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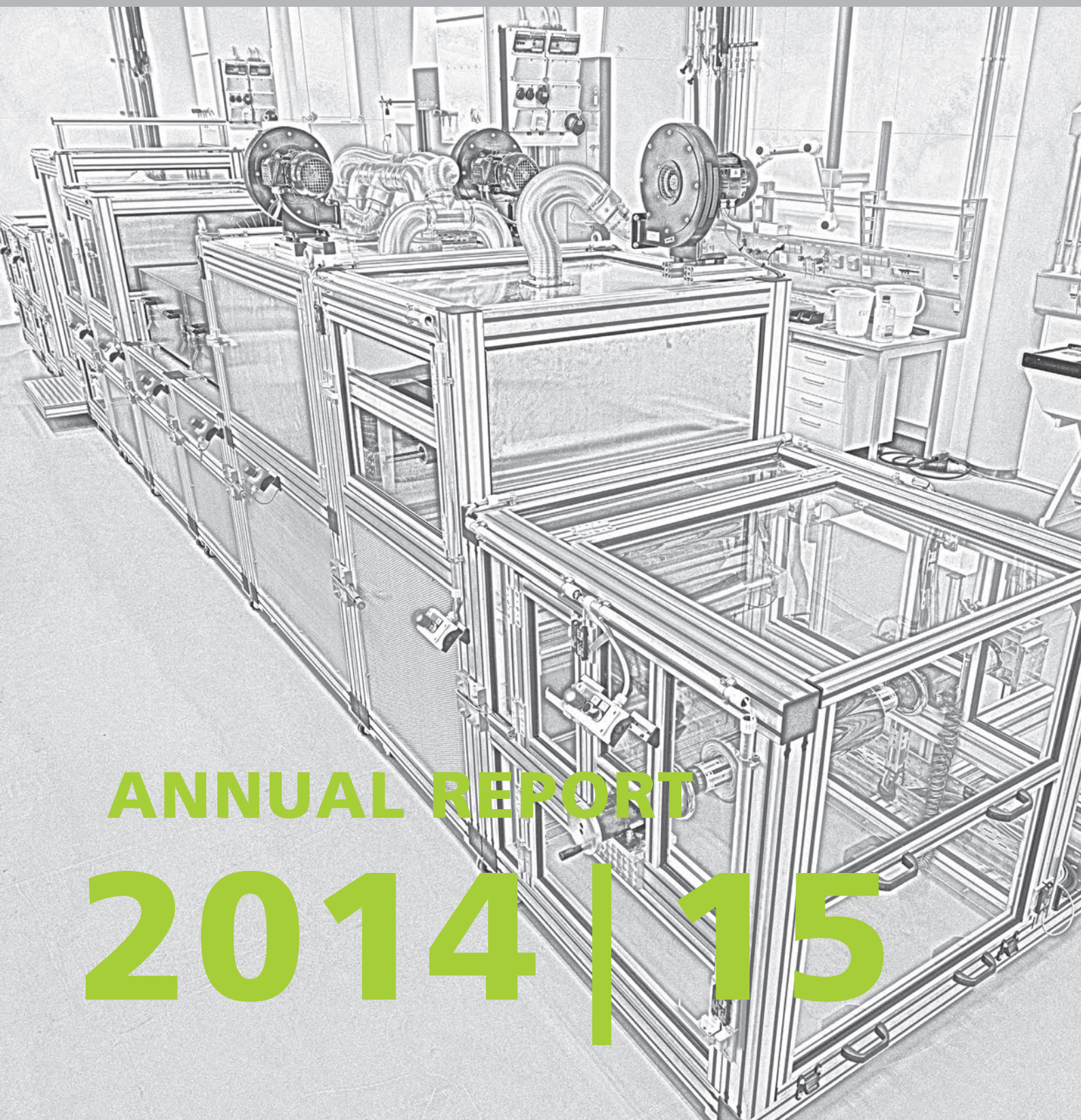
FRAUNHOFER INSTITUTE FOR SILICATE RESEARCH ISC



ANNUAL REPORT
2014 | 15

ROLL-TO-ROLL COATING FACILITY IN A CLEAN ROOM SETTING (CLASS ISO 8)

Due to its modular design, the facility can be flexibly adapted to different substrates and coating materials. Even solid substrates can be coated piece by piece. A sintering module for curing ceramic coatings is available.

PREFACE

Dear friends and partners of the Fraunhofer ISC,
Dear ladies and gentlemen,

There is no substitute for a responsible management of resources, not only due to our obligation to future generations but also under economic aspects. To ***use*** resources rather than to ***use*** them ***up***: As one of the leading materials research institutions the Fraunhofer ISC focuses on this change of paradigm. Against this background, the professional competence and production expertise of our researchers and scientists are put to use with a strong view to the efficient implementation of innovations in products and processes in cooperation with – and to the benefit of – our partners from large and small corporations alike.

We are playing a pioneering role in this change process through the initiation of new research institutions such as the Fraunhofer Project Group for Materials Recycling and Resource Strategies IWKS or the Application Centers for Resource Efficiency and Textile Fiber Ceramics at the universities of Aschaffenburg and Hof. Our research activities are dedicated to the key areas of energy, environment, and health. Our successes, such as the initiation and establishment of the European research platform »EIT – KIC RawMaterials«, the initiation of the German network »German Resource Research Institute« (GERRI), and the establishment of the German Phosphorus Platform under the tutelage of the project group IWKS in November 2013 and subsequent transfer to independent status as a nonprofit organization in March of this year with strong involvement of industrial partners, show that we are on the right track. In this context, we are particularly pleased to note that innovative and sustainable research topics are ackowledged externally as well. Dr. Karl Mandel has been awarded the »Deutscher Studienpreis« of the Körber Foundation, the most prestigious junior researcher award in Germany, for his work titled »Wasserreinigung und Rohstoffrecycling mit nanomagnetischen Helfern (Water cleaning and raw materials recycling using nano-magnetic aides)«. For the same work he has also been awarded the Hugo-Geiger prize of the Fraunhofer-Gesellschaft and the Rektor-Max-Meyer award, which he accepted in Würzburg.

Congratulations from the entire Institute to Dr. Mandel for these awards.

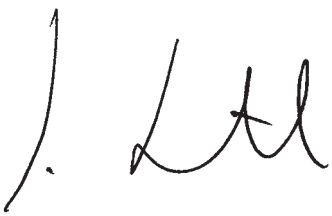
Meanwhile, the project group IWKS has grown to a staff size of 60, and successfully completed its intermediate evaluation in the autumn of 2014. It was given the green light for the continued construction and funding of the new buildings at the two sites at Alzenau and Hanau. Last June, the Fraunhofer Center for High Temperature Materials and Design HTL established the Fraunhofer Application Center for Textile Fiber Ceramics in a cooperation with the renowned textile experts of the University of Hof-Münchberg. The aim of this cooperation is to discover new pathways in the production of fiber-reinforced materials. The completion of the new building for the Center HTL in the spring of 2015 meant that the more than 60 staff members in Bayreuth reached another important milestone.

The Center for Applied Electrochemistry also passed an intermediate evaluation in the past November with flying colors. The group of external reviewers from industry research emphasized the scientific excellence as well as the pronounced orientation on applications and the excellent infrastructure that have been established at the Fraunhofer ISC in recent years concerning the development of battery materials and storage concepts and the testing of battery cells. In addition, it was confirmed that the Fraunhofer ISC is one of few outstanding institutes performing research in the field of solid body electrolytes in Germany. The steadily increasing economic returns of the young center are evidence for the well chosen topics.

Examples of the large bandwidth of the interests of the Institute include successful product developments, such as a new generation of dental materials, presented in an editorial by our long-standing cooperation partner VOCO GmbH, the ArtCut®

device for medical research, and the Gesture Control Seat that has been implemented with ISRINGHAUSEN. The range of research projects – some bilateral, some within European networks – includes the replacement of critical elements in permanent magnets, the replacement of lead in printing inks for glass or in high-tech applications, such as flexible electronics, the conversion of materials formulations to the use of renewable raw materials, such as in the development of bio-degradable barrier layers, and demonstrates the versatility of our approaches to the sustainable utilization of resources. Read more about these and other topics in the present annual report that has been designed to showcase our project work.

At this point, I would like to express my gratitude to all staff members of the Fraunhofer ISC – at the parent Institute, in the project group IWKS, and in the Center HTL – and the staff of the chair for Chemische Technologie für Materialsynthese at the University of Würzburg for their highly committed, creative, and competent work. The success of the Institute is only possible with their combined effort. I also wish to thank our partners from industry and research for the constructive cooperation and the confidence in our ideas and solutions. On behalf of all members of staff, I wish to thank the Fraunhofer-Gesellschaft, institutional sponsors and partners and, in particular, the Bavarian State Ministry of Economy, Media, Energy, and Technology as well as the Hessian Ministry for Science and the Arts for the confidence they placed in our strategic planning and for their generous funding of our projects.



Prof. Dr. Gerhard Sextl

Directors of the Fraunhofer ISC
Prof. Dr. Gerhard Sextl, Director
Dr. Rolf Ostertag, Deputy Director (retired in July 2015)

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http://www.isc.fraunhofer.de/publikationen.html

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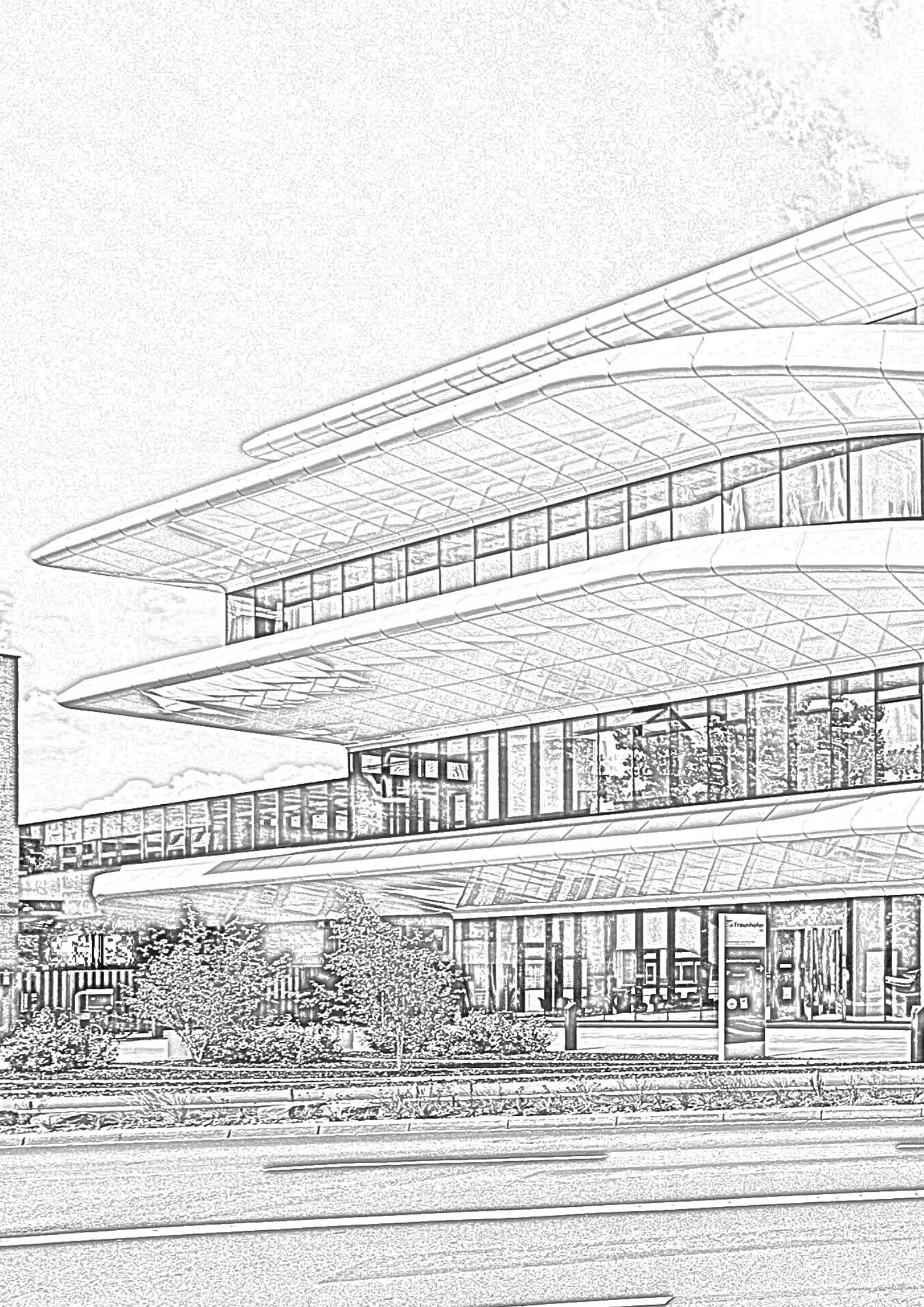

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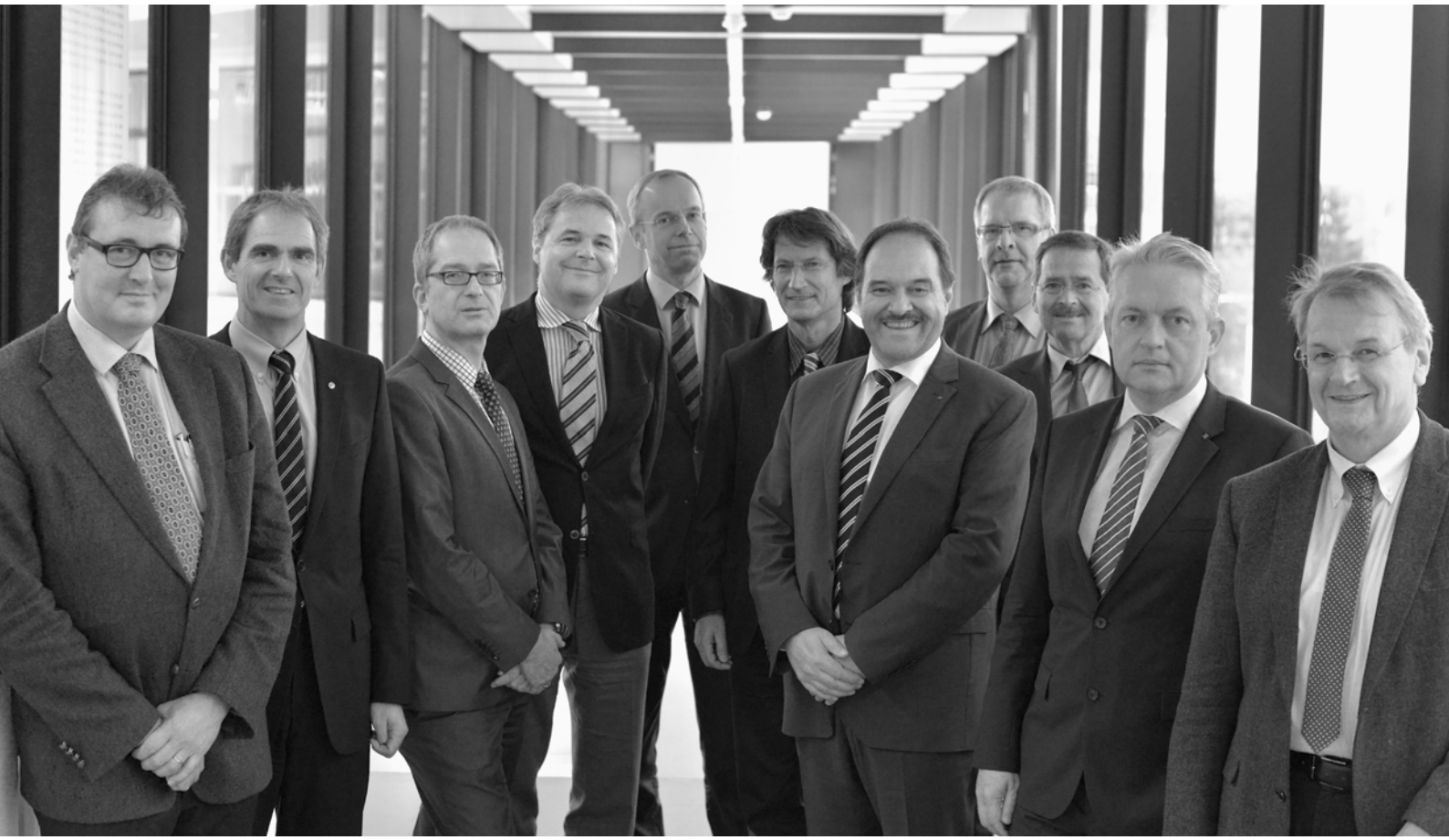


