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NEWSLETTER FOR MATERIALS SCIENCE & TECHNOLOGY

# reSOLUTION

## **Exclusive Aesthetics of Nature**

Inclusions in Gemstones

## **Steel – It All Depends on What's Really Inside**

New European Standard in Steel Industry

## **Fast Characterisation of Solar Cells**

DCM 3D Dual-Core 3D Measuring Microscope



## Pure steel, clean components and precious stones

Dear Reader,

Purity, cleanness – we all know the meaning of these words in our ordinary everyday life. In an industrial or technical environment, however, they are often a science in themselves. Terms like “technical cleanness”, “residual dirt analysis” or “purity testing” make us realise that purity is not an absolute term but depends on what is being examined and the methods used. In this issue of reSOLUTION for Materials Science & Technology we report on a wide spectrum of applications in microscopy and image analysis that visualise, measure and document “purity”. Read about steel and how it is meticulously checked for undesired inclusions, or about precision components for the automobile industry that have to be checked for tiny dirt particles which may stick to them and paralyse the whole vehicle as a system.

On the other hand, there are areas where “defects” are even beautiful and can increase the value of an object. Besides the generally familiar and coveted insect inclusions in amber, gemstones often have other inclusions that make them unique, inclusions whose beauty can only be fully appreciated under a microscope.

This reSOLUTION also features our Leica Optic Centre, which not only designs and produces objectives and optical components for Leica products, but is also extremely successful as an independent service provider for individual high-end optic solutions.

We hope you like our choice of topics. If you have requests or suggestions, we would like to hear from you.

Have fun reading!

Anja Schué  
Corporate Communications

Danilo Parlatano  
European Marketing Manager Industry

### MINERALOGY

**Exclusive Aesthetics of Nature** 03  
Inclusions in Gemstones

### QUALITY ASSURANCE

**Clean Parts – More Reliable and Longer Lifetime** 07  
Particle Measurement with Leica QClean

**Steel – It All Depends on What's Really Inside** 10  
New European Standard in Steel Industry

### TECHNOLOGY

**Fast Characterisation of Solar Cells** 14  
DCM 3D Dual-Core 3D Measuring Microscope

### RESEARCH & DEVELOPMENT

**The Leica Optic Centre** 17  
Competence for Individual Optic Solutions in High-end Quality

**YOUR OPINION IS VALUABLE!** 18

### LEICA NEWS

**Strategic Acquisitions and Organic Growth** 19  
Leica Microsystems Reports Record Sales for 2008

**EVENTS** 20

**IMPRINT** 20

## Inclusions in Gemstones

# Exclusive Aesthetics of Nature

Michael Hügi, M.Sc., Mineralogist and Gemmologist FGA, Swiss Gemmological Society

Gemstones have fascinated people for thousands of years. Rulers and kings used to demonstrate their power and wealth with jewel-studded insignia. Although fine jewellery is still a status symbol of the rich, we now tend to treasure these wonders of nature more for our own pleasure in beauty and harmony. The place where aesthetics of the mineral world and science meet is the domain of gemmologists. In modern gemmological laboratories, microscopic examination of the interior and surface characteristics of a gemstone is still the mainstay for assessing quality criteria.

Two of the most important tasks of the gemmologist are determining the genuineness and the quality of a gemstone. He identifies an unknown stone by measuring its optical and physical constants; he determines the authenticity and origin of the stone and its possible treatments to improve the optical appearance. The inner characteristics of a stone reflect the chemical and physical conditions during its growth in its natural surroundings – or, in the case of synthetic materials, in the laboratory. Such characteristics may be colour zones or natural intergrowth of two or more individual crystals according to crystallographic laws, also known as twinning. Often, foreign substances are included in gemstone minerals. Such inclusions are differentiated according to type:

- Protogenetic inclusions: Minerals already present before the host crystal formed and enclosed these minerals.
- Syngenetic inclusions: These were formed at the same time as the host crystal, e.g. fractures that have been healed. A lot of small cavities were formed in the process, so-called negative crystals, which contain remains of the aqueous solution in which the crystal grew.
- Epigenetic inclusions: Inclusions originating after the formation of the host crystal. These are mostly natural substances within fissures or exsolution products in the host crystal.

### Microscope reveals hidden beauty

It is not necessarily true that a good gemstone has to be a hundred per cent clean and that inclusions are “defects” that diminish its value. In the case of faceted diamonds, the clarity of the stone is indeed a key criterion judged by a standardised nomenclature. For all other gemstones and ornamental stones, however, inclusions do not reduce their value provided they do not impair the stone’s appearance or stability. In fact, they make the stone unique and accentuate its exclusiveness – they are, as it were, nature’s signature. Apart from their scientific significance, the unique aesthetics of inclusions can often only be seen through a microscope. In view of this hidden inner beauty, the term “defect” assumes a positive rather than a negative meaning.

The microscopic examination of gemstones places great demands not only on the gemmologist but also on the instrument. As it is mostly stones set in jewellery that are examined, possibilities of observing inner characteristics are often limited. In some cases, immersion in liquid with light refraction similar to that of the stone can help. Extreme differences in contrast against a dark background, for instance due to reflecting facets or inclusions with metallic lustre, are also a considerable challenge for the illumination.

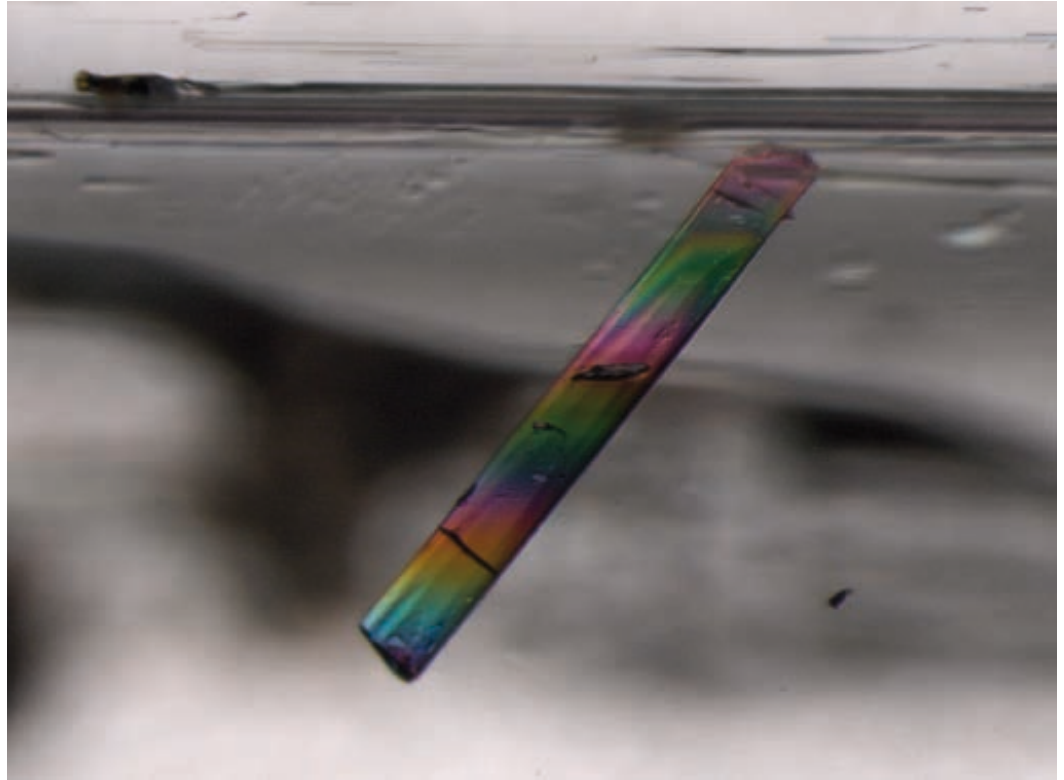
As a rule, stereomicroscopes with the following illumination techniques are used for gemmological examinations:

Fig. 1: Inclusions probably of melanterite (hydrous Fe sulphate) in rock crystal (quartz), found at Minas Gerais, Brazil. It is a pseudomorph after the iron sulphide marcasite, which is also found as inclusions in quartz in the same deposit. Transmitted light, crossed polarisers.





