



D8 DISCOVER

● The Ultimate Thin-Film System

X-Ray Reflectometry (XRR), High-Resolution X-Ray Diffraction (HRXRD), Reciprocal Space Mapping (RSM), Grazing Incidence Diffraction (GID), In-Plane Grazing Incidence Diffraction (IP-GID), Grazing-Incidence Small Angle X-ray Scattering (GISAXS) ..., all these complementary techniques allow thorough and non-destructive characterization of thin film samples.

Depending on the sample characteristics, different techniques and thus instrument setups are required in order to achieve the best possible data quality in the shortest measurement time.

The D8 DISCOVER is the most capable X-ray solution for advanced thin film investigations. Its sophisticated platform design facilitates optimized workflow from instrument setup to results.

Features

- Straightforward coplanar and non-coplanar diffraction
- Alignment-free switch between configurations
- Guaranteed goniometer accuracy and precision
- Software-controlled 0°/90° X-ray tube orientation
- SNAP-LOCK exchange of primary optics
- Push-button switch between diffracted-beam paths
- Detector guarantee

Key Components

ULTRA-GID Tube Mount

The ULTRA-GID tube mount enables switching a sealed X-ray source between two positions, parallel (0°) and perpendicular (90°) with respect to the horizontal diffraction plane. This allows exploiting the **full line focus intensity** for both coplanar and non-coplanar diffraction measurements since the line focus remains parallel to the sample surface. In addition, the non-coplanar measurements **benefit from the goniometer accuracy and precision**.

Switching between 0°/90° positions is motorized and fully software-controlled. The SNAP-LOCK bench, carrying the primary optics, turns together with the X-ray source. The beam conditioning optics consists of a Göbel Mirror, rotary absorber and an optional Channel-Cut (CC) monochromator or Soller collimator. The low divergence of the beam conditioned by the Göbel Mirror guarantees excellent control of the X-ray beam penetration depth, and thus provides **excellent depth resolution**.

The ULTRA-GID tube mount also has a motorized and software-controlled degree-of-freedom to **tune the absolute incident diffraction angle** (α_i) for non-coplanar measurements.

The motorized and software-controlled translation of the SNAP-LOCK bench automatically compensates for beam height offsets, e.g. in case of 2-bounce CC monochromators.

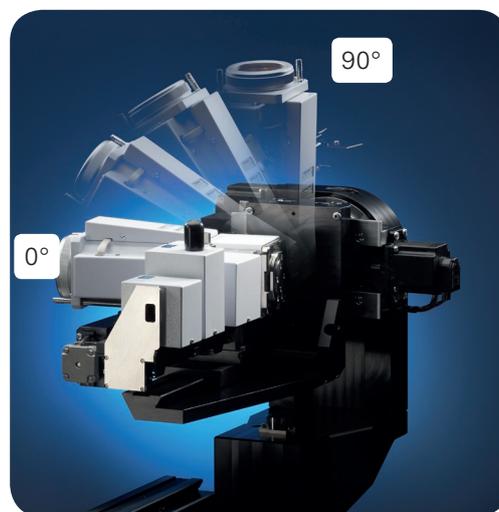


Fig. 1. ULTRA-GID tube mount

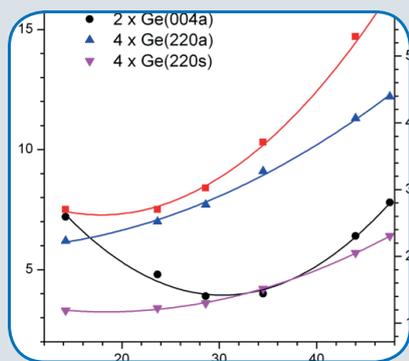


Fig. 2. Instrumental Resolution Function (rocking curve width)

