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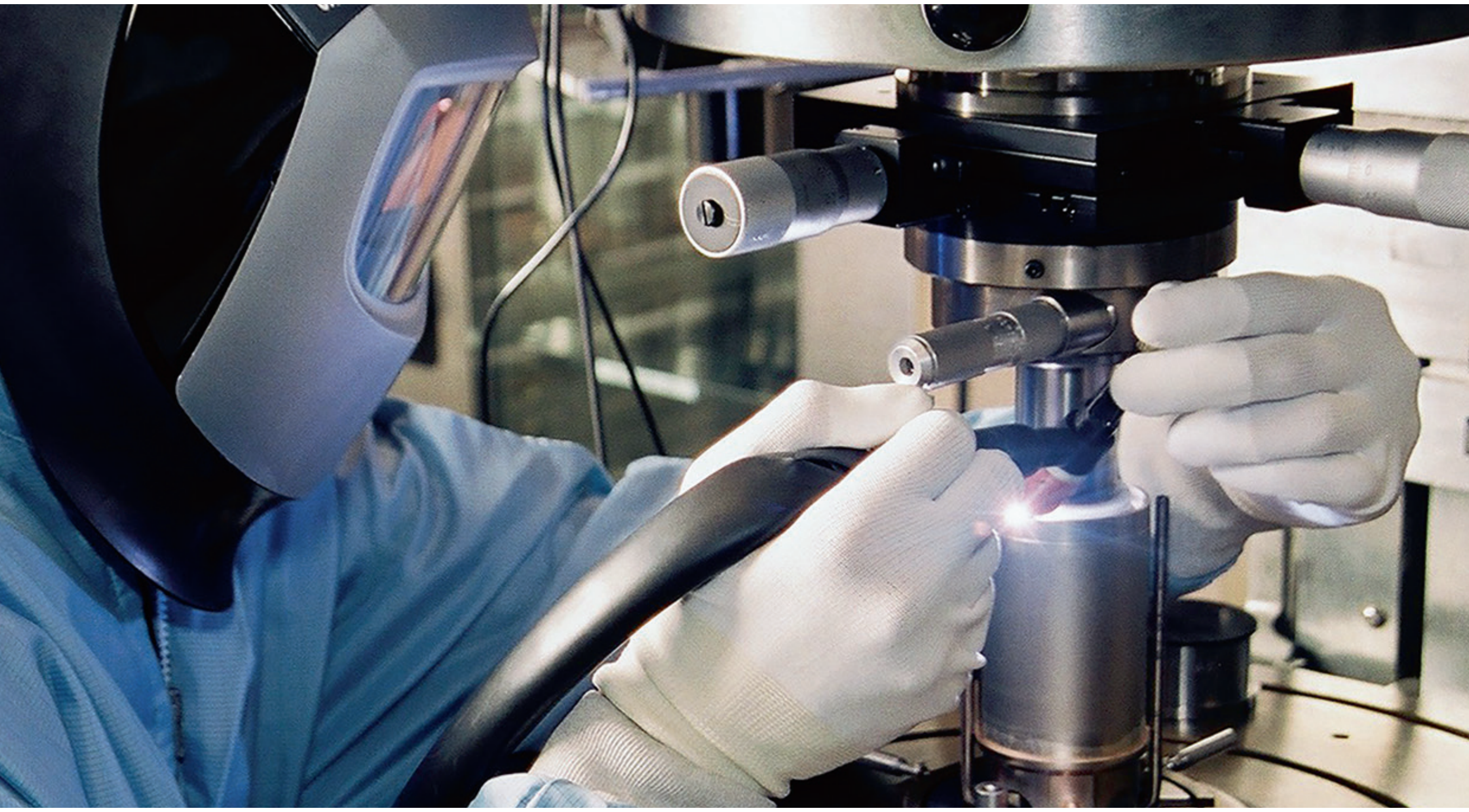


The Empyrean Tube

Advanced eco-friendly design, powerful performance

The Empyrean Tube

The industrial benchmark, redefined

PANalytical's Empyrean Tubes set the standard, both for X-ray diffraction (XRD) performance and eco-friendliness. All PANalytical X-ray tubes are developed and manufactured by the company's dedicated team in Eindhoven, the Netherlands - a facility with over 90 years experience in the field. Continuous investment in the latest technology has maintained and strengthened PANalytical's position as the world's leading producer of analytical X-ray tubes.

