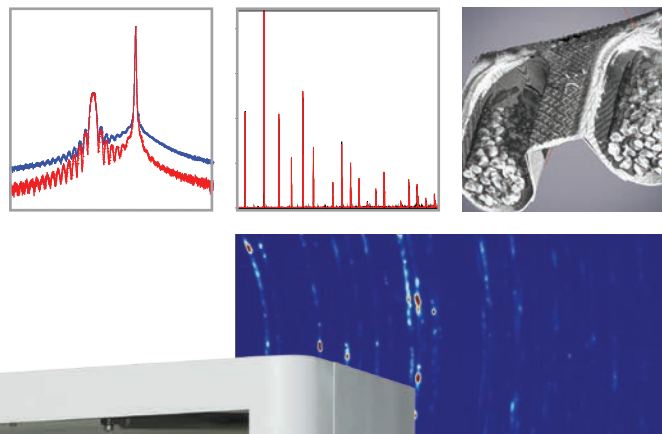


EMPYREAN

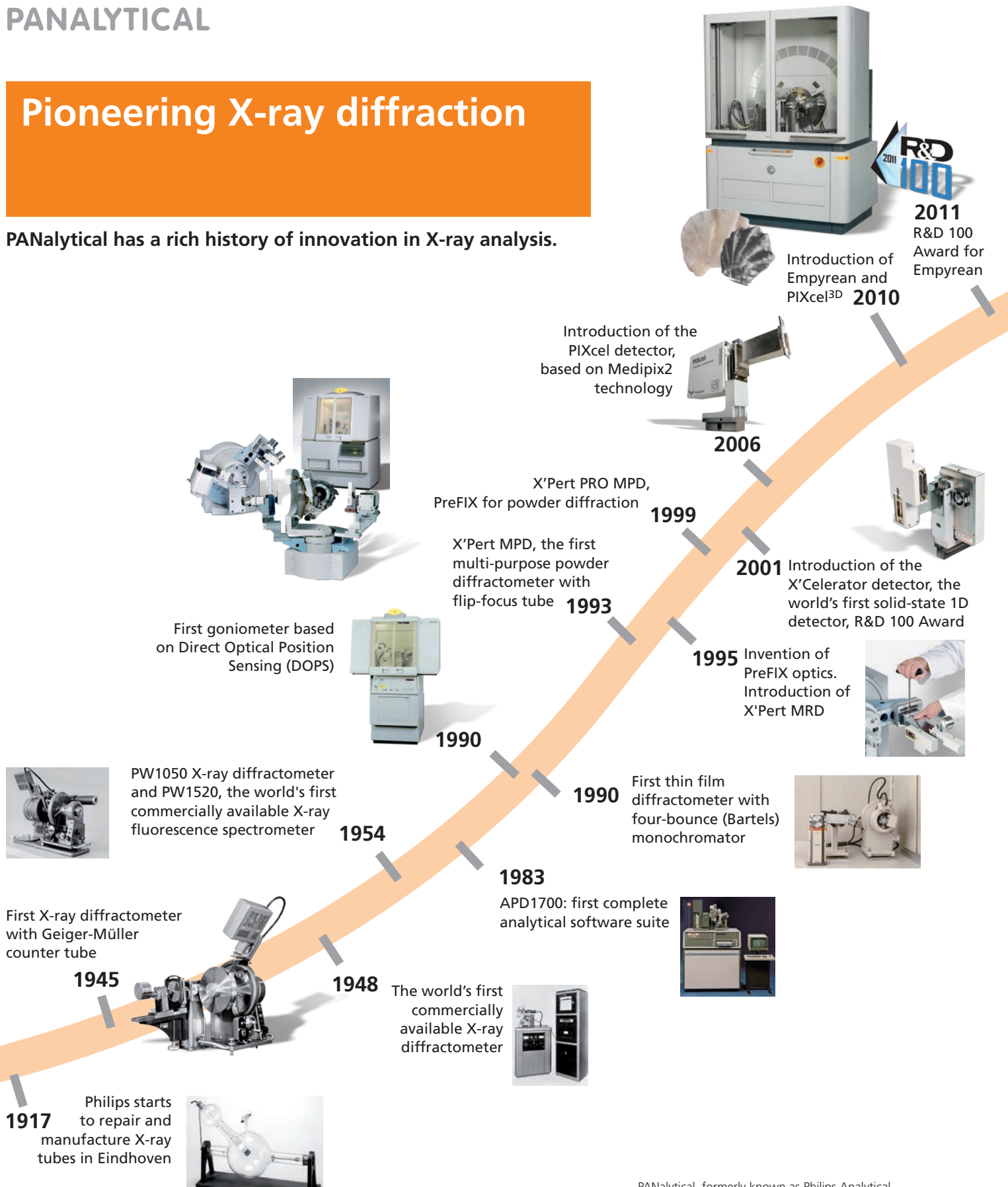
The multi-purpose
solution for your
analytical needs

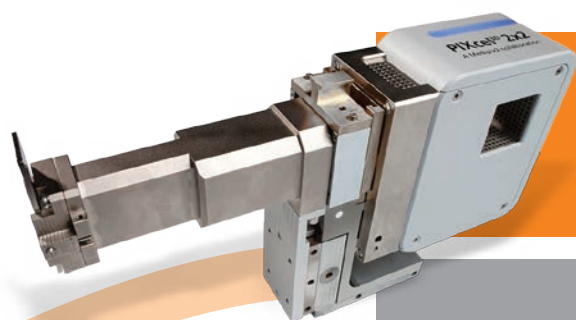


The Analytical X-ray Company

Pioneering X-ray diffraction

PANalytical has a rich history of innovation in X-ray analysis.





2012 Introduction
PIXcel^{3D} 2x2

The Empyrean from PANalytical

Truly innovative,
daringly different.



With the Empyrean, PANalytical has set the new standard for a multi-purpose diffractometer.

The market acceptance of the new platform has been great. This was confirmed by the receipt of the R&D 100 Award under the 'winning technology' category. Empyrean has the unique ability to measure all sample types - from powders to thin films, from nanomaterials to solid objects - on a single instrument.

Moreover, Empyrean not only meets the high expectations of scientists and XRD experts today, but will continue to do so as research themes evolve. The world of materials science is constantly changing and the life of a high-performance diffractometer is much longer than the typical horizon of any research project. With Empyrean, you are ready for anything the future holds.

What's in a name?

The term Empyrean is derived from Aristotelian cosmology (384 -322 B.C.) in which spherical earth was surrounded by concentric celestial spheres containing the moon and known planets¹. The highest sphere, later referred to as Empyrean in the Middle Ages, is represented by the element 'fire'. The Empyrean XRD system is the product of the fire of innovation, and is truly above all others, capable of the most analytical applications on a single XRD platform.

1. G. E. R. Lloyd, Aristotle: The Growth and Structure of his Thought, Cambridge: Cambridge Univ. Pr., 1968, pp. 133-139, ISBN 0-521-09456-9.

The only XRD platform that does it all

The highest data quality on every sample, no compromises

The highest performance goniometer

2nd generation PreFIX for optics and sample platforms

The widest range of samples

Exceptional tube performance

The widest range of sample stages and non-ambient environments

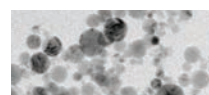
Unmatched area detector dynamic range, linearity and resolution

The world's first 3D detectors: PIXcel^{3D} and PIXcel^{3D} 2x2

Easy access for fast configuration

Flexible, high-performance software packages

Established PANalytical support



4
Sample types

6
Powders

8
Thin films

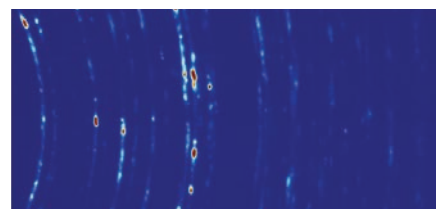
10
Nanomaterials

12
Solid objects

14
Innovation in every area

16
PIXcel^{3D} - cutting-edge technology

18
Partnership with PANalytical - ultimate commitment



EMPYREAN

The only XRD platform that does it all

Sample type

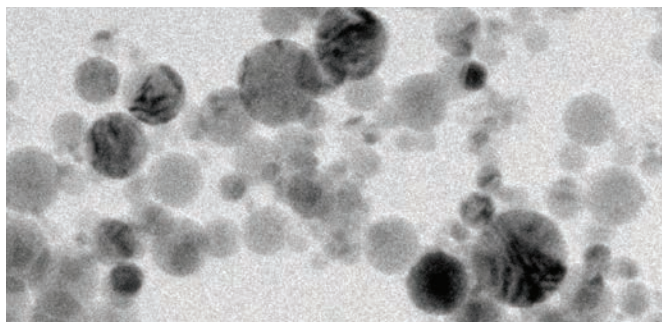
Analytical question



- What is in my powder?
- In which ratios?
- What is the amorphous content?
- What is the crystal structure?
- How do my samples crystallize?
- Can I see preferred orientation?
- What is in my well plates?
- What is the influence of temperature, pressure, humidity?