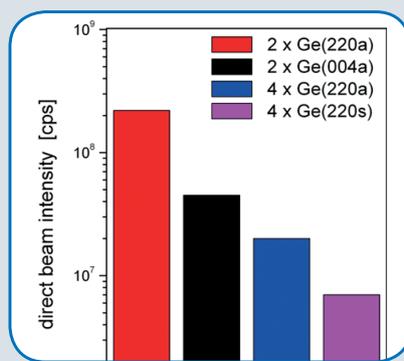


Fig. 3. Direct beam intensity for different CC monochromators

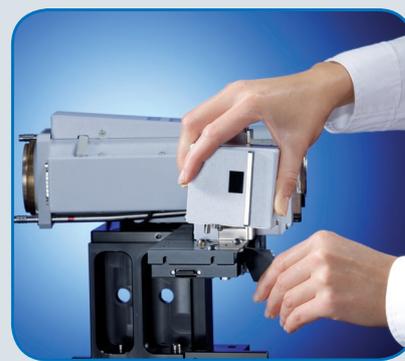


Fig. 4. SNAP-LOCK change of optics

SNAP-LOCK Bench for Primary-beam Optics

Efficient thin film analysis uses a Göbel Mirror as first optics to turn the divergent X-ray beam from the source into a highly intense, parallel beam. Various CC monochromators can be put as subsequent optics to further tune angular resolution.

Instrumental resolution (Figure 2) and direct beam intensity (Figure 3) are inversely proportional. Hence optics should be selected according to the sample properties in order to achieve best data quality in the shortest measurement time.

Bartels-type 4-bounce (4-b) CC monochromators provide the better overall angular resolution and unsurpassed spectral purity. This goes at the expense of the direct

beam intensity, which is one to two orders of magnitude lower compared to 2-b CC monochromators.

The resolution curve of the latter depends strongly upon the Bragg angle. Best angular resolution is achieved when the CC Bragg angle matches the Bragg angle of the sample. Therefore, 2-b Ge(004) CC will provide best resolution on typical SiGe or GaAs samples (Bragg angle close to 34.5°), whereas 2-b Ge(220) CC will be better suited for HRXRD measurements on oxide samples or XRR measurements.

All these high-end optics simply click into the D8 DISCOVER's unique SNAP-LOCK bench (Figure 4). And while they are in real-time identified, configured and adjusted, there is **more pure measurement time** left.

PATHFINDER with double detector arm

With our patented¹ PATHFINDER, there is no need to touch the instrument at all for changing diffracted-beam optics and thus 2Theta angular resolution

This intelligent component has built-in three different configurations that can be **switched by a simple mouse-click**.

The first configuration is a high-intensity beam path incorporating two motorized slits. This **double-axis geometry** enables fast and automatic sample alignment, XRR, and HRXRD investigations on samples with low mosaicity or samples with strongly tilted layers.

The second configuration is another high-intensity beam path incorporating a motorized slit and an equatorial Soller collimator. This setup is ideal for applications requiring low resolution, such as GID on polycrystalline coatings, residual stress measurements on thin films, XRR on very thin films, etc.

The third configuration is a high-resolution beam path incorporating a single- or triple-bounce Ge(022) channel-cut monochromator.

This **triple-axis geometry** is used for high resolution and low background HRXRD and RSM investigations.

The LYNXEYE 1-D detector next to the PATHFINDER provides further options. By collecting diffracted X-rays from a range of 2Theta angles simultaneously, HRXRD and RSM can be measured faster or with better statistics. By turning² the LYNXEYE detector 90°, even out-of-plane (α_f) data can be collected, e.g. as required for GISAXS.



Fig. 5. Double detector arm with PATHFINDER and LYNXEYE detector

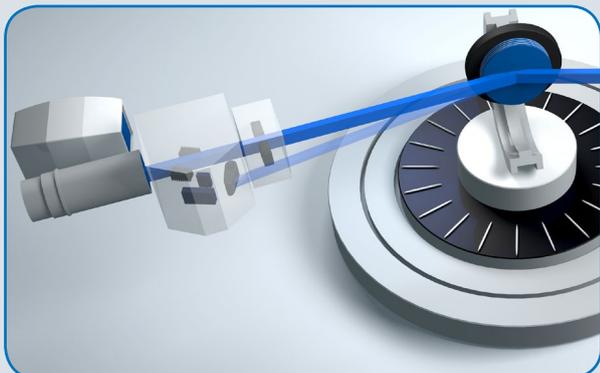


Fig. 6a. High-intensity beam path for sample alignment, XRR and HRXRD (2-axis geometry)



Fig. 6b. High-intensity beam path for GID and XRR on very thin films



Fig. 6c. High-resolution beam path for XRR on thick films, HRXRD (3-axis geometry) and RSM



Fig. 6d. High-intensity beam path for fast HRXRD and fast RSM, or GISAXS with LYNXEYE 90°

Applications: HRXRD



Analyzing epitaxial thin film samples requires a highly parallel and monochromatic X-ray beam in order to have the required resolution in reciprocal space. This is realized with 2-b or 4-b CC monochromators following the Göbel Mirror. Such HRXRD measurements allow evaluating layer thickness, chemical composition, strain/relaxation, layer mismatch, etc.