The comprehensive series of Empyrean Tubes are metal-ceramic high-power sealed XRD tubes designed for a wide range of demanding applications including: qualitative and quantitative powder diffraction; stress and texture analysis; layer thickness analysis (reflectivity), small-angle X-ray scattering (SAXS), pair distribution function (PDF) analysis as well as analysis of heteroepitaxial layers (high-resolution measurements).

Importantly, the use of lead-free solder and the incorporation of novel design elements that allow straightforward disassembly and recycling, mean that Empyrean Tubes are fully compliant with RoHS and WEEE regulations. The design follows the 'cradle to cradle' approach, aiming at a closed material loop and recycling of all components. In addition, Empyrean Tubes have two beryllium windows rather than four, and these two windows are shaped in accordance with their function (point or line focus). This not only minimizes beryllium content, but enhances ruggedness and durability, and further demonstrates the Empyrean Tube's superior environmental profile.

Improved performance

In addition to features proven in PANalytical's successful XRD-C and X'Pert tubes, the Empyrean Tube offers superior alignment reproducibility, enhanced ruggedness, easier switching from point to line focus and optimized interfacing to prevent condensation or corrosion inside the tube shield.

X-rays generate ionized air and together with the moisture in the air it generates nitric acid, which ultimately causes corrosion on the tube and shutter. Moreover, via air convection it may even cause corrosion on expensive mirrors. With CRISP (Corrosion Resistant Incident Smart Beam Path) PANalytical has virtually eliminated corrosion on these critical components with an optimized design of the tube and various parts in the incident beam path. The technology has been patented.

Empyrean Tube benefits

- **Patented, compact metal-ceramic design**
- **Fast and easy point-to-line focus switching**
- **Recognizable point and line focus windows**
- **Two different focal spots (LFF and BF)**
- **Wide range of anode materials**
- **High power**
- **Excellent long-term stability**
- **Corrosion-free tube and tube shield**
- **Superior spectral purity**
- **Low cost of ownership**

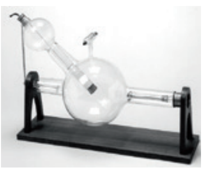
Point focus window

Line focus window

Initiators and innovators

1917

Philips starts to repair and manufacture X-ray tubes in Eindhoven.



1924

Philips develops the MetaliX X-ray tube, the world's first X-ray tube with radiation protection.

1953

Philips Analytical introduces the pioneering sealed, water-cooled X-ray diffraction tube. This technological innovation quickly established the industrial standards for spectral purity, intensity and tube life.

1993

Philips Analytical launches the next significant innovation, a compact metal-ceramic X-ray diffraction tube (XRD-C). This was the first metal-ceramic X-ray diffraction tube in the world. It combined the features of its glass counterpart with the advantages of metal-ceramic technology: high alignment accuracy and improved thermal stability.

2006

The successor to the XRD-C is introduced. The X'Pert tube adds a number of user benefits unique in the market. Specially designed for PANalytical's successful X'Pert PRO, CubiX PRO and CubiX FAST diffractometers, the X'Pert tube fulfils the most demanding X-ray diffraction applications.

2009

Building on the success of the X'Pert tube, the Empyrean Tube is the second generation of its type. It sets a new and clearly recognizable standard of industrial design, expressing PANalytical's quality and integrating form and function.

Much more than just new technology

Advanced anode
PANalytical's advanced anode technology uses target deposition by thermo-compression to create an extremely smooth anode surface. This greatly reduces absorption losses, mainly at lower take-off angles, while the 300 µm beryllium window ensures minimal absorption at all wavelengths.

Customer support

PANalytical is dedicated to providing outstanding customer support so that users realize maximum tube performance and lifetime.

Environmentally friendly
Empyrean Tubes are more eco-friendly. They comply fully with RoHS and WEEE standards by halving the number of beryllium windows, minimizing the window size, and using lead-free solder in an improved design that allows straightforward disassembly and recycling of components.

Application driven
Two different focal configurations – plus a variety of standard and special anode materials – are available to match all application requirements.