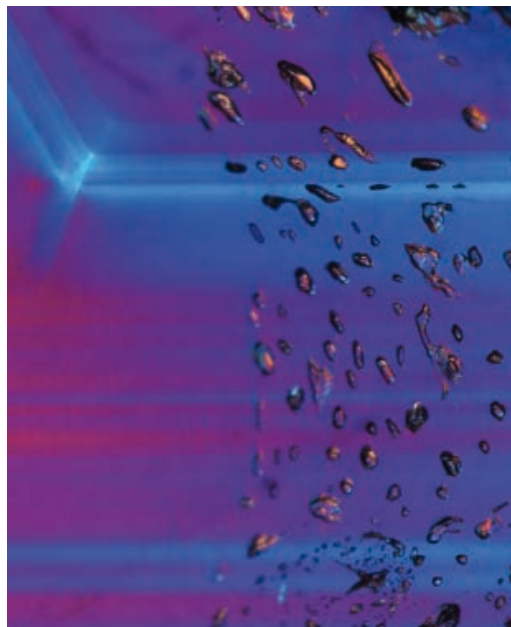
Fig. 2: Inclusion of a tourmaline crystal in aquamarine (variety of beryl), found in Northern Pakistan. The interference colours of the tourmaline needles are due to the polarised light. Width of image: approx. 1.5 mm, transmitted light, singly polarised light.



- Diffuse brightfield: This illumination enables observation of low-contrast growth structures, colour zoning and fluid inclusions. If crossed polarisers are used, it is possible to identify birefringent mineral inclusions or lamellar twin plains.
- Darkfield: Darkfield illumination shows up extremely fine structures such as needle-shaped or hairline inclusions that are not visible in bright-field.

- Glass-fibre optical waveguides: They enable a targeted darkfield illumination, or are used with incident light for the examination of surface structures.

Fig. 3: Fluid inclusions and growth zoning in aquamarine (variety of beryl), found at Mina Padre Paraiso, Minas Gerais, Brazil. During the growth of a crystal in an aqueous solution, stress can cause the formation of fractures that heal as the crystal continues to grow. In the former fissure, remains of the original aqueous solution are sealed in cavities, the so-called negative crystals. These fluid inclusions are located in the plane of the former crack. Their contents separate in different phases as the stone cools down after crystal growth. In the illustrated sample, all inclusions contain a gas bubble ( $\text{CO}_2$ ), liquid  $\text{CO}_2$  and a small amount of water. Width of image: approx. 3 mm, transmitted light, crossed polarisers, first-order red compensator.



For images like the ones illustrated in Figs. 1–6 the Swiss Gemmological Society uses a Leica MZ16 stereomicroscope with a Planapo objective 1.0x, which provides an adequate free working distance for examining even large objects. Illumination for brightfield or darkfield is supplied by a cold light source by Leica Microsystems. In addition, two glass-fibre waveguides with an external light source are used.

As many gemstones are optically anisotropic, i.e. birefringent materials, a polarisation filter (analyser) is generally used to eliminate image blurring due to birefringence.

## Digital photomicrography of inclusions

The documentation of inner characteristics of gemstones with the aid of photomicrography dates back to the 19<sup>th</sup> century. The German mineralogist Ferdinand Zirkel mentions this technique in 1873 in his book "Die mikroskopische Beschaffenheit der Mineralien und Gesteine". All the same, Zirkel regarded photomicrography with some scepticism. Compared with the technique of drawing, he was of the opinion that photography offered no possibility

# The Main Gemmological Examination Methods

As a general rule: Gemmological examination methods must not be destructive.

## Standard equipment

- Gemmological stereomicroscope
- Refractometer (determination of refraction index)
- Hydrostatic balance (determination of weight and specific gravity)
- Spectroscope (optical observation of absorptions in the spectrum of visible light)
- Polariscope: determination of optical anisotropy
- Dichroscope: determination of pleochroism (differences in colour depending on the vibration plane of the polarised light)
- UV lamp: observation of fluorescence

## Analysis techniques in modern gemmological laboratories

- Spectral photometer, UV-VIS and IR (exact measurement of absorptions in the UV to visible light range and in the infrared range)
- X-ray fluorescence (XRF): semi-quantitative analysis of trace elements
- Raman spectroscopy: analysis of molecular structures (e.g. determination of inclusions)
- Laser-ablated – inductively coupled plasma – mass spectrometry (LA-ICP-MS): highly sensitive trace element analysis
- Scanning electron microscopy (examination of submicroscopic surface structures)
- Laser induced breakdown spectroscopy (LIBS): further trace element analysis

of highlighting important parts of the image or omitting insignificant detail. Despite this supposed inflexibility, photomicrography became an indispensable instrument in gemmology as in other sciences. Gemmologists such as the Swiss professor Eduard Gübelin, one of the founders of the Swiss Gemmological Society, and the American J. Koivula played a major role in the further development of inclusion

photography with black-and-white and colour film. Their techniques are still used in today's age of digital photography. High-quality optics, careful work and a lot of patience are the preconditions for good photomicrographs, as the special characteristics of inclusion microscopy pose a considerable challenge. In particular, strong contrast, limited field depth as well as unnoticed specks of dust or scratches on the



Fig. 4: Rock crystal (quartz) and rutile needles enclosed in rock crystal (quartz), from Minas Gerais, Brazil. Because of the light metallic lustre of the rutile needles (titanium oxide,  $\text{TiO}_2$ ), the trade name of this quartz variety is "platinum quartz". The picture shows a first generation rock crystal inclusion that was enclosed by a second generation of quartz. Although the inclusion and the host material have the same refractive index, the enclosed quartz is easily recognisable due to a thin film of air at the interfaces. Width of image: approx. 6 mm, transmitted light, crossed polarisers, first-order red compensator.

Fig. 5: Fluid inclusions and inclusions of rutile needles and small siderite crystals (iron carbonate) in rock crystal (quartz), found at Minas Gerais, Brazil. The irregular cavities formed by growth contain remains of the aqueous solution in which the rock crystal grew about 500 million years ago. The bright interference colours are formed by the almost parallel viewing direction to the optical axis of the crystal in polarised light. Width of image: approx. 6 mm, transmitted light, crossed polarisers.



surface of the stone often create problems. As with microscopic analysis, different illumination techniques and, most importantly, their combination, are crucial for the results of photography.