- What is in my layer(s)?
- What is the layer thickness, roughness and density?
- Is there residual stress?
- Is there preferential growth?
- Can I get information about in-plane reflections?
- Is the layer (hetero)epitaxial?
- Is the layer strained or relaxed?
- What is the influence of temperature, pressure, humidity?



- What is my nanomaterial made of?
- What is the size of the nanocrystals? Is there micro-strain present?
- What is the nanoparticle size distribution and the specific surface area?
- Are the nanoparticles agglomerated or finely dispersed?
- What type of liquid crystalline phase is present?
- Is there some order in my mesoporous material?
- Is my sample nanocrystalline or amorphous?
- How does my nanocatalyst behave at different temperatures or pressure?



- Can I evaluate the internal (micro-)structure of my sample?
- What is in my sample?
- What is in that spot?
- Did machining induce preferred orientation?
- Is residual stress present?
- What is the influence of temperature, pressure, humidity?
- What is my polymer made of? What is the lamellar repeat distance?

Empyrean delivers the best quality data,
on every sample type

Application

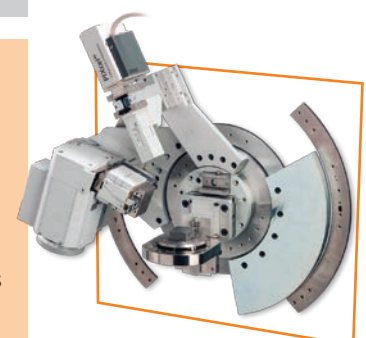
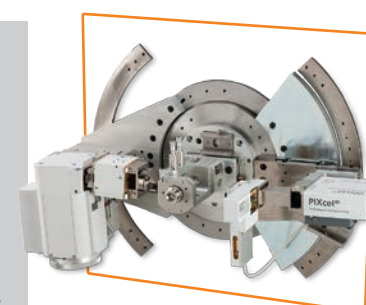
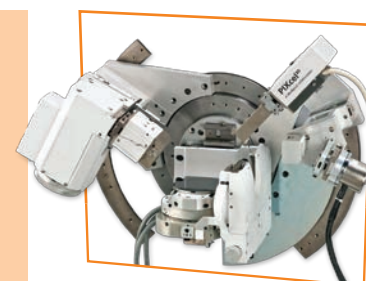
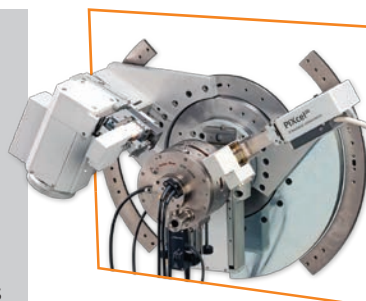
- Phase identification
- Phase quantification
- Crystallinity determination
- Structure determination and refinement
- In-situ crystallization analysis
- Direct visualization of Debye rings
- High-throughput screening (HTS)
- Measurements under non-ambient conditions

- Phase identification (and depth profiling)
- X-ray reflectometry
- Thin film stress analysis
- Orientation analysis
- In-plane diffraction
- Epitaxial layer analysis
- Reciprocal space mapping
- Measurements under non-ambient conditions

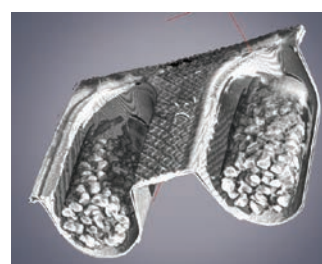
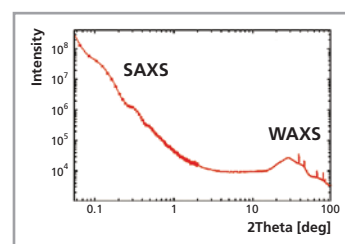
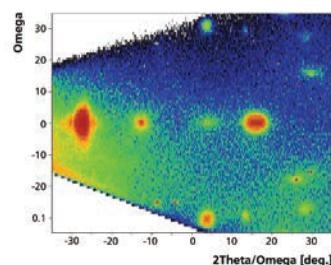
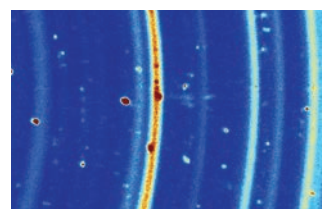
- Phase ID and quantification; XRD (WAXS)
- Size-strain analysis
- Small-angle X-ray scattering (SAXS)
- SAXS
- Transmission diffraction at low angles
- Low-angle diffraction
- Pair distribution function (PDF) analysis
- Measurements under non-ambient conditions

- Computed tomography (CT), radiography
- Phase identification (also in transmission geometry)
- Micro-diffraction
- Texture analysis
- Stress analysis
- Measurements under non-ambient conditions
- Combined SAXS / WAXS

Example configuration



Typical result



EMPYREAN

Powders – unmatched visualization and characterization

The analysis of powders and polycrystalline materials is probably the most common application of XRD.

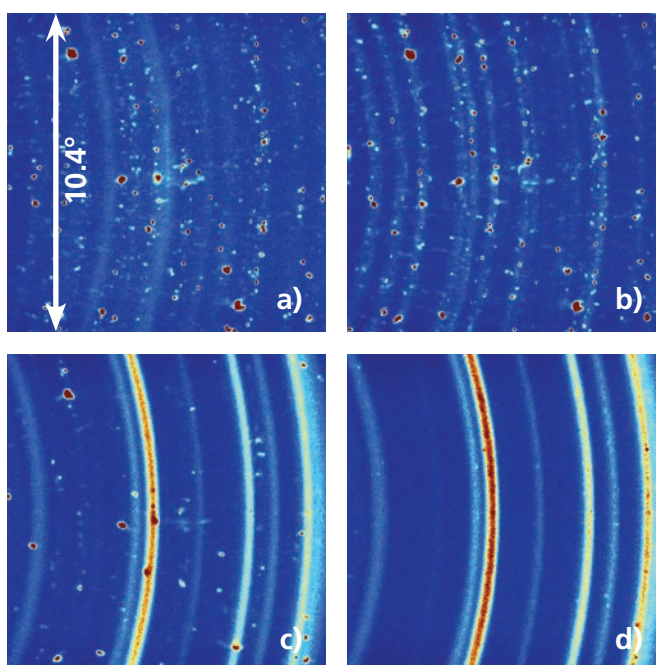
Empyrean offers you a platform to measure and identify the most complex phase mixtures. The sensitivity of the instrument and the power of the software to deal with overlapping patterns is unmatched.

Empyrean has the highest angular resolution of any laboratory powder diffractometer and delivers data closest to synchrotron quality.

Empyrean offers the widest range of non-ambient environments, under full control of the instrument software.

