



ANNUAL REPORT
2016



FOREWORD

Ladies and gentlemen,

the year 2016 was a very successful year for the Fraunhofer Institute for Surface Engineering and Thin Films IST in many ways with various innovative developments and special events such as the change of our deputy director. We provide you with a selection of the most important events and latest research developments of the Fraunhofer IST in the annual report at hand.

We would like to take this opportunity to express our thanks to all people whose hard work and commitment made our success possible in the first place: above all the employees of the Fraunhofer IST, our partners from research and development, our customers from industry, our sponsors, colleagues and friends. Thank you for a trusting cooperation.

Dear reader, we hope you enjoy reading our annual report and are looking forward to your ideas for cooperation in future.

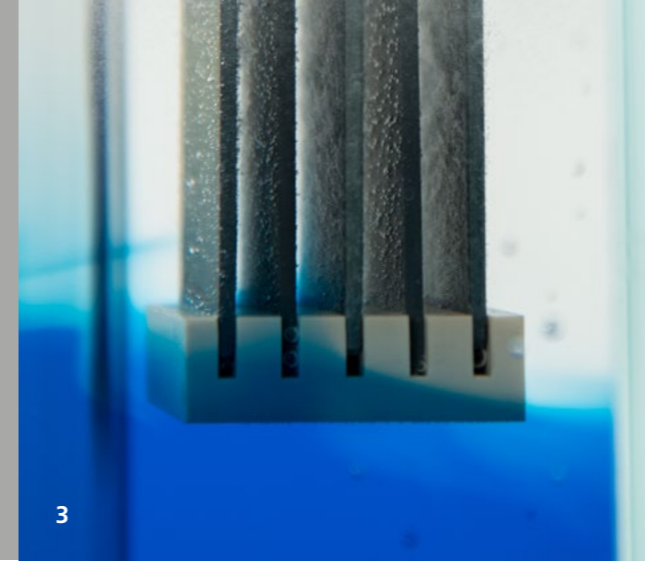
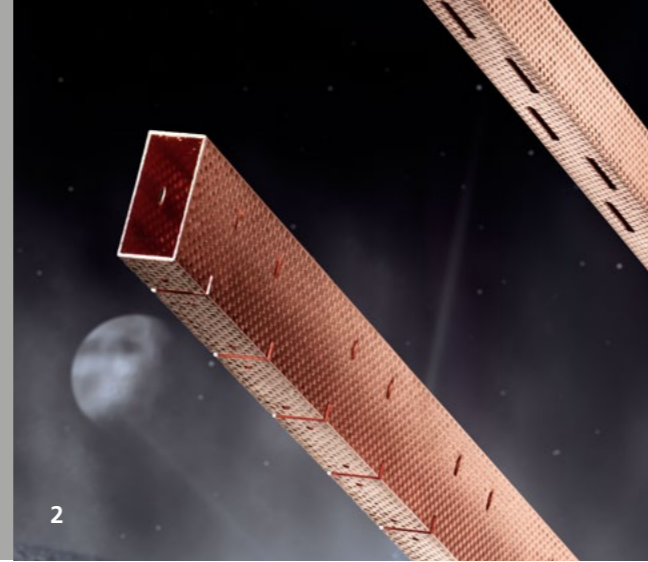
1 On the left: director Prof. Dr. Günter Bräuer, on the right: deputy director Dr. Lothar Schäfer.

Prof. Dr. Günter Bräuer

Dr. Lothar Schäfer

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2016 IN RETROSPECT

In his paper "Application perspective of plasma technology for the food industry" a Korean scientist recently showed the truth of the matter. With 1 billion US dollars for energy technologies and 15 billion US dollars for information technologies, these markets are still virtually marginal in comparison with the global foodstuffs market with a volume of about 2.5 trillion US dollars.

About 50 years ago, the significant advancement of low-pressure plasma-assisted surface treatment began. It has brought forth materials and products, which have changed our lives. At the same time, due to pressure constraints, this new technology had to restrict itself to particular inorganic materials. However new advancement in atmospheric pressure plasmas allow for the targeted treatment of organic surfaces such as seeds, fruits, vegetables, meat or human skin. Their importance for our future therefore cannot be overstated.

In accordance with its vision statement "Surfaces for our future are created here" the Fraunhofer IST continued in 2016 to expand its activities in plasma technology not only for inorganic but also for organic surfaces. One milestone in this development was the assessment of the Application Center for Plasma and Photonics in Göttingen which, following its four-year development phase, is now managed as a permanent department of the institute. Activities in Braunschweig in

the field of biofunctional surfaces have also seen a positive development. An important role here is played by young female engineers working in the field of bioprocess engineering, with two of them successfully completing the TALENTA sponsoring and development program, which supports the careers of young female scientists.

On April 22nd, some 600 waveguides electroplated at the Fraunhofer IST commenced their journey into space, now the second such occasion. Together they form the basis for the antenna of the earth observation satellites Sentinel 1B, which is to collect data for environmental and climate research. Work is already in progress on the coatings for the Sentinel 1C and 1D missions.

Some notable collaborative projects gained significant traction in 2016. In the electroplating field, ionic liquids are to open up new possibilities for the production of raw materials. While the diamond electrode plays an important role in two key projects: the Fraunhofer internal project "Power as raw material" which is concerned with using excess power for H₂O₂ production, while the EU project "SafeWaterAfrica" is to conduct research into water treatment plants for urban areas in South Africa. At the same time, diamond electronics are appearing to spark significant research interest once again.

A few years ago, we were still an unknown in the field of precision optical coatings, but in 2016 we achieved a breakthrough with the EOSS® (Enhanced Optical Sputtering System). The first orders were issued to plant constructors who work in collaboration with us as partners.

Two top politicians from Lower Saxony visited our sites in Braunschweig and Göttingen. In Braunschweig, as part of his summer tour Olaf Lies, Minister of Economic Affairs, caught up on the research activities of the Fraunhofer IST, while State Premier Stephan Weil visited in November both the HAWK laboratories and the Application Center in Göttingen. These two visits provided good opportunities for informing the politicians of the enormous importance of plasma research to Lower Saxony.

I hope you enjoy reading our activity report,

Günter Bräuer

1 *Improved stem cell growth on the surfaces of plasma-coated bags.*

2 *Metallized carbon-fiber reinforced plastic antennas for the ESA »Sentinel-Mission«.*

3 *Degradation of blue textile dye using diamond electrodes.*



BOARD OF TRUSTEES

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Dr. Philipp Lichtenauer

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Ruhr University Bochum

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FROM THE BOARD OF TRUSTEES

The initial impetus for the successful cooperation between the Fraunhofer IST and EagleBurgmann actually was technical competition. Twelve years ago, EagleBurgmann performed a comparative analysis of diamond coatings made by a Bavarian institute and by the Fraunhofer IST. This technical comparison was based on two very extensive designs of experiments with more than 2500 individual tests each. The result was clear: the silicon carbide rings with diamond coatings from the Fraunhofer IST both had a higher reproducibility and better tribological properties. This resulted in one of the greatest innovations in the mechanical seals sector and an intensive cooperation between the Fraunhofer IST and EagleBurgmann in the field of research and development, continuing to this day.

EagleBurgmann were able to present the first pump seals with diamond technology already during Achema 2006, the important industrial fair in Frankfurt. One year later, this technology was launched on the market as DiaomondFace®, together with Condias GmbH, a spin-off of the Fraunhofer IST. Since then, EagleBurgmann's technology has progressed in more than 50 other projects of the Fraunhofer IST and Condias.

Initially, mechanical seals with diamond technology were used in particular for sealing highly abrasive media, but over the years, many other applications have been added. These included, for instance, their use in highly pure demineralized water, applied as coolant in power plants, or as flushing liquid in the semiconductor industry. In these applications, the

diamond technology has solved the tribological and corrosive challenges of deionized water. Today, EagleBurgmann seals with DiaomondFace® are world market leaders.

For EagleBurgmann the quality of cooperation with the Fraunhofer IST is exemplary. In addition to the competence, reliability and flexibility of our partners in Braunschweig it is especially the shared enthusiasm, which guarantees not only concrete results but also fun during our joint projects. We have also benefited from the openness of the Fraunhofer IST regarding cooperations in the Fraunhofer Alliance, especially with the Fraunhofer IKTS in Dresden and the Fraunhofer IWM in Freiburg.

I would like to thank all institute staff members for their outstanding cooperation and commitment and look forward to many other projects in our partnership.

Michael Stomberg
Chief Operating Officer (COO)
EagleBurgmann



OUTSTANDING COLLABORATION

FHR Anlagenbau GmbH in Ottendorf-Okrilla specializes in the development of innovative thin-film technologies and installations, as well as providing services in the field of thin-film technology. It was founded in 1991 in Dresden. The FHR product portfolio includes vacuum process installations for mass production, pilot production and R&D. In addition to diverse fields of research, the thin-film technologies and FHR's products find applications, for example, in the fields of optics, thin-film photovoltaics, solar thermal energy, semiconductor and sensor technology.

In the development of innovative system technology, the FHR has been collaborating very successfully for more than a decade with the Fraunhofer Institute for Surface Engineering and Thin Films IST.

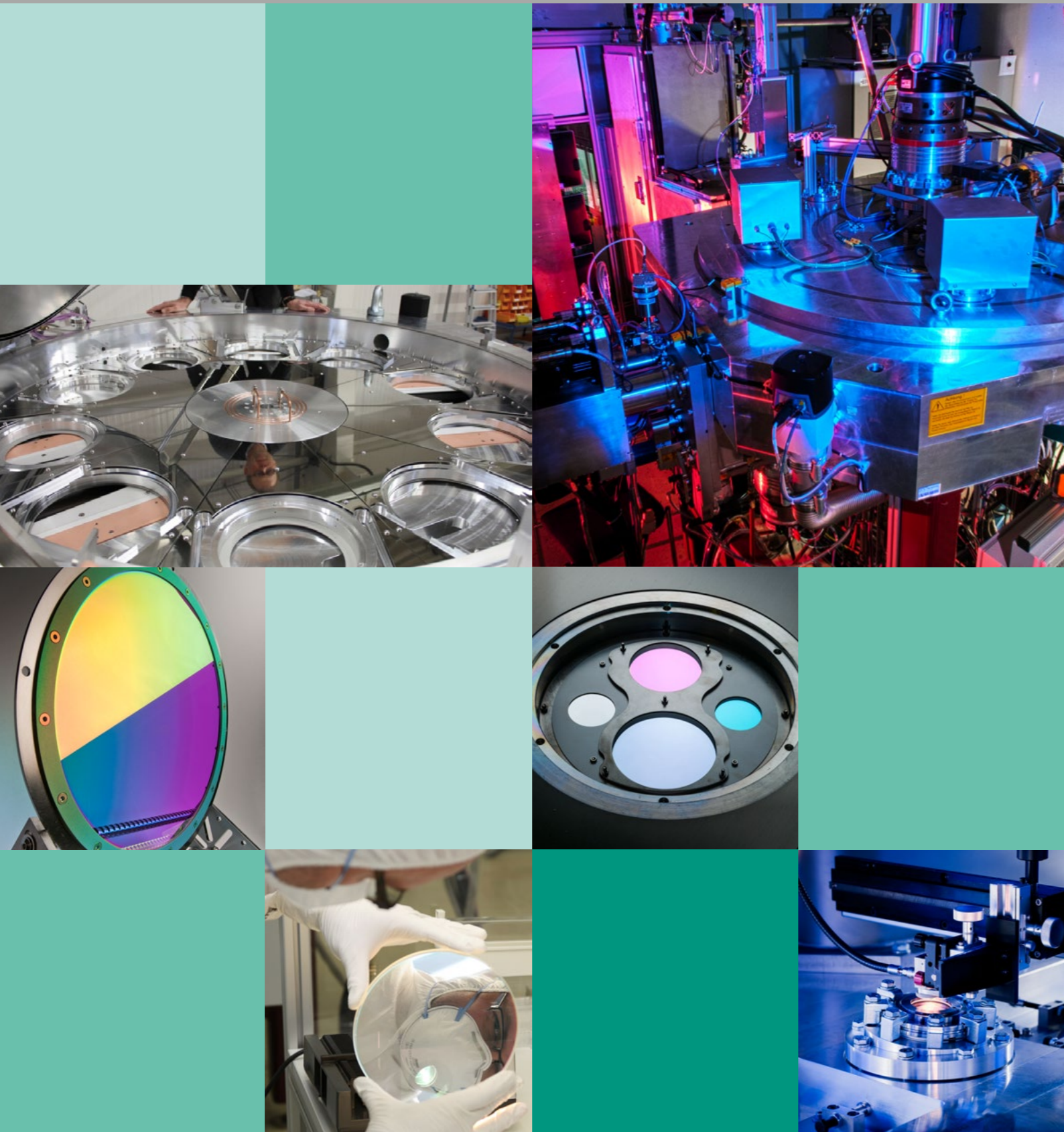
A recent example is the development of the EOSS® coating system. This is a very modern system for plasma coating with which high-precision optical interference filter systems can be produced. The machine has a carrier system which enables 10 substrates with a diameter of 200 mm to be coated automatically and simultaneously. Three chambers with cylindrical magnetron sputter sources and an innovative "sputter-up" concept make the production of extremely homogeneous and flaw-free surfaces possible.

With EOSS® a coating concept has been realized and made available on the market which as yet is entirely unique worldwide. For FHR, construction and sales form the basis for obtaining market leadership in the field of special systems for optical coatings. Users in the optics industry benefit from this innovative tool with which they can themselves produce optical coatings on large-format substrates with the deposited layers having excellent homogeneity and quality. The EOSS® coating system thus also offers our customers the technical requirements for putting their products at the head of the market.

The Fraunhofer IST with its dedicated and scientifically highly qualified staff has been and is for FHR an extremely valuable and reliable development partner in this venture. Even our customers benefit from this connection, since the IST stands out not only in its skills and expertise in the development of systems, but also in the corresponding development of coating and process.

At this point I would like to thank all employees of the Fraunhofer IST for their trustworthy and excellent collaboration.

Torsten Winkler
FHR Anlagenbau GmbH





INSTITUTE PROFILE

As an innovative R&D partner the Fraunhofer Institute for Surface Engineering and Thin Films IST offers complete solutions in surface engineering which are developed in cooperation with customers from industry and research. The IST's "product" is the surface, optimized by modification, patterning, and/or coating for applications in the business units:

- Mechanical engineering, tools and automotive technology
- Aerospace
- Energy and electronics
- Optics
- Life Science and ecology

The principle technology units at the IST are atmospheric pressure processes with the main focus on electrochemical processes and atmospheric pressure plasmas processes, low pressure plasma processes with the main focus on magnetron sputtering and highly ionized plasmas and PECVD as well as chemical vapor deposition with the main focus on hot-wire

CVD. The center of tribological coatings focusses on the friction reduction, wear and corrosion protection. The Application Center for Plasma and Photonics deals with mobile plasma sources and laser plasma hybrid processes.

The IST's expertise lies in the ability to control all of the above-mentioned processes and their combination with a great variety of thin films. Extensive experience with thin-film deposition and film applications is complemented by excellent capabilities in surface analysis using the very latest equipment and in simulating vacuum-based processes.

Choosing the optimum combination of process and coating for a particular task is one of the major strengths of the Fraunhofer IST.

Besides fundamental research activities in cooperation with universities and research centers, about 130 employees are developing tailored surfaces and processes together with service providers, equipment manufacturers, and coating users from diverse industries. For an efficient technology transfer

the IST offers a broad range of services, from development of prototypes and economical product scenarios to upscaling and even implementation of the technology at the customer.

At the site in Braunschweig the institute has an office and laboratory area of more than 4000 square meters for the 116 tenured employees. In addition, the new building of the Application Center for Plasma and Photonics provides 1500 square meters of office and laboratory area on three storeys for the 14 tenured employees in Göttingen. The service offers of the Fraunhofer IST are supplemented by the competencies of other institutes from the Fraunhofer Group "Light & Surfaces" as well as by the Institute for Surface Technology of the Technical University of Braunschweig which is also managed by the IST director Prof. Dr. Günter Bräuer. Many projects are supported by funding through the state (Land) Niedersachsen (Lower Saxony) the federal government, the European Union, and other institutions.

THE INSTITUTE IN FIGURES

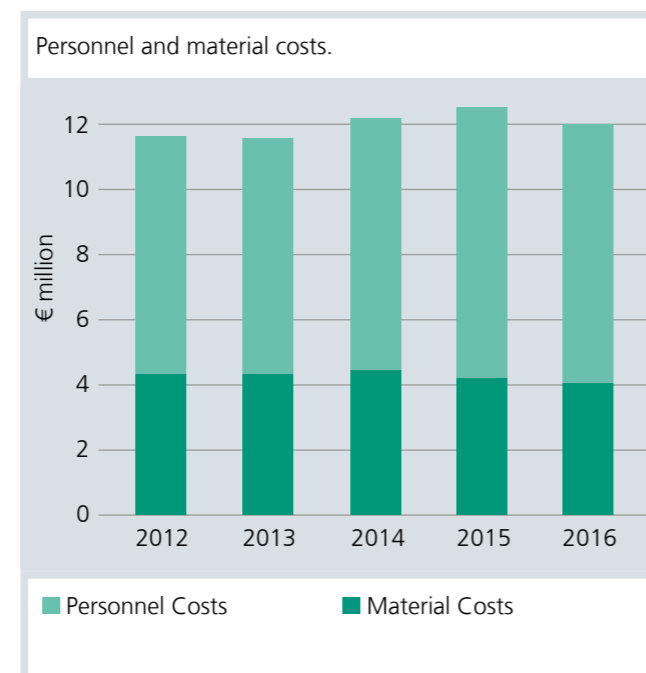
Employee development

In 2016, the period under review, the Fraunhofer Institute for Surface Engineering and Thin Films IST had 128 employees. Around 56 % are scientific personnel, doctoral candidates and engineers. Research activities were supported by technical and commercial staff as well as a large number of graduands and student assistants. Training opportunities in the vocational fields of galvanics, physics and information technology were taken up.



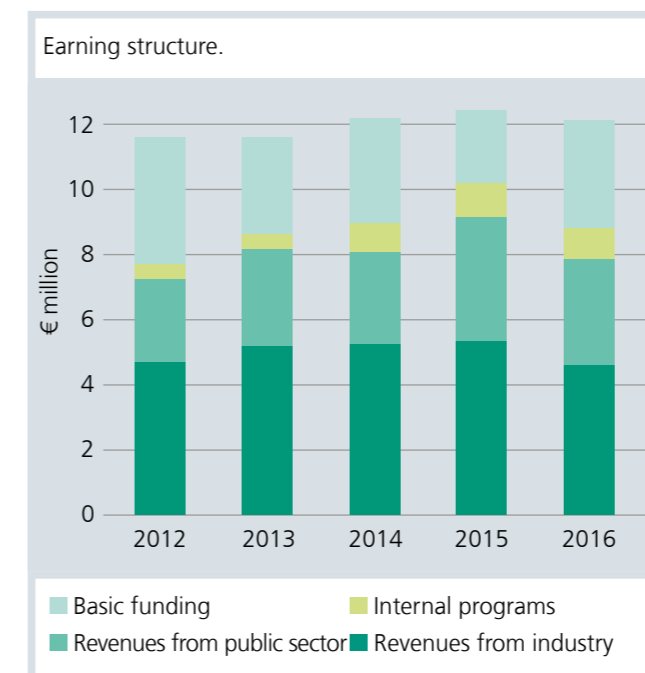
Operating budget

2016 the operating budget fell from €12.4 million to €12.2 million. The material-related costs have remained constant compared to the previous year with a budget of €4.1 million, corresponding the personal costs fell to €8.1 million and contribute 66 % to the total operating budget.



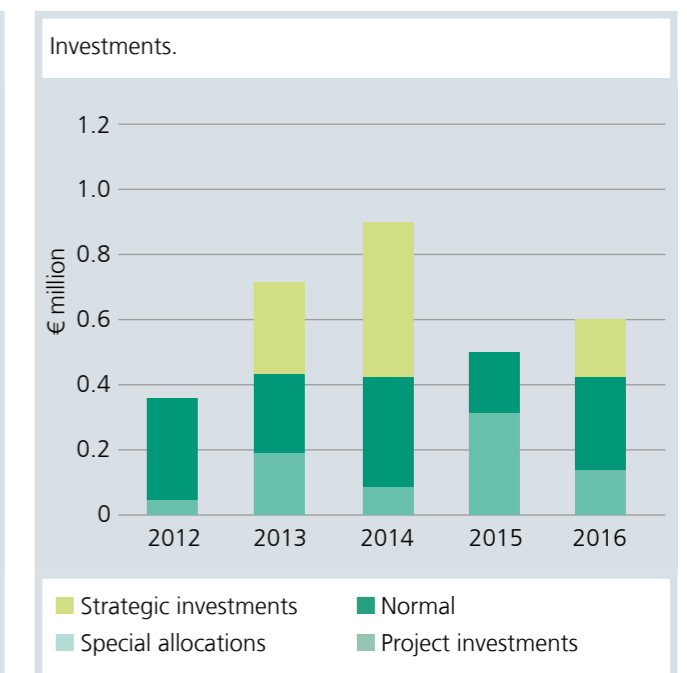
Earning structure

Due to falling revenues from industry the relative increase of the Fraunhofer Institute for Surface Engineering and Thin Films IST was 39 percent. Thereby revenues from industry contributed amounting to €4.7 million and additional €3.4 million were realized through public sector. In total, the institute achieve external revenue amounting to €8.1 million.



Investments

All in all the Fraunhofer IST dispensed on normal investment some €600,000. Approximate €130,000 could be invested through external project funds. €290,000 can be attributed to normal investments. €180,000 were used for strategic investments. This means for the Fraunhofer IST an overall budget (B+I) totaling €12.8 million.





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Optical coating systems | Process engineering | Materials engineering

Magnetron sputtering

Large area electronics | Transparent and conductive coatings | Asset and process development | New semiconductor for photovoltaic and microelectronics

Highly ionized plasmas and PECVD

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Multifunctional coatings with sensors | High Power Impulse Magnetron Sputtering (HPIMS) | Micro tribology | Electrical coatings | Hollow cathode processes (HKV, GFS) | Plasma-enhanced CVD (PECVD)

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Simulation of plants, processes and coating layer properties | Model based interpretation of coating processes

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Tools and components | Diamond electrodes for electrochemical water treatment | Diamond coated ceramics DiaCer®

Dr. Markus Höfer¹⁴

Senior Scientist
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Atomic layer deposition

Product-related system construction | Coating and process development | Highly compliant coatings of 3D structures

Photocatalysis

Air, water and selfcleaning | Product evaluation and efficiency determination | Test engineering

Hot-wire CVD

Diamond coatings and silicon-based coatings | Tool and component coatings for extreme wear resistance | Electrical applications for semiconductors, barriers | Antireflective



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Electrochemical processes

Composites | Light metal coatings | Process development | Plastics metallization | Electrochemical processes

Layer by layer

Biofunctional coatings | Polyelectrolyte coatings | Quantitativ analysis of reactive surfaces

Atmospheric pressure plasma processes

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Micro and sensor technology

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Thin film sensors | Microstructuring 2D and 3D | Adaptronic systems

Tribological Systems

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Prototypes and small volume production | Plasma diffusion | Cleaning technology | Mechanical engineering and automotive technology | Carbon-based coatings (DLC) | Hard and superhard coatings | Wetting behavior | Tool coating (forming, cutting, chipping)

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Application center for plasma und photonics

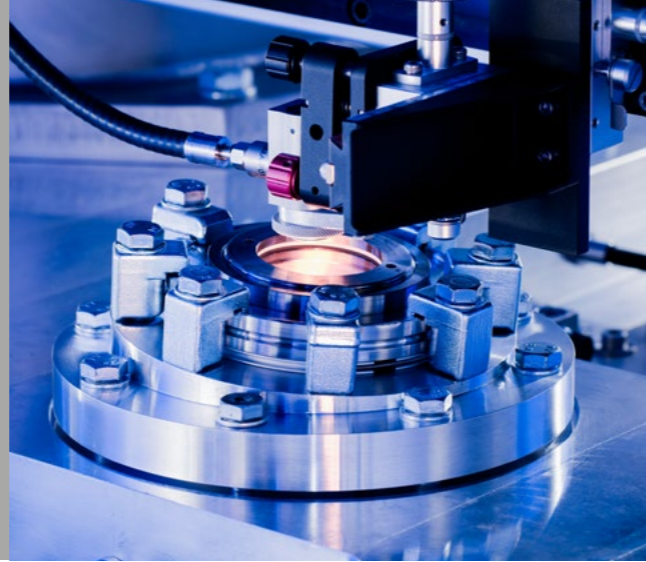
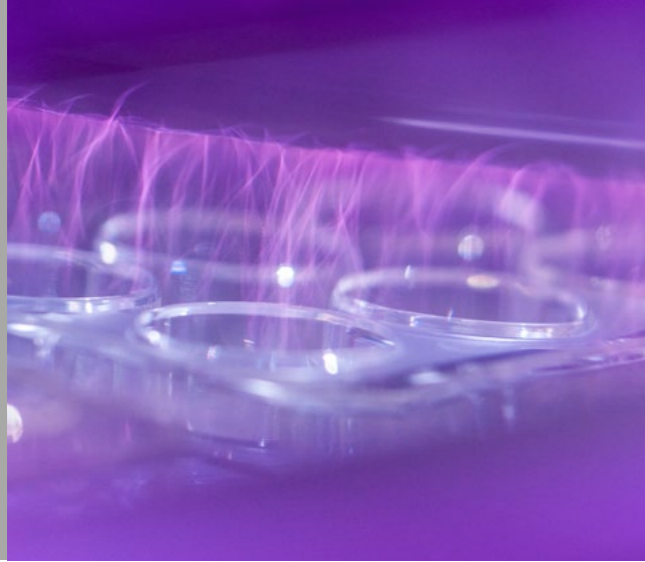
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Plasma sources conception, plasma high voltage generator, plasma toolbuilding | Plasma diagnostic and surface analytic | Plasma treatment of natural products | Plasma particle coating and cold plasma spraying | Plasma medicine, atmospheric pressure plasma-based air purification, disinfection and pest control | Laser plasma hybrid technology for micro structures and surface modification | Laser technique for material treatment and characterization | Acoustic, optical plasma supported sensors

Analysis and Quality Assurance

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Chemical microscopy and surface analysis | Microscopy and crystal structure | Test engineering | Costumer specific test engineering | Order investigation



THE SCOPE OF RESEARCH AND SERVICES

Pretreatment – We clean surfaces

Successful coating processes imply a proper surface pretreatment. Therefore we offer:

- Effective aqueous surface cleaning including drying
- Special glass cleaning
- Plasma pretreatment and Plasma cleaning
- Plasma activation and Plasma functionalization
- Wet-chemical etching pretreatment
- Particle beam

Modification and Coating – We develop processes and coating systems

Thin films and specifically modified surfaces are the core business of the Fraunhofer IST. The institute utilizes a wide range of coating technologies and surface treatments, ranging from plasma coating and treatment in vacuum and at atmospheric pressure over hot-filament CVD processes to electroplating and laser technology. Our services are:

- Surface modification
- Development of coatings and layer systems
- Process technology (including process diagnostics, modeling and control)
- Simulation of optical layer systems
- Development of system components
- Process development
- Toolbuilding and plant engineering