Dedicated design
The Empyrean Tube is specially designed for PANalytical's Empyrean, X'Pert PRO, X'Pert^3, CubiX PRO and CubiX^3 diffractometers. It integrates perfectly into the X-ray optical path, providing the best possible analytical performance.

Efficiency and long life
Using high power results in an increased X-ray intensity and ensures a brilliant focal spot. At the same time, efficient anode cooling ensures optimal heat dissipation to reduce focal spot temperature and greatly extends tube life.

Spectral purity
Each PANalytical XRD Empyrean Tube is designed and constructed to rigorous standards, providing both excellent spectral purity and precise beam focusing, for all selections of anode material and focal configuration.

Matching the tube to the application

The correct choice of X-ray diffraction tube depends on the application. Primary considerations include: optimization of peak-to-background ratios, the d-value range under study and the required penetration of X-rays into the sample. In addition, potential complications due to sample fluorescence and micro absorption need to be considered.

Anode material	Remarks	Empyrean	CubiX³
Cu	Standard anode material for most XRD applications	x	x
Co	More intensity for Fe and Co-based materials	x	x
Cu, Co, Fe, Mn, Cr	For stress analysis on various materials according to norm EN 15305	x	
Co, Fe, Mn, Cr	For mining applications which require an improved intensity and reduced background due to fluorescence		x
Mo, Ag	For applications which require a higher penetration depth (transmission through thick samples) For pair distribution function analysis (PDF)	x	

A choice of focus...

For many diffraction applications: phase identification, full-pattern quantitative analysis and thin film measurements, for example, both high resolution and high intensity are desirable. In these situations a long fine focus tube offers the best solution – combining the resolution of a fine focus tube with improved intensity. A special version of this tube exists, designed to work with high-performance X-ray optical modules such as X-ray mirrors.

For classical quantitative analysis that is based on intensity measurements of one (or only a few) diffraction peaks, instrument sensitivity may be more important than ultimate resolution. When this is the case, the best choice is a broad focus tube, as it offers the highest X-ray output. Standard versions of broad focus Empyrean Tubes are available for Cu and Co radiation; other types are obtainable upon request.

Line focus or point focus?

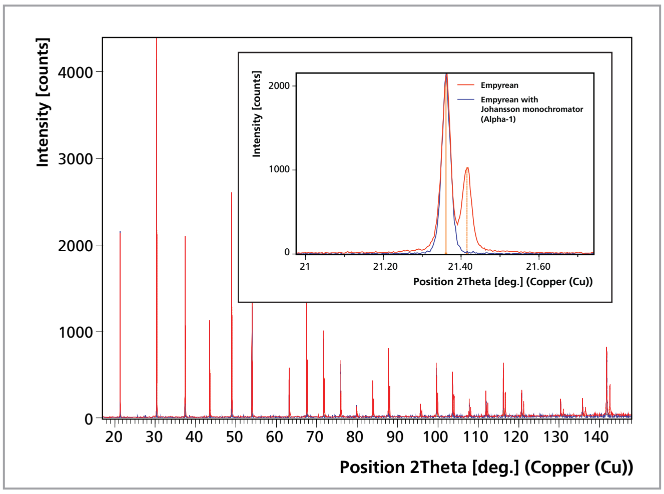
Many diffraction applications make use of the line focus position of the X-ray tube. For some specific applications, especially those where small spots are investigated, or the sample is tilted, in texture or stress analysis for example, the point focus position of the X-ray tube is preferred. PANalytical's X-ray tubes offer the possibility to quickly change the tube's focal position from line to point focus and back, without the need to disconnect the high voltage and water lines. The position of the tube is automatically detected and recorded in the XRDML file.