Figure 7 shows a typical HRXRD measurement. A sample consisting of an $\text{In}_x\text{Ga}_{(1-x)}\text{As}$ layer on top of GaAs substrate was measured in the vicinity of the (004) reflection, using 3-axis geometry. The most intense and narrow reflection is the (004) of the substrate, two orders of magnitude lower in intensity is the (004) reflection of the layer. The separation between substrate and layer peak gives the Indium concentration (Vegard's law), in this example 5.6%. The so-called fringes are interferences caused by the layer thickness of 200 nm.

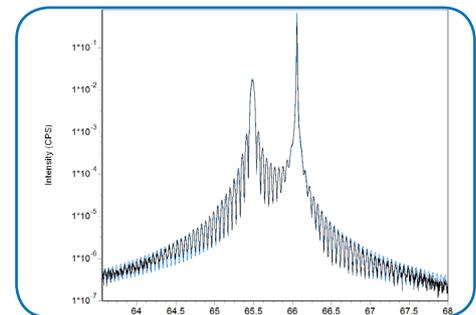


Fig. 7. 2Theta/Omega scan on InGaAs/GaAs with 3-axis geometry. Experimental data (black), data calculated with DIFFRAC.LEPTOS (blue)

Figure 8 shows the effect of the instrumental resolution by means of two rocking curve measurements on the same III/V semiconductor sample ($\text{AlInN}/\text{AlN}/\text{GaN}/\text{AlN}/\text{Al}_2\text{O}_3$) with high mosaicity. The better resolution of the triple-axis setup enables resolving the thickness fringes of the 360 nm thick AlN layer (zoomed view in the insert). Thickness fringes of the AlInN top layer do not arise due to its high mosaicity.

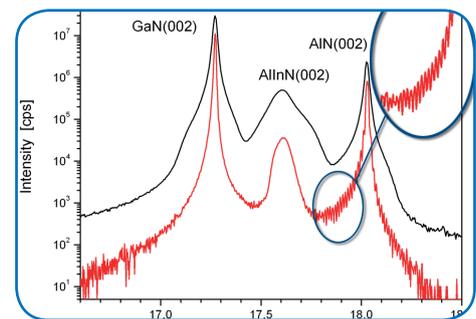
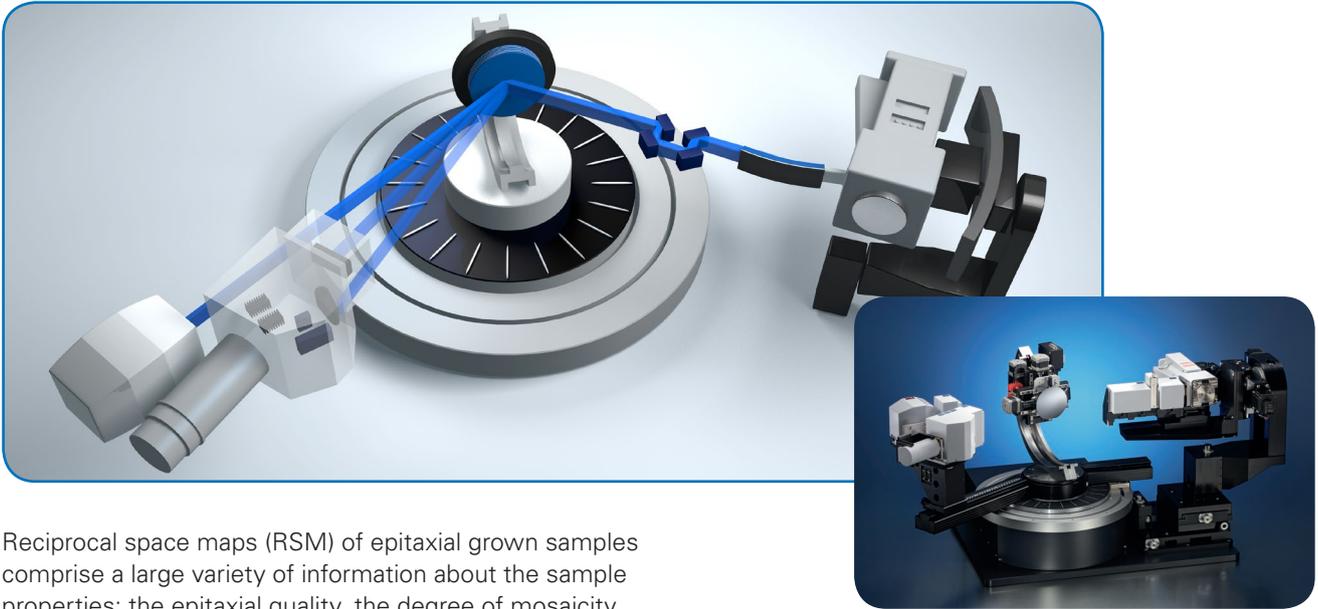