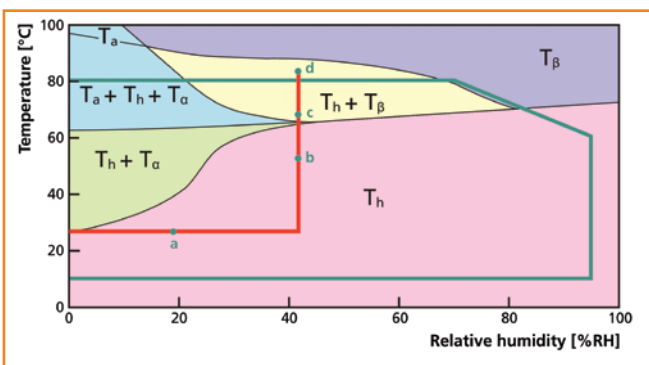
Empyrean is ideal for high-quality research and teaching purposes alike. It offers all relevant diffraction geometries (reflection, transmission, capillary, micro-diffraction, Debye-Scherrer) and is supported by the most comprehensive set of worked examples.

Non-ambient 2D diffraction



High-throughput screening

Color-coded display of samples on a well plate with similar composition or mixtures

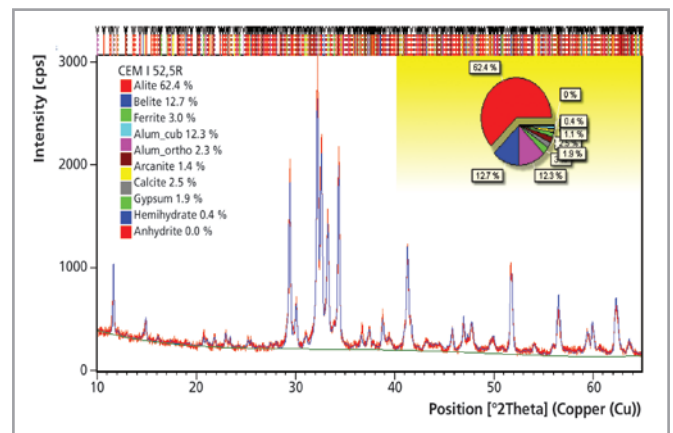


The upper images show 2D diffraction patterns of a α, α -trehalose-dihydrate sample measured in an Anton Paar CHC plus+ temperature-controlled humidity chamber. These data were collected with a PIXcel^{3D} 2x2. The lower graph shows the phase diagram of trehalose together with the temperature-controlled humidity range that can be used with the system ('green' polygon). The graph also indicates the different conditions at which the 2D diffraction patterns were collected. Additional information about the properties of the different phases (e.g. preferred orientation) is easily obtainable. These 2D data sets show the crystallization process, and thus give additional information on top of the high-speed 1D data sets that are typically obtained in this Bragg-Brentano geometry.

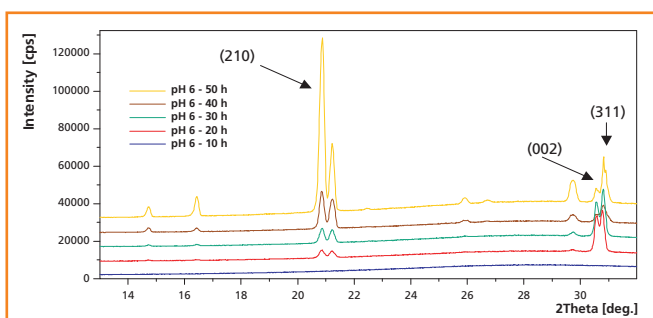


Full pattern quantification

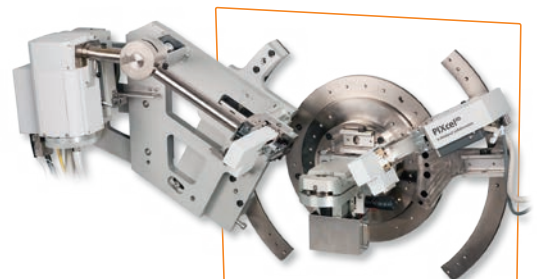
Rietveld standardless quantitative analysis has become an important method for characterizing complex mixtures.



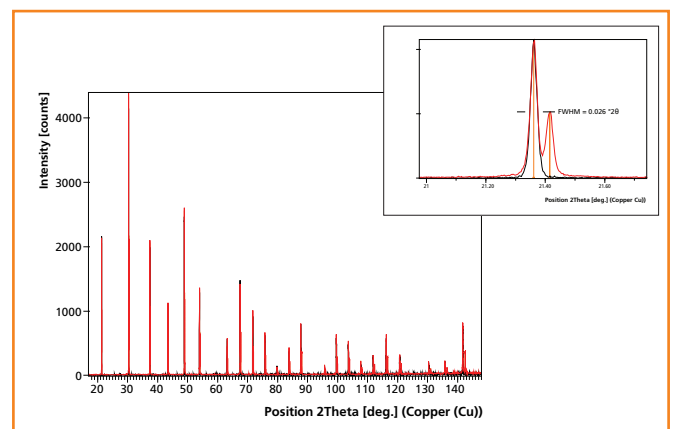
In-situ crystallization monitoring



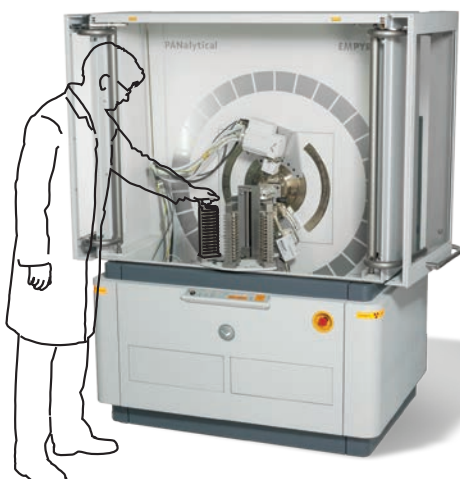
On-line investigation of DL-alanine crystallization at pH 6 measured in a slurry flow cell installed on the Empyrean. After an initial crystal growth with pronounced peaks at the (311) and (002) reflections, crystallization proceeds in the (210) direction after around 38 hours.



Highest angular resolution



LaB₆ measurement in Bragg-Brentano geometry and with Alpha-1 Johansson monochromator shows the best resolution of any laboratory system, with a FWHM of 0.026°.



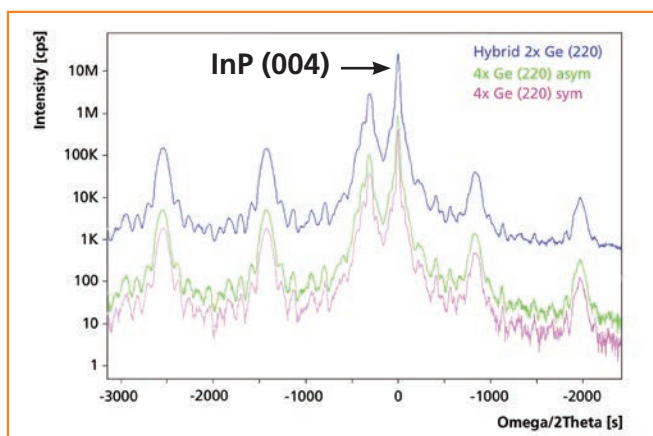
EMPYREAN

Thin films – discover all properties

Diffractometers for the analysis of thin films have typically been highly specialized for this purpose. Now, with Empyrean, the demand for high-resolution analysis has been combined with the advantages of a multi-purpose vertical diffractometer to offer you the largest range of analyses on one system.

Empyrean is the perfect analysis tool to place next to your layer growth equipment. The new Empyrean cradles allow you to fully map wafers and wafer fragments up to 2 inches, and handle up to 100 mm wafers.

