

spectrum

News from LOT-QuantumDesign Europe

Conductivity measurements with in situ AFM and SEM analysis



One of the standard measurement modes of an AFM has been the analysis of the electrical conductivity of a sample surface (conductive AFM or C-AFM). Until now, the limiting factor has been the larger radius of the specially coated, conductive cantilever

tips (20-30 nm) compared with normal, uncoated tips (< 10 nm). The result has been much poorer lateral resolution both of the topography and the conductivity signal. Our partner GETec is taking a new path by using self-sensing and conductive cantilevers which feature a different design. Fig. 1 shows a cantilever with platinum tip, applied with the FEBIP (focused electron beam induced processing) technique. Typically, these have a radius < 20 nm. In the image magnification the tip is seen as a little needle pointing upwards. During FEBIP, a nanogranular film, consisting of platinum nanoparticles embedded in a carbon matrix (Pt(C) film) is modified by using the electron beam of an SEM.

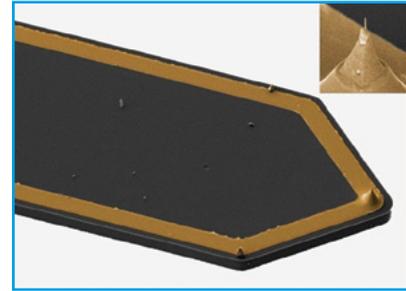


Fig. 1: Conductive self-sensing cantilever with small, conductive platinum tip (< 20 nm radius)

Under the beam radiation, carbon is oxidized by the oxygen contained in the water that remains on the surface and dissolved out of the matrix. This compresses the matrix and shortens the distance between the nanoparti-

>> Page 2

X-ray systems – composition and valence state analysis on trace level



Based on the patented tunable multi-energy and ultra-bright x-ray sources and highly efficient x-ray optics, our partner Sigray has developed several laboratory systems that are nearly as powerful as comparable synchrotron setups. This includes systems for x-ray fluorescence analysis (μ XRF), x-ray absorption spectroscopy (XAS), X-ray absorption near edge structure (XANES), and x-ray absorption fine structure (EXAFS).

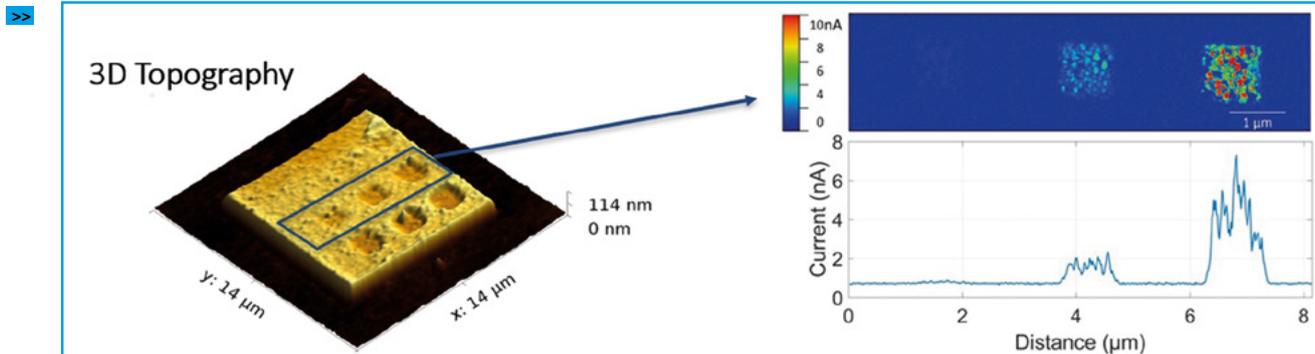
XAS, XANES and EXAFS can provide answers to important questions regarding the chemical state of the sample, like oxidation, bonding symmetry, bonding lengths and coordinative setup. The XAS system Quantum

Content

	Page
About us	5
Cryogenics	10, 11
Electron microscopy	3
Events	11
Imaging	7
Light & lasers	6, 8
Materials science	9, 11, 12
Spectroscopy	4

>> Page 2

Conductivity measurements with in situ AFM and SEM analysis



cles. Furthermore, the remaining carbon changes its structure towards a conductive, graphene-like morphology. These two processes (shortening of the distance between Pt particles and changes in the carbon structure) significantly increase the conductivity of the entire Pt(C) film. This increase can easily be monitored and controlled directly in the SEM by using the AFSEM. Contaminations are basically ruled out, as the entire process takes place in the SEM vacuum. In the end, the platinum tip (produced with

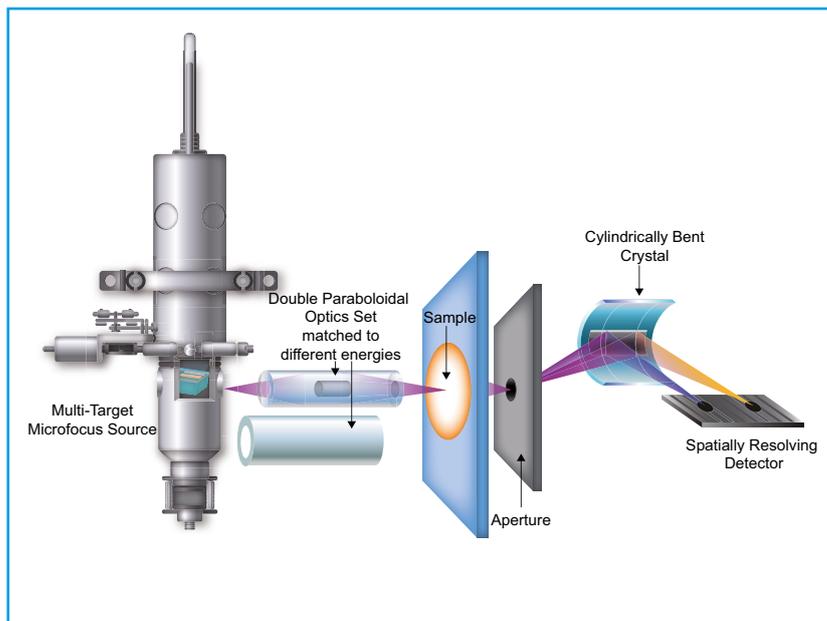
FEBIP) is connected with the current amplifier by golden conductors.