Fig. 8. Rocking curve measurements on $\text{AlInN}/\text{AlN}/\text{GaN}/\text{AlN}/\text{Al}_2\text{O}_3$ with different instrument setups: 2-axis geometry (black), 3-axis geometry (red) Sample courtesy of L.R. Khoshroo (RWTH Aachen)

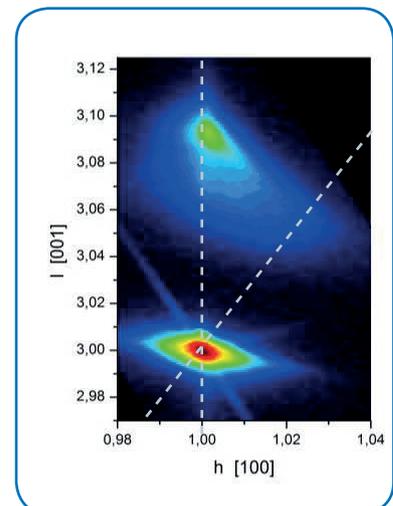
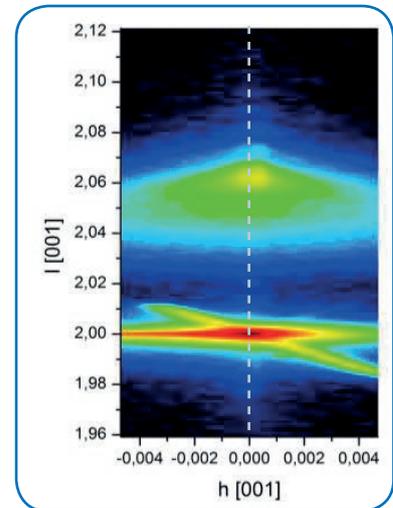
Applications: RSM



Reciprocal space maps (RSM) of epitaxial grown samples comprise a large variety of information about the sample properties: the epitaxial quality, the degree of mosaicity, miscut between layer and substrate, and many more. The instrument setup is similar to the standard HRXRD measurements, with the ULTRA-GID tube mount in 0° position. The PATHFINDER's analyzer crystal is selected to scan reciprocal space with highest q -resolution. Alternatively, the LYNXEYE detector enables speeding up measurement or improving statistics by collecting a range of 2θ angles simultaneously – obviously with reduced q -resolution.

Figure 9 shows RSM's that have been collected from a 50 nm thin LaAlO_3 layer on SrTiO_3 (STO) substrate in the vicinity of the symmetric STO(002) and the asymmetric STO(103+) substrate reflections. The symmetric RSM confirms the absence of a tilt between layer and substrate since the reflections are all aligned along the l -direction. The asymmetric RSM confirms that the LaAlO_3 layer is mostly strained, but shows some relaxation gradient towards the surface. The main intensity of the layer reflection is located at (1, 3.905) in h_l , which means that the lattice spacing for the layer is different from the substrate, but the a -direction is the same. At room temperature SrTiO_3 has a cubic structure with $a = 3.905 \text{ \AA}$, whereas bulk LaAlO_3 is rhombohedral. The crystal structure of the latter changes to cubic with $a = 3.821 \text{ \AA}$ at temperatures above 435°C . So the RSM can be interpreted as a proof that the 50 nm layer exhibits the high-temperature phase of LaAlO_3 .