MATERIALS-BASED SOLUTIONS

THE FRAUNHOFER ISC AT A GLANCE


ostics
Corrosion Protection
MOCER® s
Printed Electronics
Construction Chemistry
Battery Testing
Supercaps
trategies
GMP compliant environment
Electrochemical Analysis
Transparent Conductive Oxides
InnoSolTex®
Diagnostics
Quality Control
Packaging Technology
Surface Technology
Nanoparticles
Characterization
Colored Coatings
truction
Live Cell Imaging
Protective Coatings
Enviromental Monitoring
als Development
Special Devices
High Temperature Materials
Actuators
ion Processes + Synthesis
chnology
Upscaling
Flexible Electronics
Sol-Gel Materials
Environment
Haptics
Damage Analysis
Transmission Control
Sensors
Glass
Materials Efficiency
Functional Coatings
Pilot Production
Silicone Development
Electrochromics
cess Technology
Heat Storage
Tissue Engineering
Ceramics
Energy
Wound Dressings
Ultrabarrier Coatings
onics
crooptics
Energy Storage Concepts
Biofunctionalization
Energy-efficient Heat Treatment
ds

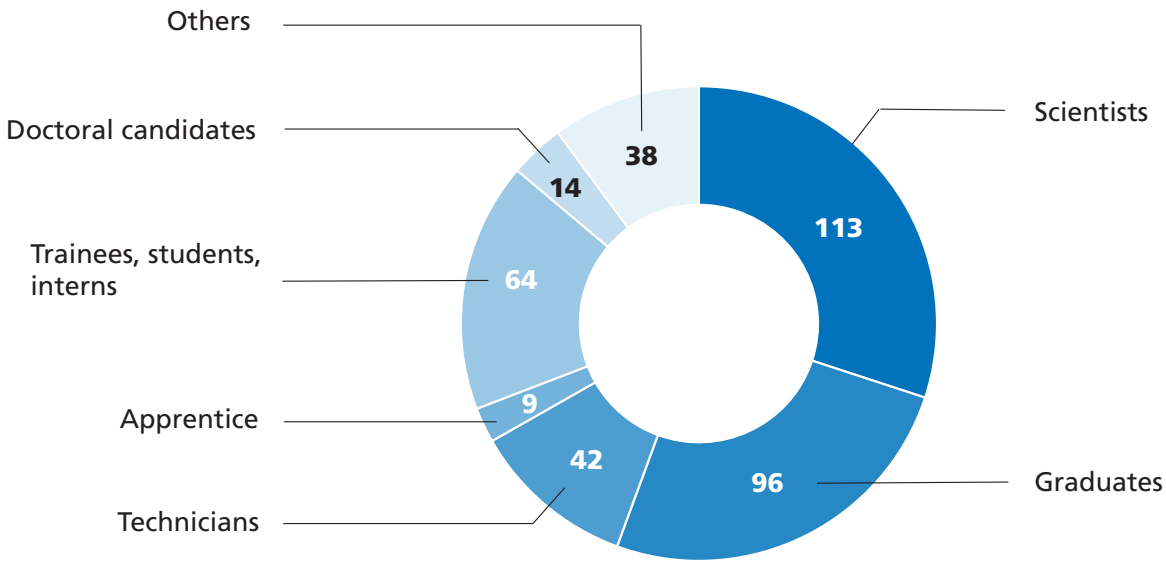
KEY FIGURES

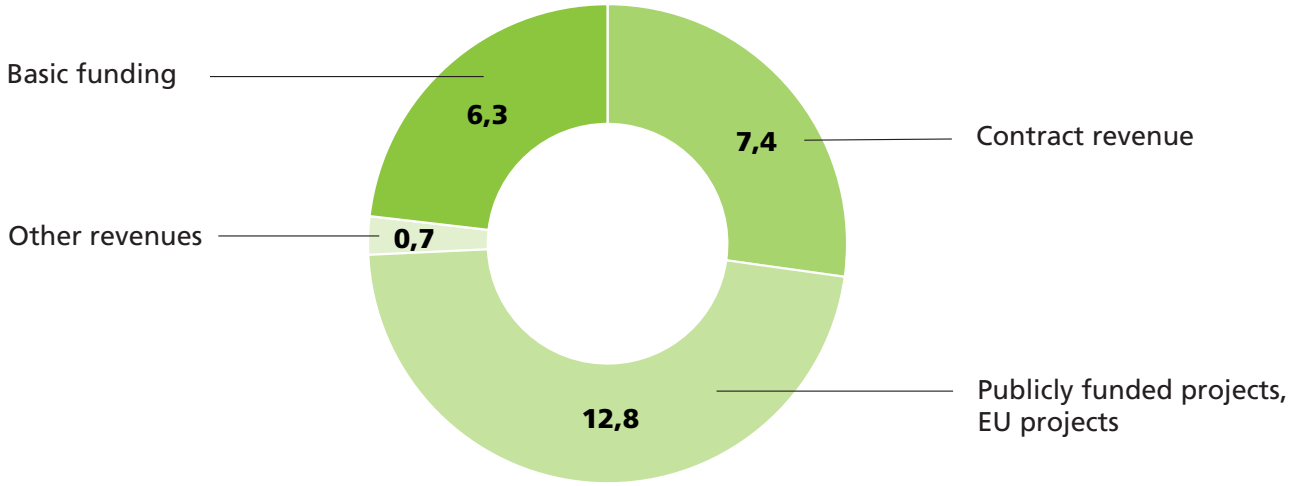
Workforce 2014	ISC	HTL	IWKS	Group
Permanent staff	181	29	41	251
Scientists	69	15	29	113
Graduates	77	10	9	96
Technicians	35	4	3	42
Other staff members	56	43	26	125
Apprentices	9	0	0	9
Trainees, students, interns (as of Dec. 31, 2014)	23	30	11	64
Doctoral candidates (*)	6/11	4	4/11	14/22
others 18	9	11	38	

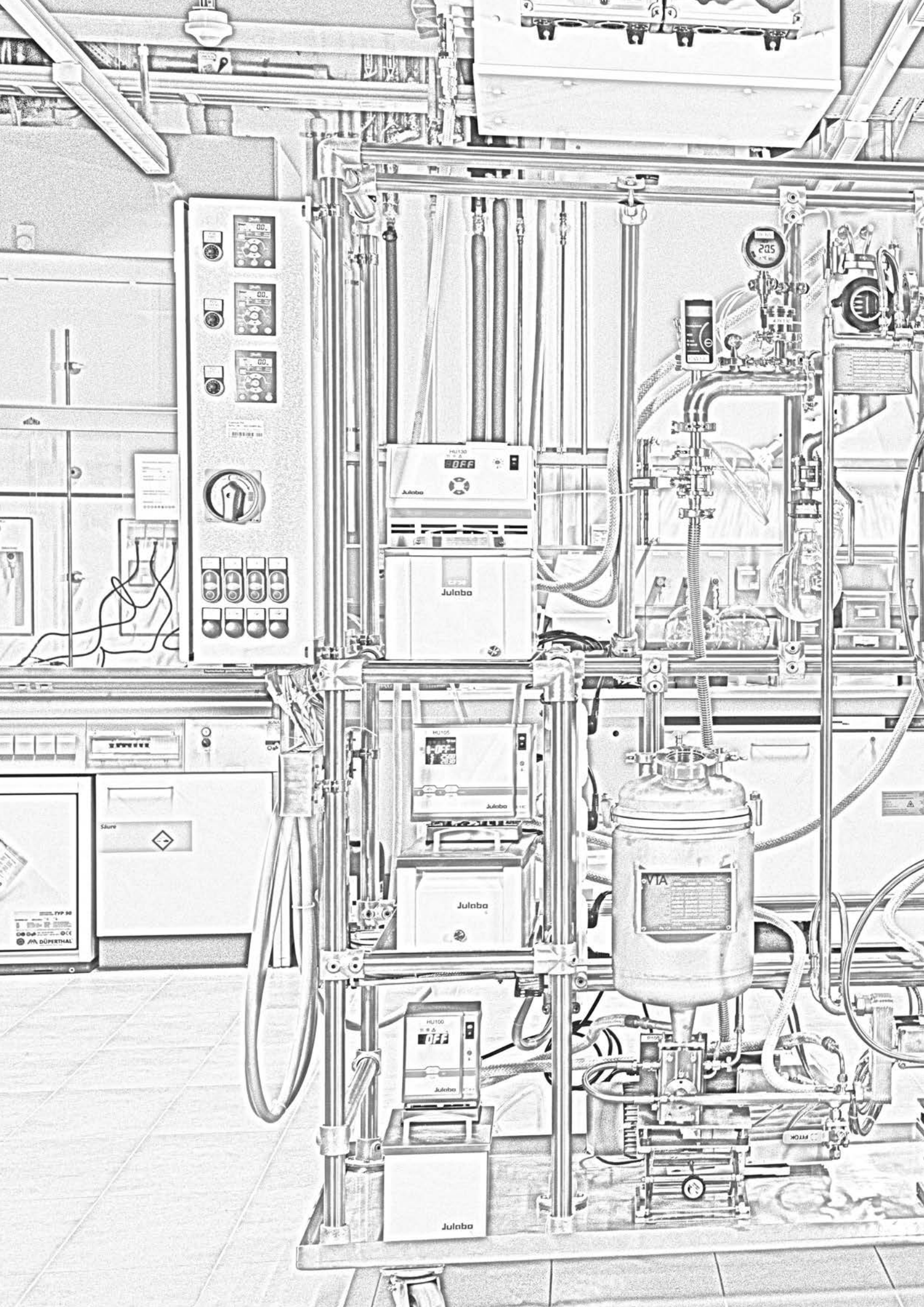
(*) in addition to doctoral candidates at the associated universities of Würzburg, Augsburg, Darmstadt, Gießen

Personnel (head count)	**376**

Financing 2014	ISC	HTL	IWKS	Group
Operating Budget Expenditure	17.3	5.0	5.3	27.6
Personnel expenses	11.3	2.3	2.4	16.0
Non-personnel expenses	6.0	2.7	2.9	11.6
Operating Budget Revenue	16.8	5.1	5.3	27.2
Contract revenue	5.4	1,3	0.7	7.4
Publicly funded projects, EU projects	6.5	3.2	3,1	12.8
Other revenues	0.6	0.0	0,1	0.7
Basic funding, internal programs	4.3	0.6	1.4	6.3
Capital expenditure	**0.9**	**0,4**	**1.6**	**2.9**
Capital revenue	**1.2**	**0.5**	**1.6**	**3.3**
Industrial revenue	31.1%	27.5%	13.5%	27.1%
Investment	**3.3**	**9.4**	**1.1**	**13.8**
Operating Budget Total Expenditures (in Mio €)	**21.5**	**14.8**	**8.0**	**44.3**


Others
38
Doctoral candidates
14
Trainees, students, interns
64
Apprentice
9
Technicians
42
Scientists
113
Graduates
96


Basic funding
6,3
Other revenues
0,7
Contract revenue
7,4
Publicly funded projects, EU projects
12,8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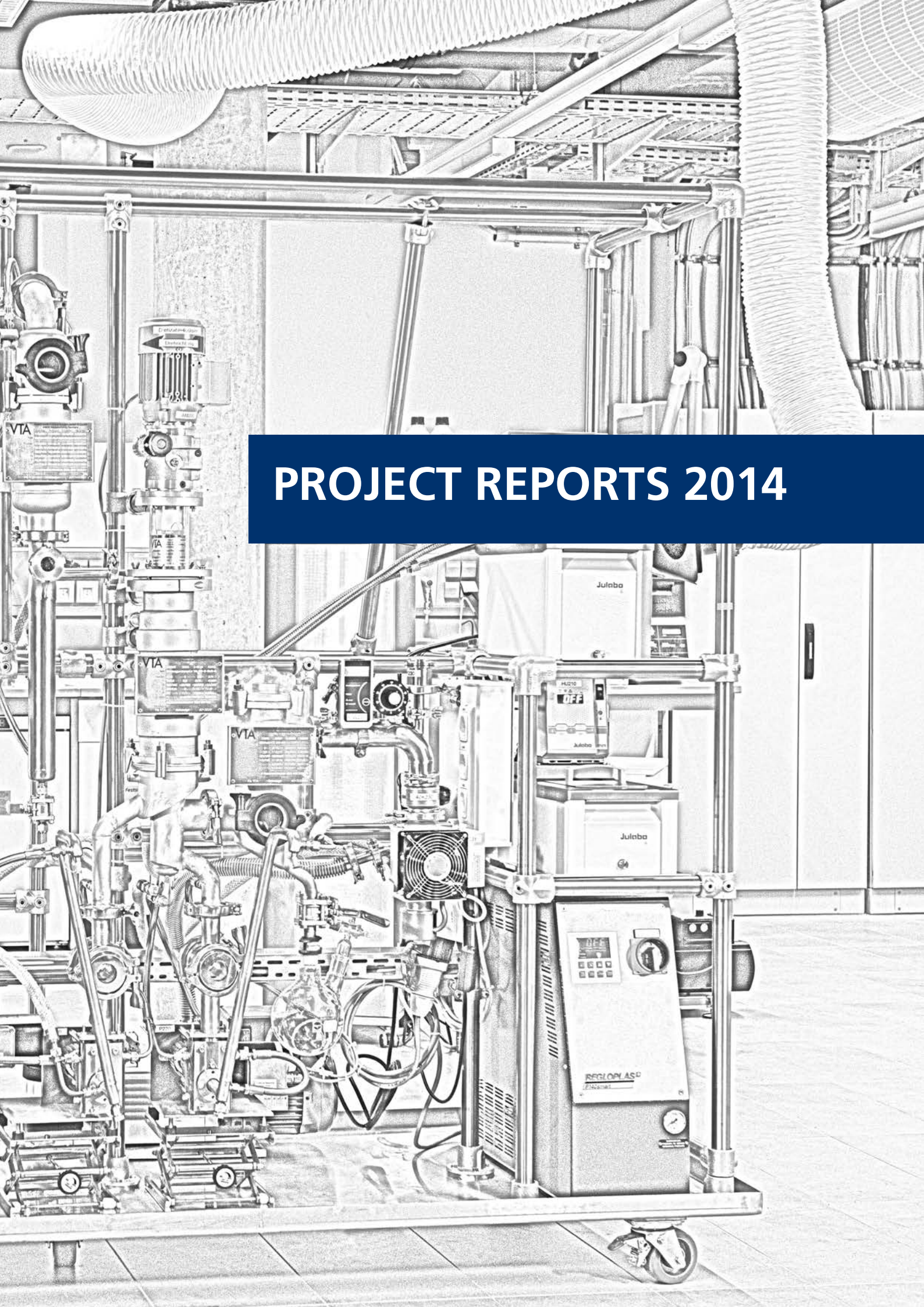


PROJECT REPORTS 2014

ADMIRA FUSION – DENTAL ORMOCER®s 4.0

GUEST EDITORIAL BY DR. REINHARD MALETZ, VOCO GMBH – THE DENTALISTS

Whenever you've got two very good materials, it is the nature of science to ask, if and how these two materials can be combined in order to pool their strengths to obtain a material with even better performance. The development of the ORMOCER®s (a registered trademark of the Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e. V.) in the 1990s made a versatile material available that also drew much attention in the field of dentistry. Used as the basis of filling materials, it afforded improved biocompatibility, reduced shrinkage, and less shrinkage stress. Unlike conventional monomers, which tend to be rather small, ORMOCER®s are based on an extensive silicon-oxygen network linked to polymerizable methacrylate units by means of linking units.