The Empyrean Tube

Performance in practice

Unmatched angular resolution

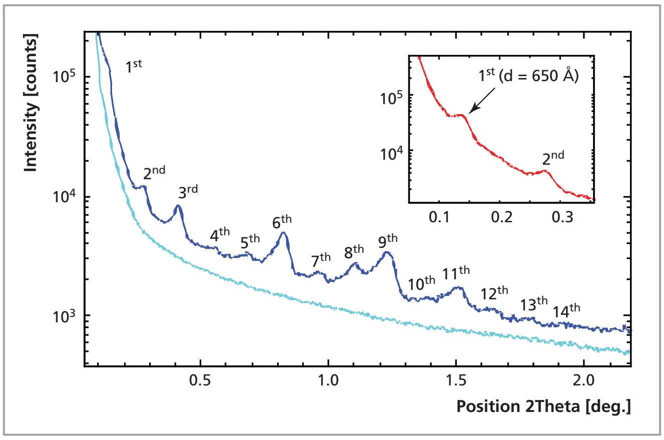
In Bragg-Brentano parafocusing geometry, the dimensions of the tube focus are important as they influence the instrumental contribution to the profile width. The true performance of the tube and the diffractometer on this parameter can best be determined with the NIST SRM660a standard for line profile: Lanthanum hexaboride (LaB_6). The figure on the right shows the full width at half maximum of the first LaB_6 reflection, recorded with a Cu LFF Empyrean Tube on an Empyrean system with X'Celerator detector.



Unmatched resolution, low background and wide angular range performance on NIST SRM660a

Unmatched low-angle performance

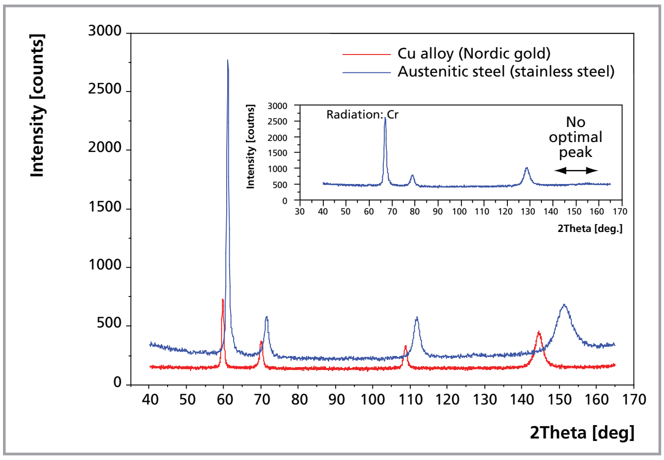
Measuring long-period reflections is a challenging task in the reflection geometry. The beam needs to be very narrow in order to avoid direct beam energy reaching the detector. In addition, the position of the reflections is extremely sensitive to sample displacement error. In transmission geometry, the displacement error at low 2theta angles is eliminated, allowing measurements at extremely small angles. The combination of tube and optics plays an important role in determining the longest period one can see on a powder diffractometer.
The figure on the right shows transmission data on rat tail collagen recorded with a focusing mirror and a hybrid monochromator (insert). Both optical modules show the first reflection at 0.14° 2θ - an outstanding result, corresponding to a period of more than 600 Å.



Low-angle diffraction from dry rat tail collagen

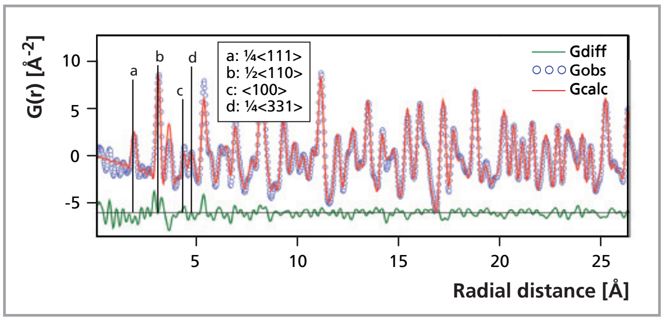
Residual stress analysis – for every steel alloy the appropriate tube

For proper residual stress analysis the choice of the X-ray tube is made such that a suitable diffraction peak is available at high diffraction angles. This improves the sensitivity of peak shifts. The European norm on residual stress analysis EN 15305 lists a number of different materials with recommended X-ray tubes and the {hkl} reflections of the material in the range from 140-160 °2θ. For example, a Cr tube is recommended for ferritic steels, while a Mn tube is specified for austenitic steels, Ni and Co alloys, as well as Cu alloys.