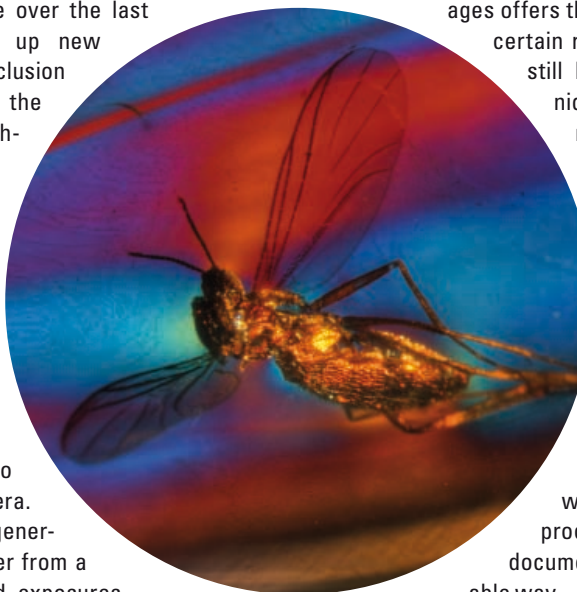
## Processing without falsifying

Fig. 6: Inclusion of a fly in Baltic amber. Amber is a fossil resin, and this sample originated about 35–40 million years ago in forests in what is now the Baltic region. Amber occasionally contains inclusions of insects, spiders, small vertebrates, etc., which stuck to the resin and were covered by subsequent resin layers. Thus they have been excellently preserved. After the tree died, the lumps of resin were washed into rivers and then into the sea, where they were embedded in sediment and fossilised. Although amber is an amorphous substance and in theory optically isotropic, the flow structures of the resin due to internal strain as well as the strain caused by the inclusions can be visualised in polarised light. The use of polarised light and the first-order red compensator lead to intensive colours in the otherwise golden amber. Combination of darkfield and incident light (glass-fibre), crossed polarisers, red-1 compensator, HDRI tone mapping.

Digital photomicrography, which has gained general acceptance over the last few years, opens up new possibilities for inclusion photography. Using the technique of High-Dynamic-Range Imaging (HDRI), photos can be produced of objects whose dynamic range of luminance between light and dark areas exceeds the limited luminance range of the photo sensor of the camera. HDRI photos are generated in the computer from a series of bracketed exposures, so that the full contrast range is stored in one 32-bit image. However, this image cannot be reproduced either on conventional monitors or with

printing techniques. To obtain a realistic image corresponding to the visual impression with distinct highlights and shadows, a second step, so-called tone mapping, is carried out by compressing the luminance range to produce an 8-bit image that can be reproduced with conventional media.

Computer-aided postprocessing of the digital images offers the opportunity to overcome certain restrictions imposed by the still limited photographic technique and to obtain a more realistic picture. However, this tempting opportunity must not lead to retouching or colour changes that deliberately falsify the information provided by the image. In this respect, Ferdinand Zirkel is still right today: The author should use his possibilities of influence – whether with a drawing pen or image processing software to help document reality in an understandable way.



**More information on the Swiss Gemmological Society: [www.gemmologie.ch](http://www.gemmologie.ch)**



## Particle Measurements with Leica QClean

# Clean Parts – More Reliable and Longer Lifetime

Kay Scheffler and Anja Schué, Leica Microsystems

In the automotive industry, the technical cleanliness of function-critical individual and system components has become an increasingly critical criterion for reliability and service life. This trend is also reflected in ISO/DIS 16232 (road vehicles – cleanliness of components of fluid circuits). Microscope systems with corresponding analytical software enable efficient and reliable residual dirt analysis of injectors, pumps, control units and other micromechanical components.

Many vehicle components cannot be checked after they are inserted or installed. Contamination from clinging particles can cause a total failure. Efficient cleanliness measurement for quality assurance of the manufacturing processes cannot be carried out directly on the complex part. Therefore, the degree of contamination is measured indirectly by means of microscopic residual dirt analysis. This is done in three steps:

- Rinsing/washing the parts
- Filtering the rinse liquid
- Optically analysing the filter

Fig. 1: The complete Leica QClean system with fully automated microscope, digital camera and software is designed for easy and reliable operation.

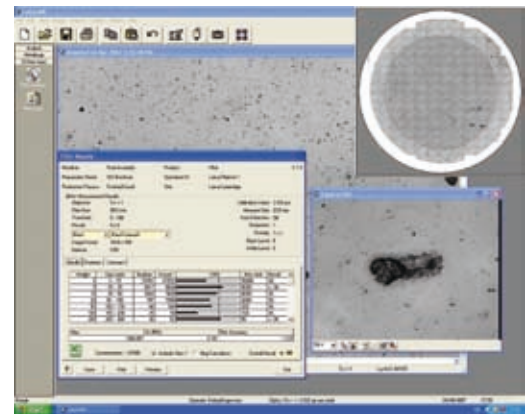
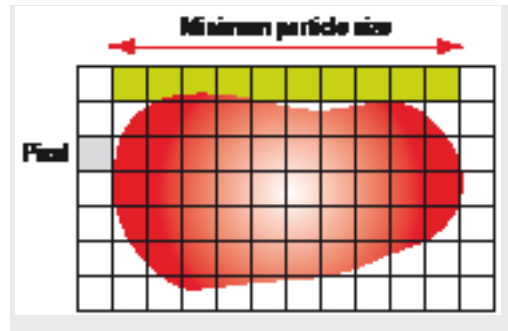


Fig. 2: The results of particle measurement are displayed automatically and can be stored in the integrated archiving system.

In close cooperation with suppliers to the automotive industry, Leica Microsystems has developed the complete Leica QClean system for residual dirt analysis. It consists of an automated microscope (Leica DM4000 M – DM6000 M) with scanning stage and digital camera and a high-performance computer with corresponding analytical software that measures the size and number of dirt particles on a filter (Fig. 1). Leica QClean analyses particulate filters according to the specifications of VDA 19 and ISO/DIS 16232. The versatile software can also be adapted quickly and easily to other industry standards or internal factory quality standards. The tester is guided through the measurement routine until the automatic particle measurement is started. The results are displayed automatically (Fig. 2) and can be stored in the integrated archiving system.

Fig. 3: A light microscope equipped with a 5x objective and NA = 0.15 attains a resolution of 2.2  $\mu\text{m}$ . This means that a calibration value of 1.48  $\mu\text{m}/\text{pixel}$  is sufficient to measure particles up from 15 to 20  $\mu\text{m}$  according to the 10-pixel criterion. In this case, a smaller calibration value does not result in better resolution.



## The 10-pixel criterion

In accordance with ISO/DIS 16232, in automated particle measurement, the length of the smallest particles should be rendered by at least 10 pixels (Fig. 3). The corresponding calibration value for the particle size depends on the magnification level of the microscope, including the factor of the camera adapter and the pixel size of the camera. The calibration value can be determined manually or calculated from the system parameters using the software.

To calculate the calibration value, the pixel size of the camera chip is divided by the overall optical magnification level. A pixel size of 4.65  $\mu\text{m}$  and a 5x magnification level with a C-mount factor of 0.63 results in a calibration value of 1.48  $\mu\text{m}/\text{pixel}$ . According to the 10-pixel criterion and the resolution

of 5x lens particles sizes up from 15 to 20  $\mu\text{m}$  can be measured. However, for small calibration values, such as 0.3  $\mu\text{m}/\text{pixel}$ , it is absolutely necessary to ensure that according to the 10-pixel criterion, particles with a size of 3  $\mu\text{m}$  are correctly detected only if an optical system with corresponding resolution is used. Details that cannot be resolved optically cannot be resolved at a later stage, either.

For manual calibration, the value is determined from a defined distance measured, if possible, down to the pixel. Generally speaking, for fixed optics, the theoretical and manually determined calibration values do not differ from each other until the second decimal place. For stereomicroscopes and macroscopes, the value should be determined manually, as the difference is greater due to the zoom optics.