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X-ray systems – composition and valence state analysis on trace level

Leap provides a high sample throughput and sub-eV resolution, similar to that of a synchrotron. As such, the system is able to provide answers regarding the functional characterization of critical materials like catalysts, battery and fuel materials, and electronic components. As a non-destructive method, it allows analyses both in vacuum and under ambient conditions. It also enables in-situ experiments like the examination of the valence state of a battery material during charge and discharge cycles. Results gained from the Quantum Leap can be compared with the results gained from synchrotron measurements.



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Self-sensing cantilevers – Seeing and Feeling in the nanoworld

Our new supplier SCL-Sensor.Tech is market leader in the manufacturing of self-sensing cantilevers.

These cantilevers are equipped with a full piezo-resistive bridge that directly measures the cantilever signal electrically. This eliminates the space-consuming requirement for an optical readout. The free space above the cantilever now enables a variety of new applications in air and vacuum conditions. Application fields include atomic force microscopy (AFM), Torque magnetometry, force measurements and gas sensing. The cantilever is bonded onto a 5.9 mm x 4.8 mm small PCB incl. connector to facilitate handling and exchange.

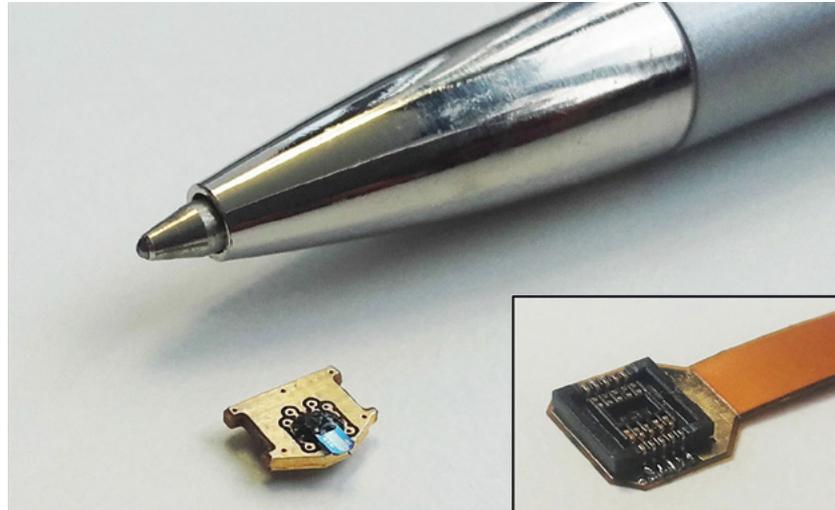


Fig. 1: REM image of a piezo-resistive self-sensing cantilever (Si-tip, $l = 300 \mu\text{m}$), proportions and connector

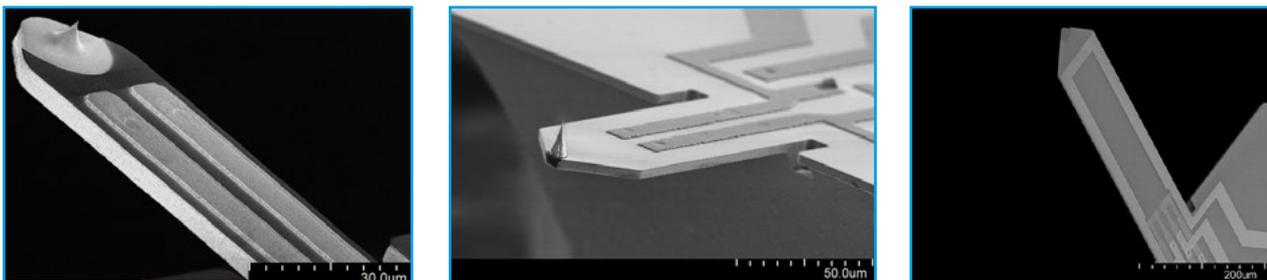


Fig. 2: Different cantilever types (Si tip, $l = 70 \mu\text{m}$, diamond dip, $l = 100 \mu\text{m}$ and tipless, $l = 400 \mu\text{m}$)

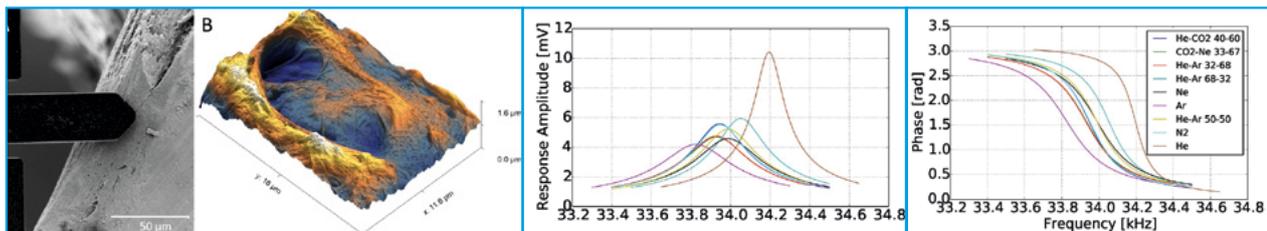


Fig. 3, left: AFSEM measurement on bone in a SEM, right: Gas analysis with SCL cantilever (Ref. <https://www.ncbi.nlm.nih.gov/pmc/articles/pmc4610587>)

The cantilevers are available in various geometries, spring constants and resonant frequencies. When larger quantities are ordered, they can also be customized.

We also offer various tips for a wide range of applications: Silicon, ultra-hard diamond (SCD) or tipless.