Combining two worlds of materials

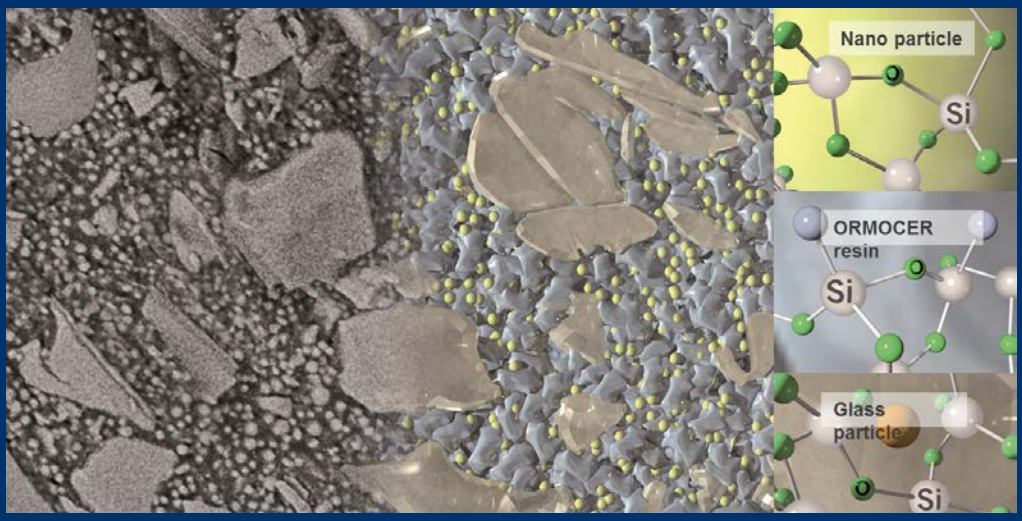
Nanotechnology complemented the group of materials that are used in dental filling therapy to include the nanohybrid composite which combines particularly high stability and pleasant handling properties due to excellent materials properties. Combining these properties with the benefits of the ORMOCER®s – the implementation of this vision became a goal of research and development. But the implementation proved to be an even larger and more time-consuming challenge than originally thought. Because it is not sufficient, obviously, to have participated in the development of these two technologies in significant manner and to have made the results real and usable in the form of filling materials (namely Admira, Grandio, and GrandioSO). In fact, many years of intense and detail-oriented research were required to develop a filling material that combines nanohybrid and ORMOCER® technologies and, at the same time, is even more strongly ceramic by nature.

Not a pure ORMOCER® base

We've come a long way: Even though the enormous potential of the substance class of the ORMOCER®s was discovered early and then developed further later on, the dental community preferred to speak of ORMOCER®-based composites in the early 2000's, and rightly so, since classical dental monomers were being added to the pure ORMOCER® chemistry to improve the processing properties (in addition to initiators, stabilizers, and pigments). This means that certain amounts of monomers were added to the resin matrix in order to be able to adjust the viscosity of the matrix appropriately such that the filling materials are easy to process. In the absence of these added monomers, the consistency of the material would have been too firm.

Monomers increase shrinkage stress

During the polymerization process, the initially mobile monomer molecules form an increasingly denser network. Initially, the material can compensate for the volume shrinkage by flow processes (pre-gel state). However, once the network becomes sufficiently dense such that the gelling point is exceeded, the resulting tension can no longer be compensated by flowing material (post-gel state). The tensions triggered in the post-gel state then produce internal tension in the material itself, which, in turn, can lead to detachment from the cavity walls and to the formation of gaps. Due to the inorganic basic structure of ORMOCER®, ORMOCER®-based filling materials contain significantly less monomers than conventional composites. Accordingly, the development of the ORMOCER® technology alone was a major step forward due to the reduced shrinkage and lessened shrinkage stress as well as the improved tissue compatibility.



Good, but not yet good enough

But, there was a notion beyond all doubt that the reduction of the shrinkage stress would be significantly larger, if the classical monomers were absent altogether.

This provided a clear description of the starting situation and the goal: A material containing none of the classical monomers, such as BisGMA, TEGDMA or UDMA, that is based on an ORMOCER® resin was not available at the time. This was to change in order to continue the development of the ORMOCER®s.

Continued development

The ORMOCER® matrix has a tripartite structure: It is composed of the inorganic-condensible molecule segment, the organic-polymerizable molecule segment, and the linkage unit. This unit is variable in terms of its length, structure, and composition. The role of the inorganic unit is to establish a nanoscale inorganic Si-O-Si network. This inorganic polycondensation is the final synthetic step of the ORMOCER® synthesis, which means it proceeds already during the production of the matrix. The organic polymerization is triggered by blue light no earlier than when the filling material is being applied in the cavity. As a result, an inorganic-organic hybrid polymer is formed that differs from the classical methacrylates, which contain organic monomers only, due to its backbone being based on an Si-O framework.

Functionalization leads to fusion

The crucial setscrew that needed to be turned here was the so-called linkage part of the resin matrix. In order to make the leap from the ORMOCER®-based composite to a filling material based on a pure ORMOCER® resin without any classical monomers being added, there was a need to have special linkage units that keep the viscosity of the resulting condensate low, yet optimize parameters such as the translucency or the water uptake. The pure ORMOCER® resin matrix is characterized, especially, by its large molecules that offer significantly more linkage options (double bonds) than classical monomers. These double bonds ensure a very firm and safe bond that is produced by polymerization. Accordingly, no matrix components can be released, there is no leachable matrix. Moreover, the very large ORMOCER® molecules provide a chemical-physical foundation for the significant reduction of the volume shrinkage during the light-induced organic polymerization (curing) as compared to a classical dimethacrylate-based composite as such.

Fusion leads to »Ceramics for filling«

The absolute absence of classical monomers is just one of the features of the filling material. Another novel aspect is that this ORMOCER® resin was combined with nanohybrid filler technology. Silicon oxide structures serve as the basis both for the filling bodies (nano- and glass ceramics filling bodies) and for the resin matrix. This is a unique procedure to date and provides the basis of the novel »Pure Silicate Technology«. This was made possible only by continuing the development of the nanohybrid technology to the sol-gel procedure, the first adaptation to this class of materials of its kind. This transfer of the nanohybrid technology into the ORMOCER® resin matrix allowed the filler content in the developed filling material to be adjusted to 84 percent by weight.

Potential put to use

As a result, Admira Fusion developed by VOCO in cooperation with the Fraunhofer ISC differs in many ways from previous commercially available filling materials based on ORMOCER®s. The changes can be described as follows: The enormous potential of the ORMOCER®e, which thus far could not be fully utilized, can now be put to use by means of the »Pure Silicate Technology« due to the absence of classical monomers and the novel successful incorporation of

nanohybrid technology into the resin matrix. In short: A good product was improved even more and a new way has been found to realize dormant potential.

The new filling material is available as a material for plugging (Admira Fusion) and as a fast-track variant for increments of up to 4 mm (Admira Fusion x-tra) and can be processed just like any conventional composite. It is compatible with all bondings, light curing equipment, and all composites. Its unequaled strengths are the very low polymerization shrinkage (1.25 percent by volume) and the low shrinkage stress (3.87 MPa), the high biocompatibility as well as the top level mechanical properties.

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GLASS CERAMIC DENTAL PRODUCTS FROM PROJECTS WITH ...

Markus Vollman
Director Development & Application Technology, DeguDent GmbH, Hanau

»An affiliate of the global dental corporation Dentsply International Inc. – DeguDent GmbH in Hanau is one of the leading companies in the dental technology industry. A novel dental glass ceramics, developed in cooperation with Fraunhofer ISC, meets the strict requirements in terms of aesthetics and materials technology and is a milestone for modern, efficient, and convenient patient management. This very innovative material and its technical implementation were achieved in about four years, a relatively short time for a development – a success that was made possible only due to the excellent cooperation with the Fraunhofer team around Dr. Durschang and Dr. Probst.«

© DeguDent GmbH, Hanau

Henry R. J. Rauter
Executive Manager, VITA Zahnfabrik – H. Rauter GmbH & Co. KG, Bad Säckingen

»We, being a family-owned business, have successfully developed products for dental prosthetics in the past 90 years. This was the foundation on which the cooperation with the Fraunhofer ISC was established aiming to develop a dental glass ceramics for a new product generation, ultimately leading to VITA Suprinity after just four years of concentrated materials and process development. We truly appreciate to have found the Fraunhofer ISC as a highly competent partner for our product development and are looking forward to further projects.«

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SKINHEAL – EFFECTIVE IN WOUND HEALING

DR. JÖRN PROBST | ✆ +49 931 4100-300 | joern.probst@isc.fraunhofer.de

Chronic open wounds are amongst the most common ailments and, with the costs adding up to approximately 8 billion euros, are one of the major burdens on the German public health system. They are caused by widespread diseases, such as diabetes or cancer, and afflict mainly the elderly. Considering the ongoing demographic change, the case numbers, and therefore the costs, are expected to keep rising in the future. The managements of chronic wounds faces strict quality and economic requirements. These treatments are protracted and the recurrence rate is high. Not least because these patients, being strongly restricted in their quality of life, are hard-pressed to comply with the specifics of the therapies.

A correct, reliable, and, most importantly, early therapy allows one of two amputations to be prevented, e.g. in the case of diabetic foot syndrome. This would not only mean less suffering by the afflicted individuals, but would also save the German public health system some 400 million euros in costs. Quality and economic efficiency would go hand-in-hand with medical progress.

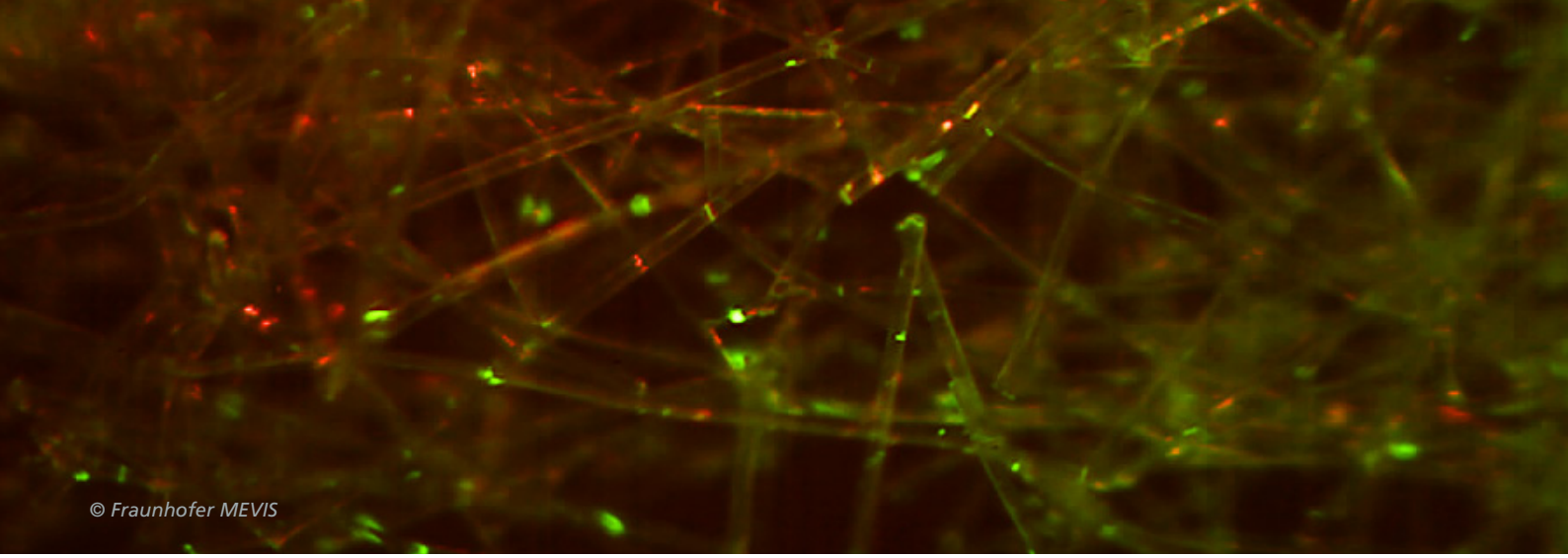
Five Fraunhofer institutes pooling their competence

In order to be able to develop and evaluate effective and cost-efficient modes of therapy for chronic wounds, researchers from five Fraunhofer institutes (ISC, IGB, IME, EMFT, and MEVIS) joined forces and pooled their competence in the »SkinHeal« project. Coordinated by the Fraunhofer ISC, the core goal of this project was to use an artificial wound model to optimize the management of chronic wounds and to keep it affordable in the future as well.

For this purpose, four developments were combined in this project, each of which pursue this goal, but can also be marketed independent of each other.

One element is the newly developed in-vitro wound model for chronic wounds. This was based on a pre-existing model of healthy skin. By injecting cytokines – i.e. proteins regulating the growth and differentiation of cells – a specific wound milieu is generated in the model. The cytokines ensure that certain macrophages of the type characteristic of chronic wounds are generated. Following this preparation, the skin model is then standardized and injured reproducibly using the ARTcut® – Artificial Tissue Cutter device developed by the Fraunhofer ISC. This results in the desired chronic model wounds, which have well-defined properties in terms of their shape, diameter, and depth.

The new in-vitro model can be used to test various modes of therapy at a very early stage. It allows wound inserts, such as the silica gel fiber fleece developed by the Fraunhofer ISC, as well as pharmaceutical agents or a combination of both to be inserted and tested for their efficacy. Moreover, models of this type are a long-sought alternative to animal experiments, right in line with the »3R rule« of William M.S. Russell and Rex L. Burch. The three Rs represent »Replacement, Reduction, Refinement«. The number of animal experiments can be minimized through the use of alternative methods, the human cell-based model systems are more suitable than animal experiments in terms of the applicability of the results to humans, and the ultimate goal of »Replacement« is to do without animal experiments altogether, depending on the specific case.



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Using actuators to monitor wound healing

Another focus of the project was on the incorporation of microfluidic actuators and sensors into wound dressings – implemented in a leading role by the Fraunhofer EMFT. These actuators and sensors allow the wound exudate to be removed without having to change the dressing. Subsequently, the wound exudate is analyzed for characteristic biomarkers to obtain feedback on the status of the healing process. Alternatively, the actuators can supply oxygen to the wound, e.g. for an oxygen therapy.

The third focus is on the modification of wound inserts that have already been approved for the market. For this purpose, e.g. special silicates encapsulating active substances, such as immuno-therapeutic agents, are incorporated into the fiber matrix. These agents are released during the wound healing process and can promote the healing. It is easy to check and confirm the proper function of the immune therapeutic agents released from the matrix using established testing methods.

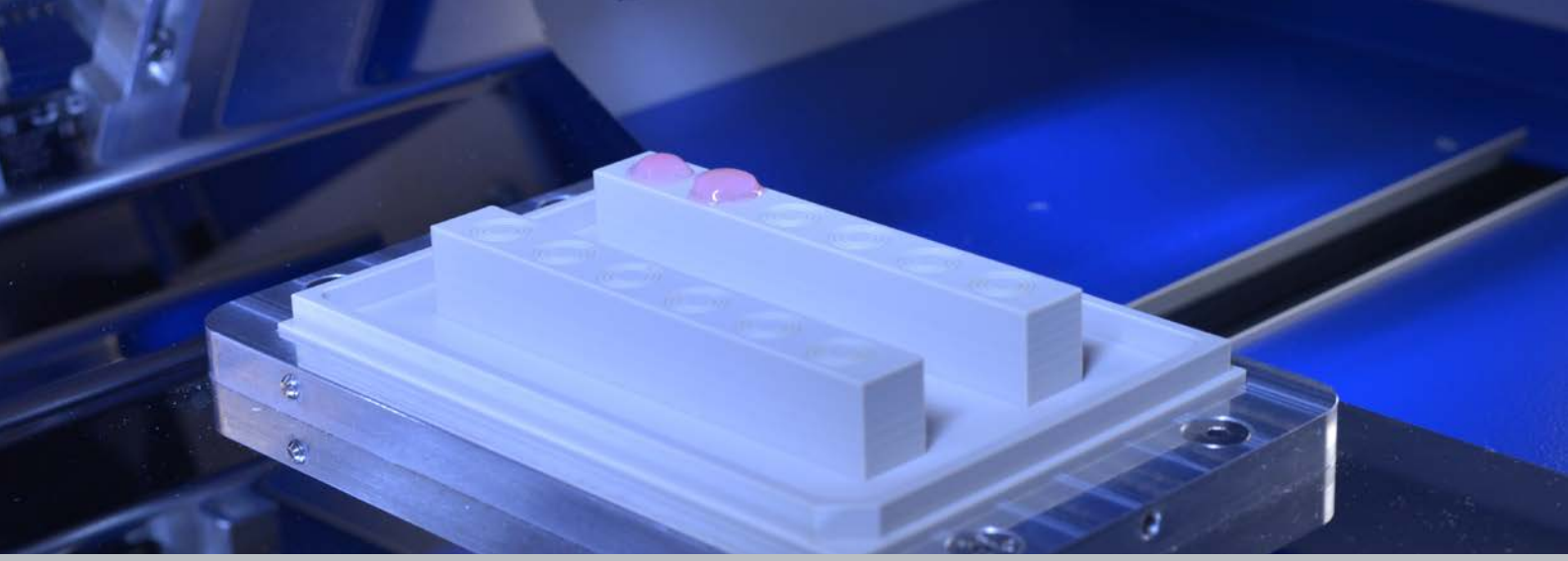
Patients document own wound status via smart phone

The fourth development in the scope of the »SkinHeal« project aims to improve fluorescence-based imaging, a method that obtains information about the progress of the wound healing process for researchers, attending physicians, caregivers and, in the long term, even the patient. Surface-modified luminescent nanoparticles developed at the Fraunhofer ISC that bind to characteristic biomarkers of wound healing, such as the types of macrophages mentioned above, are used for effective optical imaging. A specific ratio of these biomarkers identifies the status of wound healing and indicates whether a normal, i.e. healthy, or a chronic course is ongoing. Fluorescent particles that fail to bind due to the absence of biomarkers are simply eliminated by the body. But if they do bind, this is visualized by an appropriate fluorescence detector after excitation by LED light – the wound is »illuminated« in the various colors of the labeled biomarkers. In the long term, periodical imaging of the wound status by the patient or care-providing relatives, e.g. by means of a smartphone, might be feasible, making home monitoring an option. The first prototype has already been developed for this purpose and allows a diagnosis to be made by recording the macrophage distribution by means of two colored diodes and analytical software.

