University of Stuttgart

Institute of Interfacial Process Engineering and Plasma Technology

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Annual Report 2018

Editorial notes

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Annual Report 2018

Institute of Interfacial Process Engineering and Plasma Technology University of Stuttgart

Preface



Science and technology are based on facts. As scientists we are dedicated to the truth. In times which are often referred to as post-fact, a university institute has nowadays tasks to fulfill which are adding to the focus on research and teaching: We have to give an example of sincere commitment to search for the facts and to be responsible in our actions. And here we are, grounded in the scientific community which is a keeper of these values.

Our Institute of Interfacial Process Engineering and Plasma Technology IGVP is an active part of the University of Stuttgart and is dedicated to ful-

fill such timeless requirements along with more obvious ones in scientific and technical progress.

As acting director in the fourth year it is my pleasure to present our annual report on our research activities in 2018. On the following pages, we are high-lighting examples of experimental and theoretical studies in the fields of interfacial engineering and plasma technology.

As one of the key initiators, the IGVP acted as a driving force to establish the *Projekthaus NanoBioMater* as a new research structure at the University of Stuttgart over the last five years. Funding by the Carl Zeiss Foundation from 2014–2018 is greatfully acknowledged and along with continuous support by our University we were able to build a living framework for interdisciplinary research on smart hydrogels and their use for designing functional biomaterials. The IGVP is part of the core of the *Projekthaus NanoBioMater* which stands for open communication and collaboration in research. What we regard as a success story is described in this report in the section on Materials Science where many specific studies, partially being part of the *Projekthaus NanoBioMater*, are presented.

The IGVP has a strong background in Life Sciences. This report highlights studies representing the two opposite sides of viruses – friend or foe. Our studies are focussing on better understanding their role in causing infectious diseases or in developing virus-based enabling technologies to fight infections. In contrast, mammalian cell technology is used in order to deepen knowledge for cellbased screening assays and models for drug development.

At IGVP we engineer biotechnical processes and bioreactors in order to intensify the production of valuable biobased products. With these activities, we strengthen the technological basis for a competitive circular bioeconomy to become reality in the near future. We believe that biobased surfactants will find a growing market once the technological foundation is improved, which is the central of the IGVP's research within the Innovation Alliance Biosurfactants, highlighted in this report.

Along with our strong experimental approaches we have intensified our efforts in modeling and simulation. In collaboration with Prof. Dr. Bernard Haasdonk, head of our University's Institute of Applied Analysis and Numerical Simulation, we initiated a study on using machine learning algorithms in the control of microalgae cultivation and thus trying to use Artificial Intelligence (AI) along with direct modeling and simulation approaches.

Our research in plasma technology is continuously inspired by the fruitful collaboration with the Max Planck Institute for Plasma Physics in Garching and Greifswald. The report highlights numerous experimental and simulatory studies on plasma dynamics and diagnostics using our Stuttgart stellarator TJ-K, or designing components for microwave technology. Our deep expertise in plasma technology enables us to develop new plasma sources and to create and apply new processes for surface modification reactions as well as volume-oriented CO₂ gas conversion, addressing urgent environmental tasks of today.

We like to thank our partners for the trust they have placed in us and wish you a pleasant insight in our research activities.

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Prof. Dr. habil. Günter Tovar (acting director)

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Profile of the Institute

The Institute of Interfacial Process Engineering and Plasma Technology IGVP is dedicated to inter- and cross-disciplinary research and teaching in the fields of materials science and technology, life sciences, process engineering and plasma science and technology.



Key Figures

In 2018, the research budget accounted for 2.46 million euros. At the end of that year, 62 scientific, technical and administrative employees, among them 20 young scientists working on a doctoral thesis, staffed the IGVP. Additionally, 43 students were researching for their master or bachelor thesis with us.

Organization and Facilities

The IGVP is part of the Faculty 4: Energy-, Process- and Bio-Engineering of the University of Stuttgart and is organized in the two departments "Interfacial Process Engineering" and "Plasma and Micro-wave Technology".

State-of-the-art labs, technical plants and workshops are available at the IGVP for research in natural sciences and interdisciplinary engineering. They are located at the three IGVP facilities in Pfaffenwaldring 31, Allmandring 5b at the University Campus, and within the Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB in Nobelstrasse 12.

Research

The IGVP focuses on the design of functional materials and their surfaces and interfaces, on the biological interactions at these surfaces as well as on the development and engineering of interfacially driven processes.

In plasma technology, low-temperature plasmas are applied for surface activation, microwaves for stabilization of high-temperature plasmas in fusion-related plasma processes, and the dynamic properties of plasmas and electromagnetic waves are analyzed and simulated.

Collaboration

Close cooperation of the IGVP with the Fraunhofer IGB enables a dynamic collaboration between researchers and lecturers of both institutions and furthermore allows to pursue projects from basic research to application. This approach is reflected in the variety of funding received by the IGVP, including funds from the the *Land* of Baden-Württemberg, from Federal Ministries (e.g. BMBF), from the German Research Foundation (DFG), from the German Federal Foundation for the Environment (DBU), from the EU, and from various foundations as well as from industry. Since many years, the Max Planck Institute for Plasma Physics (IPP), located in Garching and Greifswald, is another key partner of the IGVP and is enabling us to maintain a leading role in plasma technology.

Teaching

The IGVP is highly active in teaching both within master and bachelor study programs at the University of Stuttgart, especially in the programs on Process Engineering, Medical Technology, Technical Biology, Energy Technology, Renewable Energy Engineering, WASTE, and Environmental Engineering.