Self-sensing cantilevers are suited for a great number of applications. They are the basis for a brand-new scan-

ning electron microscope, the AFSEM, which has been developed for the integration in commercial electron microscopes by our partner GETec Microscopy (www.getec-afm.com) (see fig. 3, left).

Correlative analysis integrates the advantages of an SEM (high depth of focus, high xy-resolution) with the advantages of an AFM (high z-resolution) and combines all data in a high-

resolution 3D image. Another possible application is gas sensing. Cantilevers can be used to distinguish and analyze various gas mixes (see fig. 3, right). Cantilevers are also used in Torque magnetometry to characterize magnetic samples at ultra-low temperatures.

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Radial velocity method in astronomy – Echelle spectroscopy

In the astronomical field, spectroscopy methods play an important role next to imaging methods. In this article, we present a well-established method to determine radial velocity: Echelle spectroscopy.

Separating the light of a star into its optical spectrum also provides information on its velocity relative to the observer. Due to the Doppler effect, the redshift for example indicates a movement away from us, while a blueshift hints at a movement towards earth. The spectra of a star during different times (of the year) disclose information on the periodic changes of the spectrum and as such can prove the existence of binary star systems. They are mostly not resolvable with direct imaging methods. Furthermore, it has been possible to prove the existence of extrasolar planets with help of this indirect method. This is particularly challenging as the typical pla-

net-to-star mass ratio is large and the mutual center of gravity lies almost in the center of the star. One of the lightest exoplanets is Gliese 581e with 1.7 times the mass of the earth (fig. 1). It was detected with this method using the ‘High Accuracy Radial velocity Planet Searcher’ (HARPS), an Echelle spectrograph at the 3.6 m telescope of the European Southern Observatory (ESO). The Gliese 581 system is about 20.4 light years away. Measuring changes in the radial velocity of a star puts high demands on the spectral resolution and light sensitivity. The spectral resolution is mainly determined by the optical setup of the Echelle spectrometer. Even though Echelle spectrometers are commercially available (for example Andor Mechelle 5000), specially adapted instruments have been developed for astronomical purposes. They feature a combination of prism and diffrac-

tion grating. One axis of the displayed spectrum corresponds to the diffraction orders of the grating, while along the other axis the light is split in each order into its wavelengths (fig. 2). The 2D separation of the spectrum allows the use of square CCDs or other 2 dimensional sensors. The great advantage of an Echelle spectrometer is the simultaneous measurement of a large wavelength range at highest resolution. The spectral resolution directly scales with the focal length. A large sensor diagonal with small pixels therefore enables a correspondingly high resolution. The instrument designed for the discovery of the exoplanet Gliese 581e reaches a spectral resolution of 120.000 and can thus detect velocity variations of the star of below 1 m/s.

Back-illuminated CCD sensors are available with more than 16 megapixels and diagonals of more than

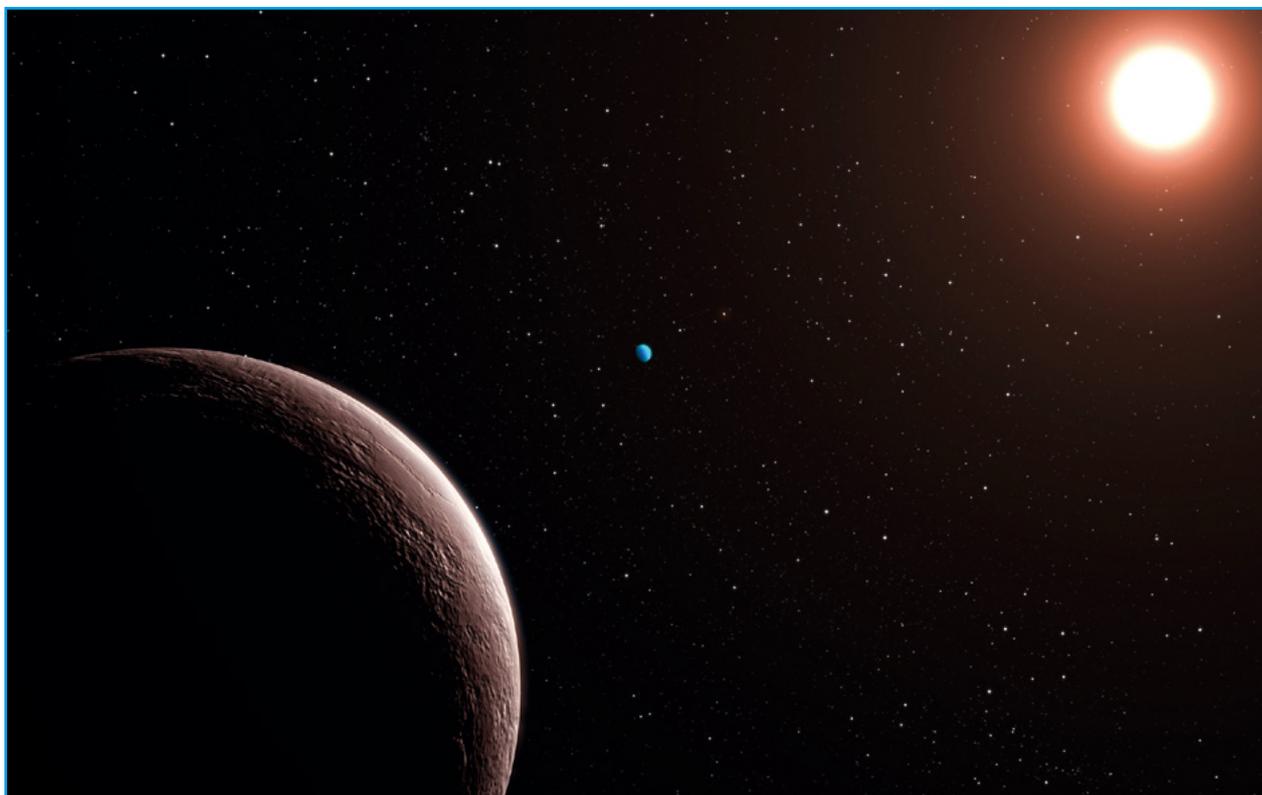


Fig. 1: Extrasolar planet system around the star Gliese 581e (artistic depiction). Credit:ESO/L. Calçada

Radial velocity method in astronomy – Echelle spectroscopy

87 mm. The low read noise of up to 2.1 e-/pixel in combination with a pi-



Fig. 2: Echelle spectrum (color display)

xel capacity of 350.000 e- justifies an 18 bit digitization. Andor Technology offer this type of CCD sensors in the iKon-XL series.