The 10-pixel criterion applies primarily when measuring function-critical particles. For other particle measurements, the requirement can be attenuated to five times the value of the resolution.

For measurements in small particle classes (CCC classes A–C or larger), we recommend using fully automated light microscopes with high imaging quality. Depending on the particle size, magnification levels of 5x, 10x or 20x can be used. For larger particle classes (CCC class D or higher), manual or motorized macroscope systems (Leica Z6 or Z16) are also suitable.

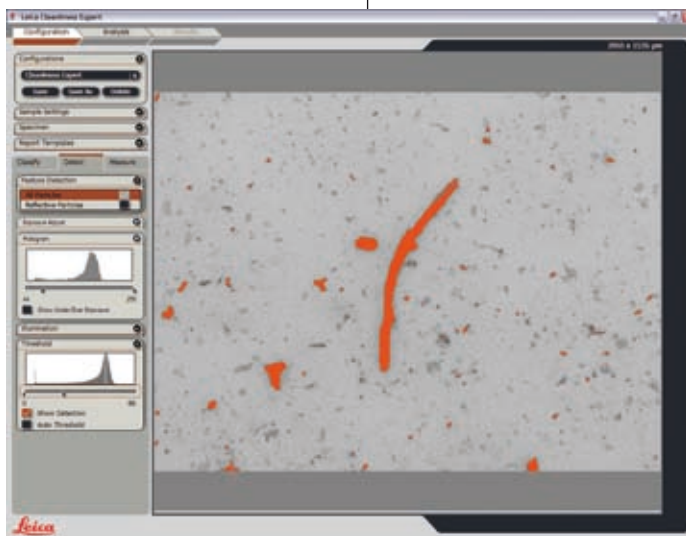


Fig. 4: Detection setting in live image

## Leica Cleanliness Expert

The successor of Leica QClean with new features

- Automatic differentiation between reflective and non-reflective particles
- Detection setting in live image
- New scanning mode
- All parameters relevant for measurement automatically adopted in the report and stored in the configuration
- Reclassification of particles/fibres and reflecting/non-reflecting particles in the list of results
- Display of measured contour line for relocalisation



# Residual Dirt Analysis in Real-world Applications

Interview with Dr. Michael Härtel, head of the materials testing laboratory at Continental Automotive GmbH, Powertrain Division, Limbach-Oberfrohna, Germany

The Powertrain Division of automotive supplier Continental AG develops and produces system solutions for the vehicle powertrain. The product line ranges from gasoline and diesel injection systems to engine and transmission control units to components and systems for hybrid drives. At the location in Limbach-Oberfrohna near Chemnitz, piezo injectors for diesel common rail fuel-injection systems are manufactured. The piezo technology not only enables diesel motors to use less fuel with lower emissions, but also helps gasoline engines with direct injection to attain significantly lower fuel consumption. Dr. Michael Härtel, head of the materials testing laboratory responsible for materials technology in Limbach-Oberfrohna, uses the Leica QClean system for cleanliness analysis of individual components and component groups.

## Dr. Härtel, why is residual dirt analysis so important?

We thoroughly check everything from parts to assembly groups for particle residue, as we now know that cleanliness is a critical factor for reliability and service life. The components are washed and cleaned several times to remove clinging particles. To do so, tools such as lances are used to clean bores from the inside, or correspondingly high pressures are used during washing. After the cleaning, we examine the particles according to their size and distribution using the microscopic analysis method. The results are not only important for verifying our high quality standards, some of which are even more stringent than the official directives. They also help us to identify the cause and origin of the contamination.

## What effect does particle residue clinging to components have?

Our components have narrow tolerances, high-precision fits and small microscopic openings. Tiny particle residues in injectors, pumps and control units can cause malfunctions with serious consequences up to and including failure of the entire fuel-injection system. Our injectors inject the fuel into the engine through bores that are almost unimaginably tiny. As you can surely understand, particles equal to or larger than a certain size at these places cause problems. The same holds true for the control system of the injectors – an interaction of different pressures which, in turn, are controlled via tiny bore holes. If a shaving gets stuck in a hole, the function of the fuel-injection system is impaired.

## Is light microscopic particle analysis adequate for your requirements?

Optical inspection is the preferred technique for routine. It provides reliable results quickly, while still being cost-effective. We are not interested in whether our parts are “clinically” clean. The questions we have to answer are whether particles are present, how large they

are and whether they can cause malfunctions. We classify the particles according to their size and assign them to functionally relevant classes. Depending on which class they belong to, corresponding scenarios exist from which, in turn, we can derive consequences for the manufacturing process. As a rule, very small particles tend to be uncritical. We record them in order to be able to detect any changes in the manufacturing processes at an early stage. In special cases, we use expensive, time-consuming scanning electron microscopic examinations – for example, if we want to study the exact composition of the particles.

## How often do you check the parts for cleanliness?

We study parts and assembly groups at regular intervals according to our inspection plans. We study fully assembled injectors as well as parts. The test frequencies themselves are affected by experimental values. If a part suddenly shows elevated values, the test frequency is increased and corrective actions are introduced simultaneously.

## What is your assessment of the analysis system?

We are very satisfied with the Leica QClean analysis system. We are particularly pleased with the self-explanatory operation. Leica Microsystems continuously develops the software and integrates things that make it easier to use. The different password-protected user levels – user, supervisor and service – make the system highly secure in everyday use. In the future, the industry wishes for a fully automatic solution for counting and classifying particles in order to increase the number of test parts per time unit and allow real-time, on-site inspections. For the time being, however, I do not see any way to implement these ideas cost-effectively.



Piezo diesel injector. Courtesy of Continental Automotive AG

## New European Standard in Steel Industry

# Steel – It All Depends on What's Really Inside

Anja Schué and Jürgen Paul, Leica Microsystems

Steel, an alloy of iron and carbon, is both stable and elastic, extremely resistant, and a permanent item in our everyday life. Today there are over 2,500 standard steel types, with new grades and applications emerging all the time. Each steel type is specially made for its purpose. It is subject to stringent quality standards to ensure that it optimally withstands the specific loads. Light microscopic determination of the content of non-metallic inclusions is one of the main metallographic examinations of industrial steel. In July 2007, the new steel inclusion rating standard EN 10247 has been introduced in Europe. Dipl.-Ing. Damian Moll, Head of Metallography of Dillinger Hütte, Dillingen/Saar, Germany, is an acknowledged expert on steel inclusion rating and, since 2002, chairman of the EN 10247 work group of the VDEh steel institute.

### How important is steel rating for non-metallic inclusions?

Inclusion rating is one of the most common metallographic examinations that industrial steel is subjected to. Non-metallic inclusions in the form of oxides and sulphides are found in small quantities in every sort of steel. The shape, composition and distribution of the inclusions are caused by production processes such as melting, deoxidation techniques, alloying constituents, casting and moulding techniques. Inclusions usually have a negative influence on the properties of the material. Depending on the intended purpose, conditions of use and re-

### Mr. Moll, how important is light microscopy and image documentation for your work?

In general, light microscopy is one of the basic steel examination techniques besides mechanical-technological tests. It accompanies the steel samples from preparation to automated image analysis examinations. Light microscopy yields information on the steel microstructure.

The structure of the samples may vary due to numerous heat treatments. With the aid of light microscopy, we are able to perform quantitative and qualitative analysis of the structure, using parameters such as grain size, shape and distribution of different phases or the content and distribution of non-metallic inclusions for sample assessment. The microstructure of the samples gives us information on macroscopic properties which is useful for the specific development of new steel qualities.