Thematic Focus

- Interfacial process engineering
- Nanomaterials and nanotechnology
- Biomaterials and infection biology
- · Renewable raw materials, industrial biotechnology, and bioenergy
- Plasma technology and plasma physics
- Microwave technology for plasmas and process engineering

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Interfacial Process Engineering



Functional Polymers with Defined Properties

Dr. Alexander Southan, alexander.southan@igvp.uni-stuttgart.de

Functional materials based on synthetic or biobased polymers are useful for various purposes, including separation of solutes, sensing, or tissue engineering. In these contexts, chemically, biologically, or physically functional groups present inside the materials or at the material surfaces drive the interaction of the materials with their environment. Therefore, our research deals with the synthesis, characterization, formulation, and processing of polymeric materials with defined functional groups or physical properties in order to tailor the response of the environment to the materials. Both the bulk and surface properties are in the focus of the research. The main objects of the research are cross-linked polymers such as hydrogels and nanoparticles.

The material formulations are based on the one hand on commercially available building blocks. On the other hand, custom building blocks are synthesized and characterized thoroughly if necessary. Also, formulation flow properties are investigated in detail to make them suitable for additive manufacturing processes like extrusion-based 3D printing or inkjet printing. Materials are characterized, e.g., regarding their mechanical properties, their adsorption behavior for solutes, or their interactions with human cells.

Examples are the synthesis of amphiphilic compounds for surface functionalization (p. 23), the synthesis of functional poly(ethylene glycols) for the generation of new hydrogel materials (p. 27), surface coating of polymer materials (pp. 23 and 29), or the functionalization of the extracellular matrix with azide groups (p. 20). Thus, our research covers the entire development process leading to functional polymers and spatially defined hydrogels.

Biomolecules from the Native Tissue Matrix

Dr. Kirsten Borchers, kirsten.borchers@igvp.uni-stuttgart.de

Object of research are fields of application for biomolecules from the native tissue matrix i.e. collagen, gelatin, hyaluronic acid, or heparin. Due to their natural origin these polymers are largely biocompatible and can be used e.g. for drug delivery, biofunctional coatings or the generation of 3D cell-culture matrices. The IGVP offers – in cooperation with Fraunhofer IGB – research and development services for application of biobased materials in (bio)medical engineering, pharmacy, tissue engineering, and cosmetics. We provide biopolymer formulations with controlled functionalities by chemical modifications and formulation, i.e. biopolymers with reactive functions for chemical crosslinking, biopolymer solutions with tailored viscosities, hydrogels with positive or negative net charge.

Investigation and Characterization of an Azide-Modified Extracellular Matrix as Potential Biomaterial Silke Keller, Alexander Southan, Günter Tovar

Biomaterials are materials that interact with biological systems in order to treat. augment or replace any tissue, organ, or function of the body. These materials have to meet certain mechanical reguirements and have to be compatible with the body. Depending on the envisaged application, it can be desirable that they affect the biological activities and responses of cells. In a natural tissue the extracellular matrix (ECM) resembels the natural microenvironment of cells. Due to its high biological activity, the isolated ECM is a promising biomaterial for the use in tissue engineering and regenerative medicine. However, the use of ECM is limited, e.g. due to the lack of specific functional groups which are often required for their use as coatings or scaffolds.



Fig. 1: Top scheme: Metabolic glyco-engineering to generate an azide-modified extracellular matrix ("clickECM"). Figure lower right-hand corner: Detection of the fluorophore-conjugated azides within the *click*ECM via laser-scanning microscopy.

Thus, our approach is to develop an azide-functional ECM which resembles the natural ECM composition of the body and which can furthermore be addressed in a biocompatible chemical reaction (azide-alkyne cycloaddition). Therefore, we incorporate azide groups as chemical handles into the ECM by metabolic glyco-engineering. This so called *click*ECM can e.g. be conjugated with alkyne-functional drugs or biomolecules or it can be immobilized on alkyne-functionalized surfaces to form stable surface coatings to enhance cell adhesion.

Publication: Ruff, S. M., Keller, S., Wieland, D. E., Wittmann, V., Tovar, G. E. M., Bach, M., Kluger, P. J. (2017) clickECM: Development of a cell-derived extracellular matrix with azide functionalities. Acta Biomaterialia 52. https://doi.org/10.1016/j.actbio.2016.12.022

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; Reutlingen University, School of Applied Chemistry, P. Kluger, Reutlingen; University of Konstanz, Department of Chemistry and Konstanz Research School Chemical Biology, Konstanz; M. Bach, University of Hohenheim, Module 3: Analytical Chemistry Unit, Stuttgart

Funding: PhD scholarship of the Peter und Traudl Engelhorn Stiftung; Baden-Württemberg Bioeconomy Research Program of the Baden-Württemberg Stiftung and the Ministry of Science, Research and the Arts ("Glykobiologie/Glykobiotechnologie", reference no. 33-7533-7-11.9/7/2; Vector Stiftung (grant no. P2015-0052); Baden-Württemberg Stiftung ("Glycobiology/Glycomics", grant no. P-BWS-Glyko/09); Fraunhofer internal Program Discover (grant no. Discover 828 355). Hydrogels and Hydrogel Thin-Films for Controlled Release of Growth Factors in Tissue Engineering Christiane Claaßen, Alexander Southan, Kirsten Borchers, Günter Tovar

Sufficient oxygen and nutrient supply of cells is a severe challenge in research on tissue engineering grafts. Controlled delivery of pro-angiogenic growth factors to cells is reported to be a promising approach to stimulate vascularization and thereby improve the nutrient and oxygen supply. Our approach relies on biobased hydrogels processed to thin films, in particular based on methacryl-modified, crosslinked gelatin and heparin, for functional storage and release of growth factors.

In order to understand the delivery behavior of such constructs, we standardized the analytical procedure used for the biobased hydrogel formulation components, resulting in well controlled hydrogel preparation. Hydrogels were obtained through thermal-redox initiated cross-linking. The effect of hydrogel composition on their physico-chemical properties and the controlled release of vascular endothelial growth factor (VEGF) were determined. The release kinetics for VEGF were mainly dependent on the amounts of VEGF used for loading. The hydrogels were cytocompatible with primary human endothelial cells. A stable immobilization of hydrogel thin films on polyethylene terephthalate track-etched membranes was achieved. The coatings are now further characterized concerning their

cell adhesion and pro-angiogenic response.