Newly developed sCMOS sensor like the ones used in the Andor MARANA cameras, are well suited for Echelle spectroscopy.

They feature:

- 2048 x 2048 x 11 μm pixel size
- Very high QE of over 95 %
- Large sensor diagonal of 32 mm
- Thermo-electric cooling to -25 °C (air) and -45 °C (water) with very little dark current

- Outstanding linearity better 99.7 %
- Extremely low read noise of only 1.6 electrons
- Pixel capacity of 85.000 electrons/pixels

We provide detailed information, technical data sheets and applications reports on research with sCMOS cameras of the NEO, ZYLA and MARANA series from Andor Technology on our website:

<https://lot-qd.com/scmos/>

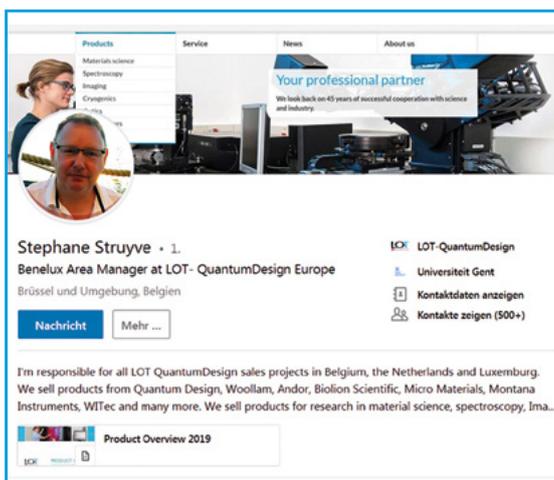
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Introducing: Stephane Struyve – Regional Sales Manager for the Benelux

After having been your LOT-QuantumDesign sales contact in the BeNeLux for over eight years now, it is high time I introduce myself to you.

Today I live in Grimbergen, close to Brussels. In my time off, I enjoy spending time with my three daughters and working on my home. You might know

Apart from that – I enjoy being your sales contact for the wide range of instrumentation we offer.



I received a master's degree in applied physics from the University of Ghent in 1992. Since then, I have been working as independent civil engineer and for various companies, the latest (but not at all least!) being LOT-QuantumDesign.

Grimbergen for its beer, but it's also known as "the pearl of Brabant"; I also truly enjoy my home town with its Abbey and the Gregorian Abbey Choir who accompanies important services and gives concerts several times a year.

Let me know your application needs and together we will find a solution!

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Arc lamps or halogen lamps? Selection of the optimum lamp type

Selection of the optimum lamp type and lamp power for your application depends on a variety of factors.

Spectral distribution

- Which wavelength do you need?
- Which wavelengths scatter the light or heat the sample, which is undesirable?

One should ideally choose a lamp with high power in the desired spectral range at and low power at wavelengths that may be light-diffusing or tend to cause other problems. Arc lamps are primarily utilized as radiation sources for light in the ultraviolet to visible range. Mercury arc lamps emit particularly strong lines in the UV range. Halogen lamps, on the other hand, are a good choice for applications in the visible to the near infrared range. The catalog contains typical spectra of these individual lamp types.

the intensity of irradiation on the receiving surface, but it cannot affect the available radiance – which, after all, is a characteristic parameter of the respective radiation source. Images of the radiation source can never exceed the radiance emitted by the source itself.

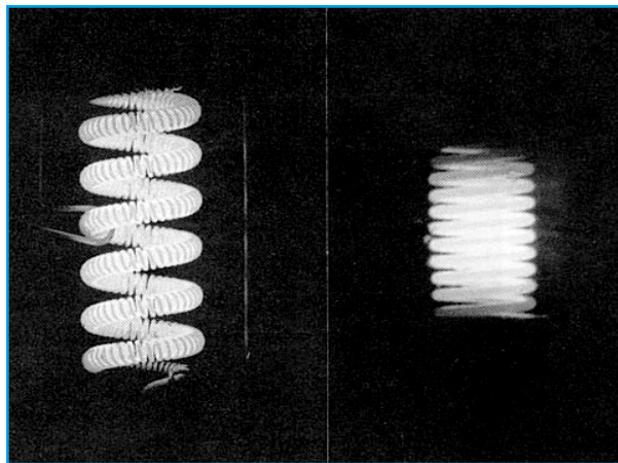
The radial intensity per unit area is an important factor wherever light is to pass through certain optical components. Small radiation sources are easily collimated and therefore easy to focus. Whenever a fiber-optical system, a monochromator slit, or a pin diaphragm, for instance, is to be illuminated - in which case the surface area to be illuminated is of the same size or smaller than the radiation source itself – then the spectral radiance at the required wavelength is of importance. At a first approximation, the value of the irradiance or the luminous flux,

If larger surface areas (several cm²) are to be illuminated, then lamps with a higher output power (luminous flux) will yield better results. The radiance of a 75 W Xe arc lamp approximately equals that of a 1000 W Xe arc lamp. Due to the larger-sized arc, however, a 1000 W light source will illuminate a surface that is 30 times as large as that covered by the smaller lamp. Irradiance curves provide a good basis for lamp selection in applications where collimation is irrelevant and only the output power of the lamp is crucial.