Testing and Characterization – We ensure quality

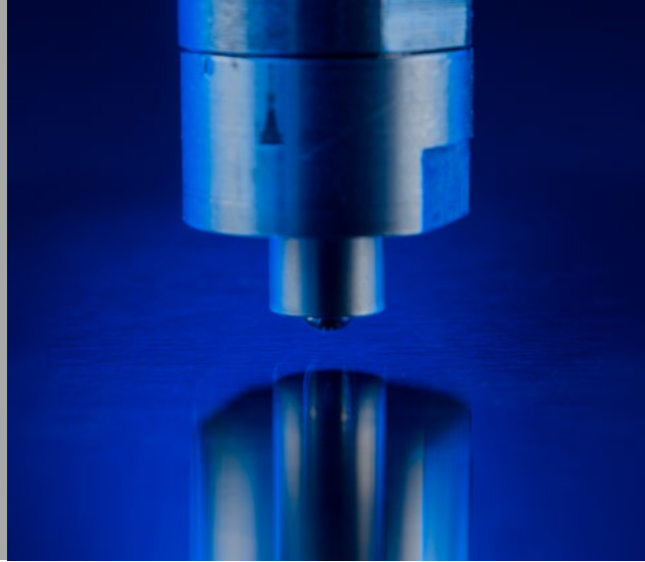
A fast and reliable analysis and quality control is the prerequisite for a successful coating development. We offer our customers:

- Chemical, micromorphological, and structural characterization
- Mechanical and tribological characterization
- Optical and electrical characterization
- Test methods and product specific quality control methods
- Rapid and confidential failure analysis
- Testing of corrosion resistance

Application – We transfer research results in the production level

To guarantee an efficient technology transfer we offer a wide range of know how:

- Cost-of-ownership calculations, development of economical production scenarios
- Prototype development, pilot production and sample coating procedures
- Equipment concepts and integration into manufacturing lines
- Consulting and training
- Research and development during production



ANALYSIS AND QUALITY ASSURANCE

Chemical and structural analysis

- | Energy-dispersive X-ray spectroscopy (EDX)
- | Electron microprobe (WDX, EPMA)
- | Secondary ion mass spectrometry (SIMS)
- | X-ray photoelectron spectroscopy (XPS)
- | Glow discharge optical emission spectroscopy (GDOES)
- | X-ray fluorescence analysis (RFA / XRF)
- | X-ray diffractometer (XRD, XRR)

Microscopy

- | Scanning electron microscope (SEM)
- | SEM with focussed ion beam (FIB)
- | Scanning tunnel and atomic force microscope (STM, AFM)
- | FTIR microscope
- | Confocal laser microscope (CLM)
- | Photo optical microscopes

Mechanical tests

- | Micro and nano indentation (hardness, Young's modulus)
- | Rockwell and scratch test (film adhesion)
- | Cross-cutting test, butt-joint test (film adhesion)
- | A variety of methods for the measurement of film thickness
- | A number of profilometers

Measurement of optical properties

- | IR-UV-visible spectrometry
- | Ellipsometry
- | Colorimetry
- | Angular-resolved scattered light measurement (ARS)
- | Integral scattered light measurement (Haze)
- | FTIR spectrometry
- | Raman spectrometry

Measurement of friction, wear and corrosion

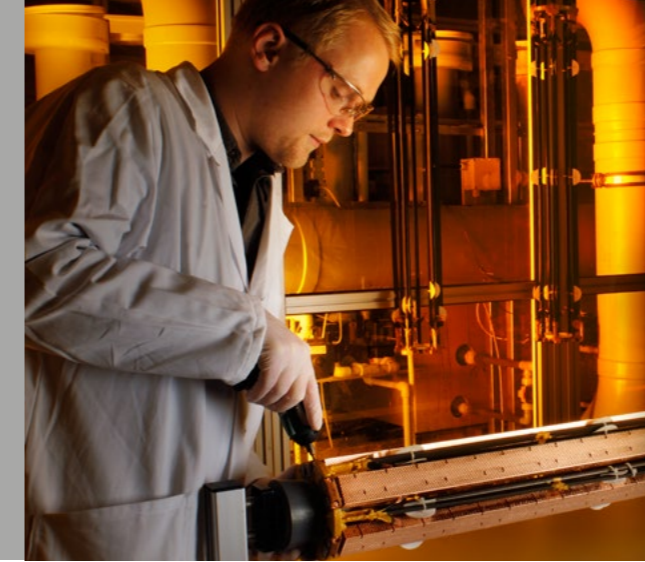
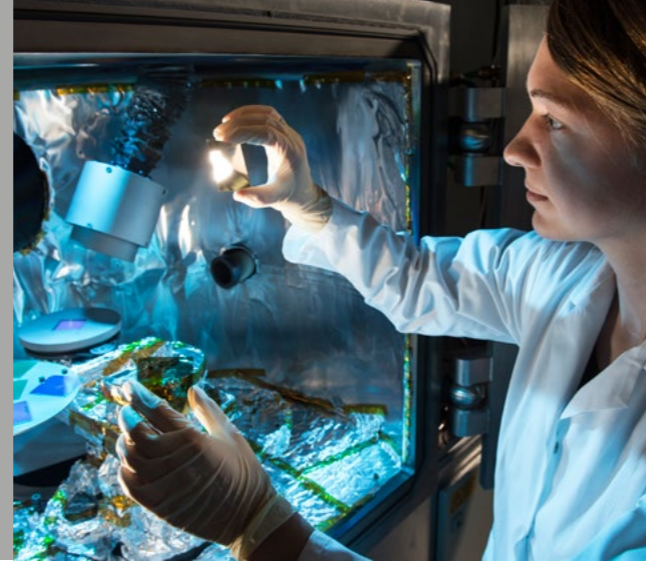
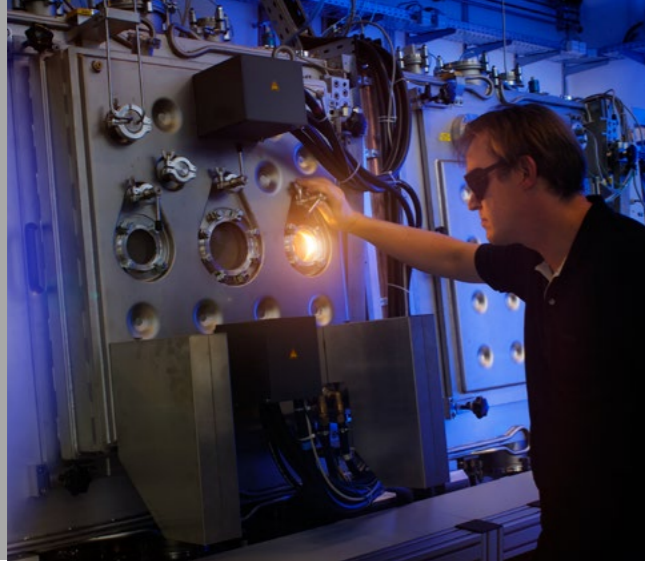
- | Pin on disk tester
- | Ball-cratering test (Calo)
- | Wazau high-load tribometer (in air, in oil)
- | CETR high-temperature tribometer (in air, in oil)
- | Plint roller tribometer (in air, in oil)
- | Taber abraser test, abrasion test, sand trickling test, Bayer test
- | Microtribology (Hysitron)
- | Impact and fatigue tester (Zwick Pulsator)
- | Salt spray test, environmental tests

Specialized measurement stations and methods

- | Characterization of solar cells
- | Measuring station for photocatalytic activity
- | Contact angle measurement (surface energy)
- | Measuring systems for electrical and magnetic coating properties (e. g. Hall, Seebeck, conductivity, vibration magnetrometer VSM)
- | Test systems for electrochemical wastewater treatment
- | Measuring stations for the characterization of piezoresistive and thermoresistive sensor behavior
- | Biochip reader for fluorescence analysis
- | Layer mapping system (0,6 x 0,6 m²) for reflection, transmission, Haze and Raman measurement
- | In-situ bondenergy measurement
- | Magnetic characterization (vibration magnetrometer VMS)
- | Electrochemical measurement stations (CV measurement)
- | Wet chemical rapid tests: colorimetric determination of Ion and molecule concentrations
- | Weathering tests: cyclical simulation of UV and rain exposition

Plasma diagnostics

- | Absorption spectroscopy
- | Photoacoustic diagnostics
- | Laser induced fluorescence LIF
- | High-speed imaging
- | Optical emission spectroscopy OES
- | Retarding Field Energy Analyzer RFEA
- | Fiber thermometry
- | Electrical performance test
- | Numerical modeling



SPECIAL EQUIPMENT

- | a-C:H:Me, a-C:H, hard coating production plant (up to 3 m³ volume)
- | Coating facilities incorporating magnetron and RF diode sputtering
- | Sputter plant for high-precise optical coatings
- | In-line coating facility for large-surface optical functional coatings (up to 60 × 100 cm²)
- | Industrial scale HIPIMS technology
- | Plants for plasma diffusion
- | Coating systems for hollow cathode processes
- | Coating plant for thermal and plasma atomic layer deposition (ALD) (2D and 3D)
- | Hot-filament-CVD units for crystalline diamond coatings (up to 50 × 100 cm²) and for internal coatings
- | Hot-filament-CVD unit for silicon-based coatings (batch process and run-through process up to 50 × 60 cm²)
- | Plasma-activated CVD (PACVD) units, combined with plasma nitriding
- | Atmospheric pressure plasma systems for coating and functionalization of large areas (up to 40 cm widths)
- | Microplasma plants for selective functionalization of surfaces (up to Ø = 20 cm)
- | Bond aligner with an integrated plasma tool for wafer pretreatment in the clean room
- | Roll-to-roll set-up for area-selective functionalization of surfaces up to 10 m/min
- | Machine for internal coating of bags or bottles
- | Laser for 2D and 3D microstructuring
- | Automated system for deposition of polyelectrolyte
- | 2 mask aligner for photolithographic structuring
- | Laboratory for microstructuring (40 m² clean room)
- | System for electroplating metallization of waveguides (11 active baths with a volume of each 140 l and 1 niche bath with a volume of 400 l)
- | Modular technical electroplating system (20 stations for active baths with a volume of each 20 l)
- | Anodizing plant (11 active baths with a volume of each 140 l and 2 anodizing baths with a volume of each 350 l)
- | 15-stage cleaning unit for surface cleaning on aqueous basis
- | Clean room – large area coating (25 m²)
- | Clean room – sensor technology (35 m²)
- | Laser structuring laboratory (17 m²)
- | Mobile atmospheric pressure plasma sources
- | Nanosecond dye laser (Nd: YAG-Laser)
- | CO₂-laser and Excimer-Laser
- | EUV spectrography
- | Semiconductor laser
- | Picosecond laser



SUSTAINABLE SOLUTIONS WITH SURFACE AND THIN FILM ENGINEERING

Sustainability is currently perhaps the most important social guiding principle of the age. Not only in the European Union but also in Germany sustainable development processes are in first place on the agenda. In the field of surface and thin film engineering the Fraunhofer IST has for a number of years now been developing solutions for sustainable products and sustainable industrial production.

A large number of research subjects at the Fraunhofer IST are oriented by urgent future-related topics and by social trends, such as the implementation of an alternative energy supply, alternatives for scarce materials and raw materials, or mobility in the 21st century. The very thinnest high-performance coatings are in addition the basis for a variety of further products and high-tech applications which are viable for the future, especially when it is a matter of saving material and energy. Some examples from our research into sustainable industrial products and processes:

Innovative materials

- At the Fraunhofer IST intensive research has been in progress on replacing indium tin (ITO) with alternative materials such as ones based on ZnO and SnO₂ and TiO₂.
- Low damage separations of indium free materials for high efficient LEDs are being developed.
- At the Fraunhofer IST alternative materials for the high-refractive-index tantalum oxide coatings used in optical industries are being developed.
- New materials like canal materials for TFIs and p-conductive materials are being developed for transparent contact films (TFTs).
- At the Fraunhofer IST a REACH-compliant plastic metallization is used as an alternative to chrome (VI).

Material efficiency

- In a combined process of atmospheric pressure plasma processes and electrochemical processes precious metals are applied to selected areas.
- Working materials with new properties are being found by combining different materials or layer and basic body.

Production efficiency

- Optimized hard-material and nanostructured coating systems for forming or cutting tools increase service lives and make more economically efficient manufacturing possible.
- Faster to the goal: simulation means ever shorter development times. For example, highly efficient production chains are made possible by model-based design and implementation of coating processes.
- Modules with sensorized thin-film systems are built into deep-drawing systems and driving machines to ensure efficient forming and machining of components.
- Hard carbon-based coatings not only stop materials such as powders from adhering to tools but also prevent deposits on or fouling of surfaces in, for example, heat exchangers or exhaust systems.

Energy efficiency

- Lower energy consumption due to the erosion protection of aero-engines: very hard multilayer coatings of ceramic and metal prevent excessive fuel consumption and falling efficiency levels.
- Broader and improved range of applications for lightweight components by means of wear-resistant, friction-reducing coatings which also protect against corrosion.
- Reduced solar radiation in buildings by the use of electrochromic windows.

Clean environment

- With the diamond electrodes developed at the Fraunhofer IST water can be conditioned electrochemically – adapted to the infrastructure on the spot and without the use of chemicals.
- Photocatalytic coatings make self-disinfecting surfaces possible and the degradation of pollutants from the air.
- The functionalization of surfaces in plasma enables adhesive to be dispensed with when, for example, bonding materials. Plasma pretreatment is also suitable as a replacement for primers and as a way of improving the adhesion of paint systems.

Health

- Plasma medicine has a great potential for the sustainable treatment of patients. With the medical device PlasmaDerm®, for example, open wounds can be treated efficiently. In the long term, this accelerates the healing, reduces the time and personnel expenditure and increases the quality of life.
- The use of atmospheric pressure plasmas allows to kill even multiresistant germs.

Mobility in the future

- Low-friction and extremely wear-resistant coatings reduce the fuel consumption of car engines and extend both maintenance intervals and service life.
- New corrosion coatings on metallic bipolar plates make possible the economic production of powerful fuel cells for the automotive industry.
- Robust thin-film sensor systems in highly stressed parts of components increase reliability and safety in many fields of application, such as, electromobility.
- Functional coatings for components of lithium ion batteries raise the efficiency and the durability of these storages for electro mobile applications.



MECHANICAL ENGINEERING, TOOLS AND AUTOMOTIVE TECHNOLOGY

The "Mechanical engineering, tools and automotive technology" business unit is primarily concerned with developing coating systems for friction reduction and also for wear and corrosion protection and optimizing these for particular applications. This covers the entire process, from pretreatment, coating and process development –including analysis and simulation–to application. Pretreatment includes not only cleaning but in particular also adjusting surface topographies by abrasive blasting or plasma processes as well as a diffusion treatment, if necessary. Examples of applications in the components field are:

- | DLC and hard coatings for motor and drive components
- | Surfaces for batteries and fuel cells in mobile applications
- | Non-stick and antifouling coatings
- | Surface optimization and corrosion protection of hybrid components
- | Metallization and functionalization of plastics
- | Highly corrosion-resistant carbon coating systems for sealing applications

Another important activity is the design of coating processes by simulating real 3D components.

In the mechanical engineering and tools field these areas of application stand in the foreground:

- | Coatings for pressure die-casting molds
- | Tool coatings for plastic molding (pultrusion, injection molding)
- | Plasma diffusion treatment (including thermodynamical simulation) and coating of forging and press-hardening tools

In addition, sensorized surfaces are developed for and successfully used in the most varied safety-related areas of application, such as:

- | Sensorized washers for continuous force monitoring
- | Pressure and temperature thin film sensor systems for highly stressed tools
- | Thin-film strain gauges
- | Magnetic functional layers

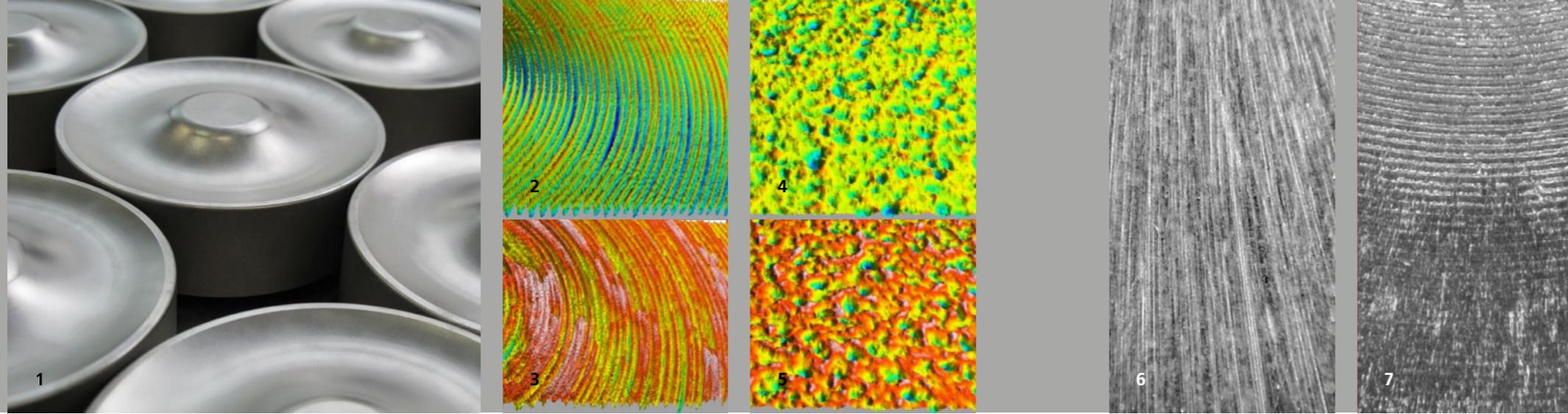
In addition to coating service providers our customers primarily include companies in the automotive sector, toolmakers, tool users and also coating users in all fields of mechanical engineering.

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STRUCTURE-PRESERVING HARD COATINGS

The structure of the surface plays an important role in hot forming processes such as forging. In the case of unstructured surfaces high tribological loads usually occur during the forming process, leading to severe loss of material. Frictional forces between the die surface and the forging are to be reduced by the use of lubricants. An additional structuring of the surface here not only ensures improved adhesion of lubricants but also a reduction in contact ratios. The surface must, however, be protected as effectively as possible against the leveling mechanisms caused by plastic or abrasive wear in the tribological contact. The conditions for an optimum design of the die surface and its successful preservation could now be defined at the Fraunhofer IST.

Topography and wear development

In a first step, different machining methods for die production – partly in combination with cost-effective abrasive blasting processes – were investigated with regard to their wear behavior. In serial forging tests the wear rate could be determined by tactile measurements of the die surfaces. With the aid of the Abbott-Firestone curve, three-dimensional surface characteristics, such as the arithmetical mean roughness S_a , were determined for the purpose of evaluation. The tests showed that, particularly in the case of tribologically highly loaded dies, there were significant correlations between the surface structure and wear development.

Successful preservation with plasma technologies

To secure retention of the specified structures a so-called duplex treatment of the dies was carried out in which the surfaces are first of all treated by plasma nitriding and then provided with a hard coating. In this way the hardness of the outer layer and of the surface could be increased and concurrently the heat resistance of the substrate. These properties influence the resistance to plastic deformation and to

a great extent abrasive wear resistance as well. Investigations also revealed that the surface structure most resistant to predominantly tribological stress also has the lowest wear values. This topography could be set to an arithmetical mean roughness of $S_a = 1.5 \mu\text{m}$ by abrasive blasting and has optimum properties for the adhesion of water-based graphite lubricant films. High frictional forces during forming and the abrasive wear thereby promoted can thus be significantly reduced by targeted surface conditioning. At the same time, this surface has sufficient load-bearing capacity not to be leveled by high surface pressures. The duplex treatment of the dies additionally supports this.

Industrial benefits

In order to evaluate the wear behavior observed in serial forging tests on model dies under industrial production conditions as well, a project partner's forging dies were processed by the wear protection method developed. Industrial dies were structured and given a combined duplex treatment consisting of plasma nitriding and the PECVD deposition of a hard coating. Despite typical fluctuations in the die service lives,

the modification yielded positive benefits. Compared to the series production dies, which have only been gas nitrided, the modified dies showed marked improvements in abrasive wear, especially in tribologically highly loaded areas. Furthermore, an increase in service life was achieved as a result of a considerable decrease in mechanically induced cracks.

Outlook

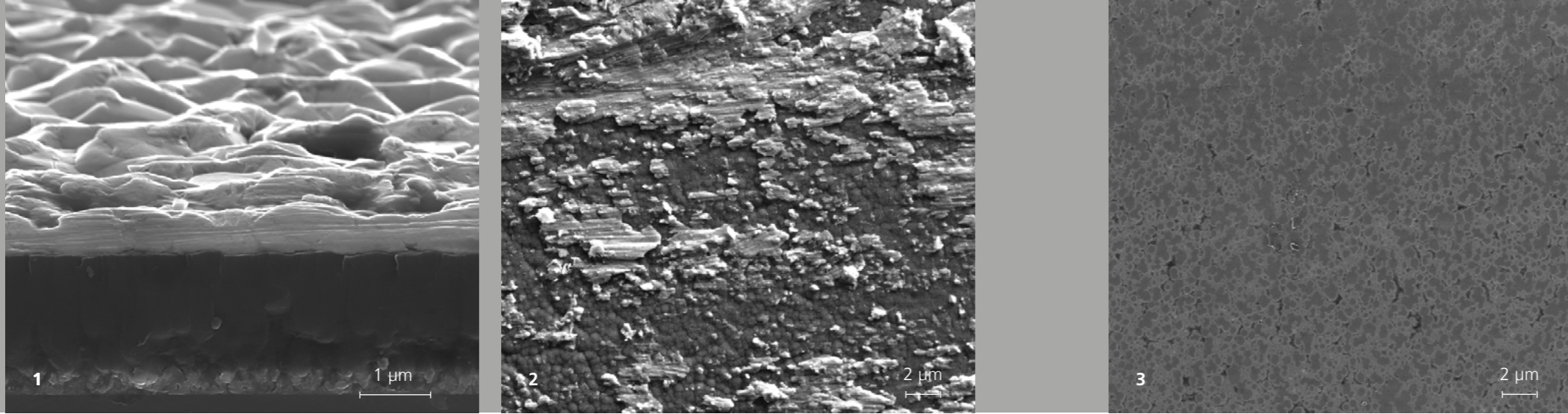
In tribologically highly loaded forging dies improved wear behavior and the associated increase in service life are currently still opposed by the increased expense of surface conditioning by the preservative duplex treatment. For this approach to be a viable alternative even as regards its cost-effectiveness, the service life of the modified dies must be significantly increased by the coating. Results, which have so far been promising, must in future be confirmed by further trials.

1-5 Tribologically loaded dies with different topographies (2) hard and (3) soft turned or (4) medium and (5) coarsely blasted.

6-7 The turned surface after 100 forging cycles shows (6) strongly abrasive wear marks in the untreated state and (7) preservation of the topography in the duplex-treated state.

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PATH TO DRY FORMING OF ALUMINUM ALLOYS

Mastering lubricant-free forming processes represents a contribution to the establishment of ecologically and economically more efficient production technologies. Dispensing with lubricants reduces for example, operating resource costs, makes expensive systems for lubricant application and component cleaning redundant while reducing production times. As one of the world's leading institutions in the research field of carbon-based thin films, the Fraunhofer IST develops coating systems which make dry forming processes possible for aluminum alloys.

Current challenges in aluminum forming

Due to their outstanding weight/strength ratio, aluminum alloys are particularly suitable for the production of the weight-optimized components which are currently in demand especially in the automotive and aerospace sector and in general mechanical engineering. In comparison with conventional tool materials, aluminum's marked tendency to adhere does however place high demands on forming technologies. At present a large quantity of lubricant is still required if rapid tool wear is to be avoided in aluminum forming. This results in a considerable drop in the cost-effectiveness and sustainability of the production process.

Coating development

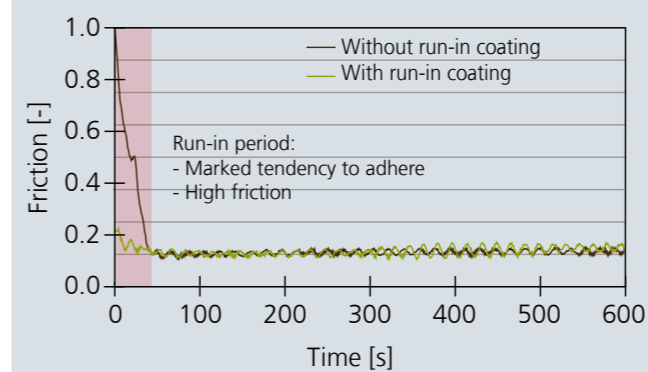
As part of the priority program SPP1676 of the German Research Foundation (DFG) the Fraunhofer IST develops anti-adhesive tool coatings to enable the lubricant-free forming

of aluminum. Amorphous carbon coatings (a-C:H) show a high tribological suitability for lubricant-free sliding contacts against aluminum. However, deposited as a tool coating, current a-C:H coating systems do not allow the dry forming of aluminum alloys. One obstacle is the run-in behavior of these coatings which is characterized by a high adhesion tendency and friction. In forming processes this behavior leads to the immediate appearance of adhesions and, associated with this, tool failure before the running-in phase is overcome. In order to optimize the running-in behavior the Fraunhofer IST is developing running-in layers for a-C:H tool coatings (see Figure 1). As can be seen in the diagram and also in Figures 2 and 3, application-related tribometer tests reveal a significant reduction of the adhesion tendency and friction. This illustrates the great prospects of success in achieving dry forming processes by using the a-C:H coating system which has been developed.

Outlook

The surface state of a run-in a-C:H tool coating can potentially be set via a specific surface treatment. In future, a method is to be developed for reproducing this state based on a plasma-chemical and thermal treatment and structuring of the a-C:H layers. The aim is to provide optimized a-C:H tool coatings for the dry forming of aluminum sheets.

Frictional behavior of a-C:H coatings with and without a run-in coating against aluminum sheets made of EN AW-5083.



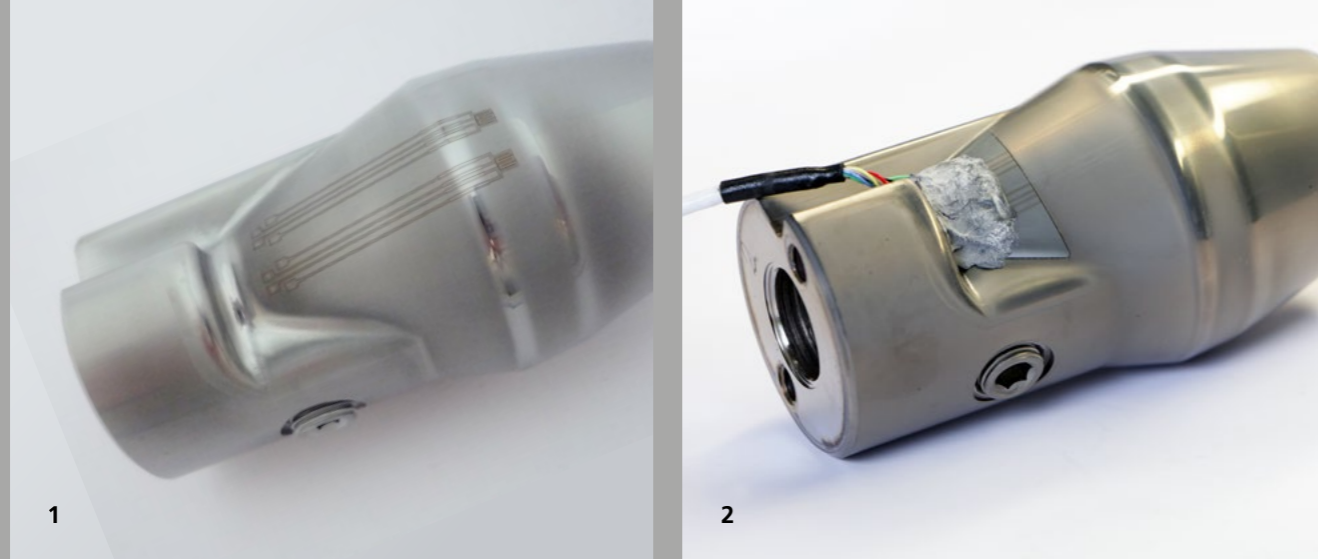
1 SEM cross-sectional image of an a-C:H tool coating with run-in layer.