This kind of self-diagnosis would spare the patient many visits to the doctor, possibly even hospital stays, which would reduce the cost of health management.

But SkinHeal can reduce other costs as well

It is self-evident that the new in-vitro wound model can be used in other fields of research as well, such as in the development of new agents and medications. Making the selection of the most suitable candidate agents more rapid and more efficient on the basis of the efficacies determined in-vitro, reduces the number of tests needed in product development and therefore saves time and costs. In addition, clinical studies can be planned much more efficiently based on the results of preliminary in-vitro experiments, and the number of required animal experiments can be minimized.



WOUNDS WORKING FOR RESEARCH

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Anybody working on modes of therapy for diseases needs to have statistically robust research and test models. For comparative wound healing studies, the reproducibility of such »in-vitro wounds« is of importance. In cooperation with the project group of the Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB and the translation center »Regenerative Therapies for Cancer and Musculoskeletal Diseases« (TLZ), the Fraunhofer ISC developed and patented the »ARTcut® – Artificial Tissue Cutter« device that can be used to produce standard wounds in artificially produced skin. This machine is unique. The focus of the Fraunhofer scientists is on chronic wounds that occur, e.g., as a corollary of age-related diabetes – type II diabetes mellitus – i.e. the so-called diabetic foot syndrome. Third only to »decubitus« (bedsores) and »venous leg ulcer«, this is one of the most common chronic wounds of the skin.

Simulating chronic wounds

To be able to research chronic wounds, artificial skin models are used to »simulate« this kind of wound by biochemical means. Cytokines – i.e. proteins regulating the growth and differentiation of cells – are injected into the skin model and cause a certain type of macrophage that is characteristic of chronic wounds to arise. Then the ARTcut® is put to use as it allows reproducible injuries to be made in the suitably prepared skin models. This is a prerequisite for research and development to obtain statistically relevant and reliable information for the evaluation of new therapeutic options.

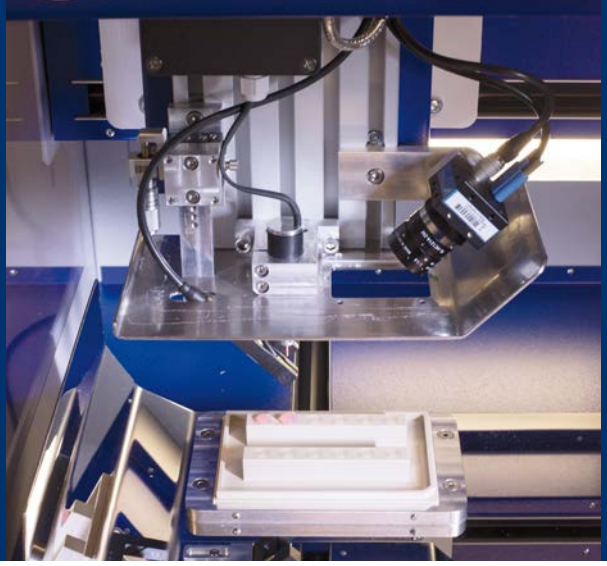
Error-free reproducibility

ARTcut® produces uniform and standardized injuries in the samples under sterile conditions. The wounds are made by a software-controlled hollow cylindrical drill. Monitored by a photoelectric barrier, the drill moves to the surface of the skin model and drills into the artificial skin at a defined rotational speed and advancement rate. The depth of the wound thus made is adjusted separately such that the type of injury becomes uniform. An additional quality control involves a camera system that detects and saves images from the drilling process. The working space inside the unit can be sterilized in time-controlled manner by means of a UV-C lamp.

ARTcut® is the first device of its kind that allows uniform wounds to be made reproducibly in all samples on a microtiter plate in a short period of time. This is a significant improvement for the throughput in sample preparation. And errors are virtually excluded. If, in contrast, a skin model of this type is manually injured by a member of the research staff, individually different injuries are produced despite all diligence as nobody can place 50 or more identical wounds of the same depth and with the same force. But for research purposes, it is crucial that the injuries are identical to allow the results to be compared later on.

Evaluation of new therapeutic options

Wound therapies can involve either wound dressings, such as the silica gel fiber fleece developed by the Fraunhofer ISC, or pharmaceutical agents or a combination of both. After the injury is made in the skin model, various therapeutics are inserted



together with diagnostic agents by means of which the wound healing process can be tracked. The latter consist of luminescent nanoparticles whose surface has been functionalized with specific antibodies. They bind to specific biomarkers from which the wound healing status can be deduced.

Minimizing animal experiments

The use of in-vitro wound models is getting more common in wound healing research. This is a first step in the attempt to closely mimic real-life conditions to allow therapies developed on this basis to be transferred to humans. The overriding rationale underlying all in-vitro models is the »3R rule« of Russell and Burch: »Replacement, Reduction, and Refinement«. In this vein, one of the goals in the development of the ARTcut® was to minimize, replace or at least limit the use of animal experiments. The more information about wound healing processes and new forms of chronic wound therapy can be deduced from a model of this type, the fewer animal experiments need to be done.

ARTcut® – flexible and modular

ARTcut® is intended to be used not only in Würzburg. The scientists of the Fraunhofer ISC aim to advance wound research throughout the world with the development they made. Since the device was designed as a highly flexible modular unit, it can be adapted and/or appropriately expanded easily to suit any given individual needs in terms of function and design. The next generation of the ARTcut® is already being planned and will perform additional routine work, such as the biochemical production of the wound milieu of chronic wounds or the defined addition of a therapeutic agent.

Long-standing expertise in the field of biocompatible and biodegradable materials

Newly established under the direction of Prof. Dr. Heike Walles from the Fraunhofer IGB, the translation center »Regenerative Therapies for Cancer and Musculoskeletal Diseases« (TLZ) at the University Hosipital of Würzburg aims to serve as a partner of industry to support the rapid translation of developments from research into new products.

The scientists at the translation center work with engineers from the Tissue Engineering and Regenerative Medicine (TERM) chair of the University Hospital of Würzburg and of the Fraunhofer ISC in Würzburg. Other closely associated institutions include the Muskuloskelettale Zentrum Würzburg (MCW), the chair for Funktionswerkstoffe der Medizin und der Zahnheilkunde (fmz), the Deutsches Zentrum für Herzinsuffizienz (DZHI), and the Tumorzentrum of the University of Würzburg (CCC).

Projects that are ongoing in cooperation with the Health Department of the Fraunhofer ISC include the development and testing of new substrate materials for tissue engineering and bio-functionalized particles for diagnostics and therapy. The Fraunhofer ISC is in possession of long-standing expertise and comprehensive equipment in the area of biocompatible and bioresorbable materials and makes laboratories available, at a close-to-GMP standard, for the development, analysis, and production of cell-based substrate systems.

FRAUNHOFER ATTRACT 3DNANOCELL

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The research aim of ATTRACT 3DNanoCell, headed by Prof. Dr. Doris Heinrich, is to investigate the influence of the environment and material surfaces on living cells to control cell functions. The migration and adhesion of viable cells are essential key factors for many processes in the body, for example for the formation of tissue, the regeneration of tissue or for immune reactions. Likewise, the incorporation of implants in the body is determined to an essential degree by these factors. These investigations are aimed to promote further developments of implant materials and their surface design as the basis for a new generation of implants.

Cell migration and cell adhesion

The term cell adhesion is used to describe the ability of cells to attach to a surface and to remain adherent. One example is adhesion of immune cells in the bloodstream to the vessel wall, if a specific signal calls for an immune reaction in this location. During immune response, specific cell types migrate into the surrounding tissue where they actively fight a pathogen. Since different cell types usually perform very different tasks in the body, they show different types of migration and adhesion mechanisms.

Cell models

Different model cell systems are available for investigation of these inherent processes of the body. The basic requirements for model systems of viable cells include easy availability, easy culturing, and the rate at which processes proceed. These model organisms are cultured specifically for research and they are well-documented, meaning that established and confirmed investigation methods can be utilized in this context. ATTRACT 3DNanoCell, for example, utilizes models of this type for initial investigations of the biocompatibility of materials and surfaces. Based on these model organisms, the use of human cell lines or primary human cells, which takes more time and is more expensive, for a confirmation of the tolerability in humans can be reduced to a low level.

Simulation of realistic model environments for cell migration

Not only the chemical composition of the surrounding milieu and the temperature, but also the topography of the cell environment plays an important role for the behavior of biological cells. While common cell cultures usually reside on a planar surface (nutrient substrate, carrier), the environment inside the body is more complex and usually has a three-dimensional structure. For this reason, various 3D substrates are being tested in cooperation with other departments of the Fraunhofer ISC for their suitability as model environments, such as TPA-written 3D scaffolds or silica gel fiber fleeces, a development of the Fraunhofer ISC for the fields of wound healing and tissue engineering.

The underlying aim of the development of model environments of this type is to induce a realistic behavior in the viable cells in order to provide the analytical prerequisites for a detailed understanding of the processes that are ongoing in the body. Moreover, new know-how for the chemical and topographic design of implant surfaces is to be developed.

New bio-laboratories with Imaging Center offer first-class infrastructure

Several new bio-laboratories, classified as Genetic Engineering S1 laboratories, have been set up at the new pilot plant building at the Fraunhofer ISC headquarters in Würzburg. These laboratories enable safe and adequate handling of living cells.

The equipment of the new bio laboratories – especially for imaging procedures – provides the basis for an accurate analysis of the interaction of living cells with their immediate vicinity, which consists of the two- or three-dimensional substrate surfaces on which they reside. As a result, the effect of new materials or agents on living cells can be observed directly.

Various specialized fluorescence microscopes are available for Live Cell Imaging – i.e. the digital recording of the dynamics of living cells. The monitoring of cell migration, i.e. of the motion of living cells on a surface or in a 3D structure, involves the use of a conventional fluorescence microscope fitted with an incubation chamber in order to be able to present ideal conditions to the cells with regard to CO_2 content, temperature, and humidity. The Spinning Disk Confocal Microscope allows very high temporal and spatial resolution in 3D to be obtained. The applications of the microscope include 3D reconstructions, the investigation of transport processes inside cells, and the analysis of the distribution of cells on scaffolds. A STED microscope (Stimulated Emission Depletion) is also available, whose special recording technique affords resolutions beyond the wavelengths of light (super-resolution) and allows for excitation of cell fluorescence by up to eight different wavelengths of light in one single spot. Access to further highly specialized analytical instruments for biological processes is made possible by a cooperation with the biophysics research group of Prof. Heinrich at the University of Leiden.

Opportunities of cooperations

Based on the Imaging Center and the expertise concerning the analysis of biological interactions of living cells with novel materials, ATTRACT 3DNanoCell provides an excellent infrastructure for in-vitro investigations on materials and agents intended for use in the human body. The expertise of the well-experienced ATTRACT 3DNanoCell researchers and a comprehensive set of instruments are available to customers and cooperation partners for a wide range of tasks, from the preparation of test samples to the planning and implementation of tests on human primary cells.

UTILIZING BIO-WASTE AS RAW MATERIAL

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Renewable raw materials are a key for industry aiming to reduce the dependence on petroleum-based materials. Biogenic waste is produced in large quantities and can be recycled for reuse as high-grade materials. Researchers of the Project Group Materials Recycling and Resource Strategies IWKS of the Fraunhofer ISC seek to develop new processes for this purpose. How can renewable raw materials be utilized as a replacement for fossil raw materials without competing for valuable agricultural areas for food production? This is the ultimate question that is being addressed by researchers of the Fraunhofer project group IWKS in Alzenau. The rationale: Use biomaterials which are present anyway, but put to no use – i.e. waste – as raw material.

The business unit Biological Materials of the project group IWKS focuses on the further processing of plant-based waste materials. These originate, as waste products, from the food or pharmaceutical industry and are the basis of new products and applications. They can e.g. serve as starting materials for barrier layers of the type used as sheathing for protection against uncontrolled leaching of nutrients from fertilizer granules. Likewise, the packaging of food items can be optimized through the use of waste substance-based barrier layers. Oxygen and water vapor permeability can be minimized by the organic material. This contributes to a longer shelf-life. In another application, the plant-derived raw materials are used to produce natural fiber-reinforced plastics. Used as adhesion promoters, they can improve the mechanical properties of the plastic material.

This allows excess quantities of plant-derived waste substances to be put to good use and contributes to the preservation of vital resources. Novel processes can be used to turn waste products into added value. Implemented on an industrial scale, this represents an enormous competitive advantage since it does not only reduce the costs of raw materials but also is a major step in the direction of a sustainable production.

Utilization of plant residues rich in fiber: Hemicelluloses are catching up

The use of biomass as a material without competing with the production of food is considered to be an important element of a bio-based economy on all levels of policy. The increased, and further increasing, demand of industry for raw materials from agricultural production and forestry is reflected on a higher level as international chemical companies are purchasing or setting up cooperations with biotechnology companies, which then contribute the processes needed for primary refining and conversion of the raw materials. Renowned chemical companies are investing in key technologies, such as enzyme production. Since the demand for plant-derived biomass is expected to be extensive, the most attractive raw materials sources are those that do not jeopardize the long-term productivity of the agricultural ecosystems, above all the utilization of plant-derived waste products from agricultural and industrial production, which have thus far been eliminated from the system anyway. In the field focusing on the utilization of biomass from plant-derived waste substances as materials, much progress has been made in recent years in the utilization of lignin, promoted by public funding, which is a side product of the paper and cellulose industry and was previously used mainly to produce energy. The hemicelluloses, a major fraction of wood and agricultural waste substances third only to cellulose and lignin, have not yet been developed for the materials market. One pioneer of the use of these substances as materials is a company that was established in Sweden in 2004 and whose barrier layers for food packages

are based on xylanes. Xylanes are structural polysaccharides that originate, for example, from agricultural side products such as the shells and hulls of cereals. In Germany, the development of technology for the utilization of fiber-rich residual substances from plant extraction has been advanced successfully by the Fachagentur für Nachwachsende Rohstoffe (FNR).

Hemicelluloses from the food industry

But the utilization of hemicelluloses from fiber-rich residual substances in bio-composite materials is still in its very early stages, although the mass of these fiber-rich extraction residues is enormous. In 2011, phytoextracts generated global sales of approximately 468 billion euros in the sectors of food supplements, functional food, and nutraceuticals. Even the sales of phyto-pharmaceuticals alone reached a substantial level of 936 million euros, whereas the cosmetics and wellness industry generated sales of approximately 1.8 billion euros from phytoxtracts. Enormous amounts of these fiber-rich extraction residues are being »disposed« at a charge, and the utilization for energy production in biogas facilities is not always economical. Some fraction is sold as animal feed supplement. One major goal of the business unit Biological Materials, Food is to develop hemicelluloses from industrial production processes as components of biologically-degradable polymer materials.

»Added value« in biologically degradable materials from hemicelluloses

The utilization of hemicelluloses in packaging materials and geo-films is attractive, since they allow the feature of biological degradability to be introduced into plastic materials and coatings. Already well-established in the market, the bio-based plastic material PLA (polylactic acid) is made interesting for additional applications by biologically degradable barrier layers. Any improvement in the profile of properties of PLA-based plastic materials contributes to the establishment of a materials cycle for PLA packaging following the pattern of the paper recycling processes. In a press release in February 2015, the Fachagentur Nachwachsende Rohstoffe (FNR) announced the start of a cooperative project on PLA recycling, which is evidence of dynamics in the field of bio-based packaging concepts.

Hemicelluloses comprise a wealth of different functional groups: Hydroxyl groups or a specific fraction of carboxyl groups and/or acrylic acid derivatives. These are the base of a wide range of options for chemical coupling to other polymers. On the occasion of the »Internationale Grüne Woche 2015« trade fair, the project group IWKS presented a coating for fertilizer granules based on a phenolic hemicellulose fraction that is obtained from the hulls of cereal grains. The coated fertilizer granules, patent pending, are more compatible with the environment, since uncontrolled leaching of the nutrients is prevented.