Metallography is basically concerned with microstructures and their illustration. Image documentation and archiving is therefore an indispensable part of our work. Our image databases contain knowledge and valuable experience we can refer to whenever necessary.



Fig. 1: Dipl.-Ing. Damian Moll, Head of Metallography of Dillinger Hütte



Fig. 2: The thirty-seventh bridge over the Seine in Paris is named in honour of author Simone de Beauvoir. The heavy plate needed, in thicknesses of up to 150 mm, and with improved deformation properties perpendicular to the surface, was supplied by Dillinger Hütte. Courtesy of Dillinger Hütte.

Fig. 3: Dillinger Hütte makes heavy plate that may be up to 400 mm thick, 5.2 m wide and 36 m long. Steel from Dillingen in the Saarland region spans bold bridges, withstands the forces of water in oil platforms, transports oil and natural gas, shapes modern skyscraper and large hall architecture. The so-called slabs from the continuous casting and blocks are rolled out on the rolling stands to the required length and thickness at a temperature of 1,200°C with a force of 11,000 tons. Courtesy of Dillinger Hütte.



## Steel Inclusion Rating to EN 10247

The method defined in the European standard is based on the analysis of a defined inspection surface of a polished, unetched metallographic section. The sample must have been taken from a prescribed part of the product. The inspection surface, which must measure at least 200 mm<sup>2</sup>, is scanned for non-metallic inclusions at a suitable magnification. The standard magnification is 100x, although 50x and 200x can also be used. The inclusions are evaluated by comparing them with a chart of standard pictures. The standard is valid for particles with a length of 3 to 1,410 µm and a width of 2 µm and above. Subject to arrangement also for with a width less than 2 µm.

quired properties of the final products, it is possible and desirable to differentiate inclusions according to quantity, shape, size, chemical composition and properties.

For steel inclusion rating we use automatic image analysis as well as manual tests as it facilitates

the otherwise time-consuming quantitative rating methods.

**What was the background for the introduction of the new European standard?**

In connection with the European standardisation of steel specifications, there was an urgent requirement to define a new standard method for steel inclusion rating. The European Committee for Iron and Steel Standardisation ECIS resolved the elaboration of the new EN 10247 as long ago as 1988, and it was published as a preview standard in 1998. The valid standard was published in July 2007. In Ger-



Fig. 4: Dipl.-Ing. Petra Arlt is responsible at Dillinger Hütte for automated image analysis examinations, including steel inclusion rating with the Leica Steel Expert.





many, the DIN 50602 could still be applied as a valid standard during a two-year interim period.

**How do you rate the acceptance of the new standard in the steel industry?**

The European standard is based on the examination technique used up to now in practice. The standard evaluation methods are similar to the previous ones, too. For users, however, it offers easier and clearer classification of the inclusions due to unambiguous inclusion definitions and rules of particle joining. The new chart of standard pictures on the basis of mathematical relationships and the physical dimensions of the measured values also make results easier to compare. Because the chart of standard pictures is based on mathematical algorithms and clear inclusion assessment rules, the new standard enables image analysis systems to be used as well. Their manufacturers were actively involved in the work of the VDEh work group.



Fig. 5: Oxidic inclusions in steel. Courtesy of Dillinger Hütte

More and more users are gradually realising the benefits of the new standard, and its application is becoming more and more common, partly to get new empirical values according to the EN 10247 standard for quality assurance. However, every new standard takes a while to get used to. Acceptance will therefore increase with every new evaluation and every exchange of experience between customer and steel supplier.



Fig. 6: Built with Dillinger steel: The impressive Millau viaduct in the heart of France, 2,460 m long and 343 m high. Courtesy of Dillinger Hütte.



Fig. 7: Dillinger Hütte supplied 23,000 t of heavy plate for the 101-storey Shanghai World Financial Center which is 492 m high. Courtesy of Mori Building.

## Leica Steel Expert – Fully Automatic Steel Inclusion Rating



Leica Steel Expert is a complete system solution that consists of high-end quality soft-

ware, a sensitive Leica DFC colour camera and a Leica DMI5000M automated inverted industrial microscope (alternatively a Leica DM4000M or DM6000M upright microscope). The system performs fully automatic classifications and analysis of non-metallic inclusions of all types of common steel, based on their colour, shade, shape and arrangement. Measurement data is available in raw, processed and histogram format all of which can be displayed simultaneously and easily compared when applied to the established international standards.

Leica Steel Expert supports all international steel inclusion rating standards including, ASTM E45 A, D, E, ISO 4967 A, B, DIN 50602 K, M, JK and JIS 0555. In addition, the latest European standard EN 10247 is fully implemented.

**Do your customers dictate the quality standards to be used or are you free to choose?**

Quality tests make sure that product properties meet the specifications of the customer – that's what the customer expects. The type and extent of these tests are either defined in binding regulations or arranged between the customer and the supplier in the contract.

**How do you see the future of steel as an industrial material?**

Steel has been the basis for technical progress for more than 3,000 years. Without steel, the pioneering technical inventions such as the steam engine, railway train, automobile, communication engineering, space travel and computer technology wouldn't have happened. New types of steel are being developed and existing types being improved all the time. To take just one example: Whereas it took 7,000 tons of steel to build the Eiffel Tower in 1889, it would only take us 2,000 tons nowadays. And there is no end to the development in sight. Not to forget that



Fig. 8: High-tech steel plate from Dillinger Hütte for the multi-storey car park at the new Exhibition Grounds in Stuttgart. This building won the 2007 European Steel Design Award.

steel can easily be recycled and is therefore a sustainable material. Steel is exciting and ultra modern and has a lot of potential for future applications.

## DCM 3D Dual-Core 3D Measuring Microscope

# Fast Characterisation of Solar Cells

Roger Artigas, Sensofar and Anja Schué, Leica Microsystems

Solar energy is becoming more and more important all around the globe. Not only is it available in unlimited supply, it also offers key advantages for protecting the climate and the environment. Every year, many thousands of solar cells are produced worldwide for new photovoltaic plants. An important criterion for quality control is 3D characterisation of the light-absorbing surface. In the past, this required time-consuming SEM analysis. The new Dual-Core 3D Measuring Microscope DCM 3D from Leica Microsystems and Sensofar offers non-contact, high-precision analysis of the surface texture of solar cells in a matter of seconds.

The sun provides about  $1.08 \times 10^{18}$  kWh of energy per year, which is roughly equivalent to 10,000 times the world's primary energy requirements. Solar energy is already used for a wide variety of applications from powering small, automated, low-consumption electronic systems to the electrification of entire regions. In Amareleja, Portugal, for example, there is a photovoltaic power station with a peak capacity of 46 megawatts. Approximately 261 million euros were invested in this plant, which is the largest of its kind to date. In December 2008 a 40 megawatt photovoltaic plant was completed in the Waldpolenz energy park near Leipzig, Germany, involving an investment of about 130 million euros.

The efficiency of the solar cell depends on the silicon dopant, the light intensity and the wavelength range used, the optical thickness and surface texture. Currently, the energy efficiency of a solar cell is about 20 per cent. Applying specific but extremely expensive surface texturing processes, the solar cell can absorb more light, increasing its efficiency by up to 50 per cent. The application that demands the greatest cell efficiency is space travel.