Epitaxial layer analysis

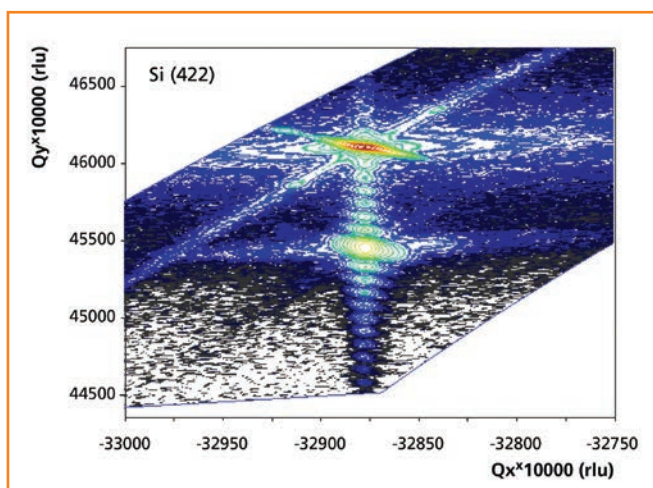


Comparison of the high-resolution performance of different incident beam monochromators on a periodic epitaxial multilayer around the InP (004) reflection. Differences in resolution and intensities are obvious. A range of modules is available that offer a range of angular resolutions matching all analytical needs.

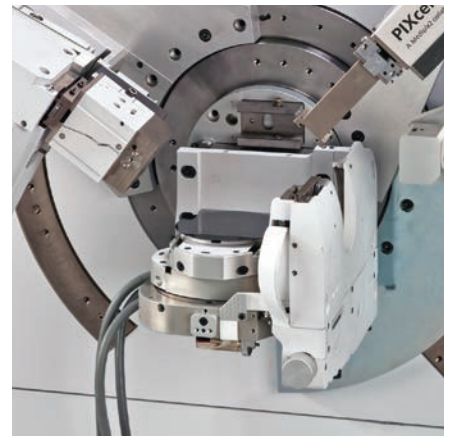
Empyrean offers it all:

- quality control of incoming materials and substrates
- analysis of the thickness and roughness of single- or multi-layer systems with X-ray reflectometry (XRR)
- analysis of epitaxial stacks using rocking curves and reciprocal space maps
- depth profiling using grazing incidence geometry
- orientation analysis of polycrystalline layers
- determination of residual stresses in layers and coatings
- in-plane diffraction

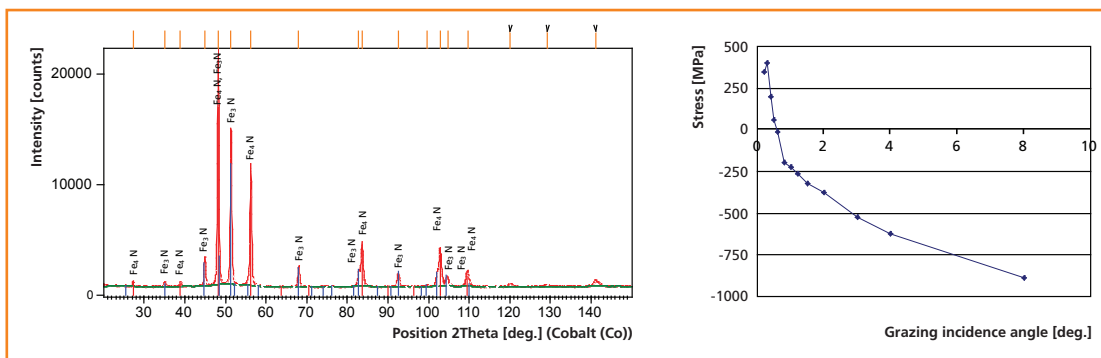
Reciprocal space mapping



Fast reciprocal space map on a Si/SiGe sample measured with a scanning PIXcel detector, allowing a 10 times faster result compared to usual setup

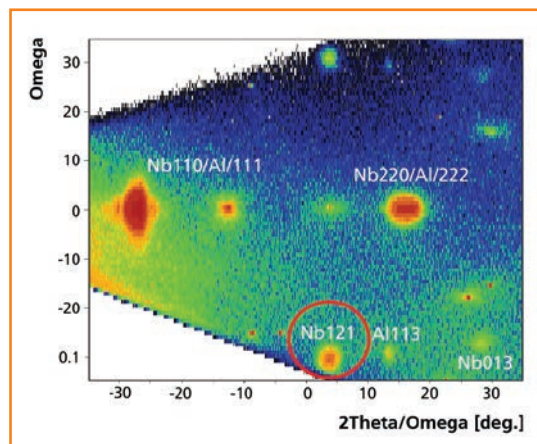
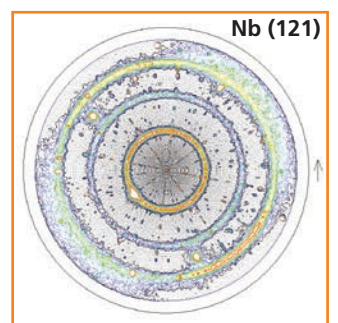
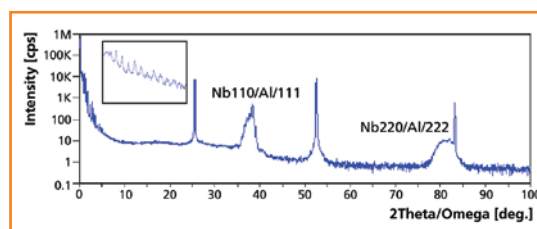


Phase identification, stress analysis and depth profiling

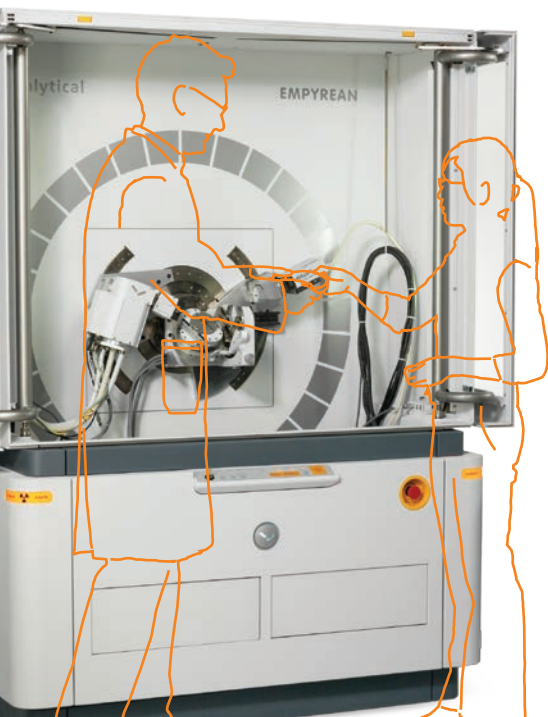


Analysis of a polycrystalline Fe_3N / Fe_4N coating on steel. The stress measurement was performed in grazing incidence. Repeated measurements with different grazing incidence angles allow to probe for the possible existence of a stress gradient.

Reflectometry and orientation analysis



NbAl thin film multilayers grown on sapphire and silicon substrates show fiber texture. Reflectivity and wide-angle scanning provide information in just 5 minutes. Reflectivity reveals information about multilayer thickness and interface roughness. Wide angle scanning can be supported with texture studies using pole figures and or 2theta/omega scans at different omega offsets. Note that the Nb(121) reflection, for example, is absent from the symmetric scan but can be observed with omega offset of 30°. A pole figure shows the Nb(121) reflection as a ring confirming that the in-plane film orientation is random.



Nanomaterials – structural characterization on different length scales

On the Empyrean a variety of complementary X-ray analytical techniques can be applied for the analysis of nanoparticles and nanostructures in powdered samples, liquid dispersions, gels, nanocomposite materials, sheets and thin films.

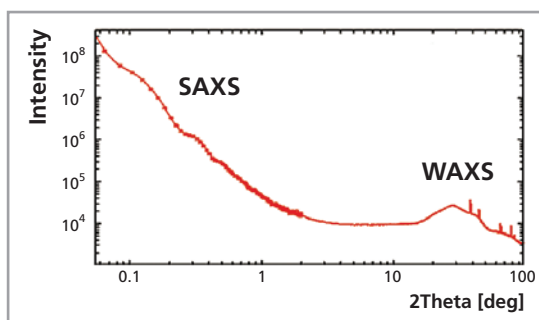
The determination of nanoparticle and pore size distribution, particle shape, specific surface area and nanoparticle agglomeration behavior can be done with small-angle X-ray scattering (SAXS). PANalytical offers a variety of highly cost-effective options for SAXS configurations on the Empyrean. The most advanced configurations allow to obtain data in a quality that so far could only be achieved on dedicated SAXS instruments.