Powerful hemicellulose analytics

The utilization of polymeric hemicelluloses requires gentle extraction techniques and powerful analytics. The project group IWKS has two chromatography systems available that are indispensable for the chemical characterization of hemicelluloses. High-performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) is used for high-resolution analysis of the sugar components of hemicelluloses. High pressure liquid chromatography with diode array detector facilitates analyses of bio-phenols, which, as hemicellulose components, are particularly important for certain applications. Some initial work has also been done on establishing spectroscopic methods by means of which bio-phenols on the surface of plant fibers can be quantified in situ by means of the fluorescence intensity.

BIOLOGICALLY DEGRADABLE BARRIER FILMS

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Most fresh products, much like the so-called »convenience food«, gets to the consumer with some kind of packaging. Hygiene, long shelf-life, and uninterrupted availability of these packaged products are major factors for our quality of life. But this convenience contributes significantly to the pollution of our environment since the preservation and packaging of these food items is often implemented by plastic packaging. As a result: Almost 3 million tons of plastic packaging accumulate as waste in Germany each year. Not even half of this amount is reused. The remainder is incinerated or ends up discarded in nature. A common plastic bag takes approximately 400 years to decompose. Plastic bottles take 450 years and nylon fishing nets take even 600 years to decompose. However, more environment-friendly alternatives will become established in the market only if their production is inexpensive and if their function is at least equivalent to the level of conventional plastic packages. This is particularly important in the area of food packaging which has to possess challenging barrier properties to meet the required shelf-life.

Biodegradable plastic packaging –
not stable enough for many applications

Biologically degradable food packaging can make a contribution not only to the reduction of packaging waste but also to the reduced wasting of fossil resources for the production of plastic packaging. Biologically degradable packaging, for example from regenerated cellulose, polylactic acid derivatives, or starch derivatives, has been known for approx. 30 years. However, these biopolymers cannot compete with conventional mass-produced plastic materials, since direct substitution is not possible yet due to a difference in their profile of properties. For this reason, bio-plastics is rarely used in packaging. It protects the goods only insufficiently from odors, oxygen, and water vapor and therefore often fails to provide the requisite shelf-life for food items. In the scope of the ongoing DIBBIOPACK project of the EU, the Fraunhofer ISC aims to develop a functional material that allows bio-plastic materials to be fitted with an oxygen and water vapor barrier which itself is bio-based and biologically degradable by composting. This affords new application options for environment-friendly packaging.

Barrier for oxygen and humidity

The Fraunhofer ISC is in possession of comprehensive expertise in the development of barrier coatings based on ORMOCER®s, ranging from the composition of the materials to the production processes involved in the manufacture of coated films. In conventional food packaging, the desired barrier effect is reached by inorganic barrier layers (e.g. SiOx) that have been deposited on carrier films in a vacuum. In the food and pharmaceutical packaging industry, improved functional coating materials based on the ORMOCER®s developed by the Fraunhofer ISC are used in combination with inorganic barrier layers to generate sterilizable packaging films with excellent barrier properties. The class of the ORMOCER® materials has good barrier properties with respect to gases and vapors and adheres extraordinarily well to a large range of plastic substrates. This makes these materials very well-suited for the coating of plastic-based food packaging. Although ORMOCER®s, as such, are not biologically

degradable, their properties can be influenced quite well by varying the chemical composition.

Biologically degradable ORMOCER®s

The development therefore focused on re-formulating the ORMOCER®s such as to become biodegradable while maintaining the beneficial properties. This was achieved by incorporating reactive bio-degradable precursors and their stable chemcial bonding to the coating components. In line with this concept, layers were developed that showed not only good adhesion and transparency, but also have excellent barrier properties. Cellulose, chitosan, and polycaprolactone triol were selected as bio-degradable starting materials and modified, to some extent, with functional organic or inorganic groups. Only methods that were non-objectionable in terms of their toxicological properties were used for functionalization. The functionalized natural substances were incorporated into the lacquer precursor of an ORMOCER®, applied to films, and cured to form a solid layer. The fraction of bio-based functionalized precursors was then successively increased and the barrier effect was tested in order to attain the desired parameter profiles, such as degradation rate, transparency of the layer, and protection effect.

Solid body magnetic resonance spectroscopic studies showed that the linking of the natural substances to the organic or inorganic network of the new bioORMOCER®s differs from system to system. This can be used to adjust the composition optimally. The layers developed by this technique show excellent barrier values, which are on a par with the current state-of-the-art of ORMOCER® coatings that are not biologically degradable.

The biological degradability was tested ahead of time using a rapid composting test with a commercial composter. Some of these layer systems showed signs of biological degradation in the test compost within just a few weeks. In addition, the biological degradability was also determined in accordance with DIN ISO 14885-1:2005 and/or ASTM D 5338:1998 revealing degradation of the coated films of up to 98 percent. The time profile of degradation can be adapted to suit the existing requirements by appropriate selection of the bio-polymeric starting compounds. The analyses of the cytotoxicity of the residues produced no negative findings.

Sustainable use of renewable raw materials rather than »Food dish versus technology«

Any competition between the utilization of renewable raw materials and the production of food, which is critically viewed in the area of bio-based fuels, was to be avoided for the bioORMOCER®s. For this reason, the search is on for inexpensive alternative raw materials in the utilization of high-quality bio-polymeric waste substances which are produced, for example, in paper production (hemicellulose) or as waste products in the production of plant-derived pharmaceutical products. The Barrier Layers department of the Fraunhofer ISC cooperates with the Biomaterials department of the Fraunhofer Project Group Materials Recycling and Resource Strategies IWKS in this field.

FLASHED! TOUCHSCREENS FOR FLEXIBLE DISPLAYS

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In the framework of the Project »Flashed« funded by the EU, Fraunhofer ISC has developed a novel sensitive material which will represent a major step towards touch screens for flexible displays.

Large surface sensors printed onto flexible films make the substrate sensitive against deformation. The current generated by the deformation of the sensor material serves as measurement signal which can be interpreted by read-out software. By that, the deformation of the film can be measured by the film itself instead of using any reference system. Prerequisite is a highly local and timewise resolution and a versatile design of individual sensors. When these sensor films are used in combination with flexible displays, the input methods are not limited to conventional ones as known from current touch screens for rigid devices but also through the deformation and motion of the film.

This signals a paradigm shift in how we use e-books, tablets, and other digital media. The partners – Joanneum Research, Media Interaction Lab of Hochschule FH Oberösterreich, FlexEnable, and Microsoft Research – of the EU project »FLASHED«, which commenced in October 2013, presented the FLEX SENSE display to the general public for the first time in October 2014 to demonstrate the technology.

FLEX SENSE is a very flexible film with a touch-sensitive surface that measures any deformation by itself. This is done by quasi-transparent piezo-electrical sensors that are printed on the film and record the deformation. This deformation can be digitalized directly and visualised spatially. In a first step, this film is combined with simple state-of-the-art tablets to demonstrate the novel input methods. When the FLEX SENSE film is combined with flexible displays that are just developed, this will enable future innovative input options to handle an electronic device much more intuitively than by buttons, switches or swiping.

Piezo-electrical printing pastes and ferro-electrical films

The team of Gerhard Domann, who heads the Optics and Electronics department at the Fraunhofer ISC, developed novel piezo-electrical printing pastes for this application that make a flexible polymer film sensitive. The printing process allows for high through-put manufacturing methods and simple assembling methods. The pressure and bending sensors needed for FLEX SENSE can be printed directly onto flexible display substrates. This results in the fact that the sensors can be manufactured with low amount of materials at lower prices.

Moreover, the printed pressure sensors can also be used as actuators and thus facilitate, e. g., haptic feedback. Combining the sensor properties with haptic feedback can improve the user-friendliness and the range of functions in many applications.

Ferro-electrical polymers are used as the basic material for the printing pastes. Due to their special molecular structure, they become oriented spontaneously. As a result, the previously needed mechanical orientation processes are dispensable and the sensors can be printed onto the substrate. These ferro-electrical printing pastes were developed by the Fraunhofer ISC in the predecessor project »3Plast-Sensors« and were presently used as the basis of the new development. Special benefit as compared to other ferro-electrical polymer pastes: The formulation from the Fraunhofer ISC does not include any toxic solvents and can be scaled-up to a large production scale without any difficulty.

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Controllable temperature behaviour

The sensors, which can be produced inexpensively from the ferro-electrical polymers, can record not only changes of the mechanical pressure – such as when the flexible display is being bent and moved – but temperature changes as well. Accordingly, they can also be used as proximity sensors. Even a small change of temperature, e. g. when a hand comes close to the sensor, triggers a corresponding signal. But the developers must also be able to suppress this effect when it is not needed. The temperature sensitivity of the FLEX SENSE display has already been reduced significantly in order to attain higher spatial resolution.

For the widespread use of sensors of this type, it is prerequisitive that their temperature behaviour can be controlled. For this purpose, piezo-electrical particles are added to the ferro-electrical printing pastes. Crucial factors for the performance of the sensors are that an extremely narrow size distribution window of the particles is maintained and that the particles can be embedded into the polymer matrix uniformly and free of agglomeration. Process know-how has been accumulated for this purpose and robust processes for particle preparation as well as a reliable mixing process for polymer matrix and particle dispersion have been developed..

A lead-free future

To date, the sensory properties are controlled by the addition of piezo-electrical particles made of lead-titanate. In the scope of the EU directives concerning the avoidance of materials classified as a health hazard, the use of lead in the field of microelectronics must cease in the future. It is therefore an important goal in the development of new materials to replace the lead-containing part of piezo-material by other piezoceramic particles without suffering significant losses in sensitivity. For this purpose, new ferro-electrical particle-matrix systems are being developed and adapted to common screen-printing.

Flexible displays – robust, inexpensive, and versatile

The major advantage of printed flexible touch displays is their freedom in design. They can be adapted to almost any surface. The display surface can be »unrolled« and thus adapted to any need. Polymer-based touch displays weigh less and are more robust than the conventional hard, glass-based touch-sensitive displays of tablets and the like. Inexpensive starting materials, avoidance of the use of critical raw materials such as lead, and the easy processing were the major requirements on which the partners of the »FLASHED« project focused in this new development. Accordingly, using the new printing pastes from the Fraunhofer ISC, a sensor can be built-up on a flexible film in just three printing steps. The results of the »FLASHED« project should contribute to the future implementation of large-sized touchscreens that are lightweight, robust, and flexible.

www.flashed-project.eu

Media Interaction Lab, Hagenberg (AT) (coordinator of the »FLASHED« project of the EU)

The outcome of the »FLASHED« project will be to give new impetus to the European display and printed sensor sectors throughout the value chain. It helps to position the emerging European flexible electronics industry in the equivalent place to that currently occupied by the Asian glass-based display industry and it will create new opportunities for European companies, not in linear improvements to existing solutions but in the creation of new and innovative products.
(Press release 14 Feb. 2014)

LEAD-FREE SPECIALTY GLASS FOR PRINTING

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Container glass for applications in pharmacy, cosmetics, consumer or laboratory often bears imprints either for decoration or for functional reasons. But the printed graphics and writings are real heavyweights: They consist of specialty glass containing a lead oxide fraction of up to 50 percent by weight.

In the production of permanent lasting printing inks, the manufacturers grind these specialty glass, mix them with inorganic color pigments and organic binders to produce printable pastes. These pastes are then applied to the glass substrate and are affixed by melting them. This makes the inks just as durable as the glass itself. Perfume flacons, beer mugs, baby bottles, beverage bottles, and ampules for pharmaceuticals are decorated and labeled by this technique.

A new EU directive aims to ban the harmful lead oxide from printing inks in the future. Easier said than done: For the printing to be durable, it must be produced from chemically resistant glass. But this type of glass usually contains high levels of silicon dioxide, which has a melting temperature above 1,600 °C – such temperature is not tolerated by the glass substrate without deformation.

The current common lead oxide additive reduces the melting temperature to less than 600 °C and thus generates more workable processing conditions without reducing the chemical resistance. As a makeshift solution, manufacturers are replacing the lead oxide by bismuth oxide. This element has its own issues: Bismuth is considered to be hazardous to health and environment. And it drives up the price of imprinting significantly. Currently, there are no heavy metal free inks on the market that would provide solutions for both low processing temperatures between 530 °C and 580 °C and high chemical resistance for use in laboratory or household.

Durable decor free of harmful substances

A new development of the Fraunhofer ISC on behalf of the Forschungsgemeinschaft Technik und Glas e. V. FTG can be a solution for these problems. The glass experts of the Fraunhofer ISC developed lead oxide-free specialty glass for the production of decor inks that contain no toxic substances at all. The new decor inks are easy to process, show high color brilliance, and are resistant both against water, acids and bases. Moreover, they contain no rare or expensive elements, which makes their use sustainable in the long term.

ZABS – Specialty glass with low melting points

Based on the strength of the existing expertise in the development of specialty glass and the know-how concerning the use of additives to influence the properties of glass, the glass group at the Fraunhofer ISC successfully developed a solution in a short period of time. The new decor inks are based on a glass that consists mainly of zinc oxide. The other ingredients are aluminum oxide, boron oxide, and silicon dioxide. The researchers named this basic material ZABS – an acronym derived from its main components. Due to its large zinc oxide fraction, this glass melts at a temperature as low as 560 °C. This substance therefore assumes the role previously played by lead oxide.

An optimal composition of the mixture for the desired low temperatures was found in tests in a gradient furnace, which allows to find exact correlations between melting temperature and wetting behavior. The obtained glass samples were used to gain a first overview over the properties profiles of the different compositions without having to resort to expensive analytical

methods. The more detailed selection of the compositions of the glass was then based on calorimetric measurements and interface analyses – the latter are important for the bonding on the base glass. The wetting behavior, an important parameter for the durability of the printing inks, was analyzed in situ in the thermo-optical measuring facilities (TOM) developed by the Fraunhofer ISC.

ZABS can be modified and adapted very well to the respective processing temperatures of the base glass by the addition of alkalis and alkaline earths. It was thus possible, e.g., to define a mixture with a melting temperature as low as 530 °C. The low temperatures simplify the processing for printing on the glass and are a prerequisite for reliable quality of the final product. At 530 °C, even very thin-walled glass like ampules can be decorated without deformation.

Adaptation to different glass types

The glass-processing industry utilizes a wide range of different glass types for the various products and applications. Depending on the application the have to meet very different requirements. Which means that the physical properties of the base glass are different as well. A particularly important parameter for printing is the characteristic thermal expansion coefficient: When glass is heated up, it expands to a different degree, depending on the glass type. An overprint should show a thermal expansion behavior pretty close to the thermal expansion of the base glass. If the mismatch is too large, it would flake off during the production processes or, even worse, at the customers site.

Prospects

It was possible to develop new glass inks for decoration of soda-lime glass and borosilicate glass containing no hazardous elements. Soda-lime glass is used for many consumer glass applications, such as drinking glasses and bottles. Borosilicate glass is used for pharmaceutical ampules, laboratory glassware or household items, such as oven dishes or tea and coffee pots. These two base glass inks are available for use in future developments. Follow-up projects are under the way in order to adapt the specialty glass to the specific requirements profiles of the industry for different glass applications, e.g. with a view to the acid and base resistance of the overprinting or with regard to different production conditions.

Ralph Hörner,
Forschungsgemeinschaft Technik und Glas e.V., Wertheim-Bronnbach

»This fundamental development of lead-free printing inks for decoration of glass made by the Fraunhofer ISC generates a solid base for further product developments.«

GESTURE CONTROL OF VEHICLE SEATS

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A professional driver sits in the driver's seat for an average of nine hours a day. This makes the seat a central component of the workplace. The comfort and safety of the seat are therefore of crucial significance to the driver. Modern seats of commercial vehicles allow the driver to travel long distances fully concentrated and free of fatigue, and make sure that harmful vibrations are reduced by a pneumatic vibration systems. An ergonomically correct design and shape as well as proper adjustment of the seat position are crucial for optimal comfort. The Center Smart Materials CeSMa of the Fraunhofer ISC and its project partner ISRINGHAUSEN developed a contact-free gesture control for driver seats in trucks that can be used to adjust the optimal sitting position just by hand motion.

Intuitive contact-free communication

Studies have shown that professional drivers adjust their individual seat position correctly only, if the driver is aware of and familiar with all adjustment functions and can use them intuitively. This means that there is a need for an operating concept that is easy and safe to learn. The new gesture control provides the driver with numerous adjustment and comfort functions that can be used to adjust not only the seat position, but also parts of the contour configuration to the individual needs and bodily prerequisites. Heating and ventilation produce a pleasant and physiologically beneficial micro-climate.

The functional principle underlying the newly developed operating concept is familiar to the user from everyday life, namely contact-free communication with technical facilities by means of intuitive gestures. The seat simply follows the natural motion of the hand – no special gestures need to be trained. Moving the hand upwards moves the seat up, while moving the hand forward moves the seat forward. Other motions directed upwards or downwards at an angle are used to adjust the backrest inclination and/or the thigh support. This implements a solution for the communication between man and machine that is easy to learn and safe to operate. In addition, operating errors can be excluded by electronic means.