Publications: Claaßen, C., Claaßen, M. H., Gohl, F., Tovar, G. E. M., Borchers, K., Southan, A. (2018) Photoinduced cleavage and hydrolysis of o-nitrobenzyl linker and covalent linker immobilization in gelatin methacryloyl hydrogels. Macromolecular Bioscience, 18(9), 1800104 Claaßen, C., Claaßen, M. H., Truffault, V., Sewald, L., Tovar, G. E. M., Borchers, K., Southan, A. (2018) Quantification of substitution of gelatin methacryloyl: Best practice and current pitfalls. Biomacromolecules, 19, 42-52 Claaßen, C., Southan, A., Grübel, J., Tovar, G. E. M., Borchers, K. (2018) Interactions of methacryloylated gelatin and heparin modulate physico-chemical properties of hydrogels and release of vascular endothelial growth factor, Biomedical Materials, 13, 055008 Claaßen, C., Sewald, L., Tovar, G., Borchers, K. (2017) Controlled release of vascular endothelial growth factor from heparin-functionalized gelatin type A and albumin hydrogels. Gels, 3(4), 35

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; B. V. Stanzel, Fraunhofer Institute for Biomedical Engineering IBMT, Sulzbach; Augenklinik Sulzbach, Knappschaftsklinikum Saar, Sulzbach

Funding: German Research Foundation (DFG) project BO 4252/1-1: Subretinal delivery and anchorage via angiogenesis of a polyester cell carrier for retinal pigment epithelial transplantation



Fig. 1: A: Alcian blue staining of a hydrogel coating based on methacryl-modified gelatin and heparin on a polyethylene terephthalate membrane; B: Cross-section of the coated membrane.

Biomimetic Hydrogels for 3D Cartilage Equivalents Lisa Rebers, Alexander Southan, Kirsten Borchers, Günter Tovar

Articular cartilage can be distinguished in three zones that differ in composition and structural organization. This superstructure is essential for mechanical properties and is maintained by the chondrocytes. Nevertheless, depth-dependent differences are considered rarely in tissue engineering (TE) approaches. Usage of biobased hydrogels as 3D-scaffolds for cartilage-TE is a promising approach. Therefore, gelatin methacryloyl (GM) is widely used. However, application of these hydrogels as cell-matrix implants is limited by poor load-bearing capacity.

Aim of this project is the strengthening of hydrogels, based on biopolymers occurring in the natural extracellular matrix (ECM) of cartilage. As a basis, we strenghtened GM hydrogels (as collagen substitute) by a sophistacted choice of raw material [1] and utilization of physical gelation in addition to chemical cross-linking [2]. Zonal differences in water content and strength of cartilage-ECM should be emulated by varying hydrogel-composition. Robotic dispensing will be used to manufacture hydrogels with a zonal structure [3]. Thereby, new functional scaffolds for 3D-culture of chondrocytes are investigated and could serve as a basis for mechanically stable cartilage-equivalents.



Fig. 1: Schematic visualization of hydrogel design as functional scaffold for cartilage tissue engineering. Three biomimetic hydrogel compositions will be investigated and processed via robotic dispensing.

Publications: [1] Sewald, L., Claaßen, C., Götz, T., Claaßen, M. H., Truffault, V., Tovar, G. E. M., Southan, A., Borchers, K. (2018) Beyond the modification degree: Impact of raw material on physicochemical properties of gelatin type A and type B methacryloyl. Macromolecular Bioscience, 18, 1800168-1800178. https://doi.org/10.1002/mabi.201800168 [2] Rebers, L., Granse, T., Tovar, G. E. M., Southan, A., Borchers, K. (2019) Physical interactions strengthen chemical gelatin methacryloyl gels. Gels, 5, 4-16. http:// dx.doi.org/10.3390/gels5010004 [3] Stier, S., Rebers, L., Schönhaar, V., Hoch, E., Borchers,

[3] Stler, S., Rebers, L., Schonhaar, V., Hoch, E., Borchers, K. (2019) Advanced formulation of methacryl- and acetylmodified biomolecules to achieve independent control of swelling and stiffness in printable hydrogels. Journal of Material Science: Materials in Medicine, 30, 35. http://doi. org/10.1007/s10856-019-6231-0

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Funding: PhD scholarship of the Evonik Foundation

Surface-Functionalization of Organosilicones and Plastics for Specific Biofunctionalization Jana Grübel, Alexander Southan, Günter Tovar

Materials like plastics and silicones are often applied in the field of medical technology, for example as implant materials. Due to their hydrophobic surfaces, proteins can adsorb unspecifically after implantation which can lead to an encapsulation of the implant. Surface-functionalization of potential implant materials is a strategy to increase the hydrophilic properties of their surfaces and improve their integration into the body. We want to generate a surface-functionalization of these materials without changing their bulk in order to maintain the mechanical properties. For this purpose we test two different strategies. For the first strategy we synthesize functionalized copolymers. For the second strategy we synthesize functional surfactants which can be covalently integrated in the cross-linking process of polymers. Providing a surrounding hydrophilic phase should enable the functional groups to assemble on the surface of the materials. These functional groups are visualized by fluorescence staining and are further used to attach coatings of bioactive substances like gelatin.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Funding: PhD scholarship of the Evonik Foundation



Fig. 1: Schematic visualization of surface-functionalized plastics. 1a, b) Functionalization of plastics with copolymers or integration of surfactants. 2) Covalent coatings of functionalized plastics. 3) Evaluation of the functionalization with cell culture experiments.