For the investigation of periodic nanostructures very fast results can be achieved by using a transmission setup for low-angle diffraction measurements.

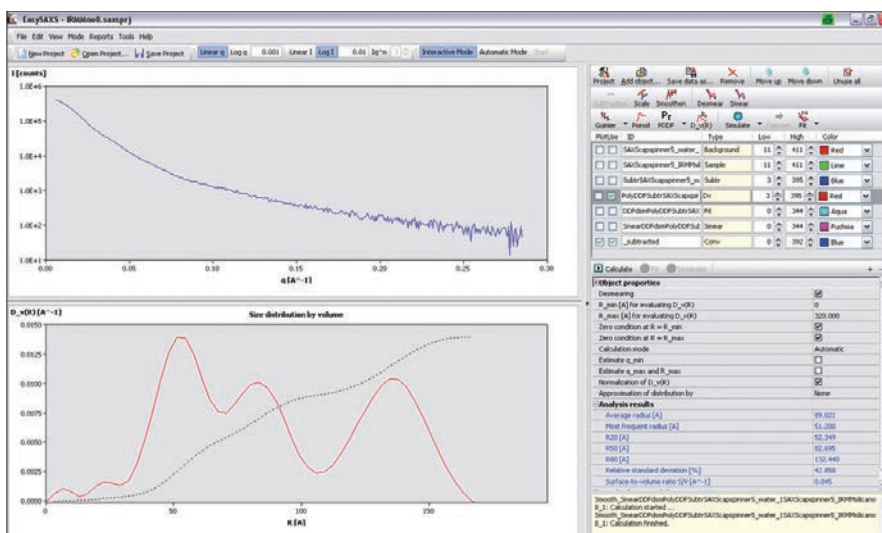
The measurements at small angles can conveniently be combined with WAXS (wide-angle X-ray scattering or XRD) to identify and quantify the crystalline phases that are present in the material, and to determine the size and microstrain in nanocrystals. These measurements can also be done under non-ambient conditions e.g. to study the catalytic activity of a nanomaterial at elevated temperatures and under high gas pressure.

The local atomic order in amorphous and nanocrystalline materials can be deduced from diffraction data that were acquired with hard radiation and up to highest possible diffraction angles. This technique, also known as atomic pair distribution function (PDF) analysis, can be readily applied on the Empyrean platform.

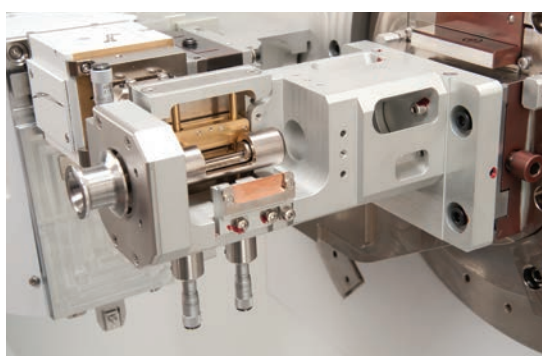
Small- and wide-angle X-ray scattering (SAXS/WAXS)



SAXS/WAXS data measured from a dilute (0.01 vol.%) colloidal dispersion of gold nanorods. The characteristic SAXS profile contains the information about the size and shape of the nanoparticles whereas the distinct peaks observed in the WAXS region confirm the presence of nanocrystalline gold particles with a cubic structure. Note the wide angular range within which the data could be acquired.



Graphical user interface of PANalytical's EasySAXS software that offers a comprehensive toolbox for SAXS data treatment and analysis. Shown in the top graph are SAXS data measured from a dilute dispersion of silica nanoparticles in water having a concentration of 0.8 Vol.%. The lower graph displays the deduced nanoparticle size distribution revealing that the sample contains three distinct size fractions (5, 9 and 14 nm).

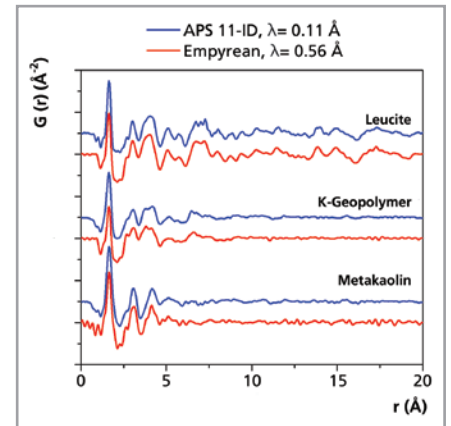
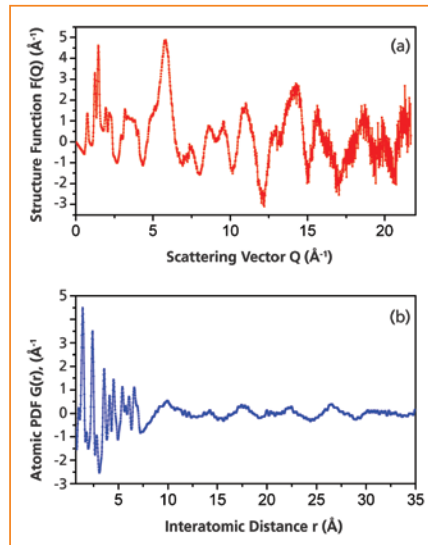


Sample stage developed for SAXS/WAXS and low-angle diffraction measurements of liquids, powders, solids, gels or fibers. The setup allows to measure scattering data at a very wide angular range from typically 0.07 up to 130 deg 2θ. A high-quality anti-scatter slit and a semi-transparent beamstop allow for optimal suppression of the background signal.



Pair distribution function (PDF) analysis

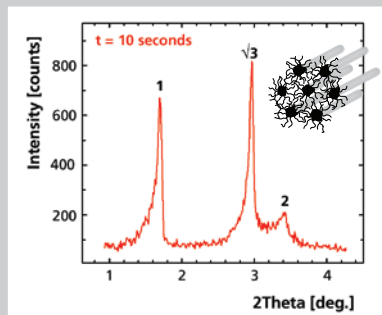
a) Structure function and b) atomic PDF of C_{60} fullerene (buckyball). The experiment was performed with Ag $K\alpha$ radiation, allowing a maximum scattering vector of 22 \AA^{-1} . The PDF data shows sharp peaks up to 7 \AA and broad features beyond this point. The sharp peaks correspond to the well-defined distance between atoms inside each C_{60} molecule. The broad features originate from the correlation of electron density between different buckyballs.



Low-angle diffraction on nanostructured materials

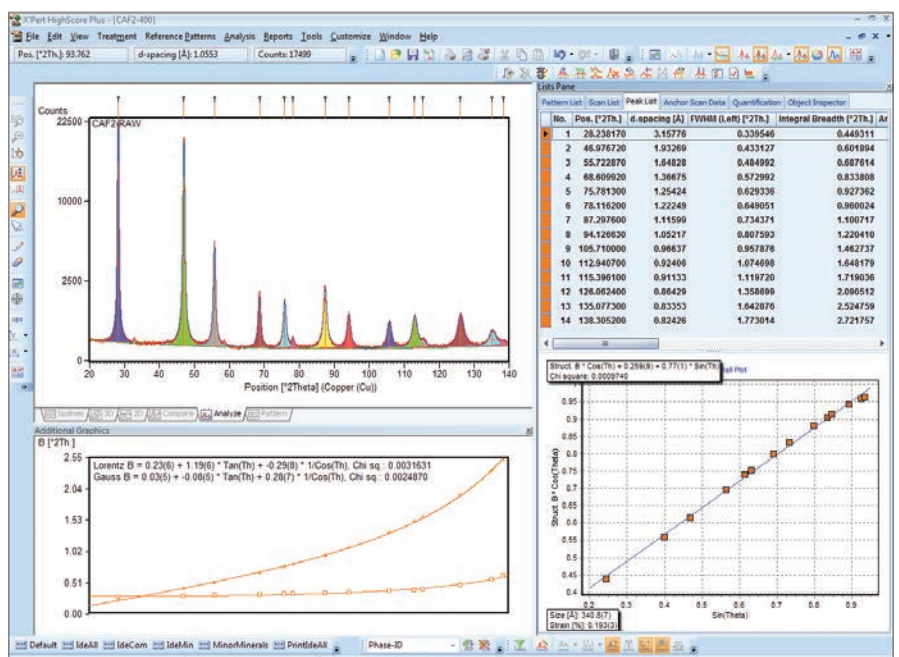
The observed low-angle peaks allow to identify periodic nanostructures and to quantify characteristic dimensions e.g. in mesoporous materials, block-copolymers, polymer-clay nanocomposites and liquid crystalline phases formed by surfactants or lipid molecules.