2 Aluminum adhesions on an a-C:H coating without a run-in layer.

3 No adhesions on an a-C:H coating with a run-in layer.

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THIN-FILM SYSTEMS FOR TEMPERATURE MEASUREMENT IN DIE-CAST ALUMINUM

During a die-casting process the molds are subjected to enormous forces and temperatures. Until now it was not possible to obtain data such as temperature curves, which are necessary for a better understanding and above all for allowing adjustments to be made with regard to process simulation. Placing sensor elements as close as possible to the surface of molds in order to achieve a better process control has therefore been a priority objective of the industry. In a project initiated and commissioned by Volkswagen AG and conducted in collaboration with the Fraunhofer IST and G. A. Röders GmbH & Co. KG, success has been obtained in implementing thin-film sensor systems as a coating directly on the mold surface.

Production of the sensorized thin-film system

In the development of the thin-film sensor system on the mold surface, the first step is to add an area to the mold cores for aluminum die-casting which will be used for contacting the measurement wires. Each mold core is equipped with two sensor structures. By means of physical vapor deposition (PVD), the insulation layer of alumina (Al_2O_3) is now added. The next step is to add a homogeneous coating with a chromium (Cr) layer 250 nm thick. This chromium layer is structured with a meander design. To do this, a photoresist is applied manually, exposed with a flexible mask and developed. The unprotected contour areas are etched away wet-chemically after development. Finally, the photoresist is removed and the metallization with sensor structures is left on the surface (see Figure 1). The sensor structures are designed in four-wire technology. The individual conductors are 100 μm wide, increasing to 250 μm locally in the curved transition.

Following application of the sensor structures, the second insulation layer of Al_2O_3 is deposited, with the contacting areas given an additional layer of copper (Cu) 1.5 μm thick.

The wear-resistant coating of chromium vanadium nitride (CrVN) is added in the next step. Finally, the measurement wires are soldered to the contact pads and the whole area sealed. A compound is used for this which adheres well, even at temperatures above 573 K, and is not electrically conductive. Figure 2 shows such a sensorized mold core. The chromium meander structures have a temperature coefficient of $1.24 \cdot 10^{-3} \text{ K}^{-1}$.

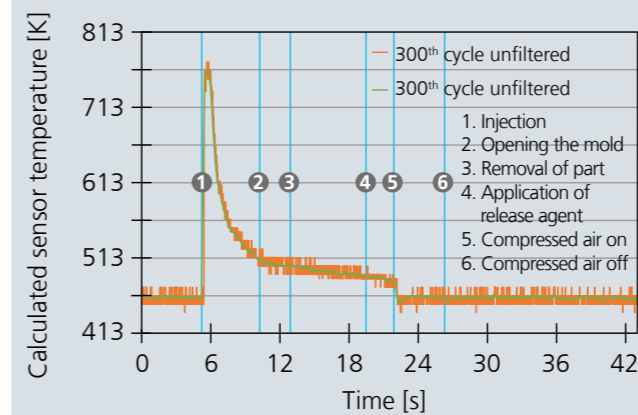
Verification of functional capability

In order to verify the functional capability of the mold core, it was installed in one half of a die-casting mold, with the measurement cable being taken out laterally. The sensor system consisting of temperature meanders made of chromium makes it possible to realistically reproduce the course of temperature development at the mold surface during the die-casting process. The coating system is functionally capable throughout the 300 die-casting cycles and even after this number of cycles no flaking or loss of sensor quality was detected. The graph opposite documents the course of the 300th die-casting run conducted with the sensorized mold core.

Outlook

This tested technology will in future be used with further molds of complex geometry. One objective here is to improve our understanding of aluminum die-casting processes to create the conditions for optimized resource-efficient production.

Temperature curve for the 300th cycle, measured with a chromium meander on the mold core.

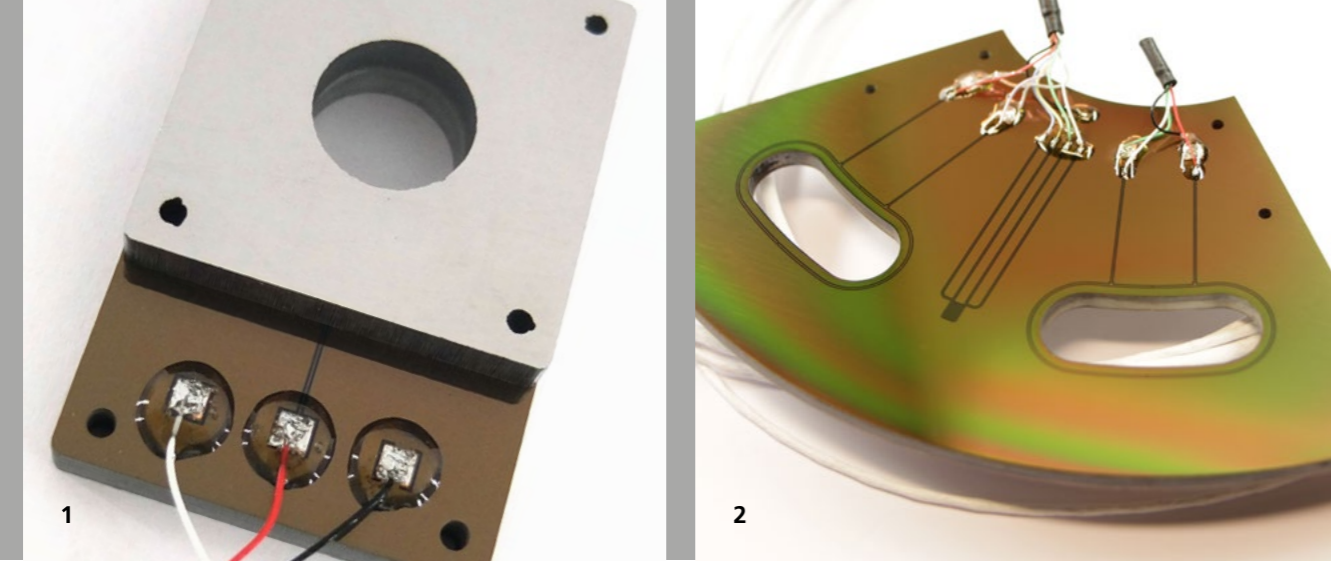


1 Mold core with two sensor structures on the surface.

2 Mold core with measurement wires soldered to the two sensor structures. The soldered area has been sealed to make it possible to carry out measurements even at temperatures above the melting point of lead-free solder.

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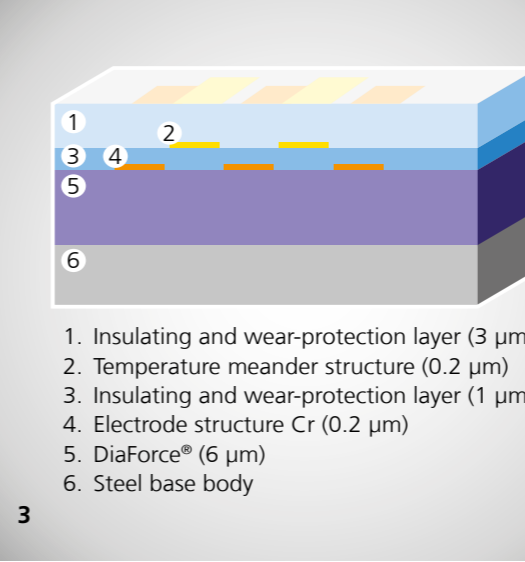
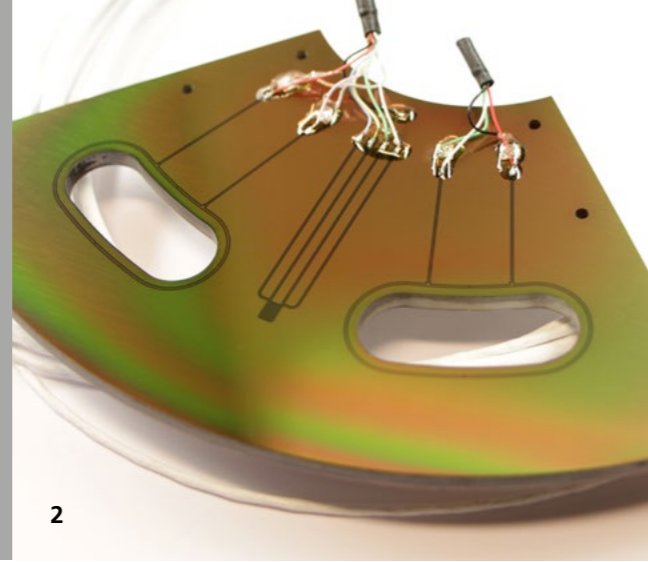
ULTRA-HIGH-PERFORMANCE SENSOR-CONTROLLED MOMENT CONNECTIONS

In timber constructions, post-and-beam connections are often used for bracing the building. However, the requirements made of these connections differ from case to case. When exposed to wind or relatively weak seismic events these connections must, for example, be as stiff as possible to minimize any resulting deformations. In the case of strong earthquakes, on the other hand, soft connections are advantageous as then, due to the possibility of deformation, no critical stresses can build up – although the building may vibrate, it does not collapse. In order to be able to monitor building structures, various modules with sensorized thin-film systems were constructed at the Fraunhofer IST which, installed in newly developed moment connections, can in direct frictional contact pick up the loads and temperatures occurring.

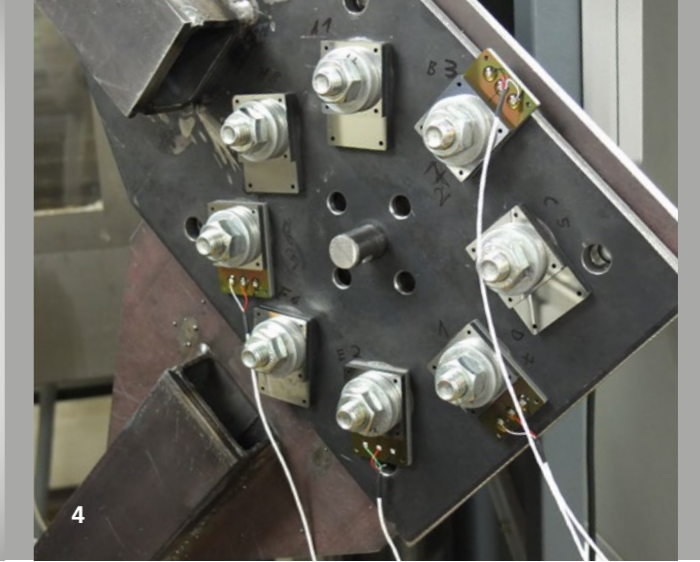
Multifunctional coating system

The sensor modules shown in Figures 1 and 2 are equipped with a multifunctional coating system on their surfaces which not only has a high wear resistance but also is equipped with piezoresistive and, in some cases, even additional thermoresistive sensor structures (see Figure 2). A diagram of the structure of the multifunctional coating system is shown in Figure 3.

The sensorized coating system is based on a development of the Fraunhofer IST, the piezo resistive amorphous hydrocarbon layer DiaForce® (5), deposited homogeneously on the polished substrate in a layer 6 µm thick. Chrome electrode structures are applied to this sensor layer in a layer 200 nm thick. This in turn is covered with an insulating intermediate layer of silicon- and oxygen-modified hydrocarbon SiCON® (d ~ 1 µm) (3). The conductor tracks and the area of the contact points are made of chromium deposited on this intermediate layer with a thickness of 200 nm (2). If local temperature measurement is wanted, additional meander structures, also made of chromium, are added for this purpose (see Figure 2). Finally



1. Insulating and wear-protection layer (3 µm)
2. Temperature meander structure (0.2 µm)
3. Insulating and wear-protection layer (1 µm)
4. Electrode structure Cr (0.2 µm)
5. DiaForce® (6 µm)
6. Steel base body



these structures are insulated with a further layer of SiCON® (d ~ 3 µm) (1) and protected against wear. The total thickness of the coating system is only around 10 µm. Vacuum-coating methods alone are used to create the layers: plasma-assisted chemical vapor deposition (PACVD) for the amorphous hydrocarbon layers of DiaForce® and SiCON®, and physical vapor deposition (PVD) for the electrode layers of chromium.

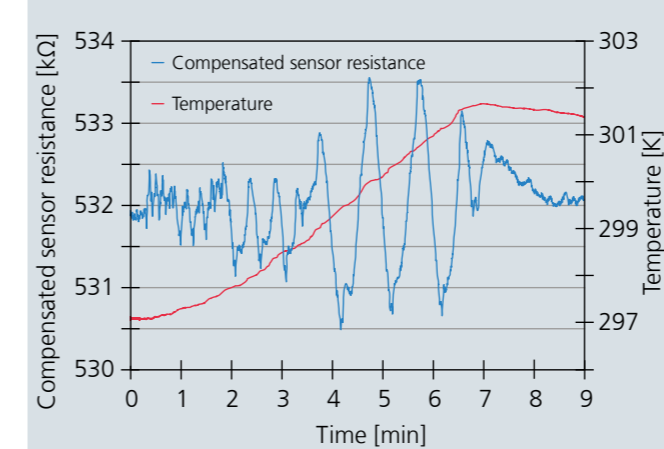
Use of the sensor modules in the experimental set-up

In order to investigate the load-bearing and deformation behavior of these innovative sensor modules, they were installed in a moment connector and, in a test rig of a project partner, the Institute of Building Materials, Concrete Construction and Fire Safety (IBMB) of the Technical University of Braunschweig, subjected to cyclic loading according to DIN EN 12512. The opposite graph shows the measurement curves for a force and temperature sensor. The shape of the force sensor curve clearly reflects the cyclic stresses. The temperature increase of 4 K which may be observed is due to the energy dissipated by friction.

The project

The sensor modules for moment connections with ultra-high-performance capability were produced in collaboration with Pitzl Metallbau GmbH & Co. KG and the Institute of Building Materials, Concrete Construction and Fire Safety (IBMB) of the Technical University of Braunschweig with support from the Fraunhofer Institute for Wood Research, the Wilhelm Klauwitz Institute WKI, and within the KF 217881 project "Development of sensor-controlled moment connections with ultra-high-performance capability, with a great energy-dissipation potential for wooden buildings in earthquake zones", which was funded by the ZIM program ("Central Innovation Program – SMEs").

Example of measurement results from piezo and thermo-resistive sensors in the multi-functional coating system.



1 Square sensor module with annular force-measuring sensor structure.

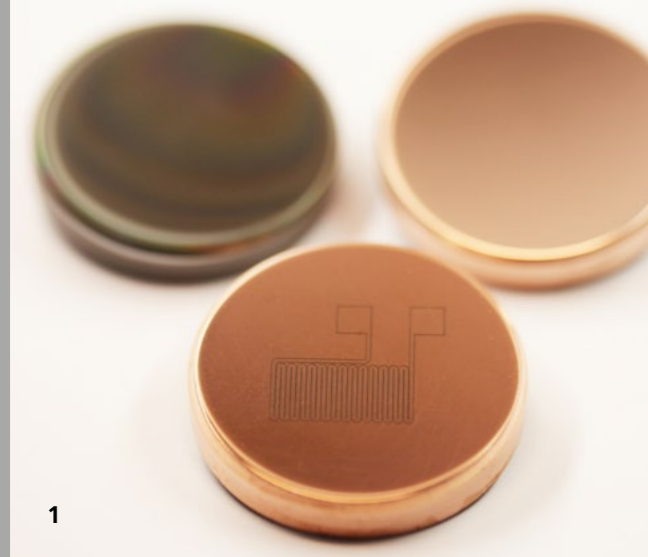
2 Sensor element with force-measuring sensor structures and additional meander structure for local temperature measurement.

3 Schematic of the multi-functional coating system.

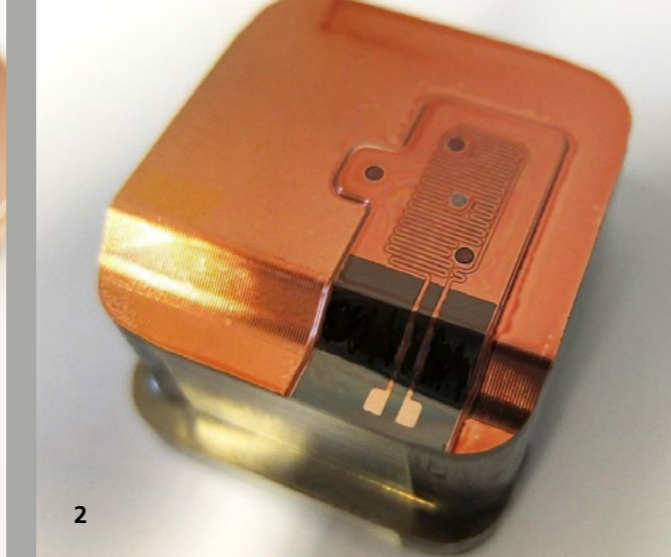
4 Arrangement of the individual sensor modules in the moment connector.

CONTACT

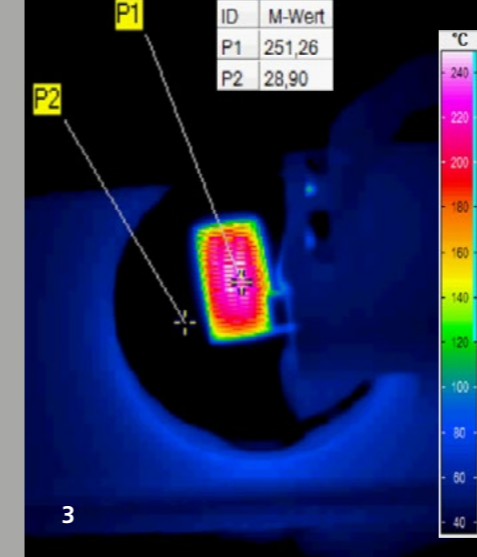
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3

HEATING CONDUCTORS FOR LOCAL TEMPERATURE CONTROL OF MICRO-SIZED PLASTIC INJECTION MOLDS

In the production of moldings by the injection of thermoplastic, thermosetting or elastomeric materials, flaws or defects can arise in the surface quality of the plastic molding. In addition, defects also frequently arise when the heated material solidifies unevenly due to contact with the colder mold wall, thereby reducing its flowability too rapidly. Since the melt by cooling too rapidly does not fill out all mold cavities, the production of optical molded parts, the accurate reproduction of microstructures, or the production of thin-walled parts is, for example, not possible. This represents a major problem especially in the field of miniature and micro injection molding. The Fraunhofer IST is therefore pursuing the approach of using thin-film heating elements specially developed for use in the injection mold in order to heat directly the area where the melt is in contact with the mold wall and thus achieve a high quality of contour reproduction accuracy during the injection-molding process.

Preparation of the sensorized thin-film system

At the beginning of the development process, the first step was to construct base bodies from ceramic material with heating structures made of copper. In a second step, steel base bodies were then used which were coated homogeneously with insulating layers of Al_2O_3 , afterwards heating structures were added. Both the insulation layers and the copper layer were deposited by the physical vapor deposition process. During the development process, heating structures with different layer thicknesses and designs were created at the Fraunhofer IST. An example of a meander-shaped heating structure is shown in Figure 1. It was found that significantly higher heating output levels could be achieved with the more complex meander design. Figure 2 shows a first micro-sized

injection mold with a heating structure of this kind. A new structuring process was developed for production, consisting of a combination of photolithography, laser structuring and wet chemical etching.

Verifying the functional capability of the heating structures

In order to verify the functional capability of the heating structures, they were provided with electrical contacts at the Kunststoff-Zentrum in Leipzig gGmbH (KUZ) and power applied to them in a test rig (see Figure 3). Here a high dynamic in the heating rates was obtained as a function of the resistance achieved, depending on the design of the heating structure. Accordingly, a temperature increase of 100 K from a tempera-

ture level of 373 K to 473 K was, for example, possible within a second at an output of approx. 120 W (energization with a protective extra-low voltage of 30 V). In a second test set-up it was also possible to carry out successful long-term tests with the aforementioned parameters involving around 60,000 test cycles. No damage to the layer was detected, even after the long-term tests. This indicated general suitability for the first utilization of heating layers under exposure to injection molding stresses.

Outlook

In the next step the Fraunhofer IST, in collaboration with the Kunststoff-Zentrum in Leipzig gGmbH (KUZ), aims to provide the heating structures with a final abrasion-protection coating and to investigate the long-term stability of the structures. As part of the "R&D funding of non-profit external research institutions in eastern Germany (INNO-KOM-OST) – Market-oriented research and development module (MF)" this project will continue to be supported by the Federal Ministry of Economics and Technology (BMWi).

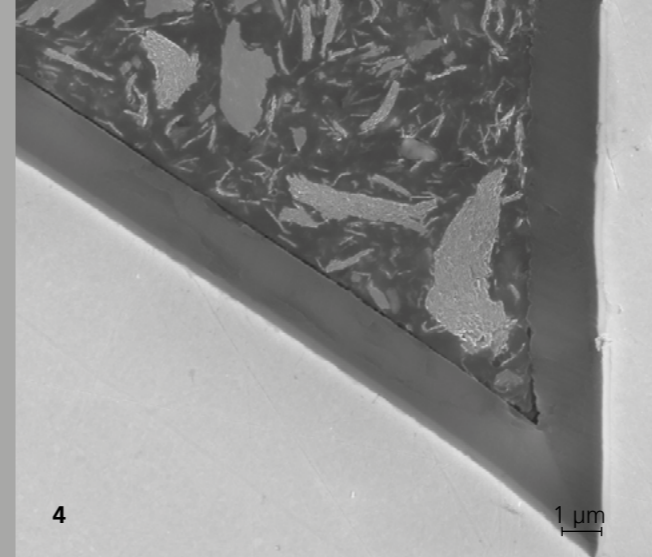
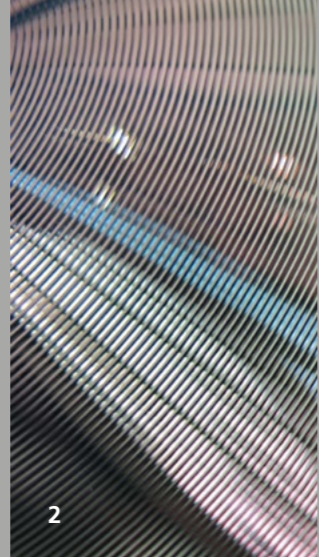
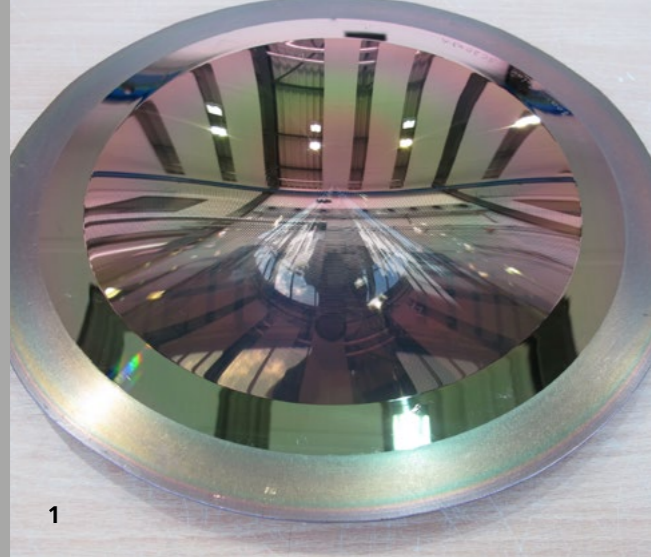
1 Flat base bodies showing the process steps: steel coated with Al_2O_3 (back left); with insulation and copper layer homogeneously deposited on it (back right); with meandering heating structure (front).

2 Micro injection mold with copper heating structure.

3 Thermographic image of a meander-shaped heating structure connected to a 40 V voltage, thereby producing a thermal output of 180 W (temperature rise of 170 K in 3 s from 353 K to 523 K).

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ANTI-ADHESIVE COATINGS WITH HIGH CONTOUR FIDELITY FOR PLASTIC MOLDING

Plastic products with defined and complex shapes and structures can be produced by injection-molding and hot-embossing methods. For the efficient production of large quantities, short cycle times are required. The tendency of many polymers to adhere to the mold surface does however restrict production as it makes demolding the plastic parts more difficult. This results in longer cycle times, compromises in process control and limited design options for feasible structures and surface finishes. In many cases release agent must be used in order to facilitate demolding or even allow it at all. External release agents are applied to the mold between cycles thereby increasing the cycle time. Internal release agents are added to the polymer and can adversely affect material properties and also represent a considerable cost factor. Furthermore, in some cases no suitable release agents are available for special molding compounds. The Fraunhofer IST is here working on cost-effective coating solutions which reduce cycle times, facilitate demolding and increase quality.

Optimized non-stick coatings for structured molds

The basis of the alternative solution proposed by the Fraunhofer IST are wear-resistant, anti-adhesive coatings which are applied to the mold surface. The coatings may not only develop low adhesion forces in interaction with the polymer, but must at the same time offer good durability for use on the mold. An additional challenge in the coating of complex shapes is their contour fidelity.

Microstructures with steep edges and high aspect ratios are increasingly required for medical and optical components. For example, fine microfluidic channels need to be molded in the production of lab-on-a-chip systems. Flat and compact optical components such as are used for head-up displays or concentrator solar cells can be fabricated with so-called Fresnel structures on the mold. For the function and efficiency of these and many other components made of plastic, precise

replication of the finest structures is necessary. Moreover, the anti-adhesive coating must follow the mold contours if the tight manufacturing tolerances are to be complied with.

From development to industrial practical trials

Within the framework of the AiF project "Contour-accurate anti-adhesive coatings for microstructured molds for efficient plastic molding processes" the Fraunhofer IST has developed PACVD processes for coating complex structured molds with high contour accuracy. The basis for this is constituted by modified DLC coatings (a-C:H:X, X = O, Si, F) with low surface energy. Subsequent investigations of these coatings carried out at the Fraunhofer Institute for Structural Durability and System Reliability LBF in Darmstadt demonstrated that a SICON® coating (a-C:H:Si:O) enables demolding forces in the injection molding of polycarbonate to be reduced by up to 90 percent. The first industrial practical trials on structurally

adapted modified DLC mold coatings conducted in collaboration with the ORAFOL Fresnel Optics company have already produced some promising results. With these innovatively coated molds, Fresnel lenses can even be made from a special polymer whose marked tendency to adhere prevents processing in the conventional way. Optical efficiency measurements indicate that a high contour fidelity and production-level quality can be achieved with the new manufacturing approach. These development results demonstrate the high potential for application of modified DLC coatings as a mold coating in the plastics processing industry.