Encapsulation of Active Ingredients by Means of Spray-Drying to Enable Controlled Intranasal Drug Delivery and Nose-to-Brain (N2B) Transport Lena Marie Spindler, Günter Tovar

More than one billion people suffer from diseases of the central nervous system. Besides inflammatory and neurodegenerative diseases, neuronal and psychiatric disorders come into account. A major problem in the treatment of the central nervous system is the shielding blood-brain barrier. High dosing is needed to reach therapeutically effective concentrations at the target site. This implies serious side effects, which burdens patients additionally to their actual illness. A promising future approach is intranasal drug delivery, which enables direct medication of the brain and the nervous system, advantageous to intravenous administration. The nose-tobrain transport is scientifically proven, but suitable dosage forms are rare yet. In this work spray-drying is utilized as formulation method. Spray-drying enables the production of micro-particles and encapsulation of active pharmaceutical ingredients (API) in one step. Due to the encapsulation in biopolymers, such as alginate or chitosan, mucoadhesive properties will be facilitated. Furthermore a specific release profile will be adjusted, using different loading concentrations and crosslinking agents. This enables enhanced bioavailability and protection of the encapsulated API against environmental conditions, such as pH and light.

Collaboration: Achim Weber, Carmen Gruber-Traub, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Funding: This work is funded by the EU project N2B-patch (grant agreement no. 721098) and a PhD scholarship of the Studienstiftung des deutschen Volkes e. V.



Fig. 1: Spray-dried sodium alginate particles containing *in situ* encapsulated active ingeredients for neurological treatment via intranasal nose-to-brain (N2B) delivery.

Surface Functionalization of Superporous Hydrogels for Additive Manufacturing Karishma Adatia, Alexander Southan, Günter Tovar

This PhD project deals with research on amphiphilic, multi-functional, water-soluble block copolymer macromonomers for hydrogel functionalization. For this purpose, anionic ring opening polymerization is applied for the synthesis of different polymers with narrow molecular weight distributions. The polymers are characterized by NMR, SEC, Maldi, FT-IR, DSC, TGA, and Langmuir isotherm experiments to investigate the structure-property relationships and to determine the capability of these polymers as hydrogel functionalization reagents. Beyond that, a hydrogel microstructuring procedure, which is compatible with additive manufacturing processes is explored, because additive manufacturing opens the possibility to print spatially resolved 3D materials. For this purpose, a tailor-made reagent for hydrogel microstructuring is synthesized which fulfills the requirements of the additive manufacturing process and the kinetic demands of the hydrogel curing process at the same time.

The combination of the two work packages should lead to functionalized 3D microstructured hydrogels for additive manufacturing. Such materials could for example be used as polymer scaffolds in tissue engineering if bioactive components are coupled to the anchor points.

Collaboration: University of Stuttgart, Stuttgart; Goethe University Frankfurt, Frankfurt am Main; Stanford University, Stanford, USA



Funding: PhD scholarship of the Evonik Foundation

Fig. 1: Schematic depiction of the surface functionalized microstructured, 3-dimensional hydrogel foam.

Hydrogels with Specific Charge Densities Tobias Götz, Günter Tovar, Alexander Southan

Hydrogels offer the possibility to tailor their physico-chemical properties for different applications by defined synthesis of their components. In this project, polyglycerol is modified with different moieties of functional groups like ally ether side chains. Via thiol-ene click reactions, polyelectrolytes are prepared which also carry groups suitable for cross-linking. For polycations amine groups and for polyanions carboxylic groups are introduced. Thus, the charge density in the hydrogels can be tuned by varying pH value. Hydrogels are prepared by cross-linking via the thio-Michael reaction.



Fig. 1: (1) Allyl-functionalized polyglycerol; (2) Side-chain functional polyglycerol with either amine or carboxy groups and photoprotected thiols; (3) Deprotection, hydrogel formulation and crosslinking; (4) Hydrogel with specific charge densitiy; (5) Step growth network of the hydrogel.



Fig. 2: Step growth network hydrogels prepared with different amounts of cross-links changing their swelling behavior. From left to right: 80%, 60%, 40% and 20% cross-linking.

Cross-linkers used can carry a positive charge, inspired by the natural crosslinker desmosin. For tailoring the mechanical properties and the biological activity of the hydrogels, the charge densities are adjusted by controlling the amount of functional side chains attached to the polymer backbone.

Publication: Götz, T., Schädel, N., Petri, N., Kirchhof, M., Bilitewski, U., Tovar, G. E. M., Laschat, S., Southan, A. (2018) Triazole-based cross-linkers in radical polymerization processes: tuning mechanical properties of poly(acrylamide) and poly(N,N-dimethylacrylamide) hydrogels. RSC Advances, 8(60), 34743-3475

Collaboration: Institute of Organic Chemistry, University of Stuttgart, Stuttgart; Max Planck Institute for Medical Research, Heidelberg

Funding: BoiMatS-011 project of the Baden-Württemberg Stiftung and the Ministry of Science, Research and the Arts of the State of Baden-Württemberg

Synthesis of Furan-Functionalized Polyelectrolytes for the Production of Double Network Hydrogels via Diels-Alder Cycloaddition

Oliver Gorke, Günter Tovar, Alexander Southan

Due to the high water content hydrogels have a very broad field of application, but in most cases the mechanical strength is very low. Double network hydrogels, on the contrary, have a high mechanical stability containing a large amount of water (50–90%). These hydrogels, by definition, consist of a rigid, brittle, polyelectrolyte network and a soft, ductile network of polymers which are interlaced.



Fig. 1: Tensile test of a calcium alginate/ polyacrylamide double network hydrogel.

For the investigation of the mechanical properties of double network hydrogels, it is important to produce standardized and defect-free samples for tensile and compression tests. The calcium alginate/polyacrylamide double network hydrogel system is used to establish a reproducible measurement method, which can be transferred to novel furan-functionalized polyelctrolyte/polyacrylamide hydrogel systems. For the preparation of the polyelectrolyte networks, furan-functionalized polymers are crosslinked via a Diels-Alder cycloaddition using a bismaleimide crosslinker.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB; M. Itskov, Department of Continuum Mechanics, RWTH Aachen University

Funding: German Research Foundation (DFG), Project: Mechanics of tough hydrogels, Funding code SO 1387/2-1.