Reflections are in the desired range for these materials when using a Mn tube

Pair distribution function analysis



Experimental (circles) and calculated atomic PDF (red line) of SiC

A diffractogram contains more information than just the Bragg peaks. Structural information about nanomaterials is present as broad, not well-defined features. Pair distribution function (PDF) analysis is a technique that enables researchers to obtain previously unobtainable information about the organization of atoms in poorly crystalline materials. PDF describes the probability of finding two atoms separated by a certain distance in the material under investigation. It can be applied to many types of amorphous and nanocrystalline materials: silicon carbide, titanium dioxide and vitreous silica, for example.

The analysis requires short X-ray wavelengths to obtain useful resolution. This has traditionally required the high-quality X-ray beams from synchrotron sources. Now, PANalytical's Empyrean Ag-anode Tube can be utilized for total scattering experiments. Using the high-energy characteristic radiation from this tube, PDF analysis can be performed in-house on an Empyrean-equipped XRPD system. Results obtained with this system compare well with synchrotron data. This lab setup offers the ideal screening tool and allows good preparation for synchrotron beam time.

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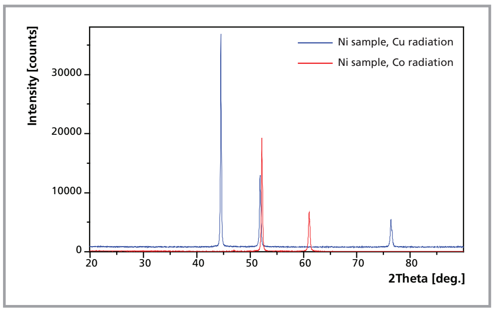
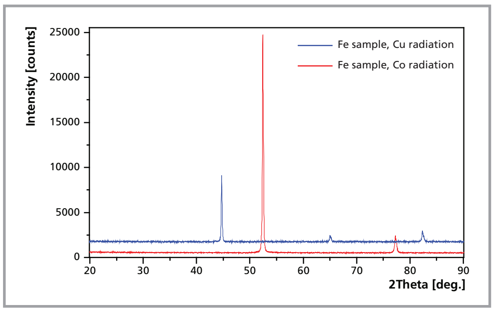
Cu or Co? A case study

The choice of an X-ray tube can have a pronounced influence on the peak intensity and the peak-to-background ratio in the diffractogram, especially when the main elements in the sample have absorption edges near the characteristic radiation.

Typically, a diffractogram recorded with Co radiation will show a lower intensity of the diffraction peaks than a diffractogram recorded with Cu radiation, as illustrated with the pure Ni sample. With the pure Fe sample, however, with Co radiation one gains a factor of three over Cu.



Cu radiation gives stronger peaks for most of the materials

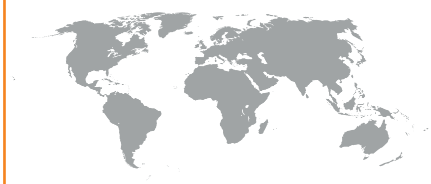


For Fe- (and Co-) bearing materials, however, Co radiation gives stronger peaks and a better peak-to-background ratio

The Empyrean Tube - specifications

Technical data			
Body construction	Metal body containing grounded anode with two beryllium windows		
Cathode isolation	Ceramic section supporting and isolating the cathode		
Anode materials	Cr, Mn, Fe, Co, Cu, Mo, Ag, others on request		
Window round	10 mm Ø		
Window oval	18 x 10 mm (l x w)		
Window thickness	300 µm		
Take-off angle	(no intensity loss over range)		
	Line (degrees)	0 - 12	
	Point (degrees)	0 - 13	
Focus types	Long fine focus	12 x 0.4 mm²	
	Broad focus	12 x 2.0 mm²	
Focal quality	To COCIR specification		
High voltage	On constant potential or self-rectified supply, -60 kV max		
Power rating	Depends on selected anode material and focus type		
Spectral purity	(typical, as % of Kα line)		
	Foreign lines on delivery	< 1%	
	Increase per 1000 hours	< 1%	
Radiation safety	< 1 µSv/h.cm² (in combination with an approved tube shield)		
Electrical safety	IEC 1010-1		
Environmental	Fully RoHS and WEEE compliant		
Cooling	All tubes are provided with a cleanable water sieve		
	Tap water	≥ 3.5	l/min
	Water pressure	≤ 0.8	MPa
	Pressure drop	0.25	MPa
	Inlet temperature	15 - 35	°C
Weight	2.5 kg		

Global and near



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