Fig. 9. RSM's around STO(002) (above) and STO(103+) (below) collected with 1-D LYNXEYE detector for superior measurement statistics.
Sample courtesy of Dr. D. Fuchs (Inst. of Solid State Physics, KIT)



Applications: XRR



XRR is a surface-sensitive scattering method, which probes variations of the electron density in depth below the surface. Hence XRR can be applied to crystalline and amorphous layers to extract information on thickness, density, surface and interface roughness.

An XRR curve quickly falls off above the angle of total external reflection. Consequently, it is important to cover several orders of magnitude in intensity. The typical instrument setup therefore uses a Göbel Mirror as primary optics for boosting intensity, and a double slit setup on the secondary side for reducing diffuse scattering. This allows resolving so-called Kiessig fringes from 0.1 nm up to 150-200 nm thick layers. Thicker layers up to 1 micron require improved resolution, which can be realized by snapping-in CC monochromators and/or analyzer crystals. Thinner films of only a few nm feature broad humps and benefit from maximum integrated intensity, as achieved with a Soller collimator on the secondary side.

Figure 10 shows an XRR measurement of a Si/W multilayer stack. The reflected intensity covers 7 orders of magnitude and was therefore collected in several ranges with different measurement time to compensate for counting statistics. Satellite peaks from the bilayer and fringes from the multilayer stack can be nicely distinguished up to an exceptionally high scattering angle of 20° (2θ). In addition, broad oscillations due to the thin W layer are superimposed.

Figure 11 shows an XRR measurement of a 1.4 nm thick HfO_2 layer on top of Si. The large density contrast helps to better recognize the broad fringes, which extend up to high scattering angles.

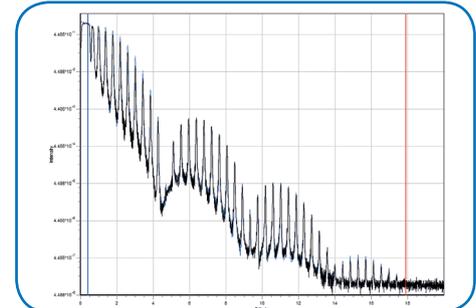


Fig. 10. XRR on a $10 \times (\text{Si}/\text{W})/\text{Si}$ multilayer stack. Experimental data (black), data calculated with DIFFRAC.LEPTOS (blue)

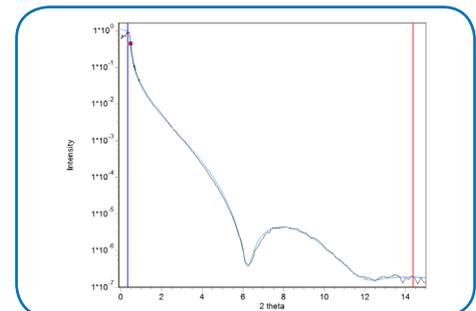


Fig. 11. XRR on a 1.4 nm thin HfO_2 layer on top of Si. Experimental data (black), data calculated with DIFFRAC.LEPTOS (blue)

Applications: IP-GID & GISAXS



Non-coplanar diffraction gives direct access to the in-plane lattice parameters, in-plane crystallite size and in-plane preferred orientation.

For this type of investigations, the ULTRA-GID tube mount is rotated to the 90° position. The low divergence of the Göbel Mirror provides excellent depth sensitivity and by varying the incident angle, depth dependent information can be extracted. Additional Soller collimators in the primary and secondary beam path control the in-plane resolution. An optional double tilt stage on the Eulerian cradle holds the thin film sample and enables orienting its surface perpendicular to the phi axis.

Figure 12 shows coplanar and non-coplanar measurements on a 10 nm polycrystalline FePt thin film. The crystallite size in the surface normal direction can be extracted from the peak width in the coplanar measurement. For this particular sample, the crystallite size covers about the total film thickness. From the peak width in the non-coplanar measurement, the in-plane crystallite size can be calculated to be about 6.5 nm. In addition, both measurements confirm strong in-plane fiber texture around [001].

Furthermore, the out-of-plane scattering signal in the vicinity of the direct beam could be measured by turning the 1-D LYNXEYE detector 90° and using a Soller collimator to control the divergence.

Figure 13 shows a GISAXS pattern of self-assembled gold nanoparticles on top of Si substrate. The side maxima indicate lateral ordering of the nanoparticles. The interparticular correlation length can be extracted from the position of these side maxima, in this example about 6.6 nm.