Outlook

These very promising coating systems and processes are being further developed for various applications in plastics processing and for additional polymer systems. Important objectives are the coating of molds with larger dimensions and the optimization of mold surfaces, including coating systems for the production of high-quality plastic products.

1-2 Mold coated with SICON® and used for the production of Fresnel lenses.

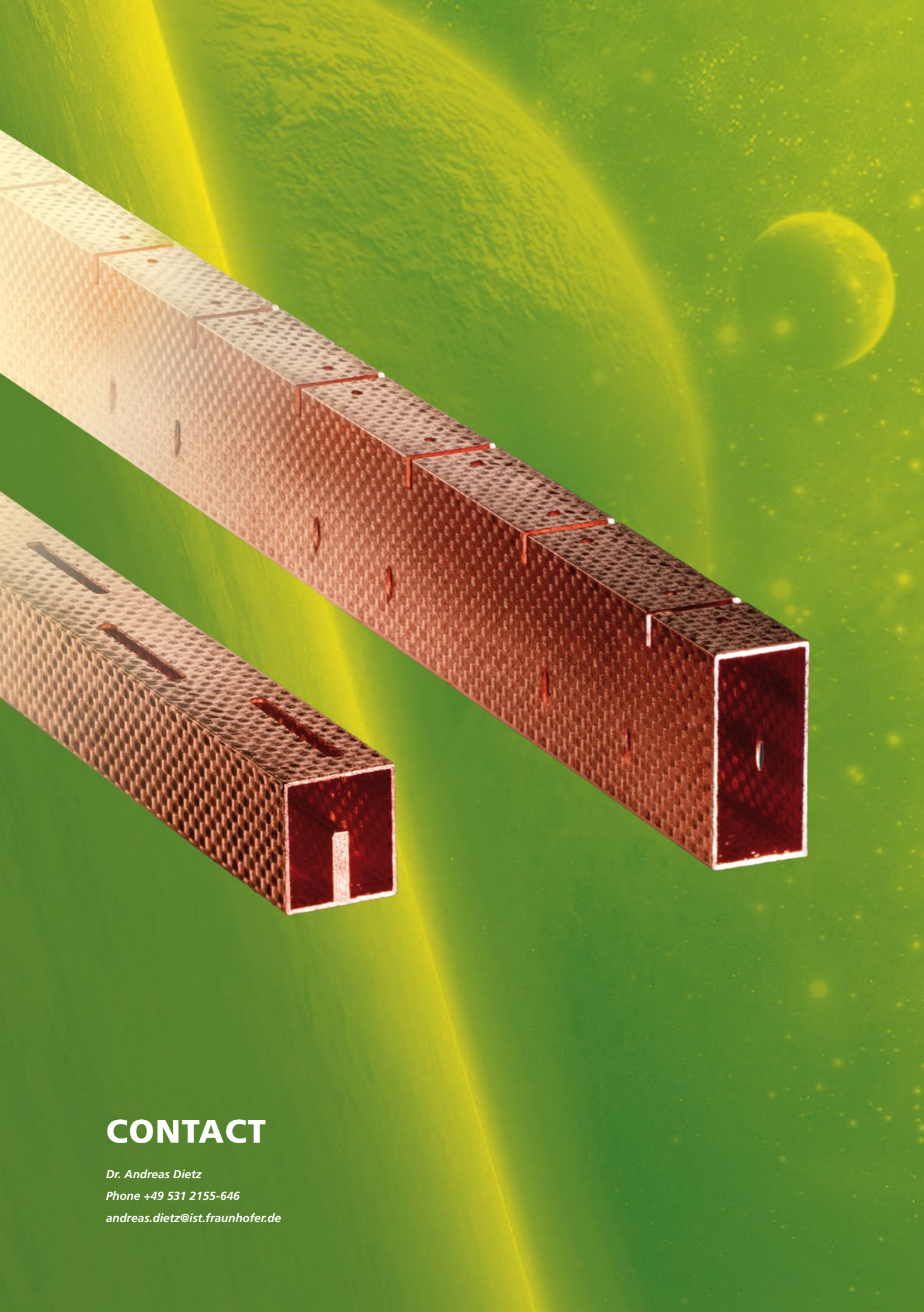
3 Fresnel lens made of plastic, produced with a coated mold.

4 Structure-adapted SICON® coating on a mold.

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AEROSPACE

In this business unit, coating technologies are developed for the aerospace sector. The focus is on functionalizing lightweight materials such as carbon fiber reinforced plastics (CFRP) or light metals. In addition, coating systems are developed for optical applications, in particular for special precision filters for space missions.

Currently the Fraunhofer IST is working on the following projects:

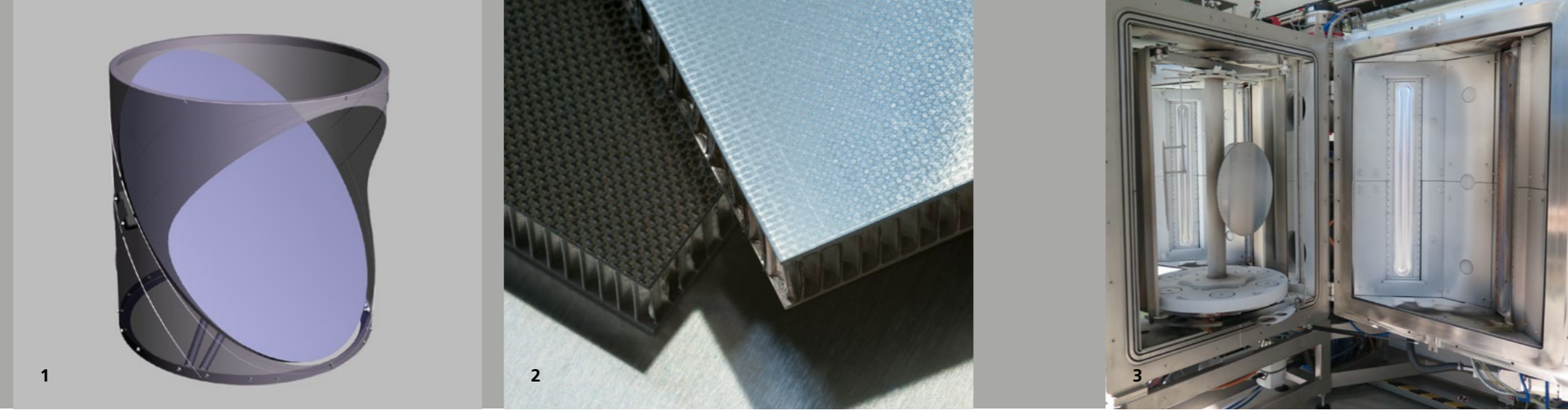
- | Electroplated metallization of CFRP components
- | Development of new environment-friendly CFRP metallization methods

- | Surface treatment of light metals e.g. titanium, magnesium, aluminium
- | Wear-protection coatings for engines in jet aircraft
- | Bearing sensor systems for condition monitoring in aircraft
- | Development of surfaces for molds free from release agents
- | Development of coating processes for precision lenses such as filters

Customers include companies from the aerospace sector as well as their suppliers.

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FUNCTIONAL COATINGS FOR WEATHER SATELLITES

Precise and reliable weather forecasts can save lives and help to mitigate the global impact of weather disasters. For this reason, Europe's second generation of weather satellites – MetOp-SG – is equipped with various innovative high-performance research instruments. Accordingly, an improved highly sensitive radiometer for measuring microwave terrestrial radiation is to be used to determine air temperatures and water vapor concentrations at different altitudes. In addition to Airbus DS and Invent GmbH, the Fraunhofer IST is also a participant in this development.

Innovative, highly sensitive radiometer

At the heart of the radiometer for measuring microwave terrestrial radiation are innovative microwave reflectors made of a special composite material, a sandwich of carbon-fiber-reinforced plastic (CFRP) and aluminum honeycomb structures, which is characterized by a very low weight and at the same time a high rigidity. Critical to the function of the reflectors are suitable metal coatings, which allow an efficient microwave reflection. At the Fraunhofer IST the necessary coating systems and coating processes for CFRP components with dimensions of up to one meter are under development.

CFRP metallization by magnetron sputtering (PVD)

The challenge in metallizing the CFRP components is to lower the process temperature into an acceptable range for the CFRP composite material and at the same time achieve good bonding of the metal coating. The process developed at the Fraunhofer IST includes a wet-chemical cleaning procedure for the CFRP components, plasma pretreatment and the actual coating deposition by magnetron sputtering. Reflectivity measurements with a positive result and the first successful system tests with a coated reflector have already been carried out.

Silicon oxide protective layers (PACVD)

Since the reflectors are exposed to changing, in some cases extreme, environmental conditions during the course of their mission – from ground-launching of the rocket to arrival at the target orbit – it may be necessary to provide the metal coating with corrosion protection as well. This must not compromise the function of the reflectors. The approach taken at the Fraunhofer IST includes the development of a thin layer of silicon oxide (SiO_x) which is applied by a PACVD process after metallization. In addition to the usual plasma excitation in the kHz range, the use of microwave plasma sources in the GHz range is being evaluated. The goal is to provide a new coating process by more intensive plasma activation and the degrees of freedom gained in process control which optimally satisfies the various requirements deriving from the special CFRP composite.

Reflector coating with hybrid processes

For the combination of metal and SiO_x deposition in a hybrid process and for coating the reflectors, an industrial coating installation is available at the Fraunhofer IST which can handle components with a dimension exceeding one meter. At the

collaboration partners Airbus DS, the new CFRP reflectors with the functional layers provided by the Fraunhofer IST initially undergo a qualification program and are subsequently to be produced for service in orbit. Data for the weather forecasting systems are expected to be ready from 2021.

The project

The contributions of the Fraunhofer IST to coating development and application to the satellite components fall within the framework of a project with partners from industry and science. The Airbus DS company is the central coordinator in the production of the high-performance research instruments for Europe's new generation of weather satellites. The CFRP composite reflectors to be coated by the Fraunhofer IST are manufactured by Invent GmbH.

1 An elliptical microwave reflector with a dimension of about 600 mm.

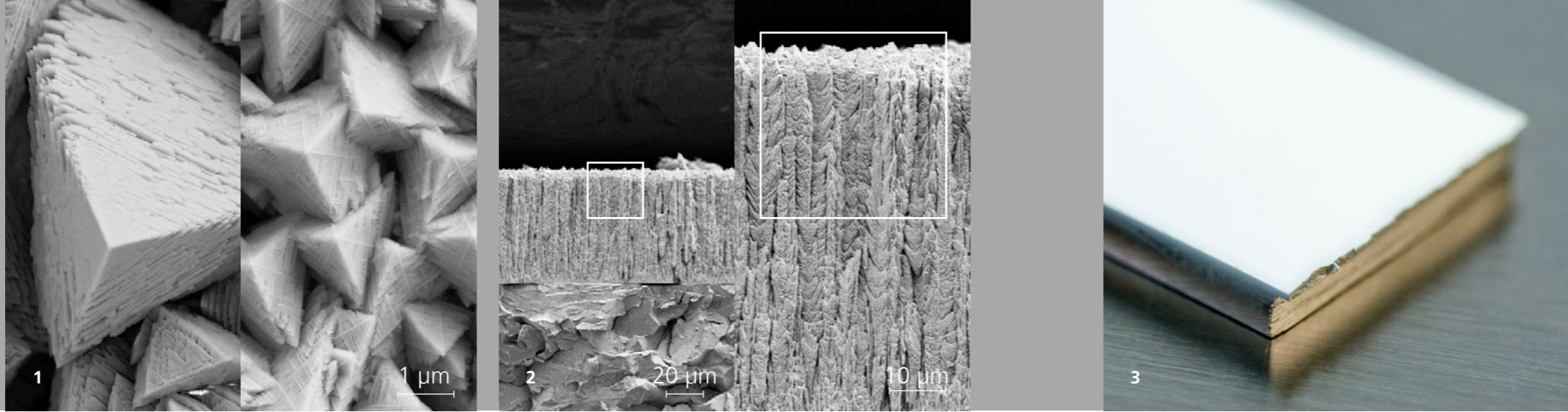
2 Special CFRP composite material for the manufacture of microwave reflectors. Left: material in a raw state. Right: material after metallization and SiO_x coating by a PVD+PACVD hybrid process.

3 Industrial PVD+PACVD hybrid system with magnetron sputtering and microwave sources. Possible component size > 1 meter.

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GAS-FLOW SPUTTERED THERMAL BARRIER COATINGS

For a more efficient operation of gas turbines at high temperatures, the metallic materials used must be protected against hot-gas corrosion and overheating. Nowadays the ceramic thermal barrier coatings employed as protection in such cases are produced by electron beam evaporation. However, novel and promising ceramic thermal barrier coatings for highly stressed components can also be produced with the gas-flow sputtering technique (GFS) developed at the Fraunhofer IST.

Thermal barrier coatings for use in gas turbines

The efficiency of a gas turbine can be significantly increased by having a high gas inlet temperature. Components in the combustion chamber of a turbine are therefore exposed to very high temperatures which even high-quality materials cannot withstand. However, with the aid of a combination of active film cooling and thermal barrier coatings, high gas temperatures of approximately 1400 °C are possible without compromising the service life.

Thermal barrier coatings usually consist of partially yttria-stabilized zirconia (PYSZ). These coatings are normally produced by thermal spraying processes such as atmospheric plasma spraying (APS) or electron beam evaporation (EB-PVD), with each method creating different microstructures. So-called columnar microstructures, as produced by electron beam evaporation, have a longer life under cyclic loading than the lamellar microstructures of plasma-sprayed coatings. An alternative production method is gas-flow sputtering, a high-rate sputtering technique developed at the Fraunhofer IST which also creates columnar microstructures with a high internal porosity.

Heat transfer through such thermal barrier coatings depends on the one hand on the material and on the other on photon and phonon conductivities. The latter are further reduced by internal interfaces which is why a specific setting for the porosity can have a positive influence on the heat resistance.

Gas-flow sputtered thermal barrier coatings

At the Fraunhofer IST, deposition parameters, which have a major influence on the resulting microstructure of the zirconia coating, were determined and investigated. With the gas-flow sputtering method, fully and partially stabilized zirconia coatings of the most diverse structures were successfully deposited on a high-temperature resistant FeCrAlY alloy. These oxide coatings, deposited at substrate temperatures in the 500 °C to 800 °C range, were characterized and tested for their suitability as a thermal barrier coating. A total of four different microstructures with different intercolumnar porosities were identified. The column diameters can be influenced by an applied bias voltage. In addition, a sufficiently high bias compresses the sputtered layer and thereby influences the mechanical stiffness of the coating and its internal stress. Another process parameter in addition to the bias voltage is the flow of

oxygen that is provided during growth, which can change the density and the preferred orientation of the resulting coating structure.

Thermal cycling tests up to a temperature of 1050 °C and up to 1300 cycles proved that the life of gas-flow sputtered coatings depends very markedly on layer stiffness and the tendency to sinter. Sintering tests with highly feather-like columns continued to reveal a high coating porosity even after a sintering temperature of 1200 °C, thus promising a low heat transfer. In addition, investigations of highly rigid layers indicated sintering tendencies between the individual columns which result in segmentation cracking. On the other hand, coatings of low rigidity do not form cracks of this kind but are rather less resistant to buckling and a subsequent spallation of the coating.

Outlook

Further investigations into the mechanisms described which limit service life are currently in progress. Depending on the selection of parameters, many of the analyzed coatings have already shown a promising durability in the tests and thus appear suitable for use as thermal barrier coatings. Assuming a rational parameter selection a balance of stiffness and the sintering tendency should be found in future so as to deliver the longest possible service life for the coatings coupled with good thermal insulation.

1 The structures of the GFS thermal barrier coatings differ according to the deposition temperature (top view).

2 Columnar microstructure of the GFS thermal barrier coatings (side view, fracture).

3 Fracture edge of the thermal barrier coating on a FeCrAlY alloy prepared for examination of the microscopic structure.

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ENERGY AND ELECTRONICS

In the "Energy and electronics" business unit the work of the institute concentrates on the following developments:

- | Functional coatings or coating systems and coating processes for architectural glass (low-E coatings, active or passive heat and sun protection, switchable electrochromic glazing)
- | Transparent conductive coating systems (TCOs) for architectural and automotive glazing, for solar cells, displays and invisible heating elements and also for solar thermal energy
- | p- and n-type TCOs as materials for transparent and flexible electronics
- | Semiconductor layers for thin film and silicon-based photovoltaics and also characterization methods for thin-film solar cells
- | Electrical contact and insulating layers, as well as barrier layers

- | (Local) plasma treatment of surfaces for wafer bonding, structured metallization and metallization of temperature-sensitive and complexly shaped substrates
- | Stable anodes and cathodes for lithium-ion batteries
- | Electrolytic coatings for high-temperature fuel cells (SOFC) and gas separation membranes for hydrogen production
- | Corrosion-protection and thermal-barrier coatings for high-temperature applications, such as in gas turbines

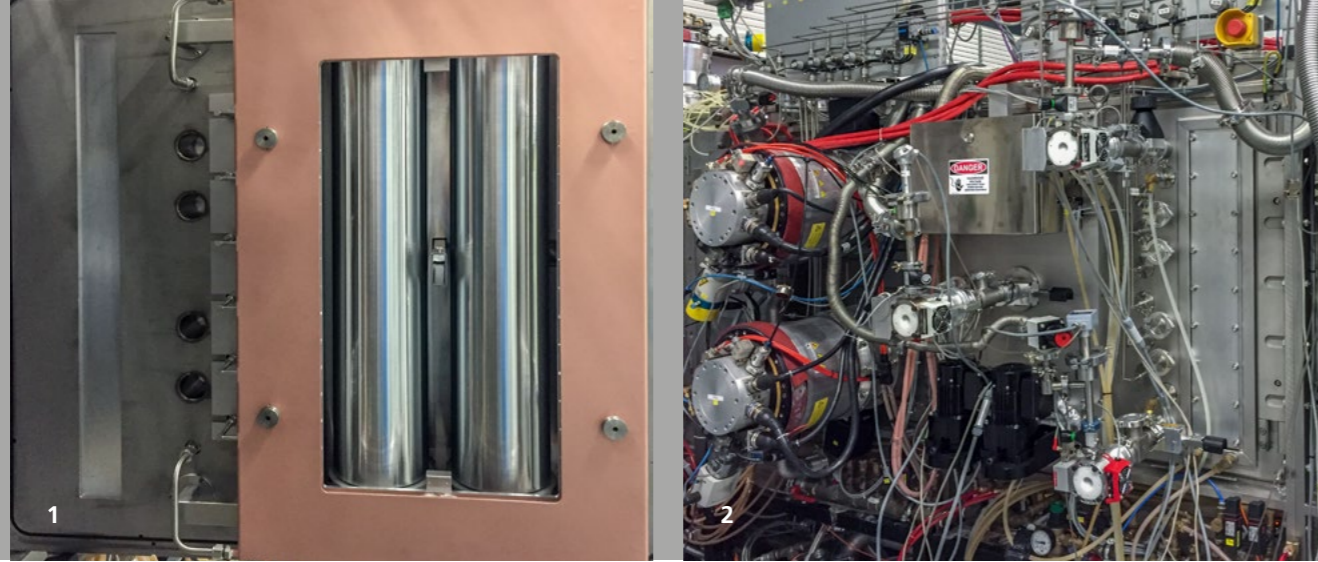
Our customers include companies in the glass, photovoltaics and automotive industries, in semiconductors and microelectronics, in the information and communications sectors, in the energy and construction industries, and also plant manufacturers and contract coating companies.

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REACTIVELY SPUTTERED ZnO:Al WITH THE DOUBLE-TUBE MAGNETRON

Transparent conductive films (TCOs) are required as front contacts in the production of thin-film solar cells. The TCO production processes used on the industrial scale today are normally based on so-called DC sputtering from ceramic tube targets. However, to improve the competitiveness of German companies both a more efficient coating process – with lower material consumption and lower costs – and a greater effectiveness and longer service life is required. As part of the »TCO4CIGS« project funded by the BMWi (the Federal Ministry of Economics and Technology) a double-tube magnetron module has been successfully constructed for this process and tested.

The Fraunhofer IST's approach to a solution

The Fraunhofer IST has at its disposal a reactive sputtering process with metal targets that has the potential to replace the conventional DC sputtering process that uses ceramic targets. For this the following developments were necessary: firstly, an upscaling, and secondly, with the aid of a double tube, the integration of this reactive sputtering process into a production process for thin-film solar cells suitable for industrial application.

In upscaling the reactive sputtering process for ZnO:Al to a double-tube magnetron the institute was able to draw on years of experience. For some years now, reactive MF processes have been carried out at the institute with its planar double cathodes. In this particular case, the amount of oxygen required for the deposition of a stoichiometric film is supplied via the gas feed. At the same time, metallic Zn:Al targets are used, whereby transparent oxides based on ZnO:Al are created and deposited in the reactive sputtering process. To ensure a stable process along the entire target (even for a relatively long period) and thereby secure uniform deposition of oxides, multiplied measures were taken. Firstly, a shared gas distributor as well as a specific gas supply were designed and integrated.

Second, a suitable shutter environment for the double tube was fabricated. Third, a short-time process control in the milliseconds range was developed, and finally, a gas-flow quadrature regulator in the seconds range was introduced.

Results of ZnO:Al production

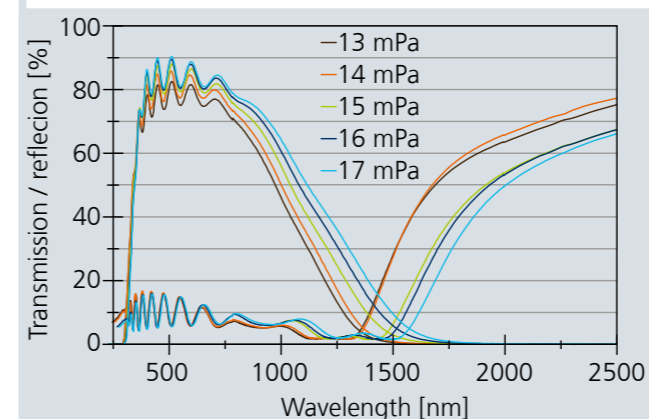
Zn:Al tube targets with an aluminum concentration of 1.75 wt.% were used in the production of the transparent oxide layers. With the aid of a lambda sensor closed-loop controller the oxygen partial pressure was stabilized at a firmly retained value by changing the power supplied. The graph opposite shows the transmission and reflection spectra of ZnO:Al films deposited on float glass for the specified values. The coating process took place at a substrate temperature of 200 °C. It can be seen that as the oxygen partial pressure rises, the charge carrier density falls. This is mainly due to a further oxidation of the aluminum but also to a reduced zinc evaporation from the substrate with increasing amount of oxygen because of the low vapour pressure of zinc. Along with the reduction of the active charge carriers, visual absorption also sinks while the specific resistance increases from 350 to approximately 500 $\mu\Omega\text{cm}$ (see the graph opposite). For the data shown, the mobility of the charge carriers varies between 20 and 22 cm^2/Vs .

Outlook

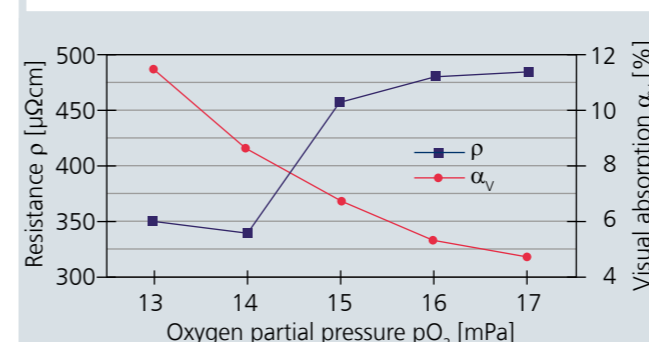
Even when first put into operation, the new double-tube arrangement resulted in outstanding coating properties for the ZnO:Al deposited. Both the target environment and the process itself are to be further optimized in the next step in order to further increase the mobility of the charge carriers. The consequent improvements in film properties, such as reducing the specific resistance while simultaneously improving transparency, should then be transferred to CIGS absorbers.

1-2 Interior and exterior view of the new installation door.

Reflection and transmission spectra measured for coatings with different $p(\text{O}_2)$ values on Borofloat glass.

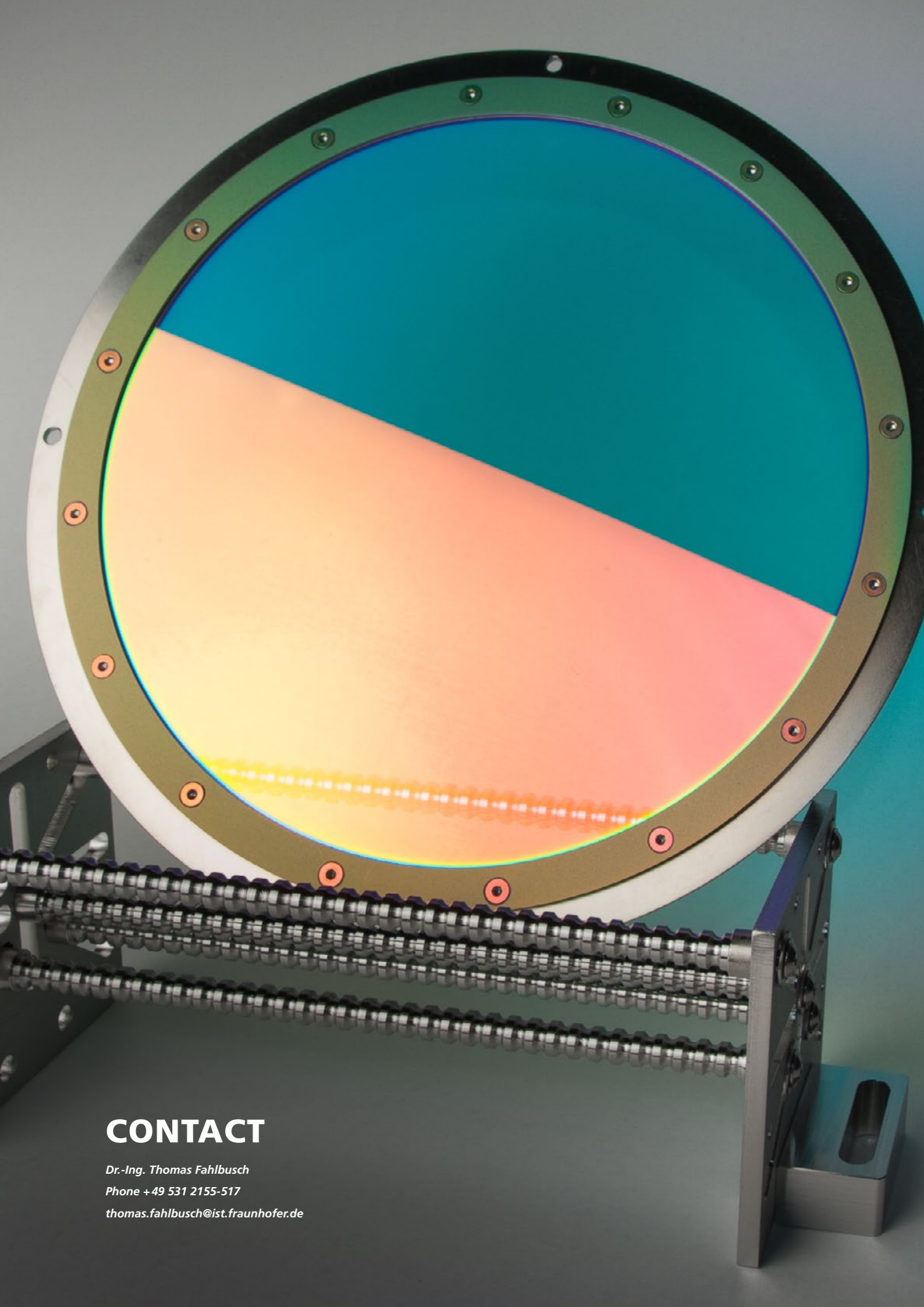


Specific resistance and visual absorption at $p(\text{O}_2)$.