Switches and buttons becoming obsolete

Another benefit: There are no elements, like switches and buttons, which might be damaged or get soiled during routine use. This simplifies the seat design and lowers production costs. In addition, if drivers change frequently, individual seat positions can be memorized by simple motions and can be recalled as needed at any time. The large control panel lacking buttons is situated in a well-accessible place to the side of the driver's seat – made invisible by a cover. It is insensitive to soiling, robust, and can be used with gloves. And there are novel degrees of freedom for seat and vehicle designers as well: Since all components are arranged behind the side cover of the seat, the external shaping and design of the lateral cladding for the operating side can be selected freely.

Approach of the hand triggers switching function

For contact-free control of the vehicle seat just by gestures, the side cladding has been provided with electrodes for a capacitive proximity sensor system based on the principle of charge transfer. The capacity between a measuring electrode and the frame potential of the surroundings is determined for this purpose. The measuring results are then averaged and used as

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basic values of the sensor field in the non-contact state. If a hand or finger approaches the sensor field under the cladding, the capacity changes and the switching function is triggered. The analytical hardware developed by the CeSMa scientists processes and transmits the information collected by the sensor system. The major challenge for the scientists in this project was to align the electrical field for the electrode of the capacitive proximity sensor system in a certain direction. In the absence of specific shielding, the proximity sensor system would be equally sensitive in all directions of space. The use of a special shield electrode allowed all requirements to be met.

Proper adjustment by software

The CeSMa scientists developed a dedicated software that allows the gestures to be properly recognized and implemented for adjustment of the driver's seat. The information from the proximity function is processed by a microcontroller. Firstly, the software control is activated by a tapping motion: Rather than using a start button, the operator only taps the sensor field once with a finger. Then, a period of time – which can be preselected – is available for operation of the seat position control. If no operation follows within the time window, the electronics is switched off again. Having this type of activation prevents the gesture control from being triggered inadvertently by a hand motion. Tapping twice activates a memory function: This automatically recalls a previously stored seat position preset. Tapping thrice stores the current seat position for recall at a later time. Multiple seat position presets can be stored for different drivers in a simple and intuitive routine. The tapping function is implemented by piezo-electrical sensors. The sensors have been developed and produced specifically for this function by CeSMa. Once the user has entered all settings, the previously determined function is terminated as soon as the hand moves out of the sensor field. LEDs provide feedback to the driver to indicate whether or not the gestures have been recognized properly.

As a result, the contact-free gesture control allows the user to make the adjustment to the desired seat position quickly and easily. No need to search for the right button, and the correct adjustment can be made much more precisely.

Obviously, the use of this operating concept is not limited to truck seats. It is well-suited also for seats in passenger cars, trains or airplanes. Likewise, man-machine interfaces in domestic technology are conceivable, like in the control of window blinds or household devices or in the control of robots and machines by motion patterns in production engineering.

Jens Rönnefahrt, Director of Advance Development ISRINGHAUSEN GmbH & Co. KG, Lemgo

»The cooperation with the Fraunhofer ISC in the development of the gesture-controlled commercial vehicle seat has proven to be very expedient for ISRINGHAUSEN GmbH & Co. KG. The professional competence and the wealth of experience of the Fraunhofer ISC concerning the integration and analysis of sensor systems were important prerequisites that allowed the desired project result to be attained timely and at full functionality.«

PEDELEC – OPTIMIZATION OF BATTERIES

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»Electro-mobile« bicycles are gaining in significance and popularity, especially with people living in or near cities. While only approximately 24,000 electrical cars are registered in Germany, figures of the ADFC bicycle club show that already some 1.6 million pedelecs (pedal electric cycle) roamed the streets of Germany in 2014. The market for low-effort electrical bicycles is booming. But neither long-term experience nor reliable data are available regarding the usage patterns of electric bicycles and batteries at realistic conditions of use. This is a shortcoming that bothers not only the users, but also the bicycle manufacturers and suppliers of battery packs. These unknown factors are the focus of an investigation of the Fraunhofer ISC in cooperation with partners from industry and science in the scope of the project titled »PEDElEc – Pendler-eBike Dauertest mit elektrischen und elektrochemischen Untersuchungen« (Commuter e-bike long-term test including electrical and electrochemical tests). Twelve staff members of the Fraunhofer ISC have been cycling to work each day since October 2013 for the purposes of this project.

Funded by the Bavarian State Ministry for Economy, Infrastructure, Traffic and Technology, this project is the first known field test of pedelec batteries that is implemented in a cooperation with partners from research and industry. This research approach focuses on the pedelec as a commuter vehicle and on the optimization of the battery for cyclists meeting this profile. A total of 19 test commuters from the Fraunhofer ISC and the municipal administration of Bad Neustadt an der Saale use pedelecs from Winora-Staiger GmbH daily to get to their workplace. The Winora pedelecs used in the field test are standard bicycles fitted with special data loggers. This additional equipment continuously records information about the distances traveled, the associated loads, and the battery status. The scientists of the Fraunhofer ISC aim to understand the aging behavior of battery packs and of individual battery cells as a function of their usage profile. This concerns not only the detection and analysis of the usage profiles, but also the electrochemical processes that might reflect not only the operability of the battery, but also possible sources of wear and/or failure. These processes, in turn, depend on the load on the batteries in routine use, e.g. on the charging and discharging cycles at high and low temperatures.

Disseminating insights to dealers and users

The investigation of the (i) commuter behavior and of the (ii) general usage of the bicycle, and the detailed analysis of the storage battery is highly relevant for the project partner, Winora-Staiger GmbH. The results of the scientific tests are taken into account directly in practical application. The results of the project thus far confirm or even exceed the previous assumptions and expectations of the bicycle manufacturer. The storage batteries are more durable than expected. The commuter data from the tests show the residual capacity to be above 70 percent after 500 charging cycles. On the other hand, it was found that the storage battery should also be checked after any crash or accident, because there might be anomalies such as reduced power or failure of the battery management system.

Battery cells being aged and tested

The motion data of the pedelecs are detected with a specially developed mobile phone app, and analyzed subsequently. The scientists use the travel data of the commuters to determine a travel profile. This, in turn, is used to develop a simulation program to allow the battery testers to run accelerated tests on the storage batteries in the laboratory parallel to the commuter rides. The specific aging is the responsibility of the project partners, BMZ (Batterien-Montage-Zentrum) and Technology Transfer Center (Technologietransferzentrum, TTZ EMO) of Hochschule für angewandte Wissenschaften Würzburg-Schweinfurt. The Fraunhofer ISC subsequently investigates the aging of the cell level based on statistical test planning.

A total of 100 cycles, and therefore three reference parameter tests per tested cell, have already been recorded since the start of the project. It has been evident that some cells lose capacity and power more rapidly than others as a function of the aging process. The use of high final charging voltages and temperatures beyond room temperature have proven to be particularly detrimental in this context. Knowing that certain weather conditions, such as rain or extreme temperatures, have a certain effect on the storage battery, is very important for Winora-Staiger.

Parallel to the aging of the storage batteries in the laboratory, which provided important information to the scientists for optimization of batteries, the batteries used in the daily commuter routine are also regularly subjected to a so-called reference parameter test. The test is used to determine the residual capacity in order to check on the aging of the battery packs. In addition to the tests on the storage batteries, the pedelecs used in the field test are regularly serviced locally by the project partner, Winora-Staiger. These local appointments are an opportunity for the commuters to ask questions and to make suggestions about the bicycles and their technical equipment. The data loggers and the app are checked regularly by the project partner, TTZ, to ensure that the data are transmitted properly.

Post-mortem analysis

The battery testing program at the Fraunhofer ISC includes x-ray diffraction analyses for elucidation of the crystal structure. The insights gained from the tests allow the aging behavior to be understood better. Based on the comprehensive tests, we meanwhile have a clear picture of the cell chemistry of new cells. As soon as the first cells show more advanced aging (EOL criteria), the study of the degradation effects on component level can be started based on the methodology and analytical baseline developed earlier.

Funded by the Bavarian Ministry of Economics in the scope of the Model Region »Electro-Mobility Bad Neustadt«, the research project is being undertaken by the Fraunhofer ISC, the bicycle manufacturer Winora-Staiger, the storage battery manufacturer BMZ and the Technology Transfer Center (TTZ EMO) of Hochschule für angewandte Wissenschaften Würzburg-Schweinfurt.

Thomas Drehmel, Product & Project Manager eBike, Winora-Staiger GmbH, Sennfeld

»The Fraunhofer ISC, engaging in pioneering research, has been a highly competent partner for Winora-Staiger GmbH. We have been working on this cooperative project since January 2013. We appreciate especially the high scientific standards and the excellent technical equipment. We are benefitting from the long-standing experience of the Fraunhofer ISC in the field of battery research.«

CLIMATE FOR CULTURE

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The ongoing climate change is one of the major challenges facing mankind today. But this goes beyond just an increase in average temperature and also concerns the overall change of the climate. In the scope of the EU project, »Climate for Culture«, 27 partners from the EU specifically investigated the consequences for our cultural heritage and developed forecasting models and tools that can be used to assess the consequences better and to set-up protective measures. The Fraunhofer ISC contributed its know-how and special glass sensors to the monitoring of the real climate consequences, and the International Convention Center for Cultural Heritage Preservation IZKK participated in the acquisition of socio-economic data and in the organization of events and workshops.

Climate models

For assessment of the climatic consequences, high-resolution regional climate models have been combined with hygrothermal building simulations for the first time in the scope of »Climate for Culture«, i.e. the profile of both temperature and humidity in buildings is calculated. This allows us to make forecasts concerning the development of the climate and energy needs for air-conditioning of the buildings in the future and to initiate suitable preventive measures for preservation of collections. Although a moderate emission scenario was selected, the results of the climate simulations demonstrate that average temperature increases of up to 5 °C are to be expected in northern Europe. Unfortunately, we already know that it will not be feasible to reduce the CO_2 concentration in the atmosphere. This concentration will keep increasing, along with the temperatures. The south and southeast of Europe will get much warmer by the end of the century. Concerning the future development of precipitations, it is evident that the drought phases in the summers will keep getting longer in the south of Europe by the end of the 21st century. In contrast, the total amount of precipitation is expected to increase strongly in northern and central Europe, mainly in the winters, promoting the growth of molds.

Impact on our cultural heritage

The changes calculated in the climate simulations are associated with the risk of building materials and sensitive objects being attacked by corrosion processes much more strongly than currently. Along with the forecasted global increase of ambient temperatures and the ensuing changes of many other climate parameters, the chemical reactions responsible for these degradation processes will proceed more rapidly than currently. This is the case, because there is an exponential relationship between reaction rate and temperature (Arrhenius equation). The following rule of thumb applies: If the temperature increases by 10 °C, the destructive power of this type of reaction doubles.

Monitoring the potential damage

The climatic conditions inside the buildings included in the case studies were tested with various methods – some of them novel – and the long-term data from the existing climate data loggers were analyzed. A particular focus was on the interplay of the various parameters, such as temperature, humidity, and pollutants with a view to the potential damage to sensi-

tive objects in exhibition rooms. Glass sensors developed by the Fraunhofer ISC were used for this purpose. These sensors consist of highly corrosion-sensitive specialty glasses that were attached to the objects in the exhibition rooms. The sensitive glasses indicate the accumulated effect of climatic conditions over the time of exposure. This can be quantified afterwards in the laboratory. In the scope of the »Climate for Culture« project, it became evident that the sensors can be used to derive very precise information about the potential damage caused by the micro-climate at the site of the exhibition objects. In addition, fluctuations of temperature and relative humidity were simulated under laboratory conditions. They resulted in increased corrosion rates on the glass sensors. It was therefore possible to clearly demonstrate that the stability of the climate has a central role for the preservation of objects of art and cultural heritage. The analysis of the more than 100 case studies confirmed that major fluctuations of temperature and humidity occur in, and cause damage to, many historic buildings. The glass sensors are therefore very well-suited for monitoring the climatic conditions, especially the stability of the climate: They are easy to use with no need for additional infrastructure (no electricity, autonomous) and allow suitable protective measures to be initiated in a timely manner.

Costs of the preservation of cultural heritage

Another aspect of the research project focused on the evaluation of the socioeconomic consequences of climate change for the preservation of cultural assets. Specifically, the acceptance level of the costs of additional preservation measures caused by the climate change was to be investigated. A questionnaire based on a cost-benefit analysis was developed in the scope of the project and was then used in visitor surveys at selected cultural heritage sites throughout Europe. In Germany, the selected sites included the Bavarian royal castles, Linderhof and Neuschwanstein, and the Bronnbach monastery. The visitor survey of the IZKK at Bronnbach revealed – much like the other surveys – the high acceptance level amongst the visitors who were willing to pay an extra fee for preservation of the sites in addition to the entrance fee. This result is highly relevant especially for policy makers.

Further results and tools

Climate simulation and building simulation models have been successfully linked to each other for the first time in the scope of the »Climate for Culture« project. As a result, data on the impact of temperature, humidity, exposure to sunlight and water vapor on the building substance are available for the first time in a networked manner for all of Europe. Expansion of the regional climate model and building simulation to Europe and the adjacent Mediterranean area allowed forecasts of the potential damage of the climate change to our cultural assets to be made. This information is being applied already in the preparation of protection concepts. For example the Swedish Church is currently cooperating with regional planners to design a sustainable climate concept based on »Climate for Culture«.

These results and new insights have been incorporated into an Internet-based software platform. As a result, a practice-oriented tool is available that provides robust and validated information concerning future climate changes and their impact on historic buildings. This information is made available in the form of climate maps, energy need maps, and risk maps as well as in a software-based expert system that suggests concrete measures for energy-efficient sustainable air-conditioning to users and/or owners of historic buildings.

http://www.climateforculture.eu/

SEPARATION OF MATERIALS IN MELTED GLASS

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On a global level, approximately 60 billion tons of resources* are consumed each year - and this tendency is on the rise. Resource efficiency is therefore one of the most important keys for sustainable development. Resource efficiency, a component of the sustainability concept, is a main element of national and international strategies, for example on the level of the UN, European Union, and the German federal government. On a technical level, resource efficiency is implemented mainly through materials substitution and recycling-based approaches in research, development, and practice.

Powerful separation processes

To this aim, the Fraunhofer Gesellschaft sponsors the »Molecular Sorting« research project as a methods-oriented development in the scope of the »Markets of Tomorrow and Beyond« program that aims, in the mean- to long-term, to facilitate the reuse and recycling of materials by novel powerful separation processes down to a molecular level following the production or utilization of products. Seven Fraunhofer Institutes joined forces in the scope of the »Next Generation Recycling« project to test new methods on selected material flows, the so-called »demonstrators«, or to ensure the transferability of the results to other materials and industries.

The focus of the Fraunhofer ISC is on the development of a method for electrochemical separation of materials in molten glass in the scope of the »Molecular Sorting« project. This underlying rationale was to improve the value of flat glass, which is green due to its iron content – for example simple window panes – in an intelligent recycling process and to separate the iron ions from the molten glass to produce a glass that is white and of the type that can otherwise be obtained only with rare highly pure raw materials, which are therefore relatively expensive. This type of »white« glass is needed, for example in solar technology.

Mobile ions in the melt

A cleaning process for iron-containing mass-produced glasses should be easy to incorporate into the conventional process technology of glass manufacturing in order to save the additional costs of separate reprocessing. Since the technically relevant glass production processes usually involve a glass melt, the development of a cleaning process right in the melt was favored at the Fraunhofer ISC.

The various ingredients making up the glass are relatively mobile in the melt. This allows the electrically charged ions to be deposited on electrodes, e.g. by electrochemical means. Electrochemical processes of this type are known for various glasses, but require, in the case of soda-lime glasses, relatively high voltages – several 10 V – between the electrodes in the glass melt for the deposition processes. These high voltages can lead to adverse effects in the end-product, for example discoloration. Therefore, it was the aim of the researchers at the Fraunhofer ISC to develop an efficient process that can be run at the lowest possible voltages.

* Krausmann et al. (2009): Growth in global materials use, GDP and population during the 20th century. Ecological Economics, vol. 68, no. 10, 2696-2705

Voltage and electrode materials

The deposition rate and the selectivity of electrochemical deposition are closely correlated to the applied voltage. But the correlations go in opposite directions. While the deposition rate increases with increasing voltages, the selectivity – for the iron to be removed in the present case – decreases. It was possible to define an optimal voltage between the electrodes in the melt to 1.5 V for the soda-lime glasses tested presently.