### Surface texture enhances energy efficiency

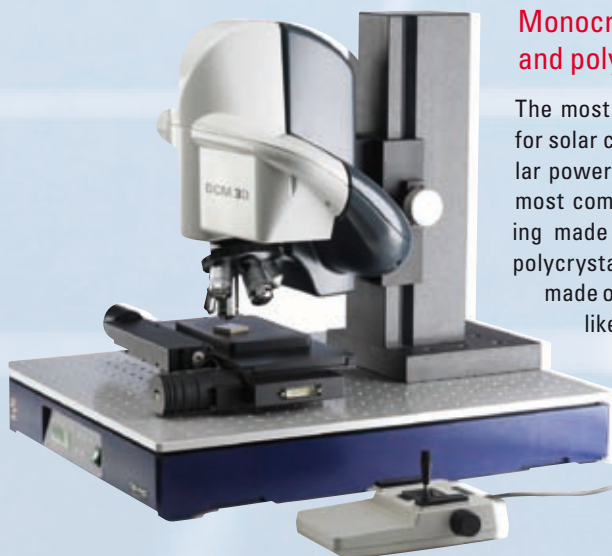
Numerous techniques have been tested for increasing cell efficiency, for instance light focusing with Fresnel lenses, solar concentrators or anti-reflection coatings. The most effective way to increase light absorption is by increasing the effective optical thickness of the silicon surface. This method, called surface texturing, depends to a large extent on the type of silicon.

Monocrystalline silicon texturing is accomplished by a wet anisotropic etching process based on sodium hydroxide solution. The crystallographic silicon plane {111} is slowly etched, and the result is squared pyramids grown randomly with equal angled surfaces. The quality of the surface and the amount of pyramids depend on the temperature and the composition of the etching solution. The light absorption of this surface texture is extremely effective by increasing the number of internal light reflections.

### Monocrystalline and polycrystalline silicon

The most frequently used basic material for solar cells is silicon. In large-scale solar power generation, thick-film cells are most common, with a differentiation being made between monocrystalline and polycrystalline. Monocrystalline cells are made of monocrystalline silicon wafers like the ones used for semiconductor fabrication. Polycrystalline cells consist of wafers with varying crystal orientation. For one thing, they can be manufactured by casting and cost less than monocrystalline cells.

Fig. 1: The Dual-Core 3D Measuring Microscope DCM 3D with confocal and interferometry technology in a single sensor head





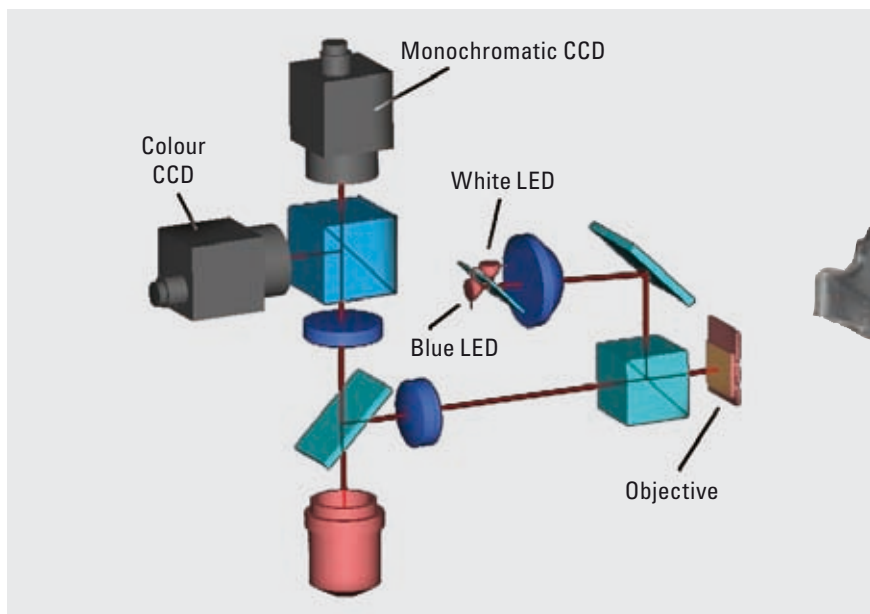


Fig. 2: The optical design of the DCM 3D

In comparison, polycrystalline silicon texturing is not quite as effective due to the fact that most of the grains have incorrect orientation. Different grains etch at different rates, causing the formation of steps at grain boundaries, which may lead to problems with soldering zones and contacting structure in the subsequent metal screening process.

### 3D surface measurement in a few seconds

Solar cell quality control is done at the end of the production chain, testing each individual cell for efficiency. The dual-core technology DCM 3D Optical Imaging Profiler offers the possibility to check silicon surface texture, roughness, pyramid statistical characterisation and metal contact in a few seconds. Unlike the time-consuming scanning electron microscope method, the wafer is simply placed under the DCM 3D and a 3D measurement taken in less than 10 seconds. The high local slope of the pyramid faces demands the use of objectives with a high numerical aperture, which are only available in confocal technology.

Figure 3 shows a 3D measurement of a monocrystalline silicon wafer after pyramid etching. For a 3D measurement of this kind, a 150x objective with a numerical aperture of 0.95 was used. As a result, the size of the visual field was reduced to a few tens of microns, which is roughly equivalent to the visual field of an SEM. The surface is scanned a few microns along the focus position of the objective, collecting the confocal images plane by plane. The result is a high-resolution image similar to that gen-

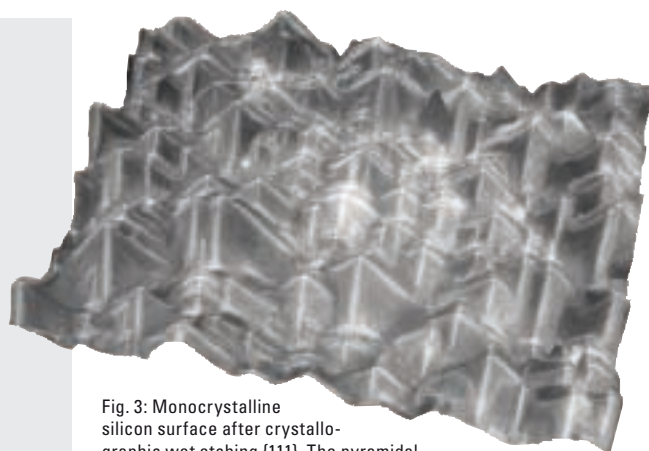


Fig. 3: Monocrystalline silicon surface after crystallographic wet etching {111}. The pyramidal surface texture increases light trapping up to 50 times, thereby increasing the cell efficiency.

erated by an SEM with infinite focus and precise 3D information on the height of the pyramids.

For the statistical pyramid characterisation, a special watershed segmentation algorithm is used (Fig. 4). This algorithm uses the height information to separate each individual pyramid into segments and then calculates the area, volume, mean and maximum height of each individual segment. In Figure 5, a profile section shows the characterisation of a single pyramid with the height, base area and face angles.