Fig. 2: Furfuryl gelatin polyelectrolyte network crosslinked via bismaleimide crosslinker.

Continuous Manufacturing Process for Composite Hollow Fiber Membranes Isabel Jesswein

Membranes with good water vapor permeability and low permeability towards air are interesting for external humidifiers of polymer electrolyte membrane fuel cells, dehydration of gases or heating, ventilation and air conditioning systems. To optimize water vapor transport through membranes, composite structures with a very thin selective layer are a preferred membrane type.



Fig. 1: Continuous dip coating of a hollow fiber membrane.

A continuous manufacturing process was designed, where hollow fibers as supporting structure are fabricated via nonsolvent-induced phase separation. Subsequently thin layers which form the selective layer of the composite membranes are made by dip coating and cross-linking. Besides evaluating different materials as support structure and selective layer, the influencing parameters of the manufacturing process are investigated. Parameters like the surface tension and viscosity of the coating solutions or the coating velocity are correlated with the resulting coating thicknesses. Furthermore, the impact of the layer thickness on cross-linking, water vapor permeability, nitrogen permeability and thermal stability are studied.

Publications: Jesswein, I., Uebele, S., Dieterich, A., Keller, S., Hirth, T., Schiestel, T. (2018) Influence of surface properties on the dip coating behavior of hollow fiber membranes. Journal of Applied Polymer Science, 135(16), 46163

Jesswein, I., Hirth, T., Schiestel, T. (2017) Continuous dip coating of PVDF hollow fiber membranes with PVA for humidification. Journal of Membrane Science, 541, 281-290

Collaboration: Thomas Schiestel, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Funding: Scholarship of the Landesgraduiertenförderung Baden-Württemberg, University of Stuttgart **Hydrogel Coatings as Selective Membrane Layers for Moisture Recovery in Automotive Fuel Cells** Andre Michele, Alexander Southan, Günter Tovar

Regarding to emissions and imminent bans on cars with combustion engines, the poly electrolyte membrane fuel cell (PEMFC) is a promising technology for automotive applications. The performance is dependent on the state of electrolyte membrane (EM) hydration. Returning excess water generated at the fuel cell's cathode to the EM, is the most reasonable way for optimal water management. This could be carried out by using a membrane with good water vapor permeability and high selectivity. The selectivity towards reaction gases may be improved using a hydrogel coating.

For the production of hydrogels, we cross-link hydrophilic polymers with a cross-linking agent. A polymer solution is mixed with a cross-linker and coated on a substrate by dip-coating or spin-coating. The network formation occurs after activation with heat or UV radiation. Optimization of the hydrogel coating is carried out by varying polymers, polymer formulations and cross-linking conditions. The hydrogels are characterized by common values e.g. the yield of the hydrogel and the degree of swelling. In order to avoid performance loss, the stability, water vapor permeation and selectivity are tested at working conditions of the fuel cell.

Collaboration: Thomas Schiestel, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; Mahle Filtersysteme GmbH, Stuttgart; Fumatech BWT GmbH, Bietigheim-Bissingen; University of Regensburg, Institute of Organic Chemistry, Regensburg

Funding: German Federal Ministry for Economic Affairs and Energy (BMWi), promotional reference 03ET6091D



Fig. 1: Spin-coated and cross-linked hydrogel film on a silanized silicon wafer as model substrate for organic membrane carrier.



Fig. 2: Dip-coated and cross-linked hydrogel film on a membrane carrier foil.

Tribology System for Cold Sheet Metal Forming Based on Volatile Lubricants Paul Reichle, Günter Tovar

Sheet metal forming is one of the most effective technologies to reduce material loss during the production process. Usually mineral oil based lubricants are utilized to reduce the friction within the forming process and to extend the tool life. For this purpose, the oil lubricants often contain toxic or long time stabile additives. Another aspect is that the components need to be cleaned costly and time intensively after the forming process for further processes like coating. One approach to reduce the environmental impacts and the production steps is to lubricate the process with a volatile lubricant such as liquid carbon dioxide or nitrogen. For the lubrication, the liquid is injected directly between the sheet and the forming tool via laser drilled micro holes (comparable to a steam iron).



Fig. 1: CFD simulation of the CO_2 velocity (m/s) inside of a simplified micro hole.

The pressure decrease of the lubricant from 60 bar to atmospheric pressure leads to a drastic cooling-down, dry ice is formed. The dry ice seems to have a positive effect for the lubrication. For a better understanding of the behavior of the fluid flow inside the micro holes computational fluid dynamics (CFD) simulations where made. The possibility to manipulate the fluid flow by the shape of the holes for producing dry ice and reducing the friction is still being researched.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; Manuel Henn, T. Graf, Institut für Strahlwerkzeuge (IFSW), University of Stuttgart; Gerd Reichardt, M. Liewald, Institute for Metal Forming Technology (IFU), University of Stuttgart

Funding: German Reseach Foundation (DFG), SPP 1676: Dry Metal Forming – Sustainable Production through Dry Processing in Metal Forming (TO211/3-2)

The *Projekthaus NanoBioMater* – An Innovative **Research Structure at the University of Stuttgart** Günter Tovar



It is our believe that research in an interdisciplinary field can be strengthened by stimulatory and beneficial structures. Thus, the IGVP is one of the key initiators of the Projekthaus NanoBioMater which is dedicated to interdisciplinary research on smart hydrogels and their use for designing functional biomaterials. Funding by the Carl Zeiss Foundation from 2014–2018 and continuous support by our university enables that the Projekthaus NanoBioMater stands for open communication and collaborative research. The IGVP operates in the core of the Projekthaus together with the Institute of Biomaterials and Biomolecular Systems of our Faculty on Energy-, Process- and Bio-Engineering and with the Institutes of Organic Chemistry and of Material Science of the Faculty of Chemistry.