Clear corrosion effects were detected at the electrodes under these conditions, i.e. electrode material was released into the melt. The corrosion properties of different materials in the melt were investigated in a series of tests. A combination of graphite cathodes and copper-doped tin oxide proved to be best suited for the deposition of iron from the soda-lime glasses.

The process temperature also plays an important role. It has an influence on the viscosity and the diffusion rate of the iron ions. A process temperature of approximately 1,400 °C proved to be particularly beneficial, not least because it is also reached in the conventional production processes of flat glass.

The investigations done with a view to the purification of the glass melt in the scope of the »Molecular Sorting« project demonstrated the feasibility of electrochemical deposition at low voltages for soda-lime glasses. In addition, it was possible to define important process parameters. A solid foundation has thus been generated for implementation in a process that can be utilized on a large technical scale.

The glass melt acting as a solvent

But even beyond the scope of the »Molecular Sorting« project, the observed corrosion effects on the electrodes are quite promising for a novel approach to the recovery of valuable raw materials: Virtually all electrode materials tested can be dissolved in the glass melt at suitably selected process conditions. Due to the high selectivity of electrochemical deposition, the dissolved substances can be separated in very pure form.

Based on the process know-how of the Fraunhofer ISC in this field, the feasibility and profitability of such deposition processes for the recovery, for example, of rare or critical raw materials or raw materials with ultra-fine distribution in the product can be investigated and assessed for customers and partners in collaboration with the Fraunhofer project group IWKS.

CRITICAL METALS COBALT AND MOLYBDENUM

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Natural resources and the ability to utilize geogenic raw materials are the foundation and the prerequisite of our successful economy and prosperity. Current developments in the energy market – especially the volatile price developments of metallic raw materials – make it ever more obvious that raw materials that are not available in unlimited quantity are critical for our economy.

The ESM Foundation (»Entwicklungsfonds Seltene Metalle« – Rare Metals Development Fund) residing in Zurich (Switzerland) is a non-profit organization that has been funding research and development related to rare metals since 1951. Under this umbrella, politicans, representatives from industry and research are jointly discussing the significance of rare metals in science and technology in view of political and society-related issues and their impact on the national economy. Two metals, cobalt and molybdenum,are particularly important to the ESM and for this reason the foundation awarded a contract concerning a comprehensive study of these two raw materials to the Fraunhofer project group IWKS in Alzenau.

A comprehensive criticality analysis was performed under the supervision of Prof. Dr. Armin Reller. The study based on »Reller's concept of material history« investigates the relevance and function of these metals in their applications as well as their impact on various industries and sectors.

Factors affecting natural raw material sources

It is apparent to an ever increasing degree that the supply with resources is becoming less certain due to the high demand and the ensuing increasing consumption as well as regional and corporate concentrations and complex and non-transparent networks. Limited options for recycling and substitution as well as short static ranges increase the supply risk and thus the vulnerability of the national economies demanding these materials.

Content of the study

- Geological availability
- Geopolitical / ecological parameters
- Recycling and substitution
- Economic relevance
- Forecasted application potentials
- Dissipation
- Degree of immobilization

Cross-industry relationships and dependencies are visualized taking into account the application fields and the function of the investigated elements. A cluster analysis by economic, technical and strategic relevance demonstrated which industries are facing similar challenges concerning the future supply for these metals, even if their interest in these metals is based on different utilization potentials. Subsequently, individual economic sectors and/or industrial sectors were investigated in order to delineate their future development and upcoming issues in a more differentiated way based on the previously derived trends. In addition, a compendium listing a selection of key players and/or other smaller companies was prepared.

Cobalt – a critical metal

The main arguments for evaluation of the criticality of the metal cobalt are, firstly, the continuous price increase in the raw ma-

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terials market throughout the past decades and, secondly, the political situation in the conflict-burdened main mining regions in Africa (mainly the Democratic Republic of Congo) in combination with long delivery times. Cobalt is used mainly in lithium ion batteries, but also in superalloys, hard metals, catalysts, magnets and pigments. Due to its good electrical conductivity and the mobility of Li ions, Li_xCoO_2 is a popular cathode material in lithium ion batteries. Cobalt-based alloys own higher melting points, better hot corrosion resistance and better weldability than Ni-based alloys. Due to the application in next generation technologies like electromobility, communication and energy, the strategic significance of this metal will remain high.

Molybdenum – a critical metal

In 2007 China surpassed the United States as the main producer of molybdenum. Molybdenum should be monitored for the future since the political situation is difficult to assess and might be associated with export restrictions. Due to the expedient combination of high melting point, low heat expansion coefficient and low density, molybdenum is a desirable alloying element for steel applications – in the form of construction steel, stainless steel, tool steel and high-speed steel as well as cast iron. The addition of molybdenum increases the strength, hardening ability, weldability, and high-temperature strenght of steel. Aside from the steel industry, molybdenum (MoS_2) plays an important role in superalloys as well as in catalysts and as a coating agent and lubricant. The economic development of molybdenum is correlated to the demand for steel; its utilization in different industries (automotive industry, machine/ plant engineering, oil and gas industry) and the limited options for its substitution will keep the demand high.

Conclusion

Both critical metals, cobalt and molybdenum, are very difficult to substitute, if at all, without suffering losses in performance. One of the reasons being that they are used as substitutes (e.g. for metals of the platinum group or nickel).

Molybdenum recycling does not occur at an elemental level from its main application field of steel refinement. On the other hand the difficulty for cobalt recycling is related rather to the logistical challenge due to its use in battery technology. Key industries that are strategically important, such as the automotive industry, the energy sector as well as machine and plant engineering and the chemical industry depend on these two metals and their industrial relevance both in Germany and in the EU should be taken into consideration in future strategic decisions.

Accordingly, there is a great interest in preventing their shortages in the future. Cobalt and molybdenum represent two critical metals that should continue to be in focus.

Dr. Margarethe Hofmann-Amtenbrink,
CEO ESM, Pully (CH)

»The Swiss foundation »Entwicklungsfonds Seltene Metalle, ESM« awarded a contract to the Fraunhofer project group IWKS in 2013 concerning a study of the »Utilization potentials of rare and critical metals across the area of interest to the ESM«. The cooperation with the Fraunhofer project group IWKS resulted in two studies in 2014 that afford a comprehensive overview of the materials cycles of the metals, molybdenum and cobalt. The lively exchange and the good cooperation between the ESM and the project management of the IWKS have proven to be very productive and goal-oriented for the purposes of the project.«

NEW PERMANENT MAGNETS FOR CLEAN ENERGY TECHNOLOGIES

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The key topics of the Fraunhofer Project Group Materials Recycling and Resource Strategies IWKS of the Fraunhofer ISC is to help secure strategically important raw materials for Germany. The availability of strategic metals, e.g. the rare earth metals, is becoming a focus of politics and industry. Production costs can be reduced, the security of the supply can be improved, and a crucial contribution to the protection of the environment can be made by new approaches to raw materials strategies, recycling, and possible substitution materials.

Rare earth metals play an important role especially in high-performance magnets. Neodymium-iron-boron magnets have by far the highest energy density of all permanent magnets known to date and account for two thirds of the global market volume. They are used, for example, in loudspeakers, hard disks, and wind energy plants (> 600 kg/MW). The demand in the automotive industry is increasing, specifically because of the rising number of hybrid and electrical vehicles. For this reason, these magnet systems are a main focus of the work of the project group IWKS. One emphasis is on the substitution of metals classified as being critical. Theoretical forecasts and proposals of novel ferro-magnetic phases that contain no neodymium (Nd) and dysprosium (Dy), but have the same magnetic properties, are made on the basis of simulations. The forecasts are used as the basis for the synthesis of new magnetic materials. To this aim, the Fraunhofer IWKS project group can utilize a modern machine park and a wide range of synthetic routes; including an electric arc furnace, the process of rapid solidification, and sintering. Modern production processes for high-performance magnets target the nano-crystalline structure, e.g. by hot pressing and hot deformation.

A second topic is the efficient use of raw materials during the production process. By developing production procedures to be near-net-shape, laborious and therefore material-intensive finishing work becomes dispensable and materials can be saved effectively. The project group IWKS also works on the optimization of the micro-structure in order to distribute critical elements, such as Dy, in the micro-structure more specifically and mainly to those sites, at which the effect on the properties of the magnet is optimal. These measures allow the use of Dy to be reduced by up to 50 percent. The project group has a leading role in the lighthouse project »Critical Rare Earths« of the Fraunhofer-Gesellschaft. The aim of the project is to reduce the demand for heavy rare earths in permanent magnets by 50 percent. This project scheduled to take about four years to complete is funded with approx. 9 million euros that have been awarded to the project consortium made up by seven Fraunhofer institutes – coordinated by the Fraunhofer IWM. Accordingly, one goal of the flagship project is the development of a substitute for Dy-containing Nd-Fe-B magnets.

Which industries will benefit?

High-performance magnets play an outstanding role mainly in the area of generators and electrical motors. Specifically, electro-mobility, the automotive industry as well as wind power plants stand to benefit from substitution materials of better availability. Since forecasts expect major growth for these technologies in the future, the demand for Nd and Dy is expected to increase dramatically. An insecure supply of these raw materials might impede the expansion of these next-generation technologies.

The lighthouse project »Critical Rare Earths« is subdivided into five sub-projects that are directed at different topics and challenges.

Sub-project 1 – Material substitution

Methods from materials science will be used to prepare theoretical forecasts and proposals concerning the synthesis of novel ferro-magnetic phases that contain no Nd or Dy, but have unchanged magnetic properties.

Sub-project 2 – More efficient processes

The processes currently involved in the manufacture of high-performance permanent magnets are characterized by substantial losses of materials. For this reason, process chains are to be established that eliminate, in particular, laborious finishing work and, taken together, render the use of materials more efficient and design the production to be shorter.

Sub-project 3 – Optimized design

The technical specifications of electrical small drives, e.g. performance-related drives in road vehicles for motor cooling and EPS (electronic power steering), are developed in line with strict packaging and weight requirements. With regard to the subsequent optimized design of the small drive, many features and design-related approaches to solutions, e.g. shape and design of the magnets or different slot design, need to be evaluated in order to specifically derive potentials regarding cost effects and magnet materials.

Sub-project 4 – Design for recycling

Since there is no 100 percent substitute for (Nd,Dy)-Fe-B magnets available anywhere in the world at this time, routes of the »design for recycling« should also be investigated by scientific studies.
This includes the following approaches:

- Development for reuse-friendly and recycling-friendly electrical motors,
- maximization of the refurbishing rate of electrical motors, for example from scrap cars in global markets, reuse of used electrical motors for the same or a different functional purpose,
- reuse of used permanent magnets in electrical motors,
- recovery of rare earth metals from disassembled and refurbished permanent magnets aiming for secondary raw materials production (recycling).

Sub-project 5 – Markets and environment

From an economic point of view, a single production country enjoying a dominant position in the availability of rare earths bears the risk of uncontrolled shortages or even disruption of the supply. Moreover, the mining of critical metals Nd and Dy is associated with major ecological problems: Radioactive waste is produced and the two metals can be dissolved from the rock only with difficulty.

The simulation of dynamic substance cycles is used to analyze the fate of the rare earths – following their progression from mine to product to scrapping. The insights thus obtained will be used to make the German economy less dependent on external raw materials supplies and to utilize the existing substitution and recycling options to reduce the Nd and Dy demand. As a result, the burden on our environment would be reduced as well.

Benefit for corporations

An assessment of the potential benefits for German companies will be completed by October 2017. Possible approaches of substitution might reduce the production costs, improve the security of supply, and the reduced mining would make a crucial contribution to preserving the environment.

CERAMIC PREPREG PROCESSING

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Fiber-reinforced composite materials based on plastics are highly rigid and stable light construction materials often used in the automotive industry, aircraft construction, and wind power plants, to name just a few. Ceramic matrix composites (CMC) are not being used as widely yet, although they are interesting alternatives to metallic materials for many technical products and have already proven their worth in some high temperature applications. One of the reasons is related to the high costs of the laborious individual »manual« production process, while ensuring the materials properties and product quality. Key factor for inexpensive production of reliably reproducible qualities is a continuous prepreg processing such as has been state-of-the-art for a long time in the field of reinforced thermoplastics – reinforced with glass fibers (GFRP) or carbon fibers (CFRP). To this aim, the Fraunhofer Center for High Temperature Materials and Design HTL in Bayreuth works on using continuous prepregs for the manufacturing of fiber-reinforced ceramics – a technology that has not yet become established on an industrial scale in the production of ceramics.

The process

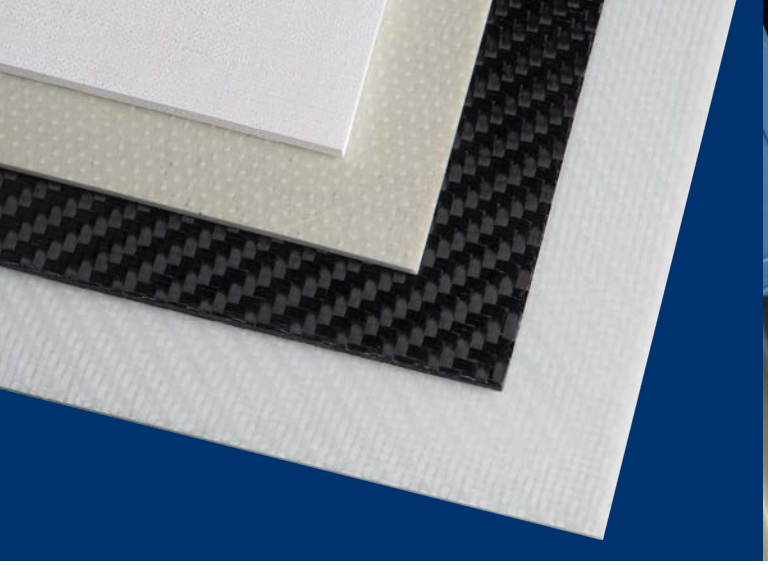
Prepregs are semi-finished products, already coated with a matrix or infiltrated and pre-crosslinked, for further processing to form fiber-reinforced composite materials. The reinforcing fibers – either ribbons or fabric material, e.g. textile fabrics made of technical fibers – are coated roll-to-roll with a polymer or ceramic slurry in the prepreg facility. The slurry consists of ceramic powders and additives as well as a thermosetting binding agent and/or of ceramic precursors. The coated fabric is protected from sticking by a separately supplied separating film and is then pre-dried and/or pre-crosslinked in a convection furnace or by IR emitters. The polymer is present in pre-crosslinked form, but it is still capable of bonding. Prepreg and separating film are subsequently reeled into a roll. These pre-stages, as blank cuttings, are easy to process and can be converted into ceramics after shaping by subsequent high-temperature steps and sintering processes.

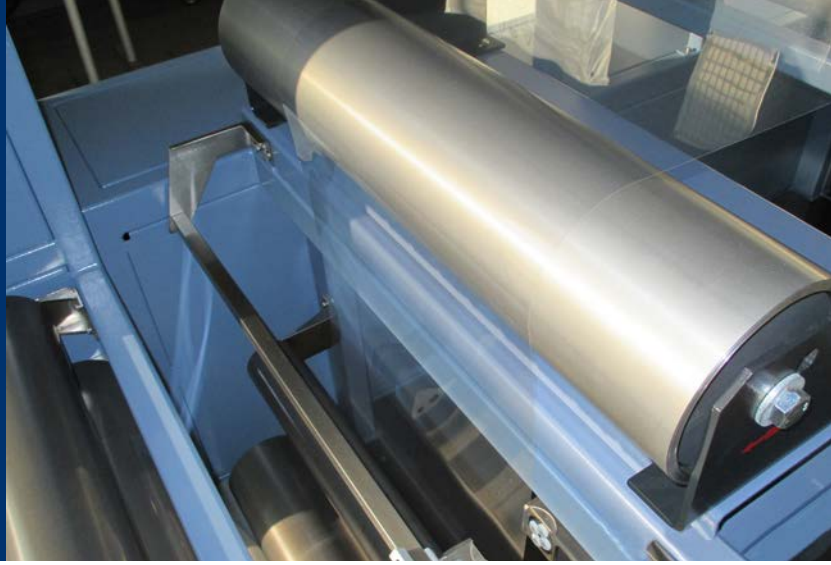
The facility

The first tests were done using glass fibers and carbon fibers as well as liquid and powdered binding agents for the production of GFRP and CFRP. On principle, various materials systems can be used on the prepreg facility of the Fraunhofer-Center HTL in Bayreuth:

- Glass fibers
- Carbon fibers
- Basalt fibers
- Oxidic ceramic fibers
- Non-oxidic ceramic fibers
- Ceramic slurries containing thermosetting binding agents or based on ceramic precursors
- Thermoplastic binding agents (for non-ceramic CFRP or GFRP prepregs)

The reinforcing fibers can be processed by the facility as unidirectional ribbons or fabrics up to 600 mm in width in a continuous roll-to-roll process.
Various coating modules are available for use with different materials systems:





- Foulard module: The reinforcing fabric moves through an impregnating bath. Subsequently, excess material is removed by rollers such that the fiber bundles and fabric are impregnated with a defined amount of slurry or binding agent.
- Doctor blade module: Predefined amounts of pastes or liquids are applied to one side.
- Free-flow module for free-flowing coating materials: This also generates a one-sided coating of predefined thickness.