### Technology with combined precision

The Dual-Core 3D Measuring Microscope DCM 3D offers a unique combination of confocal microscopy and interferometry in a single sensor head. The core technology is based on a fast-reaction microdisplay

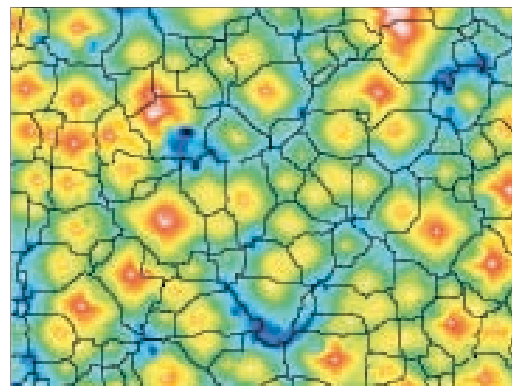
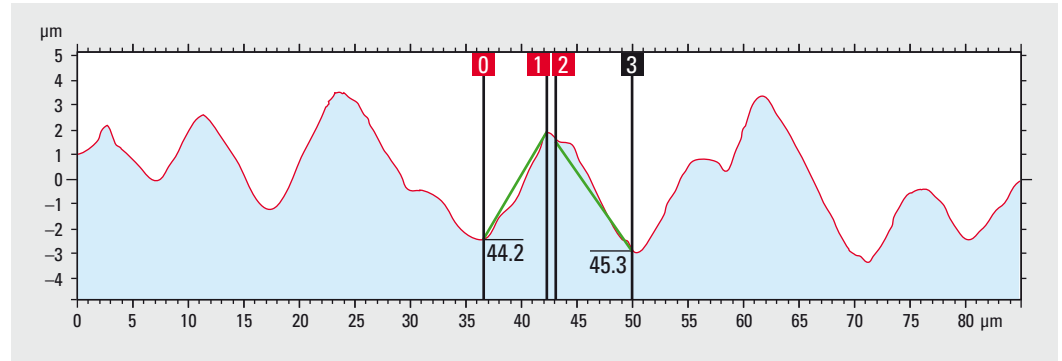


Fig. 4: Automatic segmentation using the intelligent watershed segmenting algorithm in individual pyramids

Fig. 5: The height distribution statistics of the segmentation are used for surface structure characterisation.



positioned in the field diaphragm. Brightfield, interferometric and confocal images can be produced via the microdisplay. The non-moving part concept, the confocal microdisplay (MD), two light sources and two cameras achieve high accuracy 3D measurements and unlimited depth of focus (Fig. 2).

In comparison with confocal technology, depth discrimination of phase shift interferometry (PSI) and vertical shift interferometry (VSI) does not depend on the objective's NA but on the light source properties. For a PSI height resolution down to 0.1 nm, the maximum measurable height is limited to 250 nm by optical laws. Nevertheless, the confocal + interferometry combination of the DCM 3D allows measurements from 0.1 nm to several millimetres. The system is suitable for a wide variety of measurement applications in R&D and quality control laboratories all the way to automated online process control.

**More information on the DCM 3D:**  
[www.leica-microsystems.com/products/DCM3D](http://www.leica-microsystems.com/products/DCM3D)

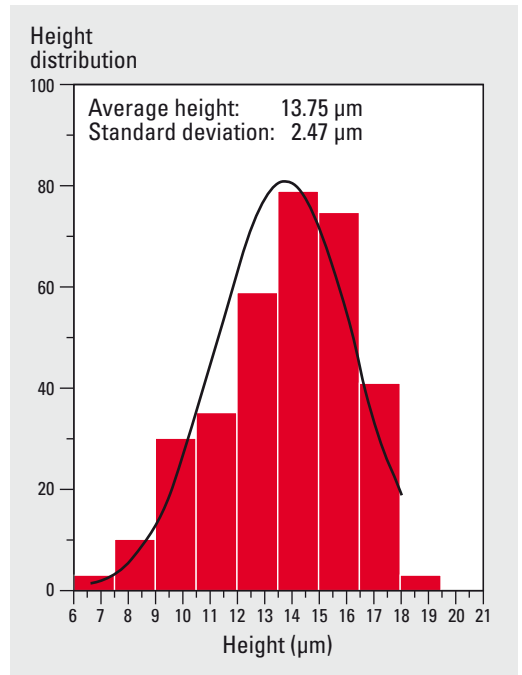
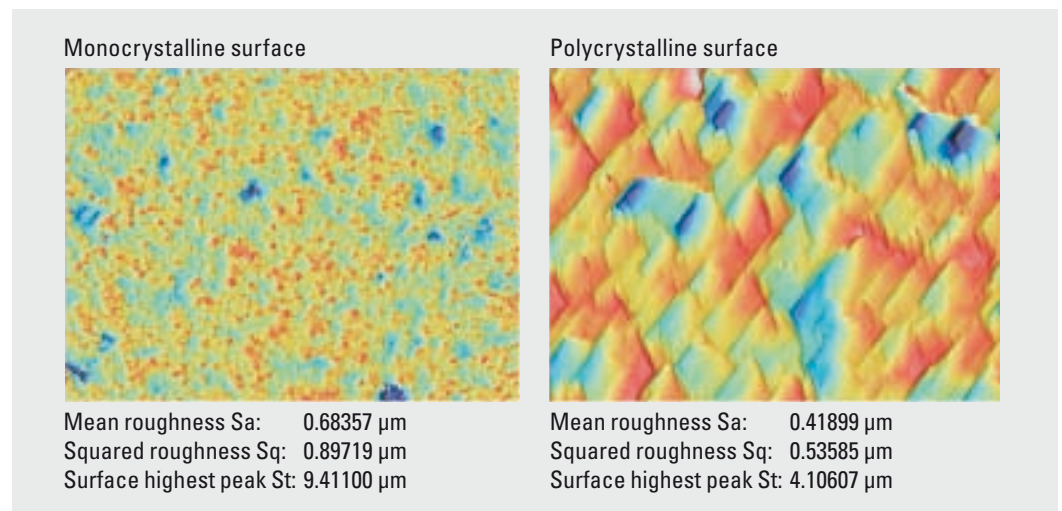


Fig. 6: Profile section of the pyramids on a monocrystalline silicon surface

Fig. 7: Comparison of the typical surface parameters (e.g. roughness) of monocrystalline and polycrystalline silicon



## Competence for Individual Optic Solutions in High-end Quality

# The Leica Optic Centre

Kerstin Pingel, Leica Microsystems

The Leica Optic Centre (LOC) at the Wetzlar plant is one of the competence centres of the Leica Microsystems company. Here, experienced and highly qualified staff, most of them precision opticians and engineers, produce high-tech optics for microscopes and microscopy systems with ultimate precision. It is also the place where Leica's proprietary optic technology and process competence for three factories are developed by physicists, chemists and engineers. Without exaggerating, the know-how at the LOC can be described as unique.

### Top precision and vast experience

The amount of precision and experience that go into objectives from the Leica Optic Centre is evident at the workplace of Rainer Cromm, where various lens component groups are assembled to make an objective. The individual parts are matched and adjusted with the precision required to make an efficient high-tech objective. After putting each of the lens component groups into the objective housing – in this case there are eight mounts with a total of 14 lens elements – the objective specialist assesses the optical axis with the aid of a microscope. Using small adjustment screws, he can make an axis offset in the  $\mu\text{m}$  range. The next step is to measure the

Fig. 1: The objective specialist assesses the optical axis with the aid of a microscope. Using small adjustment screws, he can make an axis offset in the  $\mu\text{m}$  range.