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OPTICS

In its "Optics" business unit the Fraunhofer IST is active with a variety of thin-film technologies in developing new solutions for new industrial applications. These include:

- | The development and manufacture of coatings for optical components
- | Systems for the deposition of high-grade optical coatings on flat and curved lenses
- | The EOSS® production platform for manufacturing optical filters and laser components
- | The development of new materials for intelligent coatings, such as electrically switchable filters
- | Highly durable broadband anti-reflective coatings on sapphire and glass
- | Micro-structured optical filter coatings for imaging applications
- | Optical coatings on plastic surfaces
- | Use of simulation in designing and optimizing coating processes and installations in low-pressure systems
- | Development of innovative transparent conductive coatings for lighting technology and oxide electronics

In the field optical metrology the Fraunhofer IST focuses on the following topics:

- | In-situ monitoring of coating processes with MOCCA®
- | Mapping system for measurement of ellipsometry, reflection, transmission, flare and Raman spectroscopy on 60x60 cm²
- | Defect analysis of optical layers by means of FIB REM and confocal optical microscopy
- | Testing the wear and corrosion resistance of optical surfaces and coatings

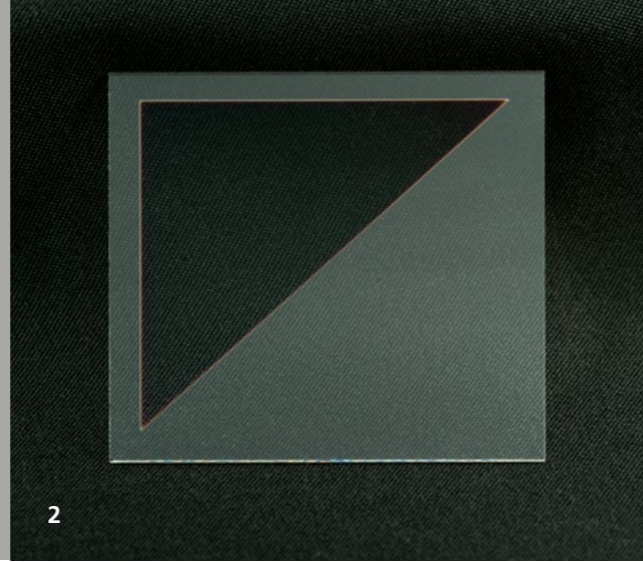
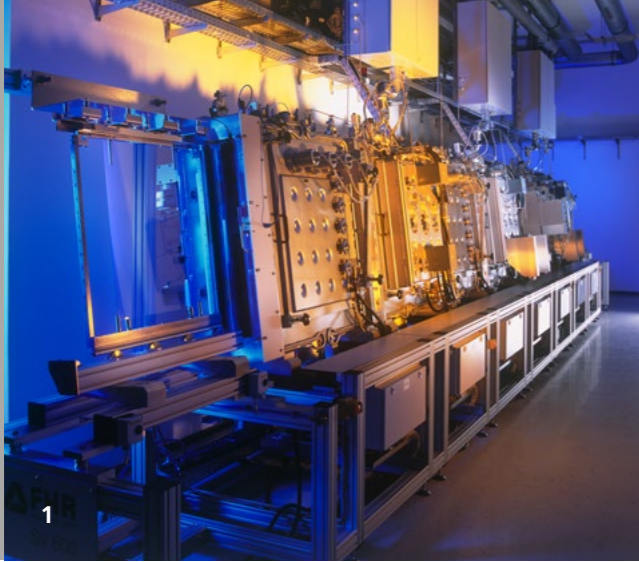
Clients of this business unit include companies in the optical industry, the automotive industry, aerospace, manufacturers of displays and data storage media as well as plant manufacturers and contract coating companies.

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HIGH-RATE DEPOSITION OF SILICON OXIDE FILMS WITH LOW INTERNAL STRESS

Compressive stresses are a major problem in the field of optical coatings. As an example, excessive pressure can bend the base body or reduce the adhesion of the coating, which can ultimately lead to the coating detaching completely. There is currently no established method of reducing the mechanical compressive stresses in the coatings, especially for silicon oxide (SiO_2), the most frequently used material in optics. Normally plasma-assisted deposition processes are used for producing optical films. Although these, as a consequence of bombardment with high-energy ions, can produce high-density layers (corresponding to a high layer stability), high compressive stresses are in most cases also created in the layer. At the Fraunhofer IST, a hot-wire chemical vapor deposition method (HWCVD) has for the first time been evaluated as an alternative production method for SiO_2 layers as part of a DISCOVER project sponsored by Fraunhofer.

Proposed solution

At the Fraunhofer IST the HWCVD process has already been developed for diamond, silicon, silicon carbide and silicon nitride materials for different applications up to industrial implementation. In addition, the HWCVD method allows for the deposition on plastics, with the capability of even depositing nitrides free from internal stresses, under virtually particle-free conditions, and at high rates. The production of oxide layers with this coating technology is however still largely unexplored and yet totally unknown in the optical industry. In the HWCVD process, electrically heated tungsten wires in the 1900 °C – 2100 °C range are used in a vacuum chamber to dissociate SiH_4 . From here oxygen is added to produce the metal oxide. The challenge now is to prevent the hot wires from becoming oxidized by the oxygen, which is essential for oxide formation.

Results of silicon oxide production

In the development of the silicon oxide layers a parameter study on quartz glass was carried out with the aid of a statistical design of experiment (DOE). This took gas composition,

pressures, and temperatures into account. Investigation of highly transparent layers (cf. Figure 1) produced with low compressive stresses revealed the following layer properties: high transparency for $d(\text{SiO}_2)=380 \text{ nm}$ $T_{250 \text{ nm}} > 89 \%$ on quartz, low compressive stress for $d(\text{SiO}_2) 2,5 \mu\text{m}$ $\sigma < 170 \text{ MPa}$ (low internal stress), low surface roughness for $d(\text{SiO}_2) 2,5 \mu\text{m}$ $r_a < 6 \text{ nm}$, coating rates $> 2 \text{ nm/s}$, tungsten contamination $[\text{W}] \leq 0.2 \text{ atom\%}$, and no substrate or layer damage caused by ion bombardment.

Following the parameter study, a low-stress anti-reflective coating system (cf. Figure 2) on a surface area of $10 \times 10 \text{ cm}^2$ was produced at the Fraunhofer IST. It consists of four alternating layers of the high-index Si_3N_4 and of the low-index SiO_2 layers developed (these being deposited by the HWCVD method). The anti-reflective coating has a very low residual compressive stress of less than 50 MPa, in other words, the risk of the coating detaching or of the base body bending can be excluded.

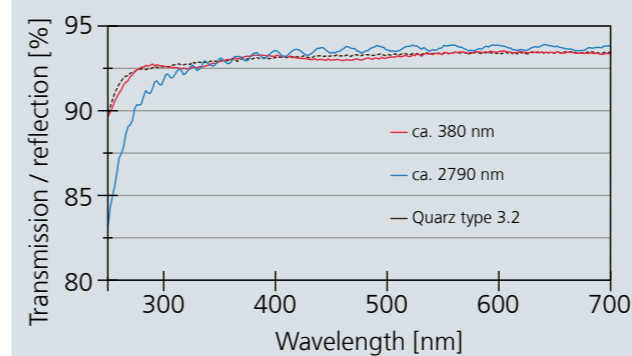
Outlook

Due to the high coating rate coupled with a high optical quality and low internal stresses, final products with a significantly longer service life will be feasible in the future at an attractive cost of production. Some examples of their planned uses are in the production of cell-phone displays, or of automotive interior components, or applications in the field of solar and architectural glass, or also utilization as barrier or decorative coatings.

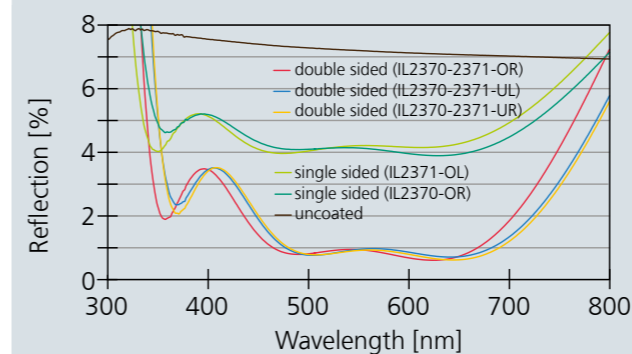
1 Seven-chamber in-line coating installation for the HWCVD method at the Fraunhofer IST.

2 Four-layer anti-reflective coating on glass: $\text{Si}_3\text{N}_4\text{-SiO}_2\text{-Si}_3\text{N}_4\text{-SiO}_2$.

Layer-related transmission for quartz glass and SiO_2 layers.



Reflection behavior of the four-layer AR coating (single- and double-sided) as compared with quartz glass.



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PIXEL FILTERS – MICROSTRUCTURED THIN FILM FILTERS FOR 3D METROLOGY

Microstructured filters are playing an increasingly important role, especially in the field of high-resolution 3D metrology or even in hyperspectral imaging. A new combined process for the production of these so-called pixel filters has therefore been developed at the Fraunhofer IST, consisting of coating and microstructuring. Here several dielectric filters are arranged side by side on a substrate, so that, for example, with the aid of a CCD sensor different wavelengths can be filtered out depending on the pixel element. So far it has been possible to achieve structure sizes of less than 100 μm which can be used both as bandpass filters and as cut-off filters.

Production of the microstructured thin-film filter

The structured filter is created in several alternating coating and structuring steps. The so-called lift-off method is used here: firstly, quartz glass is coated with photoresist (negative resist AZ4562) by the spin process and then structured by photolithography. A regular pattern of square structures with a side length of 400 μm and spaced 100 μm apart is selected.

After structuring, several of the square areas are free of photoresist while the rest of the glass surface is protected with it. In the first coating process which follows, a bandpass filter system consisting of 18 individual layers with a center wavelength of 400 nm is deposited on the exposed areas. This is a layer stack consisting of alternating low-index and high-index coating material, a so-called high-low stack. The two materials are deposited fully reactively by physical vapor deposition: the high-index material consists of niobium oxide NbO_2 , the low-index consists of silicon oxide SiO_2 . The total thickness of the filter system lies in the 2 μm range. Finally, following this coating step, to conclude the first lift-off process

the photoresist mask is removed in the potassium hydroxide (KOH) bath and the square interference filter structures are left on the surface of the glass. A sheet of glass showing the result of this process can be seen in Figure 1.

This process is repeated another three times: in the second coating step a high-low stack consisting of 15 individual layers with a target wavelength of 475 nm is deposited; in the third coating step a stack consisting of 13 individual layers with a target wavelength of 550 nm is deposited; and finally in the fourth coating step a stack consisting of 11 individual layers with a target wavelength of 625 nm is deposited. The result of this sequence of operations is an uniform arrangement of these different filter systems (see Figure 2).

Outlook

These pixel filters have a great industrial potential. Besides use in the field of hyperspectral and multispectral imaging, future examples of application for the filters will also be found in aerospace and in astronomy. In addition, concepts are already

in existence for using imaging metrology to sort waste by material type or for determining the condition of agricultural crops. The Fraunhofer IST is therefore already currently engaged in further development of the pixel filters. This work aims at further reducing the size of the individual pixels, refining the spectral properties of the pixel filter and further simplifying the entire production process, thereby reducing production costs.

The project

The research was conducted within the Fraunhofer's own internal MSE-oriented research project in collaboration with the Fraunhofer Institute of Optronics, System Technologies and Image Exploitation IOSB.

1 Filter system after the first lift-off process.

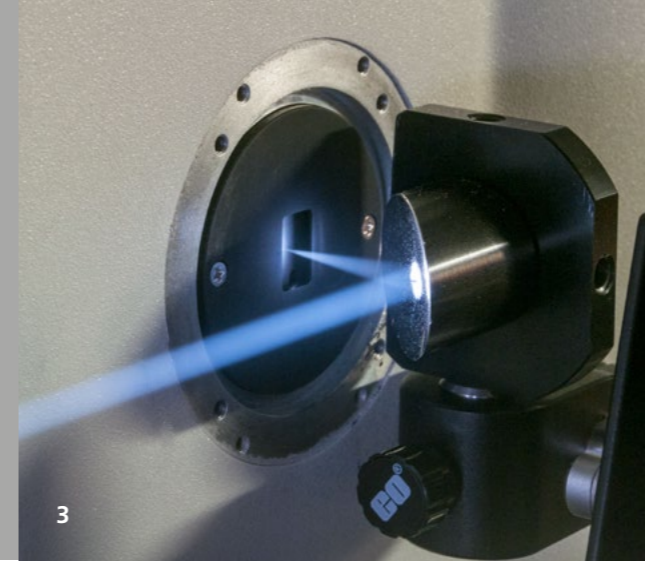
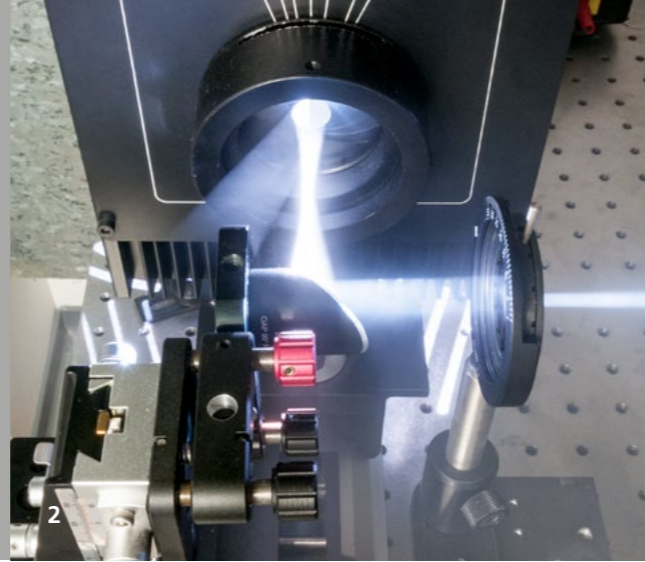
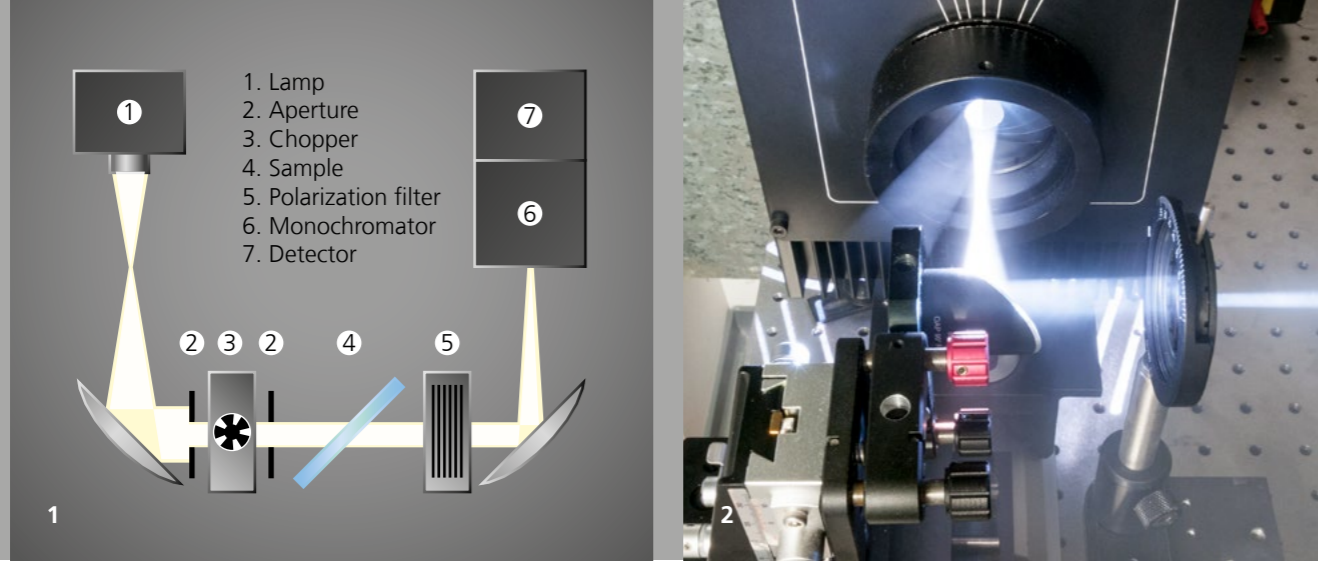
2 Filter system after the fourth lift-off process.

3 Arrangement of several pixel-filter areas on a glass substrate.

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INNOVATIVE SPECTROMETER WITH LOW DIVERGENCE

The interference filters required by industry and technology can be manufactured with the aid of thin-film technologies. The potential applications of these filters range from the simple anti-reflective coating of spectacle lenses, to digital projection technology, and on to special laser applications. However, high demands are often made of the filter spectrum, which means that characterization of the specifications, such as the full-widths at half maximum, is crucial for the quality analysis. Important spectrometer parameters for the measurement of optical interference filters include the spectral bandwidth and the divergence angle of the measuring beam. These two variables influence the width of fine structures in the spectrum of an interference filter and thereby limit the resolution of spectrally steeper edges in conventional spectrometers. Within the framework of the BMBF joint project "DAHLIA" a spectrometer with a smaller angle of divergence has therefore been built at the Fraunhofer IST.

The newly developed spectrometer in the measurement set-up

With the spectrometer featured here, transmission can be measured angle-dependently in a wavelength range of 240 to 1000 nm with an angle of divergence of 0.25° . The divergence angle set is reduced by a factor of 10 in comparison with a commercial spectrometer. In the measurements the monochromator can be set to a minimum spectral bandwidth of 0.06 nm. Since the spectrometer's measuring beam must have a maximum intensity at a low divergence angle for the measurements, a xenon arc lamp is used as the light source with this measurement configuration. In contrast to other light sources, the xenon arc lamp has a comparatively small light-emitting area and a high light density. Via a mirror and aperture system, a polychromatic measuring beam with a low divergence is generated (see Figure 2). After passing through the interference filter the measuring beam is input into a monochromator with a high-resolution diffraction grating (see Figure 3) and picked up by a low-noise detector. The measurement set-up is equipped with a chopper and can thus measure optical densities up to 4.

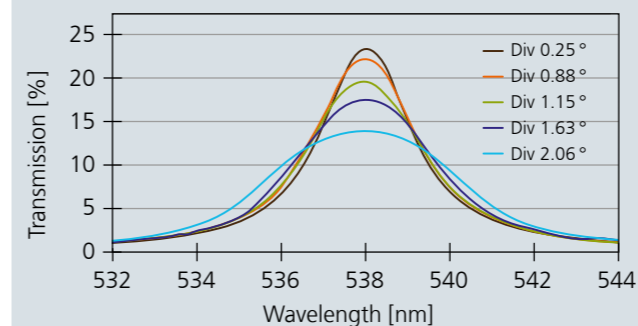
Test measurements with different angles of divergence

In order to show the broadenings of the structures as a function of the divergence angle, a sharp transmission structure of an interference filter was examined more closely. The filter was measured with a spectral bandwidth of 0.5 nm and an angle of incidence of 45° at the divergence angles of 0.25° – 0.88° – 1.15° – 1.63° – 2.06° (see top graph). During examination of the widths of the individual peaks, a quadratic relationship was found between half-width values and divergence angles. The lower graph shows the results from this test. Measuring beams with a small divergence angle were therefore required in order to characterize band-pass filters with small half-widths or long-pass filters with spectrally steep edges.

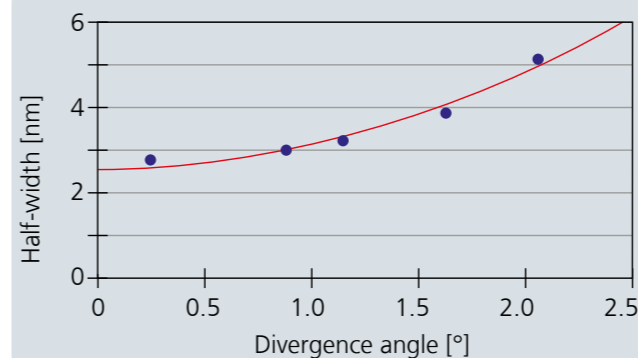
Outlook

The measuring range of optical density is to be extended in the future in order to resolve even lower intensities. This will enable the characterization, among other things, of narrow notch filters with very high blocking ($>OD 7$).

Comparison of the transmission spectra of a sharp structure at different angles of divergence (0.25° – 0.88° – 1.15° – 1.63° – 2.06°).



Comparison of the half-widths in transmission measurements of a sharp structure at different angles of divergence (0.25° – 0.88° – 1.15° – 1.63° – 2.06°).



1 Schematic drawing of the spectrometer with a small divergence angle.

2 The measuring beam from the lamp is collimated with the aid of a mirror and aperture system.

3 Coupling the measuring beam into the monochromator.

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LIFE SCIENCE AND ECOLOGY

In the "Life science" field the Fraunhofer IST develops coatings, processes and equipment for a range of application fields:

Medicine and hygiene

- | Healing support for wounds and infectious skin diseases
- | Antiseptic therapy
- | Dentistry
- | Hand hygiene
- | Sterilization of surfaces and disinfection
- | Treatment against lice and mites

Medical technology

- | Microfluidics
- | Biosensors
- | Lab-on-a-chip
- | Internal coating of tubes, bottles and bags
- | Functionalization of the surfaces of disposable articles
- | Implants

Cell culture technology and microbiology

- | Control of cell adhesion and differentiation
- | Control of protein adsorption
- | Coupling of antibodies
- | Cell transfection and transporation

Agricultural and foodstuffs technology

- | Pest control
- | Disinfection of seeds and food
- | Disinfection of packaging

In the "Ecology" field the Fraunhofer IST focuses on the following topics:

- | Water disinfection and wastewater treatment by means of diamond electrodes
- | Photocatalytic air and water purification systems
- | Self-cleaning and antifouling
- | Standardized test procedures for the neutral evaluation of photocatalytic properties of products

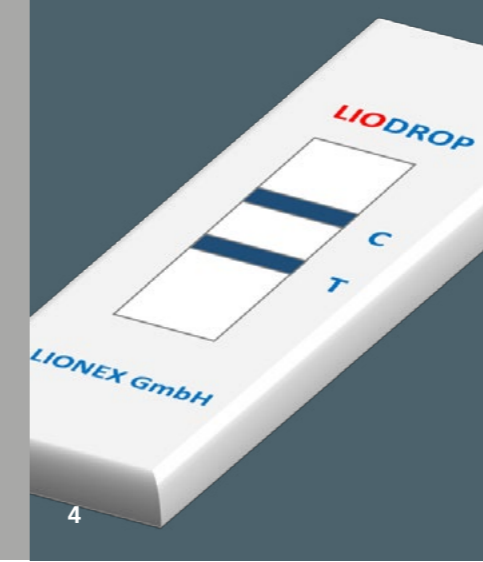
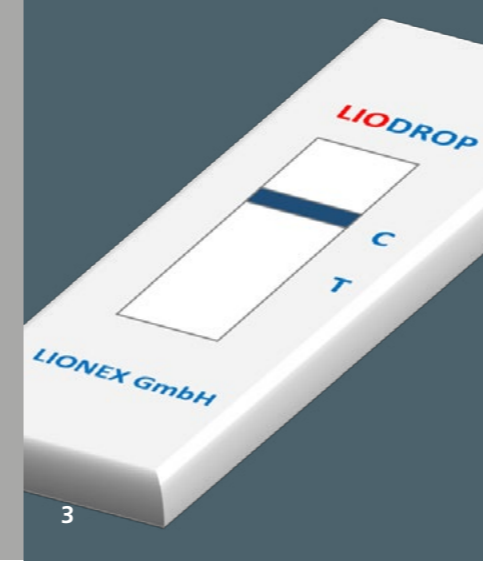
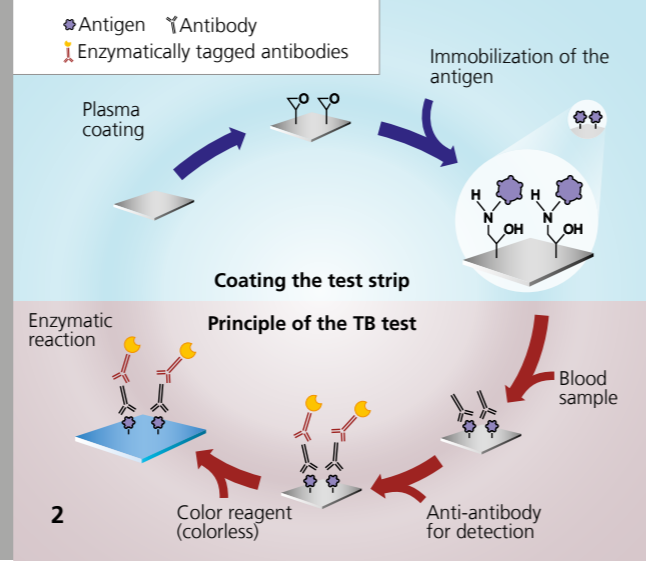
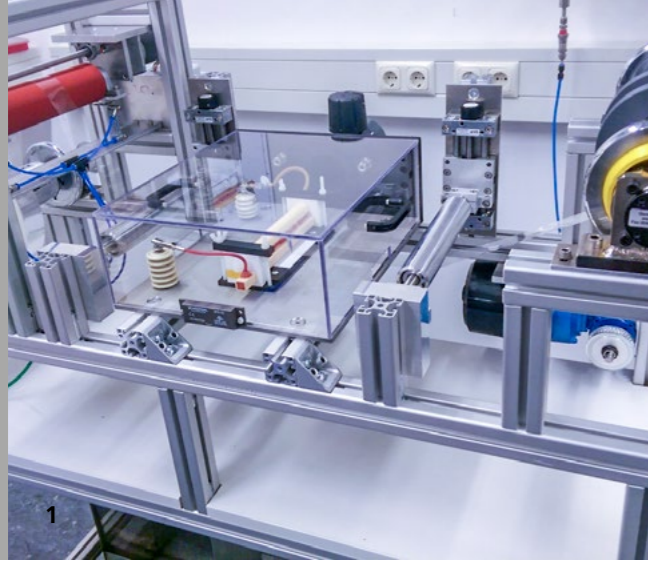
In addition to users in the fields mentioned, our customers also include manufacturers of equipment for surface modification and coating as well as contract coating companies at home and abroad.