In the NanoBioMater project house, we focus on novel hybrid materials from biological and synthetic building blocks. In an interdisciplinary approach, intelligent biocompatible and bioactive functional materials are prepared and processed in order to develop intelligent, nanostructured, biogenic-synthetic hydrogels for medical, environmental and food technology.

The Projekthaus NanoBioMater acts as a nucleus of crystallization through an experimental core team of two coordinators and three postdoctoral researchers. It facilitates interdisciplinary information exchange and networking among the participating researchers. 25 interdisciplinary dissertations (10 dissertations at the IGVP) and 129 student theses or seminar papers (58 at IGVP) were and are supervised by the project house's professors, 14 workshops, two summer schools and an international conference were organized by the Projekthaus. Prof. Dr. Günter Tovar acts as one of the two coordinators and Dr. Alexander Southan as PostDoc, thus the IGVP contributes strongly to it. 20 third-party funded projects were initiated with help of the Projekthaus NanoBioMater (8 projects with participation of the IGVP). It is currently being consolidated as the basis for future research networks.

Collaboration: https://www.project.uni-stuttgart.de/ nanobiomater/index.html

Funding: Carl Zeiss Foundation; Research Fund of the University of Stuttgart

Life Sciences

Friend or Foe – Research of Viruses Prof. Dr. Susanne M. Bailer, susanne.bailer@igvp.uni-stuttgart.de

Research in the group of Biological Interfacial Process Engineering BGVT has a strong focus on infectious diseases caused by viruses, bacteria and fungi. Herpesviruses represent an important group of human pathogens including Herpes simplex virus 1 (HSV1) and Epstein-Barr virus. With the aim to identify and characterize novel panherpesviral drug targets, we follow conserved steps of virus morphogenesis, using HSV1 as prototype. These include capsid assembly in the nucleus and release to the cytoplasm using a nonconventional membrane budding process through the nuclear envelope called nuclear egress. Analysis of conventional nucleo-cytoplasmic trafficking of herpesviral proteins through the nuclear pore is complementing these approaches.

Beyond being pathogens, viruses represent attractive platforms for virus-based technologies. Virus vaccines have successfully been applied to fight infections, as exemplified by polioviruses that are close to worldwide eradication. Advanced engineering of viruses enables the development of novel vaccines that are safe, highly potent, and multivalent. The same technology is used to program virus genomes for oncolytic tumor therapy. Modular functionalization of platform viruses is aimed to develop a combined tumor-immune therapy thereby translating virus-based research into medical application. Further efforts at the BGVT go into the development of diagnostic methods and devices. Multiplex PCRs and DNA microarrays are developed that are ideally suited for the highly parallel detection of pathogens causing human infections and for the integration into point-of-care devices.

Diagnostic Assays, Models and Sensors

Dr. Anke Burger-Kentischer, anke.burger-kentischer@igvp.uni-stuttgart.de

For more than 18 years, genetic modification and complex cultivation techniques of mammalian cells have been used in the Molecular Cell Technology group. A central aim is the development and application of cell-based screening assays and models for drug development. Various toxicity assays, antibacterial and pyrogenic/allergenic tests have been established and can be performed according to GLP (good laboratory practice). Moreover, complex epithelial 3D infection models to study host-pathogen interactions have been developed. This technological expertise is applied to identify novel immunomodulatory and antiinfective substances. A particular focus is set on reporter gene systems for the detection of immune receptor ligands (interferon- and toll-like receptors) which are also used as sensors for microbial contaminants. With our experience we are moreover developing mammalian, yeast and bacterial, as well as cell-free expression systems for the production of pharmaceutical proteins.

Characterization of the Herpes Simplex Virus Nuclear Egress Débora Marques, Susanne M. Bailer

The family of the Herpesviruses is divided into three subfamilies, alpha-, beta- and gamma herpesviruses, based on characteristics such as cell tropism, pathogenicity and the site of latency. Herpes simplex virus type 1 (HSV1), an alpha-herpesvirus, causes recurrent facial lesions, keratitis or encephalitis. Morphogenesis of herpesviral virions starts in the nucleoplasm and is completed in the cytoplasm requiring nuclear export of the capsid. Size limitations of the mature capsid prevent its transport through the nuclear pore complex. Instead, the particle uses an envelopment/de-envelopment process through the nuclear membranes called nuclear egress. This budding process, executed by the nuclear egress complex (NEC) composed of the viral proteins pUL31 and pUL34, is conserved throughout the herpesviral family and essential for viral replication. Therefore, the NEC represents a potential target for panherpesviral therapy. Numerous aspects of nuclear earess remain unclear. Using bacterial artificial chromosome (BAC) technology, a fluorescently tagged capsid protein VP26 is expressed [1]. This allows capsid tracking from the nuclear interior to the site of budding using live imaging to gain detailed insight into this highly complex process.

[1] Nagel, C. H., Döhner, K., Fathollahy, M., Strive, T., Borst, E. M., Messerle, M., Sodeik, B. (2008) Nuclear egress and envelopment of Herpes simplex virus capsids analyzed with dual-color fluorescence HSV1(17+). Journal of Virology, 82(6), 3109–3124, doi:10.1128/JVI.02124-07

Publication: Bailer, S. (2017) Venture from the interior – Herpesvirus pUL31 escorts capsids from nucleoplasmic replication compartments to sites of primary envelopment at the inner nuclear membrane. Cells, 6 (46), 1–18. doi:10.3390/cells6040046

Collaboration: B. Sodeik, Institute of Virology, Hannover Medical School, Hannover; Z. Ruzsics, University Medical Center Freiburg, Freiburg im Breisgau

Funding: Science without Borders - Conselho Nacional de

Desenvolvimento Científico e Tecnológico (CNPq), Brazil



Fig. 1: Scheme showing the generation of fluorescently labeled HSV1 capsids starting with BAC mutagenesis up to the microscopic analysis. The pHSV1(17+)Lox2 and pHSV1(17+)Lox2-CherryVP26 represent the BAC containing the HSV1 genome wild type and encoding the mCherry labeled capsid, respectively. The viral protein infected cell protein 8 (ICP8) was labeled by indirect immunofluorescence to compare its distribution with the one from the mCherry.