Shape and size of the radiation source

- What about shape and size of the object to be exposed?

The existing optical system as well as the shape and size of the radiation source determine how much light ultimately reaches the object to be illumi-



Radiance

- How bright should the image produced by the radiation source be?
- How large is the surface to be exposed?

Usually there are one or two lenses between the radiation source and the surface to be exposed (monochromator slit, optical cable, detector, sample). These lenses resp. any kind of imaging can only be used to change

divided by the surface area of the light source, affords a good reference value. The radiance of a 75 W Xe lamp, for instance, is approx. 2.7 times as high as that of a 150 W Xe lamp, since the surface area of the arc in this case is approx. 8.8 times smaller.

Total output power

- How large is the surface area to be illuminated?

An elongated arc, for instance, is a better instrument to illuminate a monochromator slit. In the case of reflecting optical systems, such as housings with an elliptical reflector, the shape and size of the image are largely determined by the optics. The chapter on ‘condensor optics or elliptical reflector’ discusses this topic.

Constant Intensity

- In how far does your application re-

Arc lamps or halogen lamps? Selection of the optimum lamp type

Arc lamps Properties

- high radiance in the UV and the visible range. Mercury lamps exhibit spectral lines with very high radiance in the UV range.
- high UV output power
- small-sized electric arc
- Xenon lamps exhibit a spectral distribution that is very similar to sunlight.

Advantages

- highest irradiance on small illumination surfaces
- simulation of daylight
- intense collimated beams due to small arcs with high radiance
- excellently suitable light sources for UV photochemistry

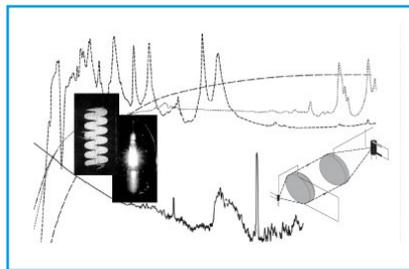


Halogen lamps Properties

- Emission between 350 nm and 2700 nm
- good stability
- high output power in the visible range
- useful as photometric or radiometric sources
- continuous emitter; i.e., relative minor spectral intensity changes

Advantages

- user-friendliness
- simpler detection during scanning
- less costly than arc lamps



quire stability with regard to space and time?

In many cases, radiation stability in space and time is so important that a double-beam approach must be employed. Halogen lamps emit a more constant intensity than arc lamps. A light regulator may further contribute to a more constant light intensity, but a good system design is equally important. Thus, free-convection flows inside arc lamps cause fluctuation around the marginal areas of the arc. A carefully designed system will eliminate these unstable zones.

Further information on ‘selecting the optimum light source’ is found on the internet under

<https://lot-qd.com/lightsources/>



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Going the next step – Microbolometer camera Gobi+

We are pleased to announce that Xenics have enhanced the well-established uncooled longwave IR camera Gobi640 and now also offer Gobi+ (plus).

What is in it for you?

The uncooled detector still features 640 x 480 pixels. However, with the new version, the temperature resolution (NedT) has been reduced to under 50 mK.

Frame rates have been increased to 60 frames per second. The so-called “windowing mode” allows even better frame rates. Still, microbolometer detectors in general are rather slow, which means that really fast rates will never be reached.

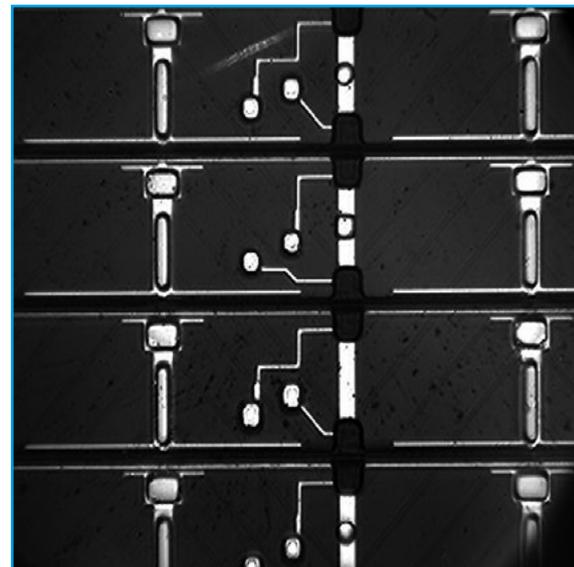
The 16-bit deep data can be transferred either via GigE or CameraLink interface. However, We recommend using GigE.

The camera can be triggered and send trigger out signals.

Like its predecessor, Gobi+ includes the comprising software package Xeneth for image acquisition and analysis.

All in all, Gobi+ is a cost-effective, compact camera with great R & D potential.

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UV process monitoring and controlling, specifications of light measurement systems in UV curing – Part 1



When monitoring a production process with UV curing it is important to validate it to ensure it works with the specified requirements. Too much of a deviation can have a serious impact. To ensure the desired quality standard and minimize downtime, many companies routinely check the UV light power of their process line.

Using a radiometer often requires an accreditation for products and components which are used in medical, automotive and aerospace. In this case you should choose an UV radiometer which is calibrated in an accredited laboratory based on ISO standard 17025. Our calibrated radiometers are NIST traceable!

Curing processes on conveyor belts or in UV chambers require that the measurement device itself is exposed to the UV light (compared with a distant sensor). A measurement sys-

tem is needed that can resist both the UV light intensity and the temperature increase during UV-exposure. A measurement system with plastic housing and exposed display would not stay intact for a longer period of time under UV exposure.

Furthermore, there are many different designs from both photo initiators and light sources, which complicates the development of a system for universal use across platforms.

So how do you choose the right light measurement system for UV curing if parameters can vary so much? First of all, you must find decide which functions are essential for correct curing.

Common names for light measurement systems are UV meter, radiometer and optometer. It is a tool that can measure the absolute (calibrated) power of light within a certain range of the electromagnetic spectrum.