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TUBERCULOSIS TEST – FASTER AND MORE SENSITIVE DUE TO PLASMA COATING

Even nowadays tuberculosis (TB) is one of the most common fatal infectious diseases worldwide. Around nine million people are infected with tuberculosis each year, of whom around two million die of it. The disease is particularly widespread in developing countries in Asia and Africa. There is therefore a high demand for simple and sensitive test systems with which the disease cannot only be detected early and reliably but also inexpensively and without great outlay on equipment in countries with a less well developed health infrastructure. In the EU-funded "IP4Plasma" research project the Fraunhofer IST is now developing such a test in collaboration with a partner from industry, LIONEX GmbH in Braunschweig.

The test strips

The principle of the test is based on the detection of human TB-specific antibodies from a blood sample which bind to TB antigens on a test strip. Immobilization of the antigens on the surface of the test strip makes a crucial contribution to the sensitivity of the test.

To secure an especially high density of binding possibilities for the antigens, the film surface of the test strips is coated at atmospheric-pressure by a plasma-enhanced chemical vapour deposition (PECVD) process. This "cold" plasma process, also known as dielectric barrier discharge (DBD), is characterized by relatively simple apparatus technology and, unlike other methods, does not use solvents. The coating is only a few nanometers thick and contains a large number of chemically reactive groups, so-called epoxy groups, which bind the antigens covalently. For each test strip two linear areas are plasma coated with epoxy groups. One line serves as the control line "C", with antigens being immobilized here

which bind non-specifically to human antibodies. The second line represents the test line ("T"). TB antigens are bound here which are used for the detection of the test subject's tuberculosis antibodies.

Test execution

The test is very simple in performance since no apparatus is required. It is suitable not only for plasma, but also for whole blood and serum, and can be evaluated with the naked eye. The person conducting the test first applies a drop of the blood sample to the test strip. Afterwards, a drop of the reagent solution is added.

If a TB disease is present or if the person already had a TB infection in the past and still has antibodies in the blood, then the TB-specific antibodies will bind by the key-lock principle to the antigens on the test line. Regardless of whether TB-specific antibodies are present or not, every blood sample will always also contain antibodies, which bind to the control

line. The reagent solution contains an enzymatically tagged antibody – a so-called anti-antibody – which binds to human antibodies. The reaction mixture is removed from the test strip with the aid of a punch and the initially colorless reagent tetramethylbenzidine (TMB) is dripped onto it. Contact with the enzyme brings about a color change to blue.

This result is already visible after a short period of time: if the control line alone turns blue, this indicates no tuberculosis disease is present. At the same time, the blue coloring of the control line shows that the test was performed correctly and also works. If both the test and control lines turn blue, in other words, the observer sees two lines, this is an indication of a possible tuberculosis infection. This test system is highly sensitive that means, even shortly after the onset of the disease a positive result is displayed.

Outlook

The tuberculosis test has already been patented and is currently being further optimized. The goal is to achieve mass-production capability. In addition, it is planned to transfer the test principle to the detection of other viral diseases.

1 System for coating the test strips.

2 Preparation of the surface (top) and the test principle of the tuberculosis test (bottom).

3-4 Possible outcomes of the tuberculosis test.

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THE NEXT GENERATION OF PLASMA TREATMENT

Wound dressings are standard in modern wound care. Their functions include the absorption of blood and wound exudates as well as protection against the ingress of bacteria and against mechanical stimuli. Plasma technology will be integrated into the next generation of modern wound dressings in order to combat micro-organisms and actively support wound healing. PlasmaDerm®, the world's first medical device in the field of plasma medicine, has been developed by CINOGY GmbH with the participation of the Fraunhofer IST.

"Cold" plasma technology and current medical technology solutions

The results from international research into the use of so-called "cold" and thus tissue-compatible plasma technology for human-medical therapies confirm a broad range of potential applications for this innovative approach. Its proven effects include antimicrobial action, pH modulation of the wound environment, stimulation of cell division and movement in human cells as well as improvement of the microcirculation, in other words, blood flow.

Currently, all of the products available on the market in this field are intended for use as stand-alone procedures and thus in the case of wound care are only applicable as a supplementary treatment when dressings are changed and also involve additional time expenditure on the part of both patient and staff.

The new solution: plasma technology integrated into the wound dressing

In the "KonChaWu" R&D project which started in 2016 the Fraunhofer IST and CINOGY GmbH are working together on the next generation of device systems. The project objective is to merge the concepts of "cold" plasma technology and wound dressings – in other words, the plasma-based dressing should be able to remain on the wound surface for up to several days beneath a bandage. Depending on the therapy modality, the air plasma can be generated for the desired period of time, typically 90 s, without a change of bandage, using an external plug-in connector routed through the bandage. If necessary, the treatment can be repeated several times a day.

From the beginning of the project, test samples made of different polymers and with application-specific geometries were prepared and tested under different electrical operating parameters. In this phase of the project the stable generation of the air plasma was in the foreground.

Outlook

In the future, safety-related process parameters, such as UV and gas species emissions, leakage current and temperature development, are to be determined so as to be able to define safe operating conditions. The project partners expect that their development work will improve the ability of the technology to integrate into medical and nursing procedures and thereby to boost acceptance sustainably.

The project

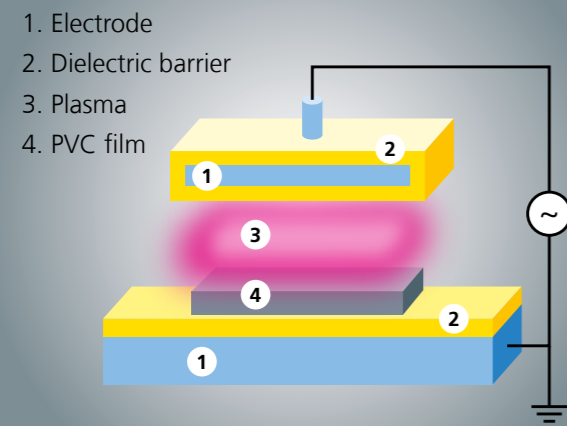
The project is funded with more than €700,000 by the State of Lower Saxony and the European Fund for Regional Development (EFRE).

1 *Olaf Lies, Minister for Economic Affairs, hands over the grant notification.*

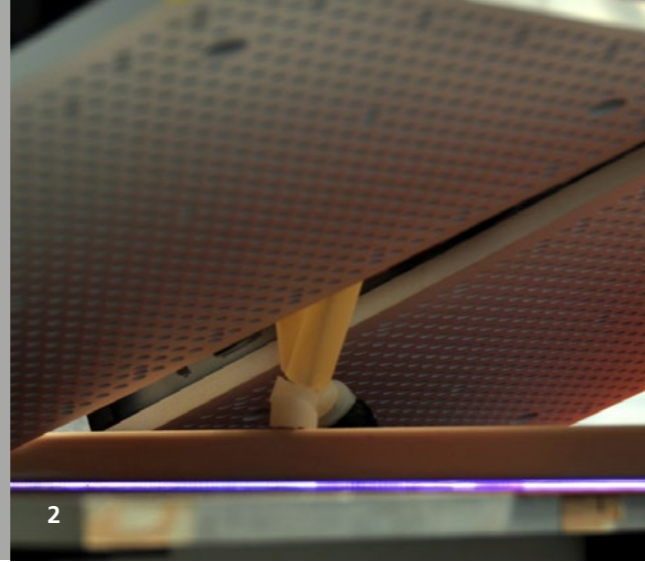
2 *The electrical signal characteristics are developed with computer support.*

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REDUCING THE MIGRATION OF PVC PLASTICIZERS

Many medical products such as blood bags or flexible tubes are made of polyvinyl chloride (PVC). This material is inexpensive, transparent, weldable and has low-temperature flexibility. To obtain the flexibility up to 40 wt.% of plasticizer has to be added to the material. In most cases these plasticizers are not chemically bound to the polymer but can move freely within the polymer matrix and also in the surrounding medium. However, the plasticizers normally used, such as bis(2-ethylhexyl) phthalate (DEHP), are considered problematic for human health. For this reason, in current research at Fraunhofer IST processes are developed, which reduce or completely prevent the migration of plasticizers from the polymer, by coating or cross-linking the PVC.

The approach

Dielectric-barrier discharges (DBD) at atmospheric pressure are used at the Fraunhofer IST for treating or coating the PVC plastics in order to reduce plasticizer migration. Here the PVC substrates are positioned between high-voltage electrodes with dielectric barriers. In the gas space between the electrodes an AC voltage generates a dielectric-barrier discharge (see Figure 1). Different effects can be achieved by selecting appropriate process gases. For example, if the process gas contains a film-forming precursor such as hexamethyldisiloxane, plasma polymer layers can be deposited in the process. On the other hand, other process gases such as pure argon generate very short-wave UV radiation, which has sufficient energy to break chemical bonds and thus can lead to a cross-linking of the polymers.

Results

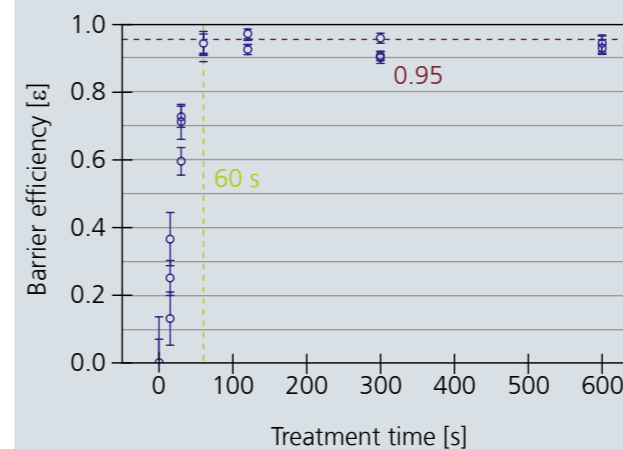
It was demonstrated at the Fraunhofer IST that the migration of plasticizers from plasticized PVC can be reduced by 95 percent by treatment in pure argon plasma (see Figure 2). Current knowledge attributes this effect to the plastic being

cross-linked by the very short-wave UV radiation of the argon plasma. It was also demonstrated that the treatment is stable in the long term (see Figure 3) and depends on the purity of the process gas. In addition to the treatment of flat film material, processes have been developed which allow even the interior surfaces of tubes and blood bags to be successfully modified and coated.

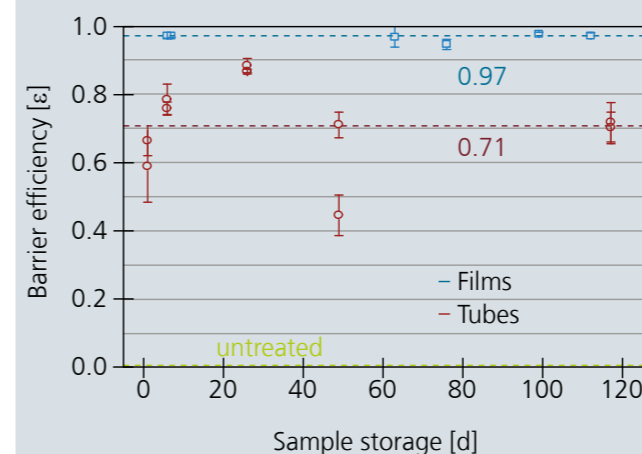
Outlook

Dielectric-barrier discharges are regarded in industry as a cost-effective way of treating plastic film before printing, gluing or painting. For this reason, current research is being conducted into the modification of film webs and the associated effects of further processing steps for medical products such as welding or gluing. In addition, various combinations of different processes are being tested with a view to reducing migration further. Future research will also look at whether results can be transferred to other polymers and the migration of other ingredients.

Barrier efficiency as a function of treatment time in argon plasma.



Long-term stability of barrier efficiency.



1 Experimental set-up for the DBD treatment of PVC film.

2 Installation for treating film by dielectric barrier discharge.

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NEW PLASMA JETS FOR CLEANING SILVER

Silver surfaces that are exposed to open air will begin to tarnish, and after a while, the well-known brownish-black discoloration of silver sulfide appears. Removing these dark coatings is a task which must often be carried out, especially in restoration work. One way of doing this is to treat the affected areas with plasma jets, operating with the use of reducing gases. Conventional, previously established systems often use a hot discharge thus making them unsuitable for treating delicate objects locally. Pen-like jet systems developed at the Fraunhofer IST use dielectric barrier discharge (DBD), making gentle work possible at low temperatures up to 50 °C and allowing for the treatment of temperature-sensitive and fragile cultural objects.

Jet development for reduction and oxidation processes

The modification of surfaces via plasma jet in the protective atmosphere of a glove box has already been a standard procedure at the Fraunhofer IST for quite a long time. With the aid of the plasma jets, oxidation products and even organic contaminants can be removed efficiently in this way, employing reactive process gases and relatively high temperatures.

By using a pulsed DBD as well as a selection of specially designed material, jet development has been optimized to the point where treatment temperatures can be lowered almost to room temperature. In addition, the special arrangement of the DBD jet along with the use of certain process and reactive gases has enabled the hydrogen atoms forming in the discharge zone during reduction treatments, as well as the ozone molecules during oxidative treatments to hit the surface in sufficient quantities to remove corrosion products and organic impurities.

Furthermore, a specially arranged flow of inert gas has been successfully developed that makes it possible to work with the jets even outside of the glove box. The steady stream of inert

gas ensures that reducing and oxidizing species in the vicinity are separated from each other and at the same time directing reduction products away from the surface. This opens up entirely new applications for the plasma jet function such as the restoration and treatment of immovable or large objects.

Cleaning textiles containing silver

The combination of metals and textiles often plays an important role in the restoration of textiles. One of the most common forms seen is silver thread, which is worked flat or spun as yarn around woven silk threads. The problem in this case is that the corrosion products of the metal are very damaging for the textile fibers since they promote their decomposition. Cleaning with conventional, mostly wet chemical or mechanical methods is however difficult to carry out on combined-material objects and often proves disadvantageous for the textile material.

The corrosion products present were reduced considerably by treating the silver with a plasma jet. Thanks to the low working temperatures and contactless treatment, this process resulted in very little damage to the silk. Investigations show

that mechanical properties of the textiles, such as tensile strength and flexural strength, have hardly changed after the plasma treatment – less than two percent in the case of tensile strength.

Coatings of pure silver sulfide can be removed relatively easily with a reducing plasma. However, since corrosion coatings often involve a complex interplay of silver and copper compounds such as oxides, sulfides and sulfates, and even organic contaminants are present, a purely reductive treatment is inadequate in many cases. Much better cleaning results are obtained by using an oxidation treatment combined with a process gas containing oxygen, and then followed by a reduction step. In addition, preliminary results indicate that the damage to the silk caused by oxidation can be reversed by downstream reduction treatment.

Outlook

In the future, the plasma jets are to be further developed so that they may be moved by hand over objects using pinpoint precision, thus enabling the treatment of even more complex object geometries. In addition, ergonomic and safety aspects must be taken into consideration since high voltages and reactive gases are involved.

1 Fragment of a stole (German Textile Museum Krefeld, Inv. No. 26071).

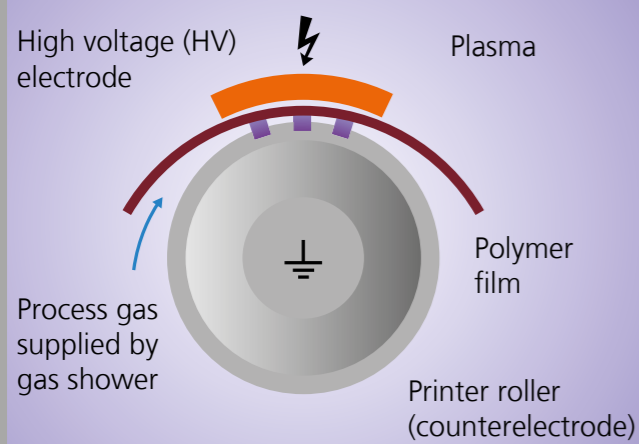
2 Detail of the stole with silver embroidery.

3-4 Flattened silver threads before (3) and after (4) a plasma treatment.

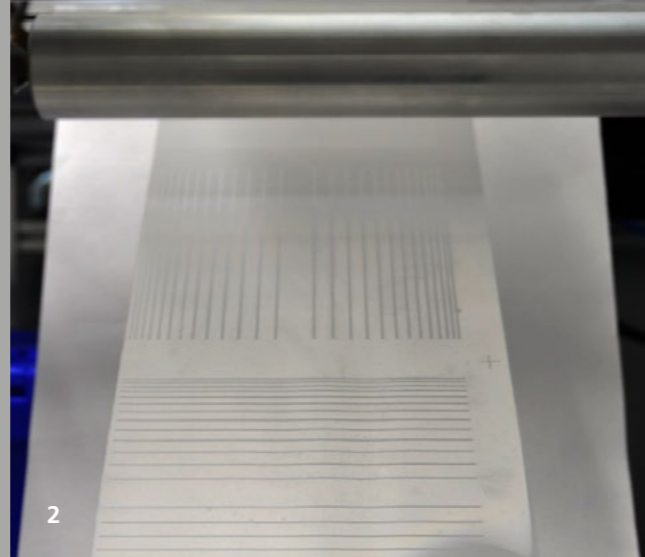
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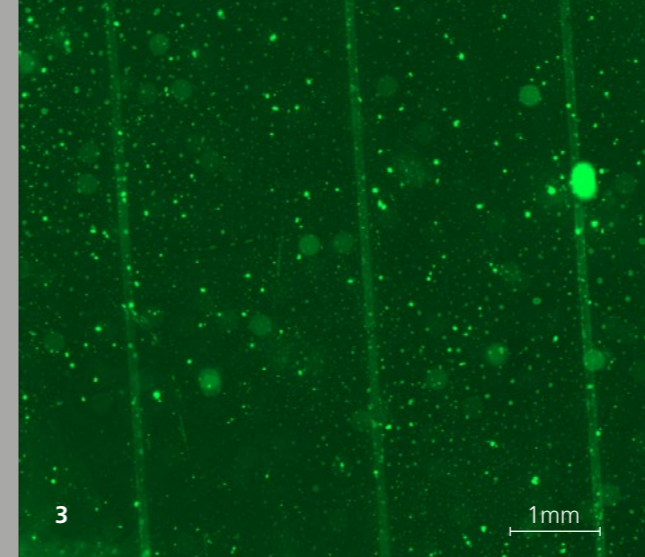
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STRUCTURED PLASMA TREATMENT FOR GRAVURE PRINTING

The aim of the BMBF project »Basic principles of location-selective wetting of nano inks« is to conduct research into new kinds of micro plasma sources for the location-selective pretreatment of film surfaces. With these new plasma sources a wetting contrast is to be created on films which will exert a local influence on the dispersion of printing inks and at the same time improve the adhesion of conductive nano inks. With the aid of various functional models from the fields of electronics and security printing, the performance of plasma printing in combination with the gravure printing of nano inks is to be investigated at the Fraunhofer IST in collaboration with our partners from industry, GRT GmbH & Co. KG and Schwarz Druck GmbH.

Influence of functionalization on wetting behavior

The location-selective structured plasma treatment of polymers at atmospheric pressure at the Fraunhofer IST is carried out by a reel-to-reel process (see Figure 1). For applications in the field of electronics or security printing, such as the forgery-proof printing of bank notes, conductive structures consisting of nano inks are to be deposited, combined with a gravure printing process. The plasma treatment hydrophilizes the surface of the polymers locally, after which the nano ink is printed into these structures. The differences in surface energy between the plasma-treated and the untreated areas makes it possible to achieve a greater contour accuracy when the nano inks are applied.

Analysis of plasma-treated films

After the plasma treatment, various investigations were carried out at the Fraunhofer IST with regard to confirm the successful structuring of the film. Here, the chemical changes on the surface, resulting from the generation of chemically reactive groups, were the main focus of analysis, but also the accuracy of reproduction and the edge sharpness of the contours created.

One way of indirectly revealing the chemical changes occurring at the surface after plasma treatment is to determine the free surface energy by contact angle measurements. Investigations showed that with a power input of at least 100 Wmin/m² the surface energy of the film could already be increased up to 40 % and in addition the long-term stability of surface activation improved to five days.

Another important aspect of the work conducted at the Fraunhofer IST is the high-definition transfer of the structure from the press roller to the film as a wetting pattern. To achieve this, selective film types were treated location-selectively with a structure width of 188 µm. The subsequent evaluation using a laser scanning microscope (LSM) revealed a deviation from the engraved structure of only 0.9 µm, which is equivalent to about 0.5 %.

In addition, an analysis of the films was carried out by means of the fluorescent marker Fluorescein isothiocyanate (FITC). Reactive nitrogenous groups, such as amines, imines or amides, were detected in the plasma-activated areas. Furthermore, the uniformity of the fluorescence signal over the labeled area

showed that the homogeneity of the plasma treatment within the structures could be demonstrated, as well as the sharpness of the boundary between the individual treated areas and the background. Figure 3 shows for instance the fluorescence of an activated line structure of 200 µm width.

Outlook

Results so far show that the wetting contrast of the films is already very high. Future research conducted in collaboration with Schwarz Druck GmbH will examine the adhesion of the nano inks for further treatments. In addition, one focus of work at the Fraunhofer IST is on further increasing the speed of the plasma treatment process.

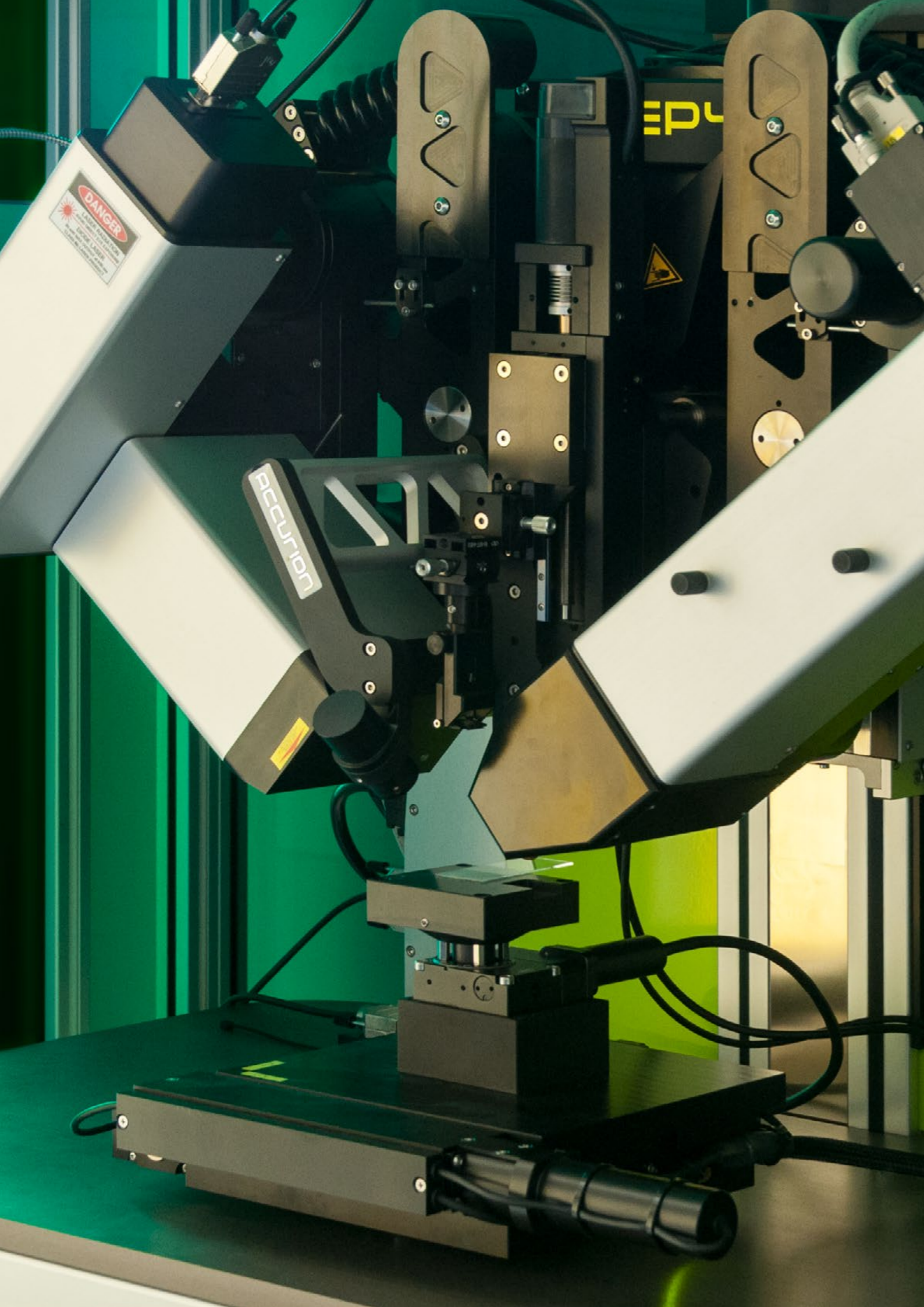
1 Schematic illustration of the reel-to-reel plasma printing process.