Screening and Identification of Endogenous Proviral Substances Eileen Arnold, Susanne M. Bailer

Epstein-Barr virus (EBV) is one of the most common human viruses worldwide. Following initial infection, EBV becomes strictly latent and contributes to cell growth and oncogenic transformation which can lead to EBV-associated cancer. Recently, a new promising therapeutic strategy for the treatment of EBV-associated tumors was reported, which is based on a targeted destruction of EBV-positive tumor cells following proviral treatment. Initial clinical studies revealed the demand of new proviral agents with higher EBV-reactivating properties and less cytotoxicity. We hypothesize that human peptides could be a promising source of new proviral agents.



Fig. 1: Schematic outline of the strategy to identify and isolate proviral peptides from a human peptide library. A proviral peptide fraction was identified with significant EBV-reactivating effects. The proviral substance was purified in multiple rounds of separation via reverse-phase chromatography and screening for proviral activity. The purified active agent was identified by mass spectrometry analysis and nuclear magnetic resonance spectroscopy. All images are illustrated exemplarily. To identify these proviral peptides, a cell-based reporter assay for EBV-reactivation was developed and a human peptide library was screened. Finally, a proviral fraction with significant EBV-reactivating effects was identified and isolated via reverse-phase chromatography. The purified substance was analyzed via mass spectrometry and nuclear magnetic resonance spectroscopy. After chemical synthesis of the substance, the unknown molecule will be comprehensively characterized for its suitability as a therapeutical drug. The identification of new proviral peptides may contribute to a promising therapy specific for EBV-associated tumors.

Publication: Funk, C., Raschbichler, V., Lieber, D., Wetschky, J., Arnold, E., Leimser, J., Biggel, M., Friedel, C., Ruzsics, Z., Bailer, S. M. (2018) Comprehensive analysis of nuclear export of herpes simplex virus type 1 tegument proteins and their Epstein-Barr virus orthologs. Traffic, 20, 12627. https://doi.org/10.1111/tra.12627

Collaboration: Pharis Biotec GmbH, Hannover

Funding: PhD scholarship of the Landesgraduiertenförderung Baden-Württemberg

Establishment of a 3D Human Oral Mucosa Model Tina Rehm, Günter Tovar, Steffen Rupp, Anke Burger-Kentischer

3D in vitro skin models are test systems based on in vivo conditions that can partially replace the use of animal models as infection models or for modelling other human skin diseases. Especially for the investigation of oral infections a human immune cell supplemented mucosa model was developed. The epidermal structure was greatly improved by optimizing the keratinocyte cultivation, their integration and cultivation inside the model. To evaluate the new models on the basis of skin- and mucosa-specific markers. the models were analyzed by immunohistochemical staining (IHC) with specific antibodies. In comparison to a normal skin model, the oral mucosa model clearly shows a different morphology and expression of specific markers and thus confirms the successful establishment of an oral mucosa model.

For integration of human immune cells in the oral model, they were isolated from Buffy Coat by density centrifugation. T cells were selected from the isolated immune cells by negative selection of CD4 or the γ/δ -T-cell receptor. Successful isolation and selection of specific T cells was analyzed by flow cytometry. The integration of T cells was successfully demonstrated.

Publications: Kühbacher, A., Henkel, H., Stevens, P., Grumaz, C., Finkelmeier, D., Burger-Kentischer, A., Rupp, S. (2017) Central role for dermal fibroblasts in skin model protection against candida albicans. Journal of Infectious Diseases, 215(11), 1742–1752. https://doi.org/10.1093/ infdis/jix153

Kühbacher, A., Sohn, K., Burger-Kentischer, A., Rupp, S. (2017) Immune cell-supplemented human skin model for studying fungal infections. In: Methods in Molecular Biology Vol. 1508, Human Fungal Pathogen Identification, 439–449. https://doi.org/10.1007/978-1-4939-6515-1

Funding: Fraunhofer Project Center for Drug Discovery and Delivery at the Hebrew University of Jerusalem, Israel



Fig. 1: Immunohistochemical staining of the new oral models. Staining for differentiation and hornification marker cytokeratin-10 (a), staining of mucosa marker cytokeratin-13 in non-basal layers (b), staining of differentiation marker involucrin in suprabasal layers (c) and staining of late differentiation marker filaggrin (d).
Stimulation of Innate Immune Receptors in a Human 3D Infection Skin Model with a TLR4 Antagonist against C. albicans Invasion Helena Merk, Steffen Rupp, Anke Burger-Kentischer

Innate immune receptors play a key role in many acute and chronic inflammatory diseases. Therefore there is a great interest in therapeutic manipulation of innate immune receptors by antagonists and agonists. Immune receptors like the toll-like receptor (TLR), C-type lectin-like receptors (CLR) family or cytokine signaling receptors are shown to be critical for immunity of fungal infections, which remain a leading cause of morbidity.

A human immune cell-supplemented skin infection model is used to validate new TLR agonist and antagonists for the inhibition of Candida albicans invasion. Therefore the model is supplemented with CD4-positive T cells,

which show itself a protective effect against C. albicans invasion (Fig. 1). By the pre-treatment of T cells with a compound active against TLR4 in an antagonistic way, we observed an increased invasion of C. albicans although immune cells are present (Fig. 1, right). This shows the successful validation of new immunomodulatory compounds in a human 3D skin model. Further studies aim to identify defense mechanisms by the addition of a TLR4 agonist to induce inflammatory response within the skin model.