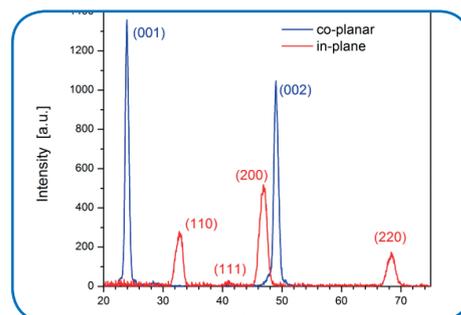


Fig. 12. Coplanar (blue) and non-coplanar (red) measurement on a polycrystalline FePt thin film.

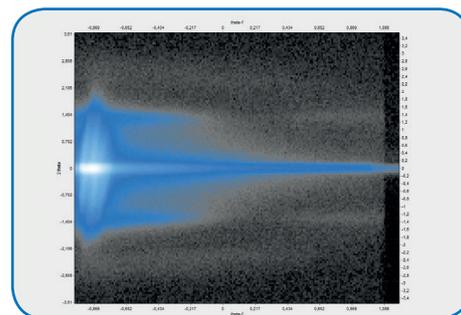


Fig. 13. GISAXS pattern of gold nanoparticles on top of Si. Sample courtesy of Dr. P. Siffalovic (Slovak Academy of Science, Bratislava)

Non-coplanar grazing incidence diffraction

In non-coplanar grazing incidence diffraction the incident and diffracted X-ray beams are nearly parallel to the sample surface.

Non-coplanar grazing incidence diffraction is used for investigating the near-surface region of samples by exploiting the high intensity of the total external reflection condition while simultaneously Bragg-diffracting from (hkl) planes that are nearly perpendicular to the sample surface. Hence it gives access to information (e.g. lattice parameters, crystallite size, texture, etc.) in the plane of the sample surface, which is why it is sometimes also referred to as “in-plane” diffraction.

Non-coplanar grazing incidence diffraction is the method of choice for measuring ultra-thin films, which can be of soft matter, poly-crystalline or epitaxial nature.

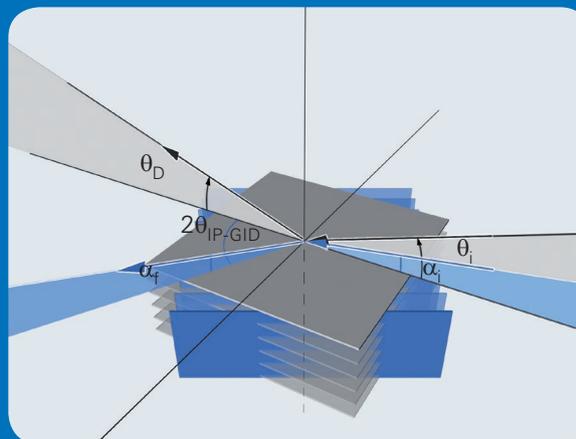


Fig. 14. Coplanar diffraction: the diffraction vector points out of the sample surface. Non-coplanar diffraction: the diffraction vector is almost parallel to the sample surface. (hkl) reflections nearly perpendicular to the sample surface (blue) are probed.

Technical Specifications

Application	ULTRA-GID	Primary beam	Secondary beam	Detector
XRR	0°	Göbel Mirror	PATHFINDER (double slit)	Scintillation counter
XRR (thick films) HRXRD	0°	Göbel Mirror with 2-b or 4-b CC	PATHFINDER (double slit)	Scintillation counter
GID (polycrystalline films) Residual stress XRR (ultrathin films)	0°	Göbel Mirror	PATHFINDER (Soller collimator)	Scintillation counter
HRXRD	0°	Göbel Mirror with 2-b or 4-b CC	PATHFINDER (1-b or 3-b CC)	Scintillation counter
Fast HRXRD Fast RSM	0°	Göbel Mirror with 2-b or 4-b CC	empty	LYNXEYE 1-D mode
IP-GID	90°	Göbel Mirror with Soller collimator	PATHFINDER (Soller collimator)	Scintillation counter
GISAXS	90°	Göbel Mirror with Soller collimator	Soller collimator	LYNXEYE 1-D mode turned 90°

Patents:

¹ PATHFINDER: US 6 665 372 and DE 10 141 958

² LYNXEYE turned 90°: EP 1 647 840 A2 patent and EP 1 510 811 B1 patent

Sealed Göbel Mirror: EP 1 503 386 B1 patent

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