When using a focusing mirror in combination with the PIXcel^{3D} detector in static mode, such measurements can be done within just a few minutes, or even within seconds.



Data measured from a hexagonal liquid crystalline phase formed by a nonionic surfactant in water. The measurement time was only 10 s.

Size-strain analysis



Information on the microstructure of crystalline materials (crystallite size and microstrain) is obtained from the width and the shape of X-ray diffraction peak profiles. HighScore (Plus) can calculate size and strain, based on a Williamson-Hall plot (see screenshot) as well as the Rietveld and Scherrer methods.

Solid objects – research without compromises

For particular applications samples must be analyzed without any sample preparation.

Examples are widespread:

- geological samples
- pharmaceutical tablets
- engineered components such as ball bearings, joints and axles
- polymers and polymer (nano) composites

Empyrean can handle all of these samples.

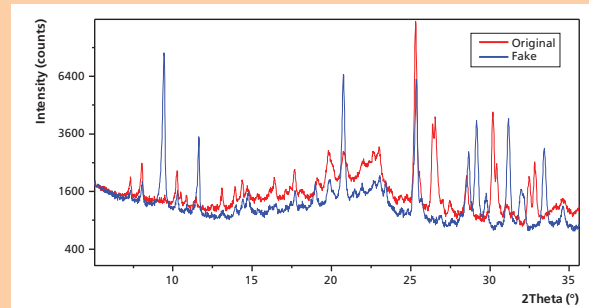
For the phase identification of rough and irregularly shaped objects, parallel beam geometry is often used. PANalytical's philosophy for this geometry is consistent with all our set-ups - optimize, not compromise.

Empyrean can also analyze large and heavy samples up to 10 kg, a unique capability for a flexible multi-purpose X-ray system. The 3-axes Empyrean cradle allows you to mount components of up to 2 kg, and measure preferred orientation (texture) and residual stress using the chi-tilt or omega-tilt method.

A further unique possibility with Empyrean is to see inside small solid objects without having to cut them, thanks to the possibility of performing computed tomography experiments.

Computed tomography and phase identification

CT can be used to visualize the fabric, defects and inclusions of a sample. As an example a combination of CT and XRD has been applied to compare original and counterfeit tablets. XRD allows to determine the phases that are present in the tablets. The X-ray diffractogram shows some differences in the composition of the two tablets and in the crystallinity of the API (active pharmaceutical ingredients).



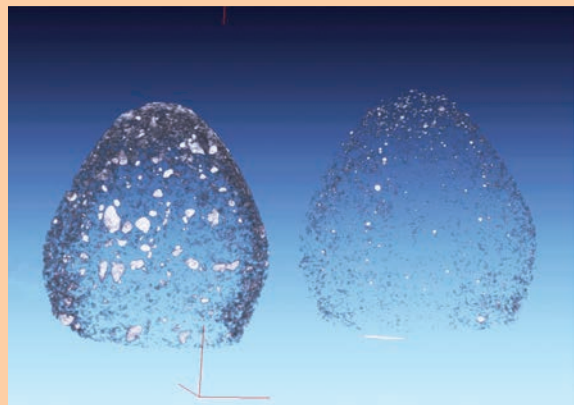
CT provides complementary information, such as relative density, inhomogeneities and granularity. The CT reveals strong differences in the inhomogeneity of the tablets. Whereas the original tablet shows a consistent product, the counterfeit tablet exhibits some bigger inclusions.

Radiography, computed tomography and micro-diffraction

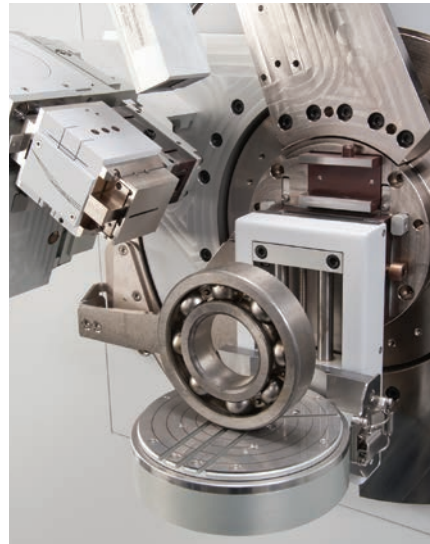
Empyrean offers unrivalled possibilities for non-destructive analysis. Non-destructive investigation of devices in microsystems is a powerful tool for quality assurance. In this example, a memory card is studied by X-ray radiography and computed tomography.

Thanks to the excellent resolution of the PIXcel^{3D} detector, it is possible to visualize small details inside the device.

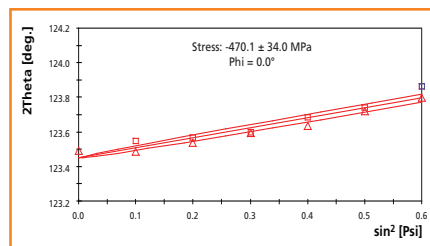




Comparison of a counterfeit (left) and a real (right) tablet. The counterfeit tablet shows more inhomogeneities.

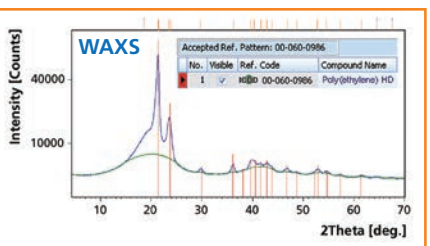
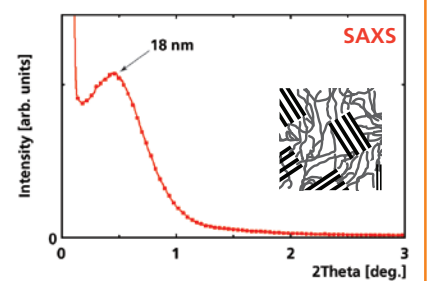


Stress analysis



Stress measurement was made on an inner ball bearing ring probing the bottom of the raceway. The graph shows the $\sin^2\psi$ plot of the data determined in raceway direction.

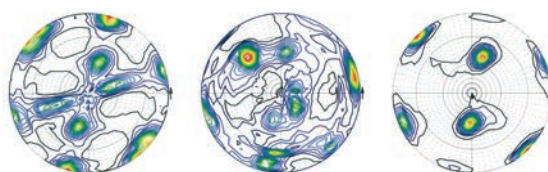
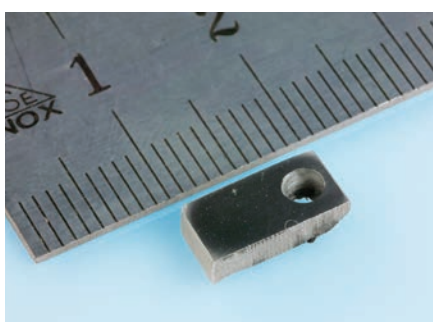
SAXS / WAXS on polymers



Small- and wide-angle X-ray scattering (SAXS/WAXS) data measured from a semi-crystalline polymer sample. From the peak observed at small angles the lamellar repeat distance can be determined. WAXS data allow to identify the type of polymer and possible filler materials, and to estimate the degree of crystallinity.