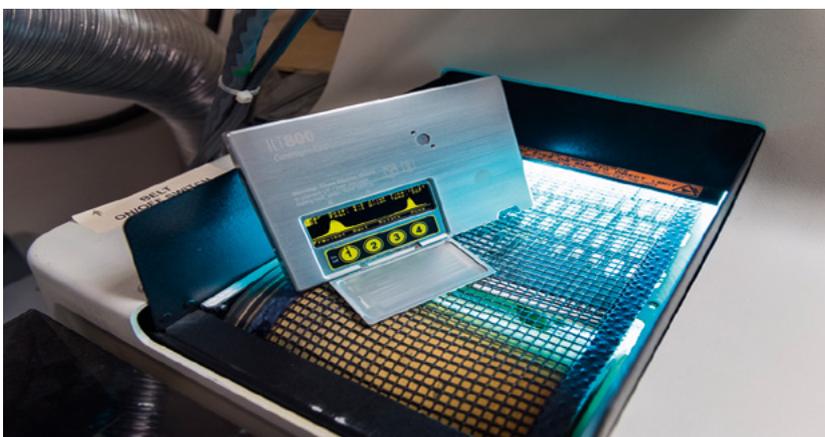
Adapting the UV spectrum to a radiometer is challenging as the number of sensor and filter materials which have low absorption and stable physical properties and can withstand long-lasting UV loads is limited. The ideal UV sensor has a flat and even reaction across the specific band that causes the curing and blocks all other wavelengths. Most UV sensors only have low and varying sensitivities in the UV range. This is why selecting the right wavelength range of a measurement system can in itself be challenging.

The first step is to determine which wavelength range is emitted by the lamp/light source. Then it is highly important to know which of these emitted wavelengths actually cause the curing.

The reaction of the photo initiators to UV light is directly related to their absorption and is highly wavelength selective. For effective curing, the wavelength of the light source must comply with the absorption peak of the photo initiator. This can range from 200 and 450 nm, depending on the application. Shorter wavelengths are selected for thinner surface applications, to allow for faster throughput. Longer wavelengths are absorbed into the substrate and provide deeper penetration into thicker materials.

Like the photo initiator, the UV sensor of the measurement system has a set sensitivity. It is important to select a system which corresponds to both the spectral power of the UV source and the reaction of the photo initiator to UV light.

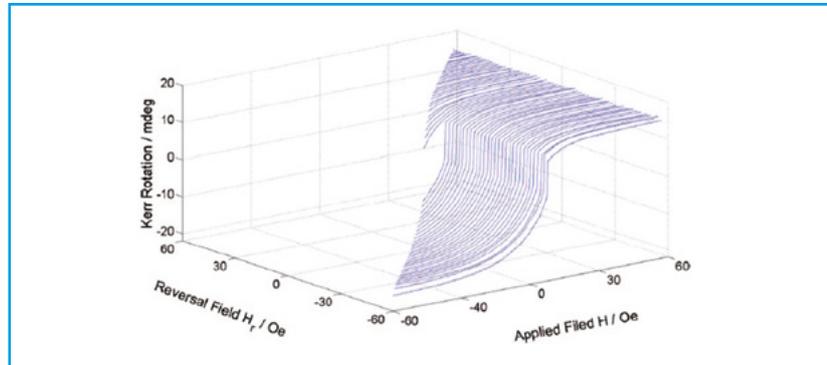
So, if for example you are curing a photo initiator with a specification for UVA (315 - 390 nm), the measurement system should be sensitive in this area. The UV light measurement system is constantly exposed to the UV light and must therefore be resistive.



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FORC measurements in just minutes with NanoMOKE3

First-order reversal curve (FORC) measurements are a powerful method to characterize magnetic samples. During FORC measurements, all magnetic states of an investigated system are distinguished by coercive field strength and internal magnetic interactions. VSM or SQUID measurements, which are normally used to determine FORC, are complex and time-consuming. Furthermore, resolution and the number of measurements are limited by time restrictions. Faster methods like MOKE (Magneto Optic Kerr Effect) can lead to stability problems due to drift effects and only relative magnetization values. The work group around Prof. Goering from the Max-Planck-Institute for Intelligent Systems in Stuttgart has developed an approach that allows FORC measurements with MOKE. It uses a special field shape that provides two anchor points for every loop.



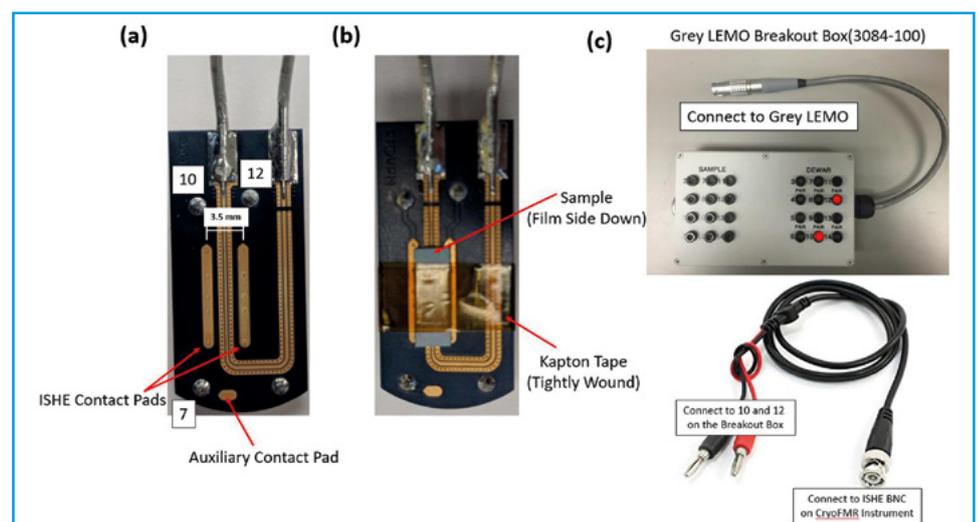
This leads to high field resolution while the measurement only takes a few minutes [1]. This method has been developed with help of the NanoMOKE3 system from Durham Magneto Optics (DMO). DMO was founded in 2002 by Prof. Russell Cowburn, one of the leading scientists in the spintronic and nanomagnetism field.