2 Wetting of BoPET film with steam after local plasma treatment; structure width 1000 µm.

3 Fluorescence of FITC-labeled film after an activation by plasma printing, width of line structures: 200 µm.

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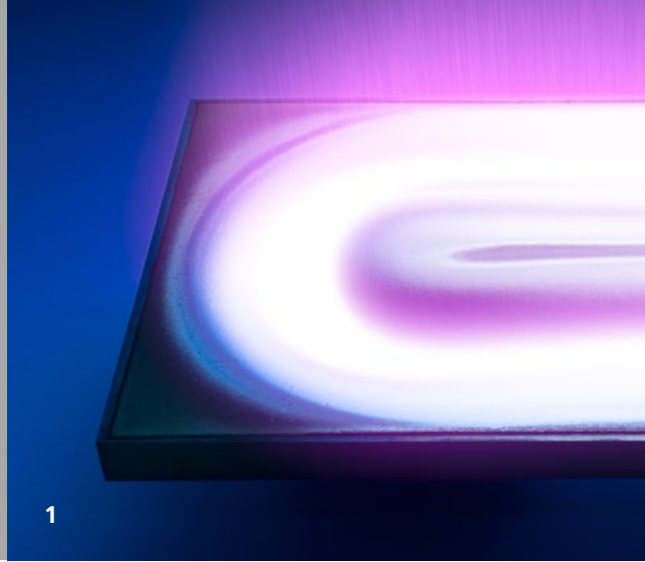


SERVICES AND COMPETENCIES

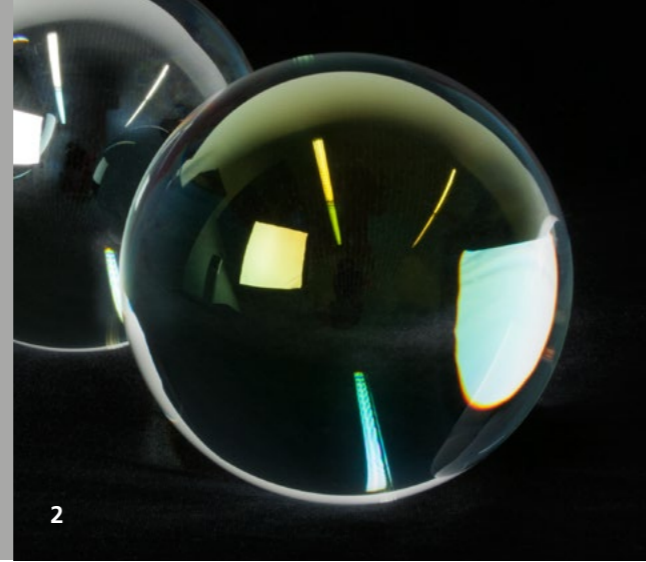
In pursuing the business units that were showcased in the previous chapters the Fraunhofer IST utilizes a wide spectrum of competencies. The focus is on these technologies:

- | Physical vapor deposition
- | Chemical vapor deposition
- | Plasma diffusion
- | Atmospheric pressure plasma processes
- | Electrochemical processes
- | Laser technology

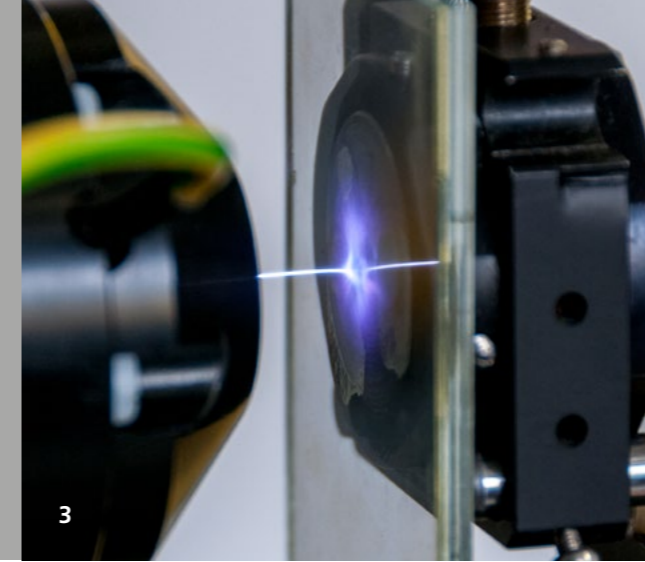
In addition the Fraunhofer IST provides recognized competencies for a variety of coating systems. The institute offers a broad spectrum of cross-sectional services: Surface pretreatment, thin film development, surface modification, process technology (including process diagnostics, modeling and control), surface analysis and thin film characterization, training, application-oriented film design and modeling, simulation, system design, device and equipment manufacturing and technology transfer.



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COMPETENCE LOW PRESSURE PROCESSES

Physical Vapor Deposition (PVD)

- | Magnetron sputtering
- | Highly ionized pulsed plasma processes like HIPIMS, MPP
- | Hollow cathode processes

Chemical Vapor Deposition (CVD)

- | Hot-wire-CVD
- | Atomic layer deposition (ALD)
- | Plasma-enhanced CVD (PECVD)

Plasma diffusion

- | Nitriding / Carbonitriding
- | Oxidizing
- | Boriding

COMPETENCE ATMOSPHERIC PRESSURE PROCESSES

Atmospheric pressure plasma

- | Micro plasma
- | Plasma printing
- | Dielectric barrier discharge/Corona treatment
- | Low temperature bonding
- | Plasma medicine
- | Plasma particle coating and cold plasma spraying

Electrochemistry

- | Multi component systems for electroplating
- | Non-aqueous electroplating
- | Electrochemical processes

Laser technology

- | Laser plasma hybrid processes
- | Laser induced fluorescence
- | Laser structuring

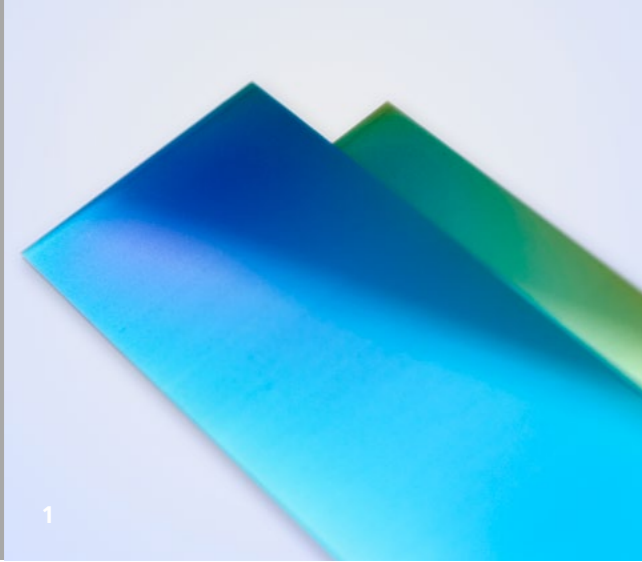
Layer by layer processes

- | Polyelectrolyte coatings
- | Biofunctional coatings
- | Chemical derivatization

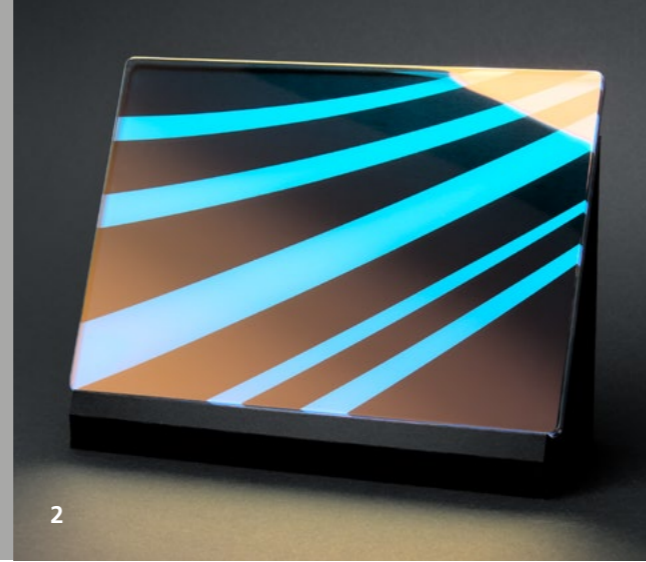
1 Planar target for coating deposition by magnetron sputtering.

2 Antireflective layer system on 3D object by atomic layer deposition (ALD).

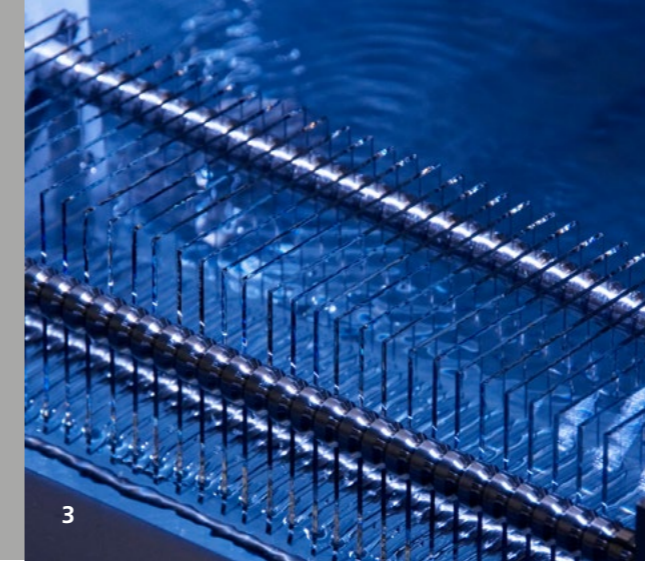
3 Discharge of a direct dielectric barrier plasma beam on glass.



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COMPETENCE COATINGS SYSTEMS

Friction reduction and wear protection

- | Amorphous carbon coatings (DLC) -----
- | Diamond coatings -----
- | Hard coatings -----
- | Nitride/Cubic boron nitride (cBN) -----
- | Metal coatings -----
- | Plasma diffusion/DUPLEX processes -----
- | Dry lubricants -----
- | Erosion protection -----
- | Corrosion protection -----
- | Anti-adhesion and antifouling coatings -----
- | Diffusion barriers -----

Electrical and optical coatings

- | Precision optics -----
- | Transparent conductive coatings -----
- | Electrochromic coatings -----
- | Low-E and sun control coatings -----
- | Diamond electrodes -----
- | Silicon-based coatings for photovoltaics and micro electronics -----
- | Semiconductors (oxide, silicon-based, diamond) -----

- | Insulation coatings -----
- | Piezoelectric coatings -----
- | Plastics metallization -----

Micro and nano technology

- | Thin film sensor technology -----
- | Micro technology -----
- | Nano composites -----
- | Control of coating adhesion -----
- | Structured surface coating and activation -----

Biofunctionalization

- | Antibacterial coatings -----
- | Adhesion and anti adhesion coatings -----
- | Chemical reactive surfaces -----

Photocatalysis

- | Air and water purification systems -----
- | Photocatalytically active coatings with antimicrobial effectiveness -----

FURTHER COMPETENCIES

Pretreatment and functionalization

- | Wetchemical cleaning -----
- | Functionalizing and coating of interfacial layers -----
- | Surface structuring -----
- | Plasma activation -----
- | Oxidation and reduction of metals -----
- | Plasma surface modification of natural products -----

Simulation

- | Simulation of plants, processes and coating layer properties -----
- | Model based interpretation of coating processes -----

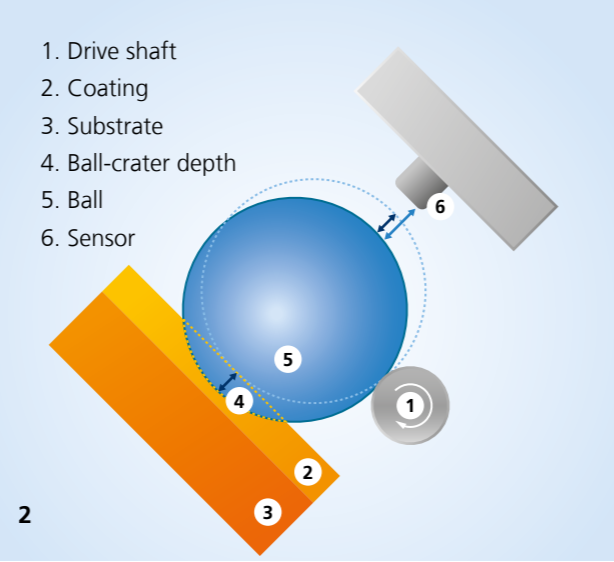
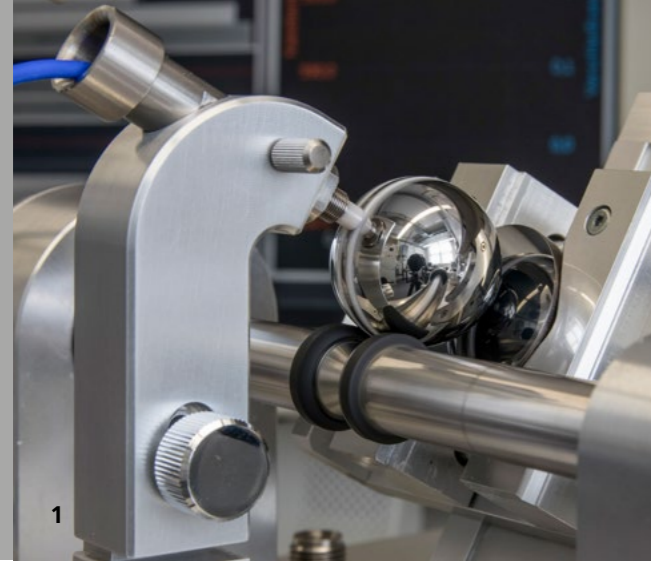
Analytics and quality assurance

- | Chemical analysis -----
- | Crystal structure analysis -----
- | Microscopy -----
- | Analysis of chemical reactive surfaces -----
- | Optical and electrical characterization -----
- | Plasma diagnostics -----
- | Tribological tests -----
- | Mechanical tests -----
- | Standardized photocatalytically measurement technology including test systems and devices -----

1 Metal strip with antifouling-coating.

2 Photocatalytic coating on a plastic substrate .

3 Ultrasonic cleaning system for precision optics.



CONTINUOUS BALL-CRATER ABRASION MEASUREMENT IN REAL TIME

For many years the abrasive wear of coatings has been measured by the well-established ball-crater abrasion test. With a modified ball-crater abrasion tester the abrasion behavior of coatings can now also be viewed as a function of time. Here the crater depth is measured continuously, thereby eliminating the additional manual evaluations hitherto necessary. The method developed at the Fraunhofer IST thus saves time as well as delivering certainty of results.

The ball-crater abrasion method

The traditional ball-crater abrasion test works on a very simple principle. A ball crater is ground into the coating under investigation with the aid of a rotating steel sphere to which an abrasive suspension is applied. After a specified test period the volume of the crater is measured and the wear coefficient obtained from this. In itself the method is very easy to handle but does have some disadvantages. For example, the abraded volume is usually determined as part of a visual evaluation and is thus dependent on the experience of the person carrying out the test. Furthermore, the wear coefficients of coating systems are determined at different surface pressures since with a specified test duration ball craters of different sizes form depending on the wear resistance of the coatings being tested. A further disadvantage of the traditional method is that it cannot describe dynamic, in other words, time-dependent wear behavior.

Continuous measurement of wear

In order to investigate dynamic wear behavior and at a future stage to determine wear coefficients under comparable surface pressures, a measurement system has been developed at the Fraunhofer IST which measures and displays in real time the ball-crater depth during grinding. To do so a ball-crater grinding device was modified to enable a displacement

sensor to continuously record the change in the position of the rotating ball – in other words, the crater depth ground into the material under test by the ball. The sensor, based on eddy-current technology, can be positioned in such a way that abrasion measurements can be carried out even with different bearing forces. The sensor can be moved laterally for handling and specimen changing.

Comparison of wear coefficients and surface pressures

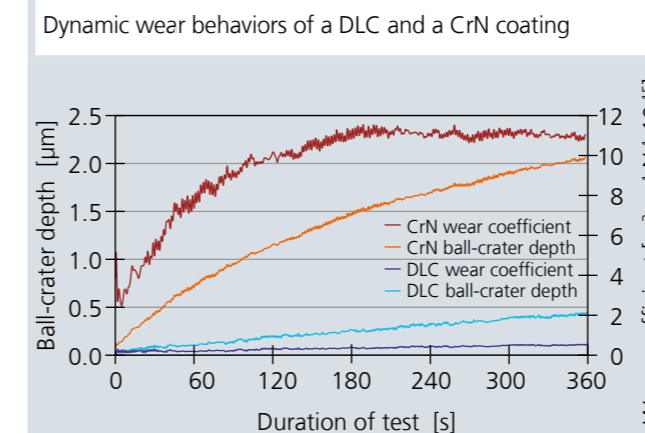
To give an example, the different dynamic wear behaviors of a DLC and a CrN coating were recorded in a test set-up. Results are shown in the graph opposite. The time-dependent wear behavior of the CrN coating here shows a marked difference from that of the DLC coating: while the wear coefficient of the DLC coating increases continuously, the wear on the CrN coating approaches a constant value.

The table shows different evaluations of the measurement curves shown in the graph opposite. With the same test duration, the traditional method yields a wear coefficient for CrN which is higher than that of the DLC coating by a factor of 20. The ball-crater depths are also different. With normalization to the same surface pressure the ball-crater depth on the other hand is identical and the ratio of the wear coefficients markedly lower (wear coefficient CrN/DLC = 10). With the

aid of dynamic measurement the wear coefficients of coatings and materials can be evaluated under uniform surface pressure and thus better compared.

Outlook

So far only the basics of the method have been tested. In further test series the as yet unsatisfactory reproducibility is to be examined more closely.



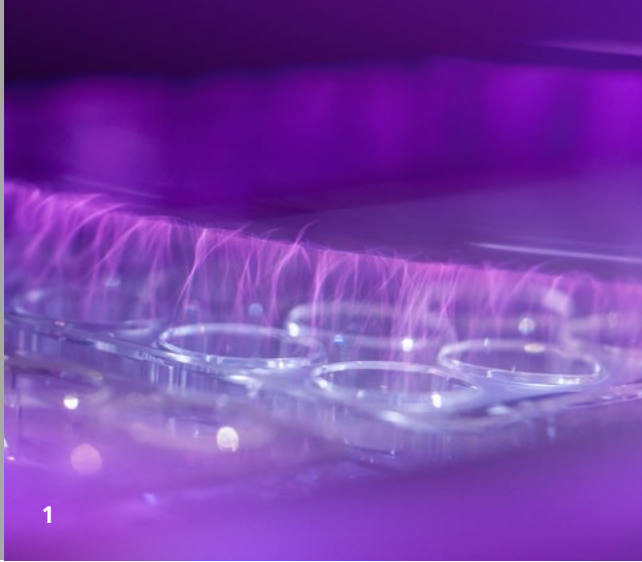
Determination of the wear coefficients	Reference value (DLC)	Traditional method Identical test duration (CrN)	New method Identical surface pressure (CrN)
Duration of test [s]	360	360	30
Ball-crater depth [µm]	0.43	2.0	0.43
Surface pressure [N/mm²]	13.3	2.8	13.3
Wear coefficient [m³ m⁻¹ N⁻¹ 10⁻¹⁵]	0.5	~ 11	~ 5.0

1 New ball-crater abrasion tester with sensor.

2 Principle of the new measurement set-up.

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3

OXYGEN-FREE PLASMA PRETREATMENT

Plasma cleaning and activation of surfaces in preparation for subsequent process steps is widely used and industrially established. Frequently used process gases are air or gas mixtures containing oxygen. Over-treatments can occur in particular in the pretreatment of plastics. Investigations are going on at the Fraunhofer IST into the use of oxygen-free, nitrogenous process gases with which high densities of nitrogenous chemically reactive groups such as amino groups can be created, especially on organic surfaces.

State of the art

Conventional plasma pretreatments of plastic surfaces on the one hand creates polar groups on the surface and on the other the highly reactive oxygen species generated in the plasma can damage the polymer, even superficially, which can lead to adhesion problems during subsequent processing. However, since oxygen-free nitrogenous process gases are used during pretreatment, oxidation of the surface of the plastic is avoided. This means that with an appropriate choice of parameters, nitrogenous chemically reactive groups form, which during subsequent coatings have a mostly positive effect on adhesion.

Surface functionalization

In contrast to a treatment at low pressure, the composition of the process gas is not so easily controlled in the case of pretreatment with atmospheric pressure plasma. Special devices are required, which prevent oxygen from the ambient air getting into the treatment area.

This is why atmospheric pressure plasma processes and the corresponding equipment are being developed at the Fraunhofer IST and being adapted customer-specifically, offering the possibility of treating substrates in a previously defined process gas atmosphere. In addition, methods for surface characterization are under development with which the various chemically reactive groups created can be detected and their density determined. The investigations showed that plastic surfaces which have been treated with process gas mixtures of nitrogen and small quantities of hydrogen contain high densities of chemically reactive groups.

In addition, not only can surface properties be directly detected at the Fraunhofer IST but the adhesion of paints, adhesives or printing inks can also be characterized. In this way, surface pretreatments are being further optimized.

Treatment of customer samples

In the case of pretreatment with atmospheric-pressure plasmas the substrate geometry plays an important part in the design of equipment. The Fraunhofer IST has various laboratory facilities for very different substrate geometry, such as sheeting or 3D substrates. In addition, plasma systems are also available for a local treatment, such as plasma printing or plasma jets. Due to the wide range of different systems, the processes can be transferred with good results to customer samples and customer requirements fine-tuned. Lambda sensors and optical emission spectroscopy are used for process monitoring, in particular to ensure the absence of oxygen. A final characterization of the surfaces using derivatization, fluorescence labeling, infrared spectroscopy or even XPS makes a comprehensive and promptly surface characterization possible.

1 RotoTEC system for the treatment of 3D plastic parts.

2 Pretreatment of substrates for subsequent bonding.

3 Treatment of a plastic sample with a plasma jet in a glove box.

CONTACT

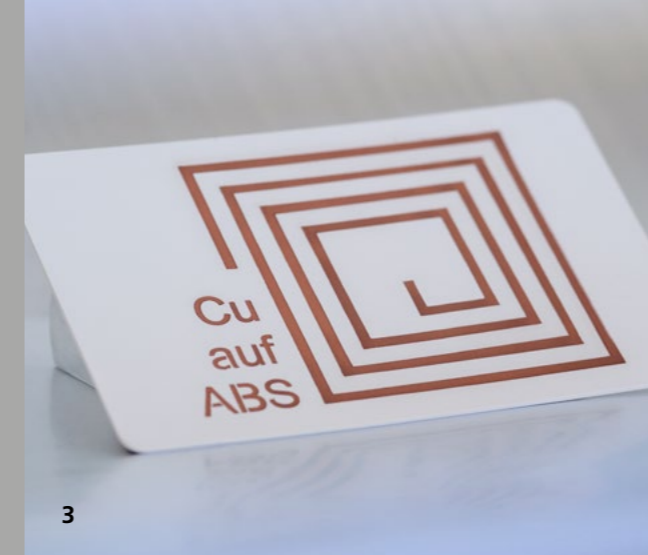
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NAMES, DATES, EVENTS 2016

In 2016 the Fraunhofer IST once again appeared on various platforms. An overview of the most important events and activities of 2016 follows:

- | Trade fairs, exhibitions, conferences
- | 11th International Conference on Coatings on Glass and Plastics–ICCG11
- | Events, colloquia, workshops
- | Prizes and awards



TRADE FAIRS, EXHIBITIONS, CONFERENCES

Hannover Messe 2016

Hannover, April 25–29, 2016. In recent years industry has increasingly felt the need for a sensor system which is used directly on component surface. A multifunctional thin film system was therefore being developed at the Fraunhofer IST for the local measurement of pressure and temperature distribution over the surface. Furthermore the system which was presented at the Hannover Messe, has a high wear resistance. As part of the Fraunhofer Adaptronics Alliance the scientists of the Fraunhofer IST also presented their latest developments in the field of thin film sensor technology

International Conference on Metallurgical Coatings and Thin Films ICMCTF 2016

San Diego, CA, USA, April 25–29, 2016. Several scientists of the Fraunhofer IST took part at the International Conference on Metallurgical Coatings and Thin Films ICMCTF in San Diego and participated with a variety of lectures.

SVC TechCon 2016

Indianapolis, IN, USA, May 11–12, 2016. The Fraunhofer IST presented their new research results of the coating technology HIPIMS (High Power Impulse Magnetron Sputtering) and latest developments in the field of sensory and optical functional films during the exhibition of the SVC TechCon. Furthermore the Fraunhofer IST participated in the program as in the years before with numerous lectures.

International Air Show ILA 2016

Berlin, June 1–4, 2016. Already for the second time the Fraunhofer IST presented its activities in the field of aerospace as a part of a joint Fraunhofer booth at the ILA Berlin Air Show. Particular focus was placed on metallized antennas of carbon-fiber-reinforced plastic (CFRP) which can be used in space even under extreme temperature conditions.

Optatec 2016

Frankfurt, June 7–9, 2016. As part of a joint Fraunhofer booth the Fraunhofer IST presented different high precision optical filters produced with the innovative sputtering system EOSS®. Furthermore this year also the Application Center for Plasma and Photonics of the Fraunhofer IST showed its latest developments in the field of laser plasma hybrid technology. The process combines both named technologies – laser and plasma – for a surface optimization and treatment. This approach allows to deposit a variety of materials homogeneously with high precision as well as productivity and low costs on large areas. Also optical components can be microstructured precisely and efficiently.

7th International Conference on High Power Impulse Magnetron Sputtering (HIPIMS)

Sheffield, UK, June 27–30, 2016. The Fraunhofer IST organized the annual International Conference on High Power Impulse Magnetron Sputtering (HIPIMS) in cooperation with the Sheffield Hallam University and INPLAS e.V. competency network. In the meantime, it has established itself as a rendezvous for scientists from all over the world who exchange

information about the latest results and trends in the field of innovative plasma surface engineering. Representatives from the Fraunhofer IST were once again participating in the conference program with numerous presentations this year.

glasstec 2016

Düsseldorf, September 20–23, 2016. Jointly with other Fraunhofer institutes and its Application Center for Plasma and Photonics the Fraunhofer IST presented the latest developments and research results related to glass materials at glasstec 2016. Focal points included ALD coatings on glass objects, glass treatment using atmospheric pressure plasma and the innovative laser plasma hybrid treatment of glass.

International Conference on Space Optics ICSO 2016

Biarritz, France, October 18–21, 2016. As part of the Fraunhofer Space Alliance the Fraunhofer IST was also represented at the ICSO in this year. Diverse optical filters for space produced with the innovative sputtering system EOSS® (Enhanced Optical Sputtering System) were presented. Such high precise optical filters can be used in spectrometers which in turn can be used in satellites for example to examine the earth vegetation.

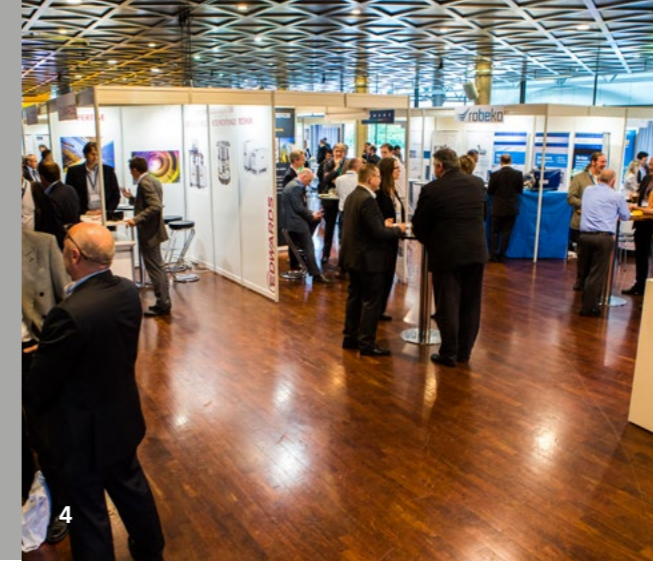
K2016

Düsseldorf, October 19–26, 2016. The Application Center for Plasma and Photonics of the Fraunhofer IST was participating for the first time at K – the largest plastic trade fair in the world – where it presented the newly developed “cold plasma spraying” technology among other things.

1 Prof. Wolfgang Diehl, former deputy director of the Fraunhofer IST, during the ICMCTF 2016.

2 View of the joint Fraunhofer booth at the K2016.

3 Copper conductor track produced by cold plasma spraying, presented at the K2016.



11TH INTERNATIONAL CONFERENCE ON COATINGS ON GLASS AND PLASTICS–ICCG11

From June 12th to 16th 2016 the International Conference on Coatings on Glass and Plastics ICCG was once again being held in Braunschweig, offering experts from science and industry an important platform for finding out about future trends, new technologies and the latest developments and applications in the field of glass and plastic coatings. ICCG 11 took place this year for the third time in Braunschweig and was organized by the Fraunhofer IST in cooperation with the international organisation committee.