Texture analysis



Typical ECAP-processed sample and pole figures determined for three different reflections

Texture analysis of aluminium processed by equal channel angular pressing (ECAP) – an effective method to reduce grain size and enhance mechanical properties of metals and alloys.

EMPYREAN

Cutting-edge technology in every aspect

In order to make the ultimate X-ray diffractometer for powders, thin films, nanomaterials and solid objects, all essential components have been newly developed by our experienced R&D team.

2 Universal PreFIX

The Empyrean system is equipped with universal PreFIX optics, stages and accessories, PANalytical's proven proprietary kinematic mounting concept for Pre-aligned Fast Interchangeable X-ray modules. The robust hardened tool steel mounts allow a reproducible positioning of optics and stages in three dimensions with microns precision eliminating any need for re-alignment.

1

PANalytical's Empyrean **Tubes**: robust exchange of tube focus position and largest variety of tube anode materials



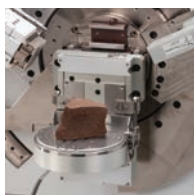
3

The unique family of **PIXcel detectors**, co-developed with CERN and other leading European scientific institutes - more on next pages



4 Quickly interchangeable sample stages

Unique alignment-free exchange



5

Meets all relevant **worldwide regulations**, for electrical, mechanical and X-ray safety, with all anode types, no power restrictions.



6

19" rack mounts for non-ambient controllers and integrated vacuum system

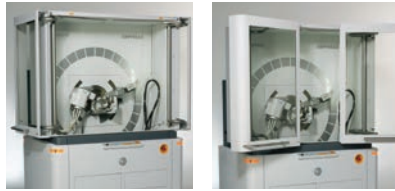
7

Enclosure is **on wheels** for easy installation and relocation.



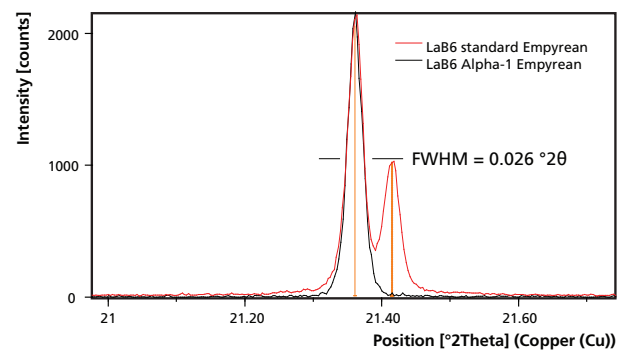


9 Wide opening cabinet gives easy access to the experimental area.



9 The world's most accurate high-resolution goniometer

Step size 0.0001°, 2 θ linearity $\pm 0.01^\circ$ over the whole angular range.



Empyrean sets the new bar for laboratory X-ray system resolution, with a FWHM of 0.026 degrees 2 θ for the first reflection of NIST SRM660a LaB₆.

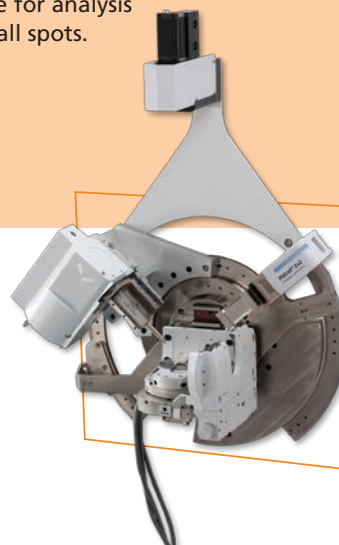
"We combined long-time experience and the latest technology to make a high-performance workhorse with ultimate flexibility for today's and future applications".



8 Comprehensive software suite for data collection and analysis. Supports multiple users, unattended and remote operation, and automatic data collection, analysis and reporting.



10 Alignment camera allows the user to visually position the sample for analysis on small spots.



A heart with a brain

The heart of the Empyrean is a the revolutionary high-resolution goniometer evolving upon a long PANalytical tradition in precision goniometers. It makes use of the next generation of Direct Optical Encoding System (DOPS2), featuring precisely aligned Heidenhain encoders and Path Tracking Technology (PTT) for continuous advanced motion control based on Digital Signal Processing (DSP). Due to this advanced motion control the goniometer offers highest resolution and differential accuracy and positions its arms faster and more precisely than ever before.

PIXcel detectors – cutting-edge technology

The introduction of the PIXcel detectors (PIXcel^{1D}, PIXcel^{3D} and PIXcel^{3D} 2x2) adds a new dimension to XRD analysis.

These unique photon counting detectors are the most advanced in the world. The combination of smallest pixel size, point spread function of one pixel and high stopping power of the sensor results in the highest resolution available on the market and together with the highest dynamical range and low noise sets a new standard of performance.

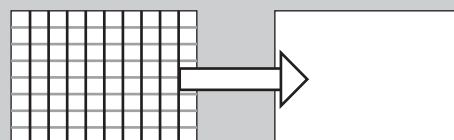
One of the advantages of the high resolution is that it allows to bring the detectors closer to the sample, increasing the angular range to more than 28 degrees (for the PIXcel^{3D} 2x2), without compromising on data quality.

In addition, the 2D scanning possibilities, available for the PIXcel^{3D} and PIXcel^{3D} 2x2, make them the most versatile detectors ever available on a commercial diffractometer.

Properties:

- Smallest pixel size (55 µm x 55 µm)
- Point spread function = 1 pixel
- High dynamic range (>10¹⁰)
- Window energy discrimination
- True photon counting detector
- No calibration required

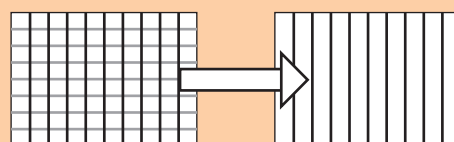
Detector mode



0D mode: All pixels are added up to give one intensity value as a function of time

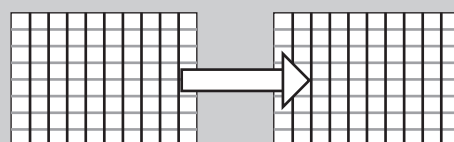
PIXcel^{1D} and PIXcel^{3D} advantages

The point detector with the highest dynamic range, highest maximum count rate and lowest background



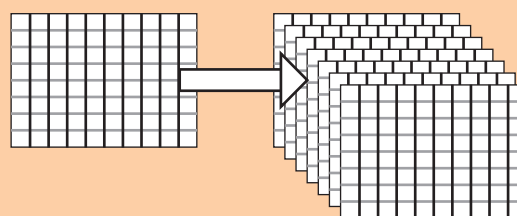
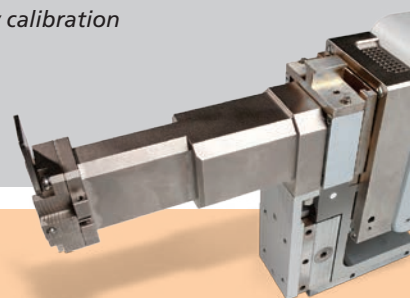
1D mode: All pixels in one column are added up to form a position-sensitive detector in one direction

The line detector with the highest dynamic range and the smallest strip size



2D mode*: All pixels are read out independently: direct observation of the X-ray image

The area detector combining the advantages of highest spatial resolution, high dynamic range per pixel and low noise - not requiring any calibration