[1] Eberhard Goering et. al., Max Planck Institute for Intelligent Systems, Heisenbergstraße 3, 70569 Stuttgart, Germany, Review of Scientific Instruments 85, 023901 (2014), <http://dx.doi.org/10.1063/1.4865135>

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Measurement of the inverse Spin-Hall-Effect (ISHE) with the PPMS/DynaCool/VersaLab

In the last Spectrum editions, we have described the Inverse-Spin-Hall-Effect (ISHE) in great detail. It is a transversal DC voltage that appears in ferro/non-magnetic double layers with spin-orbit-interactions. It is measured with a special coplanar waveguide (CPW) which has additional electrical contacts that allow the measurement of the generated voltage. Quantum Design have published a comprehensive application note on the setup and measurement with an ISHE-CPWs for low temperatures. The measurements were taken in a PPMS/DynaCool/VersaLab.



<https://www.qdusa.com/sitedocs/appNotes/general/1087-203.pdf>

Searching for hydrogen impurities in liquid helium

Alex Chuquitarqui from the group around Prof. Conrado Rillo of the University of Zaragoza is searching for hydrogen traces in liquid helium.

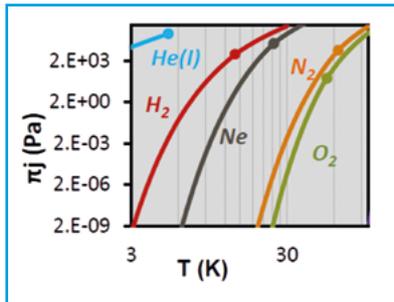


Fig. 1: Steam pressure of He(l), H₂, Ne, N₂ and O₂ as function of temperature

Hydrogen vapor pressure as a function of temperature proves that these exist. At 4.2 K the steam pressure of hydrogen is still high enough to contaminate liquid helium substantially (see fig. 1). There are various reasons for contaminations: Traces of hydrogen can already be present in the helium source, oil degradation in pumps and compressors during recovery can lead to hydrogen formation just like the outgassing of metallic tubes and tanks and the diffusion of plastic tubes and gas balloons. This means that almost every dewar with liquid helium also contains a considerable amount of hydrogen.

Generally, dissolved hydrogen does not cause any problems. When liquid helium is pumped through capillaries, however, it cools off locally and the dissolved hydrogen freezes out as solid and acts as sealing. [1]. A capillary is used for temperature control for example in NMR and LT-STM devices and in the PPMS and MPMS3 systems from Quantum Design. When blocked, the base temperature of the system cannot be reached any more. In this case, the system must be warmed to release the blocked capillary.

Hydrogen impurities in liquid helium are easy to detect with a special sensor developed by Prof. Rillo from the University of Zaragoza. It consists of a test capillary that is connected to a vacuum pump (see fig. 2).

This project is supported by the Spanish Ministry of Science, Innovation and Universities.

The project number is MAT2015-64083-R. Alex Chuquitarqui can be reached at the following e-mail address: achuquitarqui@unizar.es

[1] M. Gabal et al., Hydrogen-Free Liquid-Helium Recovery Plants: The Solution for Low-Temperature Flow Impedance Blocking, *Phys. Rev. Applied* 6, 024017 (2016).

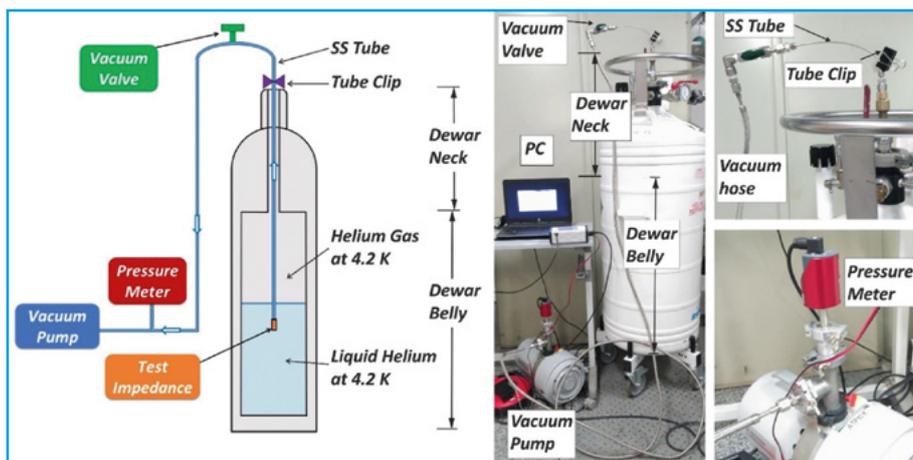
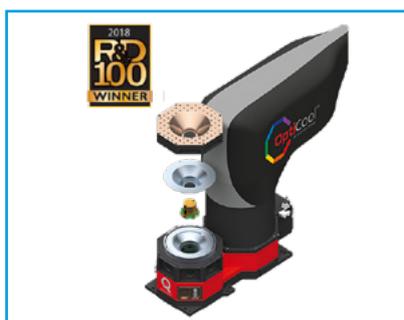


Fig. 2: Schematic of the hydrogen sensor (left) and real setup (right).

Alex Chuquitarqui is looking for cooperation partner who are willing to test the H₂-detector. If you are interested, we will send you one of the detectors for free so you can detect the hydrogen content in your liquid helium and quantify it until the capillary is blocked. This way you can avoid using hydrogen-contaminated liquid helium for delicate applications.