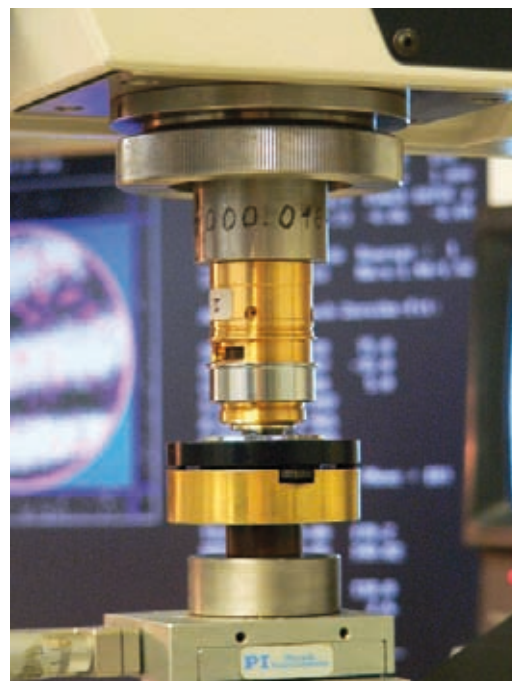


Fig. 2: An interferometer plus software was specially designed and produced at the LOC to meet the high requirements of Leica objectives.

objective with an interferometer. This measuring instrument plus software was specially designed and produced at the LOC to meet the high requirements of Leica objectives.

### Each objective is unique

Various parameters, such as the sphere, the outer and inner coma, and wavefront areas, are crucial for the quality of the objective. On "his" objective type, the HCX PL APO 100x/1.40–0.70 Oil, there are four assembly spacings between the mounts, which Rainer Cromm alters by hand depending on the result of the measurement. These alterations are in the region of only a few  $\mu\text{m}$ . For some corrections, however, there is no clear solution. In such cases, analytic thinking and a great deal of experience are required to track down the nanometre-sized "error" by repeated alteration of various parameters and repeated measurements.



On average, Cromm works on an objective for six hours. What are the essential skills for assembling a good objective? "Patience, years of experience and the ambition to always strive for the best possible quality – even if the objective has to be taken apart several times," says Rainer Cromm, who has 30 years of experience working for Leica Microsystems. "After all, every objective is unique, even in series production."

### Individual solutions for individual customer applications

The Leica Optic Centre also offers individual solutions for measuring, analysing or processing microstructures, going by the basic principle of the company's founder, Ernst Leitz I. "With the user, for the user": From the original idea through planning and production all the way to delivery, a team of experienced design engineers, technicians and highly qualified scientists work out the product that exactly meets the customer's needs to Leica's first-class quality standards.

### Transparent high-end quality

To achieve this, the LOC implements state-of-the-art manufacturing technologies. Permanent quality checks and efficient and transparent processes are a matter of course. All work is thoroughly documented – to a standard far exceeding that prescribed by the DIN ISO 9001. Component quality is examined with the latest methods after each stage of produc-



Fig. 4: The LOC is the place where Leica's proprietary optic technology and process competence for three factories are developed by physicists, chemists and engineers.

tion. For example, the LOC documents every single optical surface and the final system as a whole with interferometric and spectrophotometric techniques to check adherence to the agreed specifications. For Dr. Claus Gunkel, Manager of the Leica Optic Centre, quality is not an empty word: "For me, High-end Quality means working with highly motivated and qualified employees who leave DIN and ISO standards far behind – day in, day out."

**More information on the Leica Optic Centre:**  
**Raimund Bayer, Project Manager LOC**  
**[loc-sales@leica-microsystems.com](mailto:loc-sales@leica-microsystems.com)**



Fig. 3: Dr. Claus Gunkel, Manager of the Leica Optic Centre

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## Leica Microsystems Reports Record Sales for 2008

# Strategic Acquisitions and Organic Growth

Dr. Kirstin Henze, Leica Microsystems

For the first time in its history, Leica Microsystems' annual sales volume for 2008 exceeded the billion US dollar mark. "Over the last two years, we have seen a dramatic increase in the demand for our products throughout the world. In most of the markets in which we operate – including biomedical research, clinical applications, industry, microsurgery, and histopathology – we have achieved double-digit organic growth rates. Moreover, we have substantially expanded our product breadth through a number of strategic company acquisitions," comments Dr. David Martyr, President of Leica Microsystems.

### Numerous company acquisitions expand product range and benefit customers

Leica Microsystems was purchased by Washington D.C. based Danaher Corporation (NYSE: DHR) in

the summer of 2005. Since that time, Leica Microsystems has acquired and integrated eight companies in Australia, Europe, the US, and Asia. With these acquisitions, Leica Microsystems has significantly broadened its product offering and now provides one of the most comprehensive ranges of microscopy and histopathology products on the market. Leica Microsystems' histology offering now includes consumables for use with its instruments. This allows histology customers to obtain all needed products from a single source. "I'm pleased to say that we've not only expanded our product portfolio through strategic acquisitions, but we have also gained significant market share as a result of innovation within our existing segments," says Martyr.

### Innovative strength drives organic growth

An important pillar of the success of Leica Microsystems, according to Martyr, is its innovative strength. In 2008 alone, the Life Science, Biosystems, Industry and Surgical Divisions launched over 50 new, and in some cases, breakthrough products. As a result of its recent product launches, Leica Microsystems is now at the cutting edge of technology. Examples of innovation in life science include the super high-resolution STED technology, the macro confocal Leica TCS LSI, and stereomicroscopes with FusionOptics™.

### Leica Microsystems is owner of the Leica brand

Leica Microsystems owns the rights to the Leica name and the Leica brand and controls its use through licensing agreements. Leica Microsystems, Leica Geosystems, and Leica Camera are financially, legally, and operatively independent companies, operate in different markets, and belong to different owners.



Dr. David Martyr,  
President of Leica Microsystems

# Events

**Control**

May 5–8  
Stuttgart, Germany  
[www.control-messe.de](http://www.control-messe.de)

**SMT Hybrid Packaging**

May 5–7  
Nürnberg, Germany  
[www.mesago.de/en/SMT](http://www.mesago.de/en/SMT)

**22th National Symposium of the Italian Association of Metallurgy**

May 6–8  
Salsomaggiore, Italy  
[www.aimnet.it/tt2009.htm](http://www.aimnet.it/tt2009.htm)

**EPHJ – Professional Watchmaking – Jewelry**

May 12–15  
Lausanne, Switzerland  
[www.ephj.ch](http://www.ephj.ch)

**Intertech**

May 14–16  
Dornbirn, Austria  
[www.messedornbirn.at/intertech](http://www.messedornbirn.at/intertech)

**National Electronics Week**

June 16–18  
London, UK  
[www.nationalelectronicsweek.co.uk](http://www.nationalelectronicsweek.co.uk)

**Congreso Ibérico de Tribología**

June 17–18  
Lisbon, Portugal  
[www.upv.es/trib2003](http://www.upv.es/trib2003)

**Goldschmidt**

June 21–26  
Davos, Switzerland  
[www.goldschmidt2009.org](http://www.goldschmidt2009.org)

**Annual Conference of the German Mineralogical Society**

September 13–17  
Halle/Saale, Germany

**MesurExpo**

October 6–8  
Paris, France  
<http://en.mesurexpo.com>

**SFGP – Société Française de Génie de Procédés**

October 14–16  
Marseille, France  
[www.sfgp.asso.fr](http://www.sfgp.asso.fr)

**Parts2Clean**

October 20–22  
Stuttgart, Germany  
[www.parts2clean.de](http://www.parts2clean.de)

**Trends in Nano Technology**

November 7–11  
Barcelona, Spain  
[www.tntconf.org/2009](http://www.tntconf.org/2009)

**Productronica**

November 10–13  
Munich, Germany  
[www.productronica.com](http://www.productronica.com)

**Milipol**

November 17–20  
Paris, France  
[www.milipol.com](http://www.milipol.com)

**RichMac**

November 25–27  
Milan, Italy  
[www.richmac.it](http://www.richmac.it)

**Scanlab**

November 25–27  
Stockholm, Sweden  
[www.scanlab.nu](http://www.scanlab.nu)

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