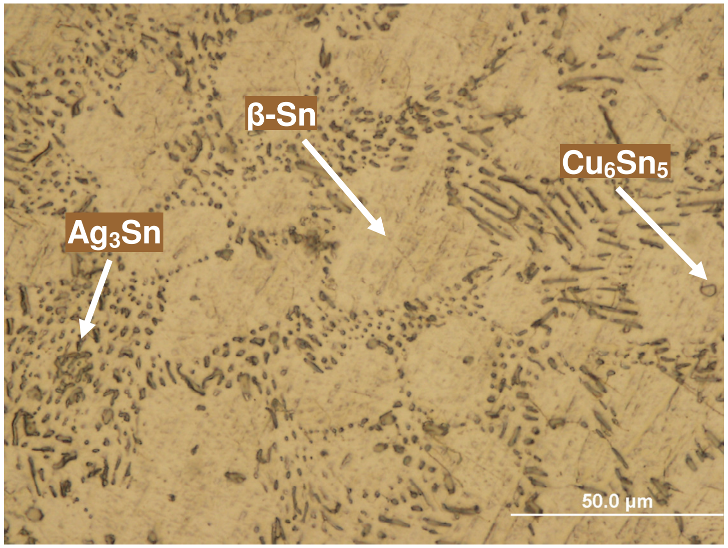


Fig. 20. Microstructure of the solder alloy [IMR SAS]

Beamline G3: Solder Alloy Measurement

1. step

- point scintillation detector,
- **Bragg-Brentano geometry**,
- 2θ range: 30º-105º, Δ2θ=0.04º, λ = 1.54187 Å (Cu K_alpha),
- the solder spots positioned on the sample table of 4 circle diffractometer,
- **Time reduction of CCD camera measurement.**

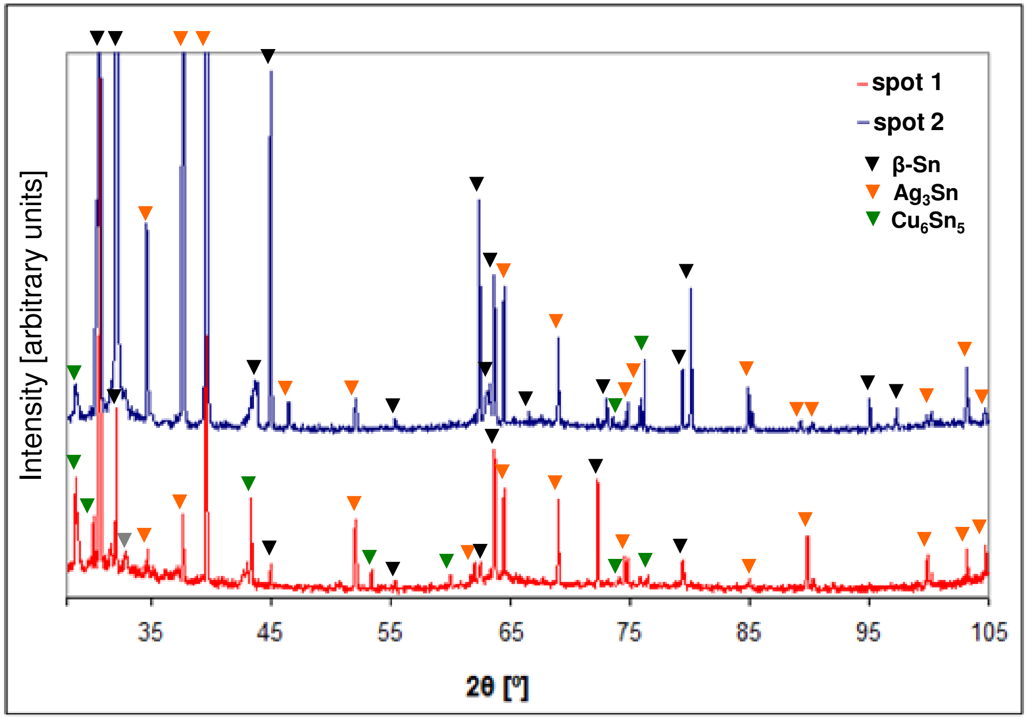


Fig. 21. Diffraction patterns of 2 solder spots

Beamline G3: Solder Alloy Measurement

2. step

- CCD camera (detector),
- λ = 1.54187 Å (Cu K_alpha),
- 2θ range selection: 2θ=60-100º, but measured was only in limit of **width of Bragg peaks at their basis**,
- Δ2θ=0.04º ≈ 300 seconds.