Please contact us
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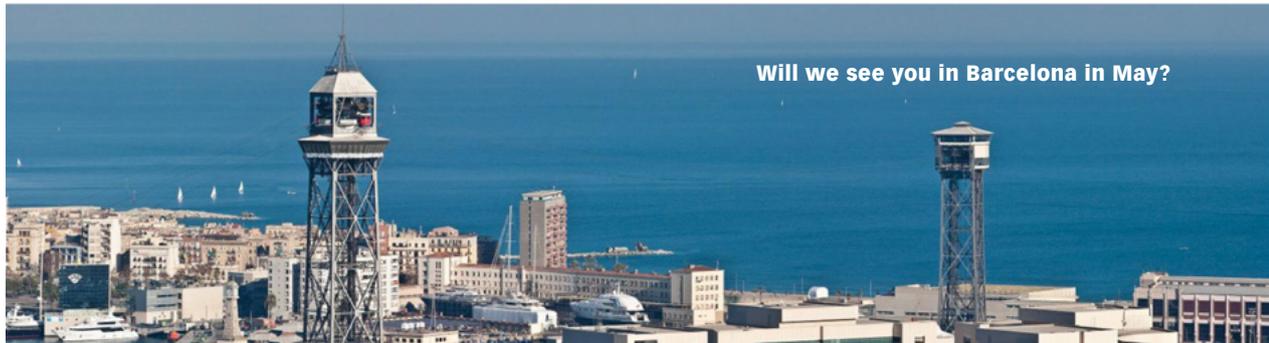
Quantum Design OptiCool wins prestigious "R&D 100" Award!



OptiCool, Quantum Design's optical cryostat with closed helium cooling cycle and 7 Tesla superconductive magnet, has just been awarded the R&D100 Award as one of the top 100 technologically most exception-

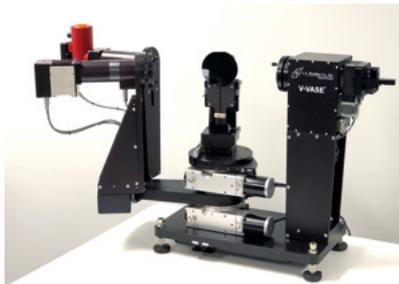
nal products in the field Analytic/Test. We congratulate the OptiCool development team and thank all Quantum Design staff members that have contributed to pushing analytical research forward!

International Conference of Spectroscopic Ellipsometry in Barcelona directly at the sea



The next International Conference of Spectroscopic Ellipsometry – ICSE – will take place in Barcelona from 26 - 31 May 2019.

Our ellipsometry experts will be there together with the J. A. Woollam Co. team. We look forward to interesting discussions and fascinating presentations. Last but not least, we will present our brand-new spectral ellipsometer.



Come see for yourselves!

New flash: VASE as base system now with 250 - 2500 nm range!

Since 2019, the VASE base system is available with the NIR detector to 2500 nm. And the best thing about it is: you pay the old base-system price! Note that the system can be extended to 193 nm with the DUV option and to 3200 nm (4000 nm resp.) with two different XNIR options.

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2018: ATL helium liquefier workshop in Zaragoza, Spain

With 15 participants from all over Spain, last year's Advanced Technology Liquefier (ATL) user workshop was a great success! We discussed topics like the technology behind the helium liquefiers ATL160 and ATL160+ and disclosed why higher liquefaction rates are achieved with higher helium gas pressures. Furthermore, Prof. Rillo, our host from the University of Zaragoza, gave us insights into the state of his research and offered a glimpse of the further development of

helium liquefiers and helium gas purifiers. For now, all we know is that soon higher liquefaction rates will be possible with the same cold head. In the second part of our workshop, our service engineer Alexander Isanin presented details on service and maintenance. Our ATL liquefiers are easy-to-use and can be maintained without the help of a service technician. Last but not least, we demonstrated the exchange of a cold head on one of the many liquefiers in Prof. Rillo's lab.



Workshop participants in Prof. Conrado Rillo's lab at the University of Zaragoza

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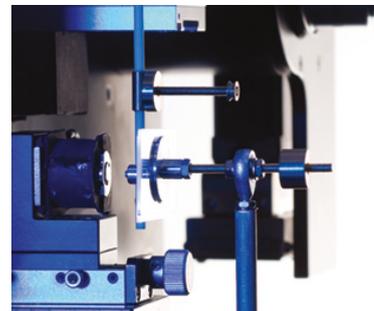
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For sale: Nanoindenter demo system

We offer our Nanotest Vantage demo system. The system is equipped with a passive vibration isolation table and nano load head and comes complete with indentation and scratch software and indenting tips. More options are available upon request and can be offered together with the demo system. We have been using the system in our demo laboratory in Darmstadt since 2012.

Please contact me, if you are interested – We are ready to sell at highly attractive conditions.



Dr. Tobias Adler
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Introduction seminar in Poland: Woollam ellipsometry – 16.05.2019

This seminar offers a short introduction and an overview of the current status of application and research in ellipsometry.



We will demonstrate the power of the Woollam spectroscopic ellipsometers by undertaking various sample measurements.

Date: 16.05.2019
Location: Instytut Materiałów Inżynierskich i Biomedycznych Politechnika I ska ul. Konarskiego 18a Gliwice

Overview

In the last few years the areas where spectroscopic ellipsometry is used are permanently growing due to the increasing need to characterise multilayer and multicomponent systems. This technique allows very sensitive measurement of film thickness, optical constants, composition, surface and interface roughness and many more. Increasingly applications close to production are developing alongside its traditional use for research.

This requirement has been addressed by the development of several in-situ solutions for process monitoring and control. During the seminar we will present basic J.A. Woollam system; Alpha-SE and showcase latest developments to address the requirements of photovoltaic applications.

System will be available to demonstrate sample measurements.

You can register on our webpage:

https://lot-qd.com/introduction_ellipsometry_pl/

Editor: Joachim Weiss, PhD
Please note that not all products are available in every country.