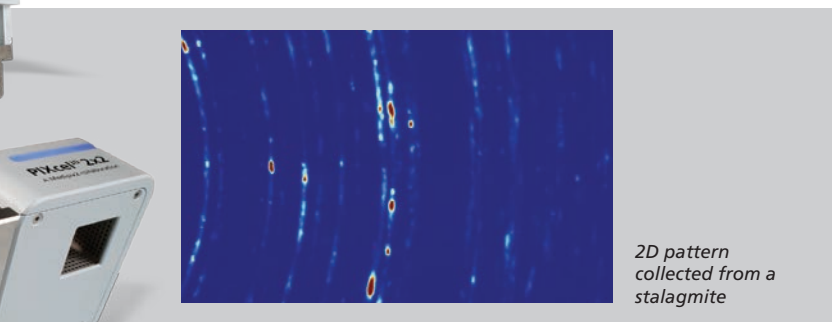
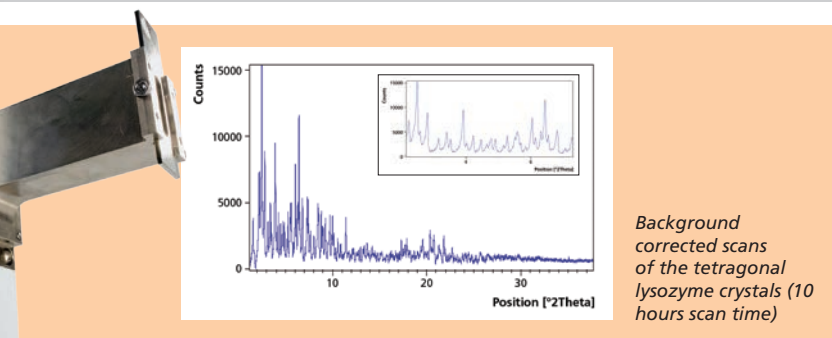
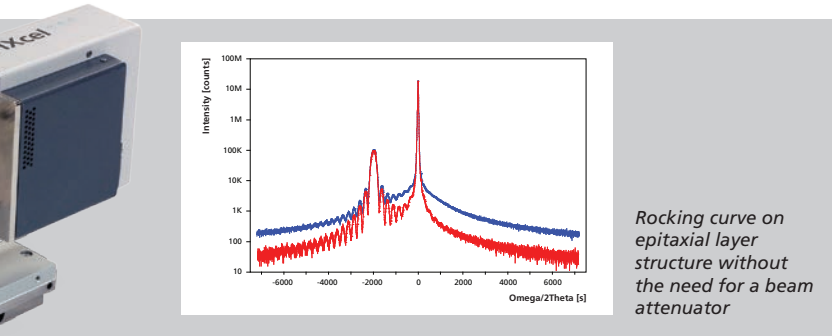


3D mode*: All pixels are read out independently: many 2D images are combined to reconstruct the voxels of the object: computed tomography

The computed tomography detector with the highest dynamic range and lowest noise

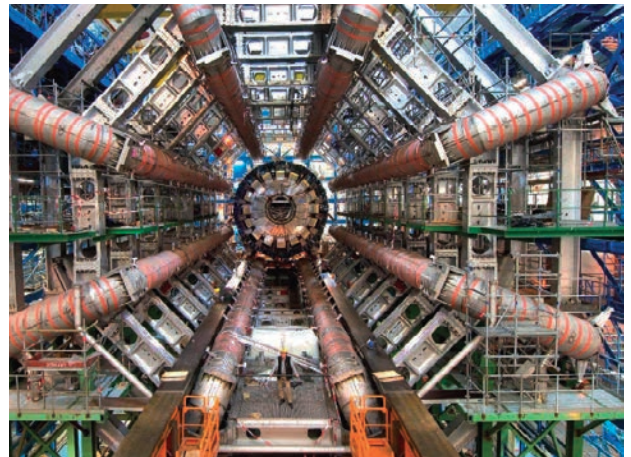
* only available on PIXcel^{3D} and PIXcel^{3D} 2x2

Typical applications



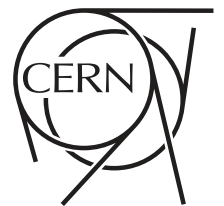
A strong partnership with CERN

PIXcel^{3D} is the result of PANalytical's partnership in the Medipix2 collaboration – a consortium of more than 16 leading particle physics research institutes across Europe, headed by CERN. As state-of-the-art detector developments have become more complex, only the largest groups active in fundamental research can deal with the planning and investment involved.



The ATLAS detector during its construction phase. Note the person in the front

PANalytical is an industrial partner to this collaboration and has secured the exclusive rights to commercialize the resulting technology for analytical X-ray applications, putting you at the forefront of detector developments for many years to come.



"The Medipix2 chip technology combines high spatial resolution, high dynamic range and low noise. The Medipix2 collaboration is happy that this leading-edge technology is being exploited by PANalytical for materials analysis."

M. Campbell, Spokesman, Medipix2 collaboration

"PANalytical adopted the technology early and has been an invaluable Technology Transfer partner for CERN and the Collaboration."

B. Denis, CERN Technology Transfer

Partnership with PANalytical - ultimate commitment

Your decision to invest in Empyrean marks the beginning of a relationship that will last for many years. A critical goal in our development of Empyrean was to do more than provide a solid platform for each user to build their perfect XRD system. Together with our technical specialists and applications scientists, each diffractometer will be developed into the perfect instrument for your analysis needs. Your input guides our development efforts, and jointly we will get the most information out of your challenging materials.



Access to expertise



PANassist

- Always connected to PANalytical's customer care network
- Remote diagnostics
- Instrument certification
- Automated alert and monitoring
- Shared desktop support



Training courses, instrument familiarization sessions, application workshops and remote learning channels provide important information for new and existing users. PANalytical facilities around the world deliver these, alongside more general scientific meetings on a variety of X-ray diffraction and scattering topics.



Extending your capabilities

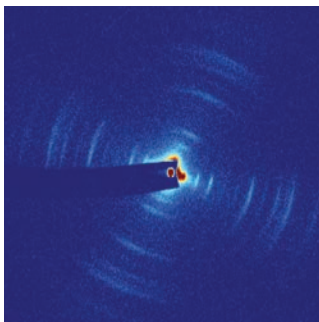
PANalytical's track record of adding new technology and new applications to existing systems is unrivalled, thanks to consequent use of PreFIX modules over many years. Empyrean will build on this heritage.

This page shows a few examples of the technologies we are working on for further expansion of Empyrean's capabilities:

- GISAXS (grazing incidence small angle X-ray scattering)
- Evacuated beam path for SAXS/WAXS
- 2D SAXS
- Topography
- Microfocus X-ray sources and optics



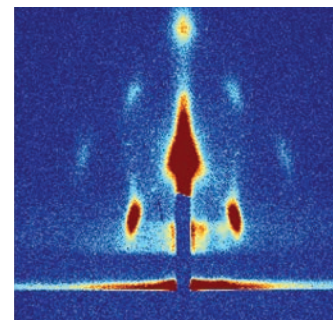
Microfocus X-ray source



2D SAXS pattern measured from two crossed rat tail collagen fibers



Attachment for SAXS/WAXS measurements with evacuated beam path (patented). For highly dilute and low-contrast samples and for increased data collection speed.



GISAXS measurement of a thin film of mesoporous silica. The distinct reflections indicate a high degree of order in the spatial arrangement of the pores.



X-ray topographs of potassium alum single crystal

Global and near



PANalytical

PANalytical is the world's leading supplier of analytical instrumentation and software for X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF), with more than half a century of experience. The materials characterization equipment is used for scientific research and development, for industrial process control applications and for semiconductor metrology.

PANalytical, founded in 1948 as part of Philips, employs around 1000 people worldwide. Its headquarters are in Almelo, the Netherlands. Fully equipped application laboratories are established in Japan, China, the USA, and the Netherlands. PANalytical's research activities are based in Almelo (NL) and on the campus of the University of Sussex in Brighton (UK). Supply and competence centers are located on two sites in the Netherlands: Almelo (development and production of X-ray instruments) and Eindhoven (development and production of X-ray tubes). A sales and service network in more than 60 countries ensures unrivalled levels of customer support.

The company is certified in accordance with ISO9001-2008 and ISO 14001.

The product portfolio includes a broad range of XRD and XRF systems and software widely used for the analysis and materials characterization of products such as cement, metals and steel, nanomaterials, plastics, polymers and petrochemicals, industrial minerals, glass, catalysts, semiconductors, thin films and advanced materials, pharmaceutical solids, recycled materials and environmental samples.

Visit our website at www.panalytical.com for more information about our activities.

PANalytical is part of Spectris plc, the precision instrumentation and controls company.

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