Over five conference days more than 300 expected participants had the opportunity to attend numerous presentations by speakers from all over the world. The large variety of topics in a program put together expressly by a panel of experts and covering current issues and trends in the field of glass and plastic coating ranged from the latest developments in many technologies, such as, for example, the HIPIMS process, the atmospheric pressure plasma process or atomic layer deposition, to a variety of application examples from inter alia the fields of flexible electronics, photovoltaics, displays, architecture and automotive engineering. The program was rounded off by a panel discussion entitled 'Architectural glazing—quo vadis', short courses, background information about materials in use, various coating processes or individual processes, and also a poster exhibition. In addition, around 40 international companies and research institutes showcased their latest developments and products in the accompanying exhibition. A brief insight into the topics of the ICCG11:

Direct metallization plastic materials

Today, metallized plastics are used in various fields of application. The chrome fittings for the sanitary area are classic. In this case, the chromium VI ban is expected to lead to an increase in demand for vacuum based processes as an alternative to electroplating. Reflectors in the automotive sector are already coated with the latest sputter technology.

With the increasing complexity of the components the need for an adherent precision-contoured coating grows. The layer bonding still poses a special challenge. Processes that allow a significant increase in layer adhesion without additional process steps are extremely in demand.

Such a process is High Power Impulse Magnetron Sputtering HIPIMS. This technology's potential documents current research results in the field of direct metallization of plastics. A first screening of various plastic substrates showed that a very good layer bonding by means of HIPIMS is possible without any pretreatment. Specifically for the combination of aluminum on acrylic glass (PMMA) it has been shown that optimized HIPIMS parameters allow a direct metallization, although PMMA is typically damaged under the UV radiation of the plasma and only a defective layer bonding is possible with pure plasma based processes. Dr. Ralf Bandorf, head of group for "highly ionized plasmas and PECVD" at the Fraunhofer IST gave an insight into the current state of the art of plastic metallization using HIPIMS at the ICCG11.

Laser plasma hybrid technology – the combination of laser and plasma technology

Laser processes have a number of advantages for a wide range of applications. These include amongst others micro structuring, surface and coating modification or the removal

of impurities and coatings. Here, laser processes are particularly characterized by the possibility of being able to produce area-selectively precise structures. However, laser processes are also subject to certain limitations and can be very energy and time consuming depending on the application.

At the Application Center for Plasma and Photonics of the Fraunhofer IST in Göttingen has therefore developed a new process combining two technologies: laser technology and atmospheric pressure plasma processes. In this so called laser plasma hybrid technology plasmas are coupled into the laser beam in order to increase its effects for microstructuring considerably. This process can significantly reduce the required laser energy. Additionally the plasma enhanced effects of the laser treatment leads to several advantages such as precise structures as well as lower production costs due to shorter treatment time.

Dr. Christoph Gerhard from the Application Center for Plasma and Photonics presented the laser plasma hybrid technology at ICCG11 and explained the possible applications by means of two cases. Using the example of plasma-assisted crystallization of amorphous layers by laser radiation, he approached the achievable efficiency improvement of such a process. In addition, Gerhard explained the fundamental interactions and effects between laser radiation, plasma and surface for a laser plasma hybrid removal of lacquers on glass surfaces.

1 The 11th International Conference on Coatings on Glass and Plastics ICCG took place for the third time in Braunschweig.

2 A view in the conference hall.

3 Dr. Volker Sittinger, local Chairman of the ICCG11, welcomes the conference participants.

4 More than 40 international companies and research institutes present their latest developments in the technical exhibition.



EVENTS, COLLOQUIA, WORKSHOPS

New professorship in South Africa

Prof. Wolfgang Diehl, deputy director of the Fraunhofer IST, was appointed Professor of the Faculty of Engineering and Built Environment by the university. The university is thus honoring the years of good cooperation and, above all, the commitment of Prof. Diehl, who has been an associate professor at TUT since 2011. Previously, he had already been actively involved in the academic process through guest lectures and his involvement in setting up the RETECZA program.

The economics minister of Lower Saxony visited the Fraunhofer IST

During his annual summer journey the economics minister of Lower Saxony Olaf Lies visited the Fraunhofer-Institute for Thin Films and Surface Engineering IST in Braunschweig for the first time. His visit accompanied by Klaus-Peter Bachmann, vice president of the Lower Saxony Parliament, Dr. Christos Pantiazis, SPD provincial council delegate, and Matthias Wunderling-Weilbier, regional representative for regional development Braunschweig, on July 29, 2016 was focused on the topic plasma surface technology in Lower Saxony.

New deputy director for Fraunhofer IST

On September 23, 2016, Fraunhofer IST said farewell to its long-time deputy director, Professor Wolfgang Diehl, in the course of a celebratory symposium. It focused primarily on the institute's international further development in the preceding years, steadily driven by Diehl. The numerous guests recognized his outstanding performance in this field in technical presentations and the subsequent greetings. Dr. Lothar Schäfer will assume the position of deputy director going forward. He has extensive expertise in the thin film segment.

3rd International Workshop

Plasma Science & Entrepreneurship

Braunschweig, November 30 – Dezember 1, 2016. The third international workshop in the field of plasma science and entrepreneurship took place in Braunschweig this year. The Fraunhofer IST participated with numerous lectures.

Visit of the Lower Saxony Prime Minister Stephan Weil

On November 1st, 2016 the Minister President of Lower Saxony Stephan Weil visited the Fraunhofer IST at its location in Göttingen, the Application Center of Plasma and Photonics. The focus of the visit was on plasma research which is becoming more and more relevant for everyday use. Plasma technology is fascinating because it is a cross-section technology that can be used in almost all areas, the Prime Minister emphasized. "We will continue to support this as a federal state", explained Weil.

PRIZES AND AWARDS

Supplemental award to EFB Project Award 2016

Dipl.-Ing. Martin Weber, long-standing employee of the Fraunhofer IST, received the supplemental award to the EFB Project Award 2016 for his successful work on the project "Fast Tool Wear Test for Deep and Stretch Drawing of Sheet Steel". It was presented on April 12th during the congress of the European Research Association for Sheet Metal Working (EFB).

2nd place in the competition INNOspace Masters 2016

At the INNOspace Masters conference on Mai 4, 2016 in Berlin, Dr. Andreas Dietz, manager of the business unit aerospace at the Fraunhofer IST, was honored by the German Center for Aerospace (DLR) in the framework of the competition INNOspace Masters 2016. Together with his project partners Prof. Enrico Stoll from the Institute of Space Systems of the TU Braunschweig and Stefan Linke from INVENT GmbH he took the second place in the category "DLR Space Management Challenge" with the "OCULUS" project.

3rd Poster prize at the ICCG11

Employees of the Fraunhofer IST were honored with the third place in the poster prizes awarded during the 11th International Conference on Coatings on Glass and Plastics from June 12 – 16, 2016 in Braunschweig. Felipe C. Carreri, Holger Gerdes, Ralf Bandorf, Michael Vergöhl and Günter Bräuer from the Fraunhofer IST were awarded for the contribution "Deposition of ITO films by reactive high power impulse magnetron sputtering from a metallic rotatable target".

1 *Change of the deputy institute management of the Fraunhofer IST: Prof. Wolfgang Diehl, Prof. Dr. Günter Bräuer und Dr. Lothar Schäfer (f.l.t.r.).*

2 *Visit of the Minister President of Lower Saxony Stephan Weil at the Fraunhofer IST's location in Göttingen: f.l.t.r.: Prof. Dr. Günter Bräuer, Prof. Dr. Wolfgang Viöl, Prof. Dr. Christiane Diemel, Ministerpräsident Stephan Weil.*

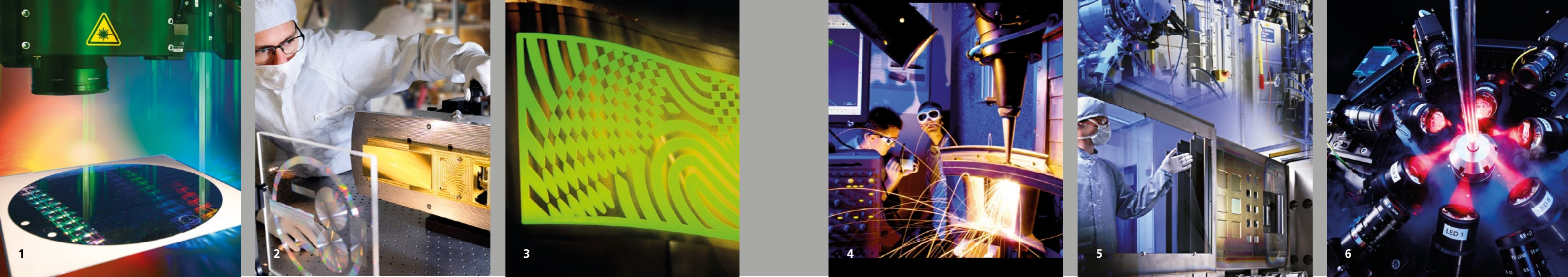
3 *The prize winners of the EFB project prize 2016.*



THE FRAUNHOFER IST IN NETWORKS

With its research and development activities the Fraunhofer Institute for Surface Engineering and Thin Films IST forms a part of various internal and external networks which function with different points of emphasis in the field where business, science and politics interact and even clash. Within the Fraunhofer Society the institute pools its competences with those of other Fraunhofer institutes in, amongst other things, the Fraunhofer Group for Light & Surfaces and in various Fraunhofer alliances in order to be able to offer customers and partners optimal - and even cross-technology – solutions for their specific tasks.

In addition the Fraunhofer IST also keeps an eye open for future scientists and researchers. For this reason the institute networks intensively with educators, students and schoolchildren in order to arouse an enthusiasm for the natural sciences and engineering at an early age and to encourage the upcoming generation of scientist.



FRAUNHOFER GROUP LIGHT & SURFACES

Competence by Networking

Six Fraunhofer institutes cooperate in the Fraunhofer Group Light & Surfaces. Co-ordinated competences allow quick and flexible alignment of research work on the requirements of different fields of application to answer actual and future challenges, especially in the fields of energy, environment, production, information and security. This market-oriented approach ensures an even wider range of services and creates synergetic effects for the benefit of our customers.

Core Competences of the Group

- | Surface and coating functionalization
- | Laser-based manufacturing processes
- | Laser development and nonlinear optics
- | Materials in optics and photonics
- | Microassembly and system integration
- | Micro and nano technology
- | Carbon technology
- | Measurement methods and characterization
- | Ultra precision engineering
- | Material technology
- | Plasma and electron beam sources

Business Areas

- | Ablation and cutting
- | Imaging and illumination
- | Additive manufacturing
- | Light sources and laser systems
- | Lithography

- | Material testing and analytics
- | Medical engineering and biophotonics
- | Micro systems and sensors
- | Opticals systems and instrumentation
- | Tooling and mold making

Fraunhofer Institute for Applied Optics and Precision Engineering IOF²

The Fraunhofer IOF develops innovative optical systems to control light from the generation to the application. The service range covers the entire photonic process chain from optomechanical and opto-electrical system design to the manufacturing of customized solutions and prototypes. The institute works in the five business fields of Optical Components and Systems, Precision Engineering Components and Systems, Functional Surfaces and Layers, Photonic Sensors and Measuring Systems and Laser Technology. www.iof.fraunhofer.de

Fraunhofer Institute for Organic Electronics, Electron Beam and Plasma Technology FEP³

Fraunhofer Technology FEP works on innovative solutions in the fields of vacuum coating, surface treatment as well as organic semiconductors. The core competences electron beam technology, sputtering, plasma-activated deposition and high-rate PECVD as well as technologies for organic electronics and IC/system design provide a basis for these activities. Fraunhofer FEP continuously enhances them and makes them available to a wide range of industries: mechanical engineering, transport, biomedical engineering, architecture and preservation, packaging, environment and energy, optics, sensor technology and electronics as well as agriculture. www.fep.fraunhofer.de

Fraunhofer Institute for Laser Technology ILT⁴

With more than 400 patents since 1985 the Fraunhofer Institute for Laser Technology ILT develops innovative laser beam sources, laser technologies, and laser systems for its partners from the industry. The technology areas cover the following topics: laser and optics, medical technology and biophotonics, laser measurement technology and laser material processing. This includes laser cutting, caving, drilling, welding and soldering as well as surface treatment, micro processing and rapid manufacturing. Furthermore, the Fraunhofer ILT is engaged in laser plant technology, process control, modeling as well as in the entire system technology. www.ilt.fraunhofer.de

Fraunhofer Institute for Surface Engineering and Thin Films IST⁵

As an innovative R&D partner the Fraunhofer IST offers complete solutions in surface engineering which are developed in cooperation with customers from industry and research. The IST's "product" is the surface, optimized by modification, patterning, and/or coating for applications in the business units mechanical engineering, tools and automotive technology, aerospace, energy and electronics, optics, and also life science and ecology. The extensive experience of the Fraunhofer IST with thin film deposition and film applications is complemented by excellent capabilities in surface analysis and in simulating vacuum-based processes. www.ist.fraunhofer.de

Fraunhofer Institute for Physical Measurement Techniques IPM⁶

The Fraunhofer IPM develops tailor-made measuring techniques, systems and materials for industry. In this way the institute enables their customers to minimize the use of energy and resources while at the same time maximizing quality and reliability. Fraunhofer IPM makes processes more ecological and at the same time more economical. Many years of experience with optical technologies and functional

materials form the basis for high-tech solutions in the fields of production control, materials characterization and testing, object and shape detection, gas and process technology as well as functional materials and systems. www.ipm.fraunhofer.de

Fraunhofer Institute for Material and Beam Technology IWS¹

The Fraunhofer Institute for Material and Beam Technology is known for its innovations in the business units joining and cutting as well as in the surface and coating technology. Across all business units our interdisciplinary topics include energy storage systems, energy efficiency, additive manufacturing, lightweight construction and big data. Our special feature is the expertise of our scientists in combining the profound know-how in materials engineering with the extensive experience in developing system technologies. Every year, numerous solutions with regard to laser material processing and coating technology have been developed and have found their way into industrial applications. www.iws.fraunhofer.de

CONTACT

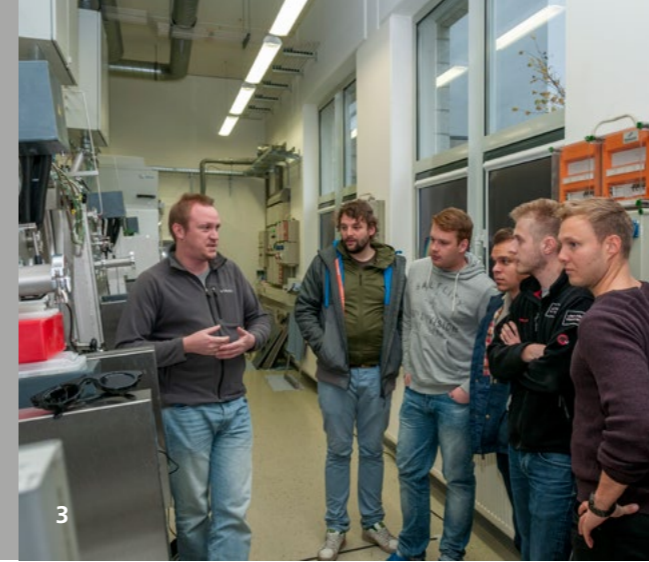
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SUPPORTING AND TRAINING YOUNG SCIENTISTS AT THE FRAUNHOFER IST

Supporting young scientists: for the Fraunhofer Institute for Surface Engineering and Thin Films IST this means not only being active as supervisors and in an university context, it means introducing young people to scientific topics, removing their initial reservations, and giving them the chance to become interested in industrial research. The support and supervision of school children and students who are interested in the research areas covered by the Fraunhofer IST was as important in 2016 as ever, and an indispensable part of work at the institute.

Bona SZ vocational training fair

This year, the Fraunhofer Institute for Surface Engineering and Thin Films IST was represented at the BONA SZ vocational training fair for the first time. On March 8–9, 2016 interested pupils could find out about the various professional training programs. The IST offers a wide range of choice: In addition to a vocational training for office management clerks the IST provides training as a physics laboratory technician, an exciting profession in which the trainees pass through several stations within the institute and therefore acquire extensive knowledge in the field of surface engineering and thin films.

Future day for boys and girls at the Fraunhofer IST

It has already become a tradition at the Fraunhofer IST that once a year pupils, equipped with lab coats and protective goggles, explore the institute at the "Future Day". With the aim of inspiring these 8 boys and 14 girls for science, the IST opened its doors in collaboration with the Fraunhofer WKI. The young researchers did not only have the opportunity to

see luminous plasma flashes or huge coating systems, but in many participative actions they could also for example pre-treat plastic cars by atmospheric pressure and afterwards electrolessly metallize them with copper. At the end of the day they were allowed to take their coated works home with them. In addition, this year for the first time, some boys and girls also had the opportunity to take a look behind the scenes of the research institute at its location in Göttingen.

A visit from the Technikakademie of the city of Braunschweig

This year, students of the Technikakademie of Braunschweig visited the Fraunhofer IST for the first time. After a brief introduction of the institute and the working areas engineering technology interested were given an insight into the possibilities for entry and employment within the Fraunhofer Society. Finally, the students received an insight of the Fraunhofer IST during a tour through the institute.

A visit from the TU Braunschweig's Career Service

This year scientifically interested students and graduates from the Technical University of Braunschweig visited the Fraunhofer IST once again. After a brief presentation of the institute the topic "Tribological Systems at the Fraunhofer IST" was the focus of the event organized by the Career Service of the Technical University of Braunschweig. TU graduate Tim Abraham presented his work and his tasks at Fraunhofer and answered questions about the career at the institute. In a subsequent guided tour the students and graduates were given the opportunity to get to know further fields of activity within the Fraunhofer IST.

1 Two trainees of the Fraunhofer IST presented their vocational training program as physics laboratory technician in more detail at the BONA SZ vocational training fair.

2 The participants of the "Future day for boys and girls" at the Fraunhofer IST.

3 Employees of the Fraunhofer IST showed the large coating systems of the Fraunhofer IST to the students of the Technikakademie Braunschweig.

50 INPLAS-Mitglieder

Kompetenznetz
INPLAS Stand: November 2016



THE NETWORK OF COMPETENCE INDUSTRIAL PLASMA SURFACE TECHNOLOGY E. V.–INPLAS

The INPLAS e. V. network of competence, accredited as a network by the Federal Ministry of Economics and Technology (BMWi) in the “go-cluster” program, has its business offices at the Fraunhofer IST. Overall, the network consists of 50 members from industry and science, and about 200 people participating in the activities of the network of which 74 percent come from industry.

INPLAS can once again look back on a successful year of network activities in 2016. The aim of its work is to raise further the profile of plasma technology and to support, promote and act as moderator for development in numerous fields of application in their various levels of complexity. Some highlights of the numerous activities, projects and events of 2016 are presented in the following section:

11th INPLAS General Assembly at Schaeffler AG

The 11th INPLAS General Assembly was held on November 17, 2016 at Schaeffler AG in Herzogenaurach, Germany. Following a tour of the plant offering an interesting insight into the company’s daily production activities, the official part of the general assembly started with a review of the year. This clearly revealed the numerous activities of the network: from diverse events, working group meetings and political involvement to public relations work. It was also important to present an overview of future activities and to discuss these collectively. The general assembly always offers the possibility of exchanging views and of more intensive work for the network.

Award presented to Dr Bernhard Cord, Singulus AG

After 10 years as head of the INPLAS working group “Innovative plasma sources and processes” Dr Bernhard Cord of Singulus Technologies AG entered upon his well-earned retirement. On the November 18, 2016 he chaired for the last time the working group meeting at Schaeffler AG in

Herzogenaurach and at its close was given the INPLAS Award for his long-standing commitment. We would like to thank Dr Cord once again for his exemplary and exceptional commitment to INPLAS and great teamwork and wish him all the best in the years ahead.

INPLAS joint stand at the international PSE conference in Garmisch-Partenkirchen

At the 15th International Conference on Plasma Surface Engineering held on September 12–16, 2016 in Garmisch-Partenkirchen, INPLAS was represented at the industrial exhibition with a joint stand. The following members participated in the network’s appearance at the conference: W&L Coating Systems GmbH, Advanced Energy Industries GmbH, FHR Anlagenbau GmbH, Fraunhofer FEP, Fraunhofer IST, IOT of the TU Braunschweig.

INPLAS series started in the VIP technical journal

This year, thanks to the dedication of members and management boards, an INPLAS series with technical articles started in the VIP journal (“Vacuum in research and practice”). The series consists of the following topics: cleaning, functionalizing and coating, optimizing products with atmospheric-pressure plasma; 3D coatings; product costs with plasma processes; large-area coatings. The first of a total of four parts has already been published in VIP’s October issue.

Active work of the working groups

Members and in particular a large number of partners from industry come together in the INPLAS working groups to discuss and bring forwards topics of common interest. The “Innovative plasma sources and processes” working group headed by Dr Bernhard Cord of Singulus Technologies AG met twice in 2016 – at Singulus Technologies AG in the spring and at Schaeffler AG in the autumn – to exchange ideas in the topics of target materials, power supplies, simulation, process control and monitoring.

The “Tool coatings” working group, headed by Hanno Paschke of the Dortmund Surface Technology Center DOC/ Fraunhofer IST met in Austria at Boehlerit GmbH & Co. KG and in Freiburg at the Trumpf Hüttinger GmbH & Co. KG. The project kick-off for the “Eco-Clean” project, pretreatment processes for complex-shaped cutting tools, and power supplies were in the foreground here.

In the “Combined surface technologies” joint expert panel (GA) the topics of Industry 4.0 and additive manufacturing were tackled in discussions at the German Research Association for Surface Treatment (DFO) in Neuss and at IGOS GmbH & Co. KG in Solingen.

In addition, the first meeting of the “Plasma4Life” working group was held in Göttingen, clearly showing the diversity and the potential of the topic of life science in plasma surface technology. Almost 40 participants from industry and research participants from industry and academia came together to discuss current issues in the fields of plasma technology and the life sciences. Interesting presentations from the fields of pharmacy, medical technology, hygiene, cell culture, microbiology, medicine, nutrition and the environment gave an impression of the enormous range of subject areas and pointed out current issues.

Further press and publicity activities / service projects:

- IP4Plasma, FAST, SafeWater, EU projects: responsible for the “Dissemination” work package
- 35th meeting of the “Tool coatings and cutting materials” industry working group with partners the Department of Machine Tools and Factory Management (IWF) of the Technical University of Berlin, the Fraunhofer Institute for Production Systems and Design Technology IPK and the Fraunhofer Institute for Surface Engineering and Thin Films IST
- PlasmaGermany sessions and workshops
- “Plasma science and entrepreneurship” workshop at the Fraunhofer Institute for Surface Engineering and Thin Films IST
- Participation and engagement in the strategy for photonics

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Arbeitsgemeinschaft Wärmebehandlung und Werkstofftechnik e. V.
www.awt-online.org

Deutsche Forschungsgesellschaft für Oberflächenbehandlung e. V.
www.dfo-online.de

Deutsche Gesellschaft für Elektronenmikroskopie e. V.
www.dge-homepage.de

Deutsche Gesellschaft für Galvano- und Oberflächentechnik e. V.
www.dgo-online.de

Deutsche Gesellschaft für Materialkunde e. V.
www.dgm.de

Deutsche Glastechnische Gesellschaft (DGG)
www.hvg-dgg.de

Europäische Forschungsgesellschaft Dünne Schichten e. V. (EFDS)
www.efds.org

European Photocatalysis Federation EPF
www.photocatalysis-federation.eu

Fachverband Angewandte Photokatalyse
www.vdmi.de/deutsch/produkte/angewandte-photokatalyse.html

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www.faps.uni-erlangen.de/mid

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www.light-and-surfaces.fraunhofer.de

German Flatpanel Display Forum DFF
www.displayforum.de

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Göttinger Research Council
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www.iccg.eu

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- Thomas, M.: Labbag® – WISA: Labor im Beutel – Labbag®, Netzwerktagung (eingeladener Vortrag), München, Deutschland, 24. Februar 2016.
- Thomas, M.: Functionalization and coating by plasma technology (eingeladener Vortrag); Plasma and Corona Surface treatment, Seminar Kopenhagen, Dänemark, 13. April 2016.
- Thomas, M.: Fundamentals and applications of plasma and corona surface treatment (eingeladener Vortrag); Plasma and Corona Surface treatment, Seminar Kopenhagen, Dänemark, 13. April 2016.
- Thomas, M.: Trends and applications – Plasma medicine and microplasmas (eingeladener Vortrag); Plasma and Corona Surface treatment, Seminar Kopenhagen, Dänemark, 13. April 2016.
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- Thomas, M.: PSE-Tutorial 2016: Fundamental and Trends of Plasma Surface Processing – Surface engineering with atmospheric-pressure plasmas, Tutorial, 15th International Conference on Plasma Surface Engineering (PSE), Garmisch-Partenkirchen, Deutschland, 11. – 16. September 2016.
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- Viöl, W.: Plasma-Hybrid-Verfahren, Kolloquium des Leibniz-Instituts für Oberflächenmodifikation, Leipzig, Deutschland, 28. Januar 2016.
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DISSERTATIONS

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Köhring, M.: Mikrostimmgabel-Chips-Photoakustik: Der Weg zum optisch integrierten Gassensor, Clausthal, Technische Universität, Dissertation, April 2016.

Stein, Christian (2016): Entwicklung von nanostrukturierten Nitriden und c-BN als Verschleißschutzschichten für Zerspanwerkzeuge. Zugl.: Braunschweig, Technische Universität, Dissertation, 2015.

DIPLOMA THESIS

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MASTER'S THESIS

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Barati, V.: Elektrische und optische Charakterisierung von technischen Plasmen mit unterschiedlicher elektrischer Anregung, Technische Universität Braunschweig, 2016.

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Menzel, R.: Charakterisierung des Verschleißverhaltens bei der Online Tiefenmessung im Kalottenschleifverfahren, Technische Universität Braunschweig, Dezember 2016.

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Schneider, E.: Tribologische Analyse von Ventiltriebskomponenten, Technische Universität Braunschweig, August 2016.

Schulze, S.: Realization Possibilities of Technical Changes for the Surface Protection Concept in Aircraft Industry, Technische Universität Braunschweig, August 2016.

Spreemann, D.: Charakterisierung von Hochleistungs-Impuls Magnetransputterprozessen im Labor- und Industriemaßstab, Technische Universität Ilmenau, Dezember 2016.

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Vogtmann, J.: Korrelation zwischen den Prozessparametern und der entstehenden Randschicht beim Plasmaborieren von Nickelbasiswerkstoffen, Technische Universität Braunschweig, Juli 2016.

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BACHELOR'S THESIS

Brandes, Y.: Elektrochemische Herstellung von H₂O₂ mit einer Mikrokanalzelle, Ostfalia Hochschule für angewandte Wissenschaften, 2016.

Britze, C.: Aufbau eines Spektrometers zur Vermessung von Interferenzfiltern mit hoher Flankensteilheit, HAWK Hochschule für angewandte Wissenschaft und Kunst Hildesheim/Holzminden/Göttingen, 2016.

Brückner, T.: Weiterentwicklung und Charakterisierung ternärer PACVD-Ti-B-N-Dünnschichtsysteme für den Einsatz im Aluminiumdruckguss, Fachhochschule Dortmund, August 2016.

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PATENT APPLICATIONS

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Josten, S.; Wittland, F.; Brückner, S.; Gerhard, C.; Viöl, W.: Verfahren zur Herstellen einer einbrennsilikonisierten Injektionsspritze.

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