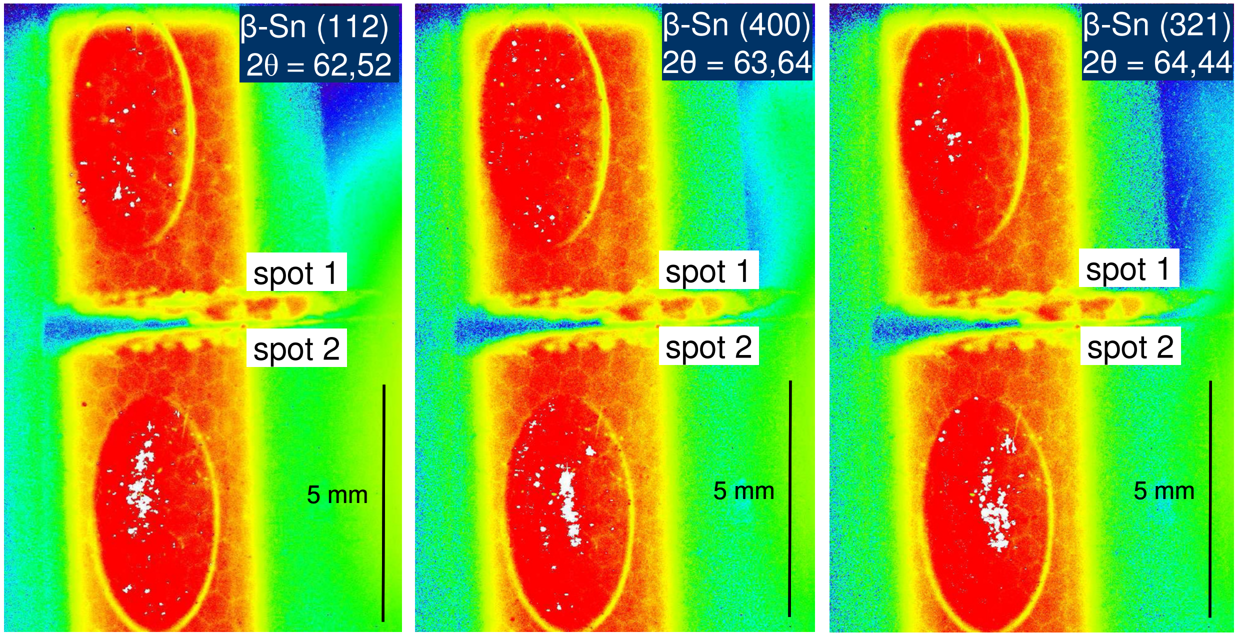


Fig. 22. Diffraction imaging of solder spots at selected angles

Beamline G3: Solder Alloy Measurement

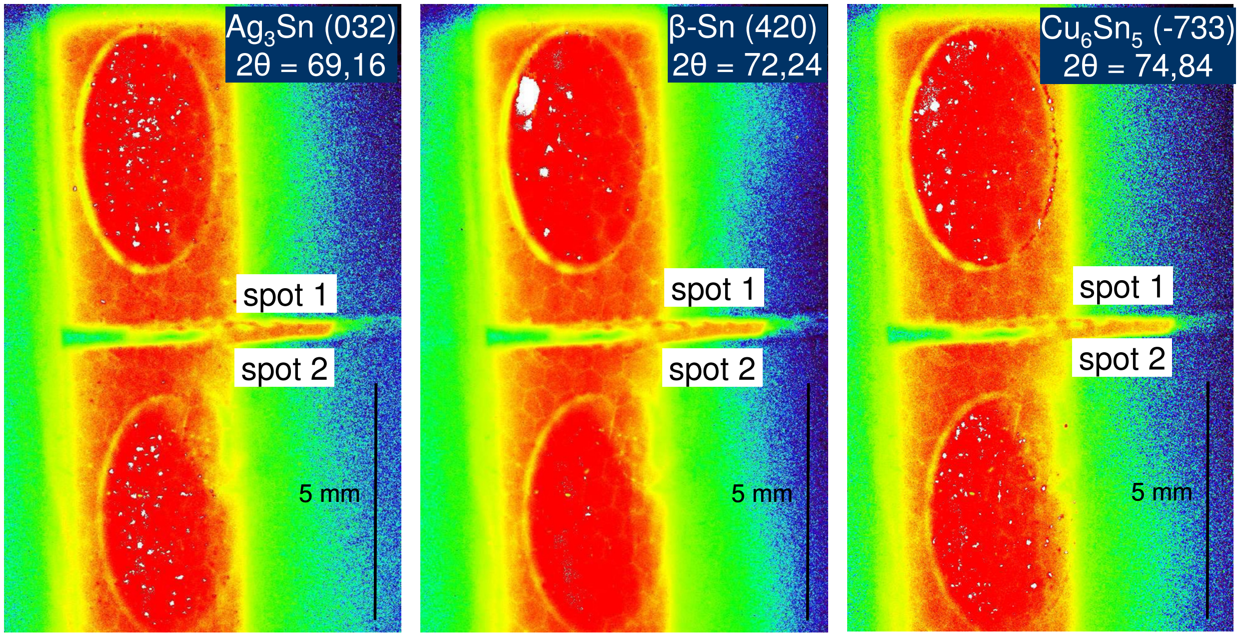


Fig. 23. Diffraction imaging of solder spots
at selected angles

Beamline G3: Solder Alloy Measurement

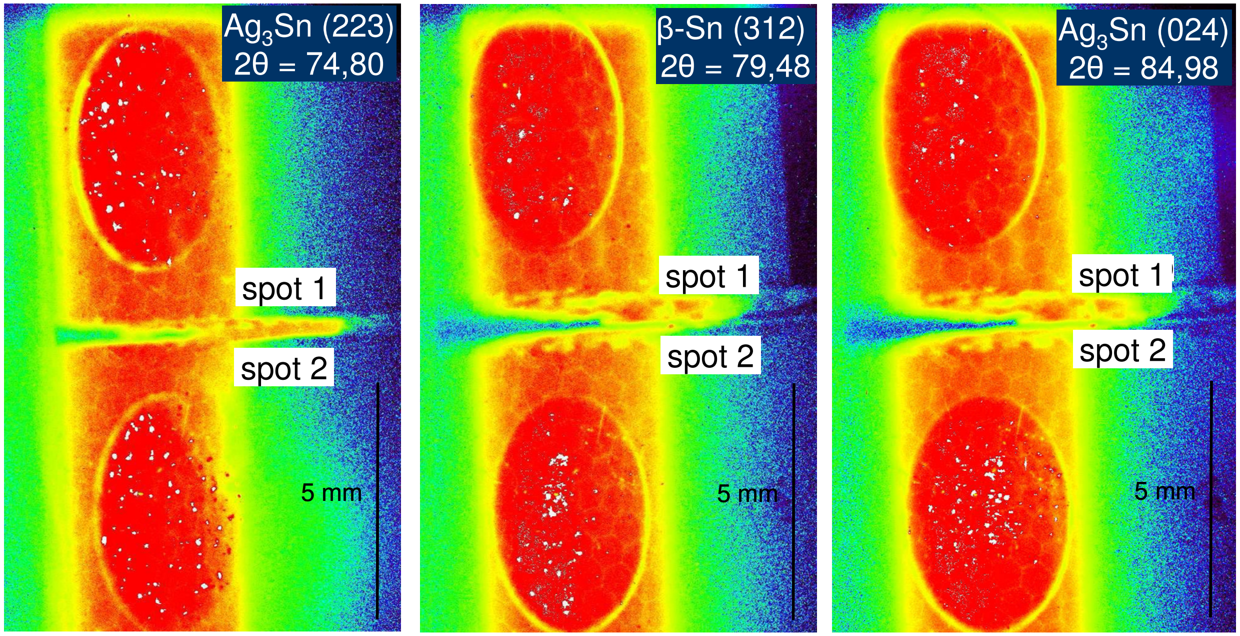


Fig. 24. Diffraction imaging of solder spots at selected angles

Beamline G3: Solder Alloy Measurement

Brief summary

- **Non-uniform distribution** of β-Sn crystals (if crystal orientation taken into consideration),
- size of β-Sn crystals: $\approx 10^1$-10^2 μm,

- **uniform distribution** of intermetallics – Ag_3Sn, Cu_6Sn_5.

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