In addition, batch processes are feasible as well, for example for tests of various systems and parameters while using only small amounts of material.

Accordingly, this facility provides versatile options for the production of ceramic – or non-ceramic – prepregs. It can be adapted to different customer specifications rapidly and flexibly. For example, materials systems of customers can be tested with the facility.

Online quality control and testing of the materials properties

A continuous determination of mass is integrated into the prepreg process. An ultrasound sensor moves transverse to the fabrics across the coated material and measures the mass distribution. This integrated online monitoring ensures uniform application of the polymeric resins and/or ceramic slurries – as the foundation of the reproducible quality of the composite components. The uniform pre-crosslinking of the polymers after the drying process can be monitored by means of infrared spectroscopy. Prepregs from the test runs done thus far have been processed into components by means of hot pressing and their materials properties have been tested. Accordingly, sheets made of GFRP and CFRP have been produced by stacking, pressing, and curing at approx. 200 °C, and the interlaminar shear strength has been tested by standard assay. The microstructure of sections was analyzed by light microscopy and the homogeneity of the sheets was analyzed by an air-coupled ultrasound procedure. These analytical imaging procedures allow the homogeneity of the microstructures of the fiber-reinforced composite materials to be determined, from which insights for specific adaptation and optimization of the process parameters can be obtained.

Prospects and supply

One advantage of the continuous procedure is the controlled and reproducibly uniform distribution of the specific fractions of fibers, matrix, and additives in the final prepreg. The infrastructure required for translation from plastics production to the production of ceramic fiber-reinforced composite materials has been established at the Fraunhofer-Center HTL. Initial tests on the easy-to-handle GFRP and CFRP materials systems were successful and demonstrated the feasibility. The transfer to ceramic materials systems is scheduled for the ongoing year 2015. Complex ceramic materials systems are included in the procedure according to actual need. This is aimed at the provision of small amounts of prepregs for prototype construction or small-batch production. Moreover, special substance systems can be tested for the procedure as requested by customers. Not only the process infrastructure, but also comprehensive processing expertise is available at the Fraunhofer-Center HTL. The center also accepts orders for further processing into components as well as standards-based testing and characterization of prepreg-based materials for a range of applications.

ADDITIVE MANUFACTURING – CERAMICS, METAL

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Additive manufacturing of ceramic components via stereolithography

In recent years, the Fraunhofer Center HTL has established manufacturing competence and an excellent infrastructure for small-batch production via ceramic 3D printing. Especially small, highly complex, and dense components, which are exposed to high mechanical, thermal or tribological stresses, or which have to fulfill strict requirements concerning the dielectric strength, are now produced at the center via 3D printing in the form of prototypes or small batches.

In these applications, a printing procedure based on stereolithography is used, which allows for a high density and surface quality of the components and further works largely without finishing steps. The application range of the printed parts varies from complex components for power plant and mechanical engineering, automotive engineering, measuring technology, process technology, and medical engineering up to the jewelry industry.

The Fraunhofer Center HTL also offers comprehensive expertise regarding component construction, design, and production, as well as thermal post-treatment of complex structures built-up by the 3D printer. For example, the duration of the debinding process was reduced by more than a half compared to the original temperature program via specific analysis and optimization by means of thermo gravimetry and thermo-optical measurement devices – generating enormous savings of time and energy.

In addition, the structure of the printed bodies parallel and perpendicular to the printing plane was analyzed by means of cross-section polishing, thermal etching, and scanning electron microscopy. These results are being correlated to the mechanical properties and printing parameters of the components.

Characterization and analytical methods such as, for example, thermo-optical measurement procedures or high resolution micro-computer-tomography are used for quality assurance at the Fraunhofer Center HTL. These services are available to external partners as well.

The advantages in terms of freedom of design offered by this manufacturing procedure are connected to the fraction of organic binding agents in the ceramic slurries used for printing, which is substantial for reasons of process technology. This condition is associated with special challenges during the transfer of the freshly printed green body into the final printed ceramic component via debinding and sintering. However, the competence acquired at the Fraunhofer Center HTL allows the thermal processing of such binding-agent-rich systems to be safely controlled.

The progress of 3D printing technology therefore opens up interesting opportunities for the additive manufacturing of ceramic components. Building up components layer-by-layer enables geometries and design principles which could either not or only with a relatively large effort be fabricated by conventional ceramic shaping methods, such as, e.g., hollow bodies having complex internal structures or undercuts.

Additive manufactruring utilizing powder printing

Powder printing processes are used at the Fraunhofer Center HTL to manufacture metallic and metal-ceramic components. In this process, the powder material is applied layer-by-layer onto a powder bed and impregnated selectively with a binding material by an inkjet printhead. During the binding agent curing process, the powder particles are cross-linked to each other at the impregnated sites and thus form a stable molded body. This mold is then subjected to post-curing after the printing and the powder particles are finally joined after debinding by sintering in a tempering step.

In addition, liquid metal can be infiltrated into the printed components to increase the density. The unused powder can be purified, recycled, and returned to the printing process.

In order to increase the surface quality, the printed parts can subsequently be finished by sandblasting or polishing. The process of thermal post-treatment is monitored in this manufacturing procedure as well, and is analyzed and optimized by methods including e.g. thermo-optical measuring procedures.

In the course of the further process development at the HTL, the material system will be expanded to various ceramic materials. In addition, the process is to be adjusted with regard to the production of high-quality ceramic components and metal-ceramic composite materials, and the process parameters have to be adjusted to the different material systems. Methods including micrographs, high resolution micro-computer- tomography, and mechanical testing procedures are used for quality assurance and process optimization.

The binding-agent-based powder printing process is comparatively fast and low in emissions. Another advantage of the process is that no thermal stresses are caused which could cause the components to deform. The described printing principle can be used for the processing not only of ceramic powders, but also of some reactive metals and metal alloys as well as sintered metals. The process is therefore well-suited for small-batch production of high-grade metallic and ceramic components and metal-ceramic composite components.

PREPARATION PROCEDURES – CRYO-CSP

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In recent years, ion beam methods have become established in the high-quality preparation for electron microscopy in the field of materials analysis. The lower tendency to produce artifacts is one advantage of this method as compared to classical preparation methods, such as cutting, fracturing, grinding, and polishing. The classical mechanical methods modify the microstructure of the test samples significantly and introduce contaminations; freshly-prepared surfaces might immediately react with oxygen such that the material is oxidized. This leads to faulty analyses and can make it virtually impossible to find correct answers to certain questions. Target preparations in the micrometer range, as often needed for damage analysis, are very laborious and sometimes cannot be implemented depending on material and damage; likewise, porous or non-solidified material can be extremely difficult to prepare by classical methods.

Precise and artifact-free target preparation by ion beams

In contrast, ion beam methods used properly allow virtually artifact-free samples for scanning and transmission electron microscopy studies to be produced very efficiently, even from materials that are difficult to prepare, such as ceramic green bodies. Various ion beam preparation methods have been established at the Fraunhofer ISC in recent years.

One of these methods, the so-called »Focused Ion Beam (FIB)« method, involves precise target preparation with a gallium ion beam in a scanning electron microscope (SEM) – specifically for preparation of very thin lamellae for investigation by transmission electron microscopy and for the preparation of highly oxygen-sensitive samples that can be analyzed further right after the preparation in the vacuum of the SEM.

Another method yielding very clean and fault-free cross-sections through the sample material is called »Cross Section Polishing (CSP)«. An argon ion beam is used in this method to cut an artifact-free cross-sectional surface at micrometer-accuracy through the preparation target – e.g. a void in the material.

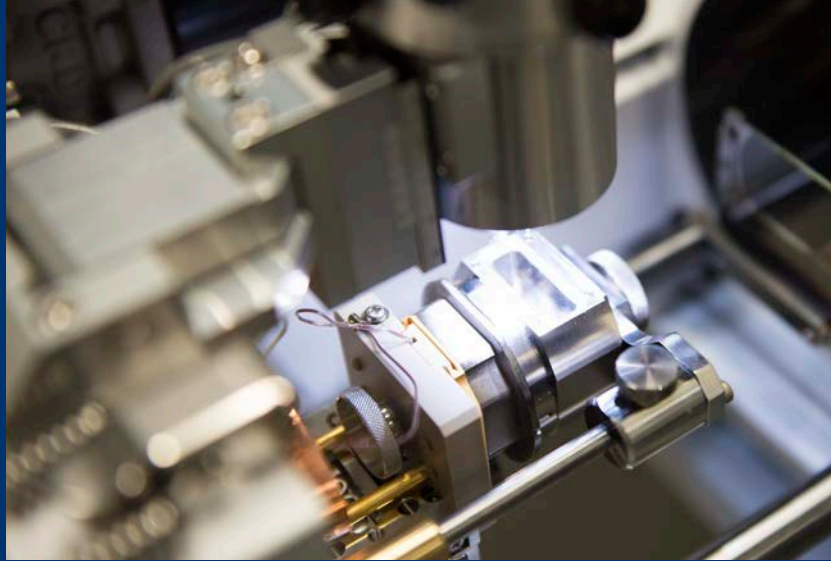
Both preparation methods have proven invaluable with a large number of materials and allow precise statements to be made concerning the composition, microstructure, and interface phenomena in the material. Yet, there actually is one interaction of the material with the ion beam used as the cutting instrument.

Heat protection for sensitive samples in Cryo-CSP

Although the noble gas ions impinging on the material in CSP ion beam preparation do not cause direct changes in the chemistry or microstructure, they do in fact introduce energy, which causes the sample material to heat up locally to up to 120 °C depending on the duration of preparation. While this heating phenomenon is insignificant in many cases, it is undesirable for some materials. For example in the case of hydrate phases or elements like lead and zinc, evaporation or a phase transition might already be triggered.

The heating phenomenon is also a problem in the preparation of organic materials or plastics and wax-like substances.

For this reason, a new preparation device, called Cryo-CSP, was set up in the past year – the first facility of its kind in Europe. The samples are cooled to -160 °C during the preparation in the



Cryo-CSP. This allows the heating during ion beam preparation to be prevented and even temperature-sensitive materials can be prepared free of artifacts for subsequent analysis in the electron microscopes. In addition, the samples can be processed and transferred in a defined atmosphere, e.g. free of oxygen – which is of particular significance for tests on battery materials.

Well-chilled - even during TEM preparation

During the preparation of thin lamellae (approximately 100 nm to be transparent for electrons) for ultrahigh resolution TEM analyses, up to an atomic level, the materials are heated up by bombardment with the ion beam. An ion slicer facility equipped with nitrogen cooling system has been used to expand the preparation methodology to improve the preparation of temperature-sensitive materials for TEM studies. This is important, for example for analysis of nucleation processes in glasses and for analysis of cements or other hydrate phase-containing materials and of lead- and zinc-containing materials. In addition, the device facilitates the preparation of large TEM sample lamellae with a surface area of up to 160,000 µm² (for comparison: the surface area of FIB lamellae is approx. 200 µm²), which means that the TEM analyses become significantly more representative and there is no longer a need to produce large numbers of small FIP lamellae for the same surface area.

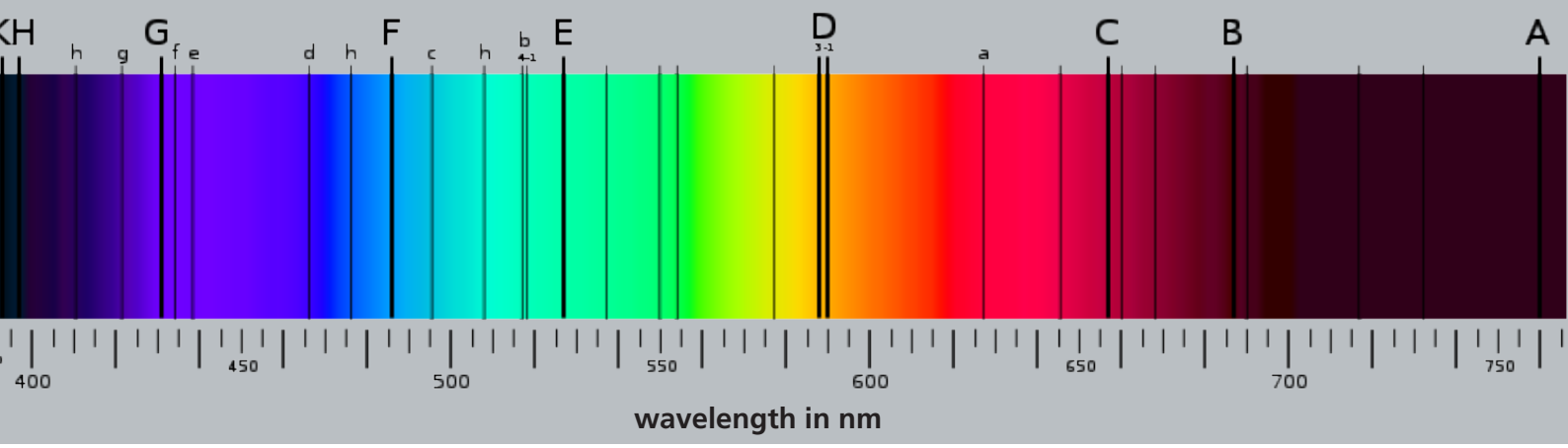
Sample preparation, analytics, expertise in materials – a unique combination

Based on the FIB procedures, the standard CSP preparation as well as the cryo ion slicer and the Cryo-CSP, the Fraunhofer ISC has a set of methods and equipment available for artifact-free preparation of a wealth of different materials for electron microscopic investigations that is unparalleled in Europe. Based on the combination of the ultra-high resolution analytical imaging procedures (also established at the institute) and the profound expertise in preparation, analysis and materials, the Center for Applied Analytics offers its clients optimal prerequisites for efficient quality assurance during the production process and for the materials-based further development of products and production processes.

Roland Och,
ITW Automotive Products GmbH, Röttingen

»The Center for Applied Analytics already supported us with regard to several questions of quality assurance in the production. We are benefitting, time and again, from the detailed knowledge of materials, the profound analytical expertise, and the comprehensive, customer-oriented consulting provided by the Fraunhofer ISC on questions related to materials, in our own product development.«

FRAUNHOFER-GESELLSCHAFT



Research of practical utility lies at the heart of all activities pursued by the Fraunhofer-Gesellschaft. Founded in 1949, the research organization undertakes applied research that drives economic development and serves the wider benefit of society. Its services are solicited by customers and contractual partners in industry, the service sector and public administration.

At present, the Fraunhofer-Gesellschaft maintains 66 institutes and research units. The majority of the nearly 24,000 staff are qualified scientists and engineers, who work with an annual research budget of more than 2 billion euros. Of this sum, around 1.7 billion euros is generated through contract research. More than 70 percent of the Fraunhofer-Gesellschaft's contract research revenue is derived from contracts with industry and from publicly financed research projects. Almost 30 percent is contributed by the German federal and Länder governments in the form of base funding, enabling the institutes to work ahead on solutions to problems that will not become acutely relevant to industry and society until five or ten years from now.

International collaborations with excellent research partners and innovative companies around the world ensure direct access to regions of the greatest importance to present and future scientific progress and economic development.

With its clearly defined mission of application-oriented research and its focus on key technologies of relevance to the future, the Fraunhofer-Gesellschaft plays a prominent role in the German and European innovation process. Applied research has a knock-on effect that extends beyond the direct benefits perceived by the customer: Through their research and development work, the Fraunhofer Institutes help to reinforce the competitive strength of the economy in their local region, and throughout Germany and Europe. They do so by promoting innovation, strengthening the technological base, improving the acceptance of new technologies, and helping to train the urgently needed future generation of scientists and engineers.

As an employer, the Fraunhofer-Gesellschaft offers its staff the opportunity to develop the professional and personal skills that will allow them to take up positions of responsibility within their institute, at universities, in industry and in society. Students who choose to work on projects at the Fraunhofer Institutes have excellent prospects of starting and developing a career in industry by virtue of the practical training and experience they have acquired.

The Fraunhofer-Gesellschaft is a recognized non-profit organization that takes its name from Joseph von Fraunhofer (1787–1826), the illustrious Munich researcher, inventor and entrepreneur.

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