Collaboration: EMC microcollections GmbH, Tübingen, Germany; The Hebrew University of Jerusalem, Israel

Research (BMBF), promotional reference 031L0124A



Fig. 1: Infection of skin models with Candia albicans in the presence of immune cells (middle picture), absence of immune cells (left) and incubation of immune cells with a TLR 4 antagonist (right).

Funding: German Federal Ministry of Education and

Process Engineering

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Interfaces in Bioprocess Engineering Dr.-Ing. Matthias Stier, matthias.stier@igvp.uni-stuttgart.de

Interfaces often play a decisive role in bioengineering. In processes for wastewater and exhaust air treatment microorganisms or enzymes are often immobilized on carriers in order to increase catalyst density. The microbial organisms in aerobic and anaerobic production systems are strongly affected by the ambient conditions in fermenters. For this reason there is a need for studying the respective processes in their entireties. Therefore we use on the one hand analyses in the laboratory such as next-generation sequencing and on the other hand high-tech process analysis such as mass spectrometry. We use standard fermenters but in addition also specific reactor systems such as the flat-panel airlift reactor (developed at Fraunhofer IGB) and self-developed membrane reactors in particular for the use of gaseous substrates such as carbon dioxide and methane instead of sugar. In this manner new products for a sustainable biobased future are developed.

For new products an effective downstream processing is also necessary. For example microalgae contain a broad range of ingredients that can potentially be used in the food and feed sector. From microalgae and other plants biosurfactants can be produced by microorganisms and be tailored in their surfactant performance. Therefore, we investigate various cascading extraction methods in order to harvest the products efficiently from the biomass.

The transfer of innovations from research to the market is a key objective of our activities in this field. Product visions and business models are methodically developed. Since 2015, two start-up projects have been pursued.

Industrial Biotechnology

Dr.-Ing. Susanne Zibek, susanne.zibek@igvp.uni-stuttgart.de

Bioprocess engineering and industrial biotechnology are becoming increasingly important within the area of bioeconomy. The IGVP is working on topics such as the treatment of renewable resources (lignocellulose, plant oils), enzyme screening, process development, and scale-up of fermentation processes. Our focus is currently mostly on optimizing fermentation processes for the microbial production of biosurfactants and polyhydroxyalkanoates from 2nd generation feedstock. Another important topic is the purification of these fermentation broths. Here we work on different downstream processing techniques such as extraction, chromatography or crystallization in order to produce high-quality products being tested by industrial partners.

Laminarin Production with Microalgae: Process Development for the Mixotrophic Biomass Production and Extraction

Konstantin Frick, Matthias Stier, Günter Tovar

The aim of this dissertation is to establish a biotechnological process for the production and processing of laminarin derived from microalgae. The poly carbohydrate laminarin is composed of β -1,3-linked glucose molecules. It can be found in macro- and in microalgae, which use it as a storage molecule for energy and carbon.



Fig. 1: Change in biomass composition of *P. tricornutum* during nitrogen depletion.

Laminarin is similar to a compound located in the cell walls of plant pathogenic fungi. Therefore the non-specific immune system of vascular plants reacts to a contact with laminarin. This activation of the defense mechanisms of the plant can prevent infections with plant pathogenic fungi. For example, common grape wine leaves treated with laminarin are less likely to be infected with downy mildew (*Plas-* mopara viticola) or Botrytis cinerea. The number of infections with downy mildew dropped by 75% and with *B. cinerea* by 55%. Beside its effect as an elicitor in plants, laminarin has an immunomodulating effect in animals and humans. Therefore it can be used to substitute antibiotics for example in animal breeding.

The biomass for testing of applications of laminarin is currently obtained using *Phaeodactylum tricornutum*. Under nitrogen depletion a *P. tricornutum* culture accumulates laminarin up to 25% of its dry biomass. At the moment other diatoms are being investigated to find out whether they are better suited for producing laminarin. Also the influence of specific cultivation conditions on laminarin accumulation in the cells is tested.

Collaboration: Ulrike Schmid-Staiger, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Automation and Optimization of Microalgae Cultivation with Machine Learning Control Yen-Cheng Yeh, Matthias Stier, Günter Tovar

The requirements of climate and environmental protection, as well as the world's growing population, are helping to bring the predominantly fossil-based economy to its natural limits. Especially after the Paris Agreement in 2015, the world has reached a consensus to reduce greenhouse gases and environmental degradation. One way to tackle these problems is to use algae because of the use of carbon dioxide as a raw material and the ability to conserve agricultural land.

Microalgae are suitable as raw materials for the production of food, cosmetics, chemicals, pharmaceuticals, and biofuels. For economic and environmental reasons, microalgae should be cultivated using natural daylight as an energy source on a large scale outdoor production. However, the major challenge is that a robust and proven fully automated control system for algae culture has not yet been well established. This is primarily due to missing models that can control algae growth and product formation in the algae.



Fig. 1: Simulation of the light profile in the FPA photobioreactor.

The goal of the project is the introduction of data-based algorithms based on machine learning to control algae cultivation in order to develop an economical, ecological and robust algae production process on an industrial scale. The focus will be on the cultivation of microalgae in the flat panel airlift (FPA) bioreactor to provide data-driven models for the efficient production of algae biomass and for the accumulation of an intracellular product, as exemplified by laminarin.

Collaboration: Ulrike Schmid-Staiger, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; Fraunhofer Center for Chemical-Biotechnological Processes CBP, Leuna; Prof. Dr. Bernard Haasdonk, Institute of Applied Analysis and Numerical Simulation IANS, University of Stuttgart

Funding: Scholarship of Deutsche Bundesstiftung Umwelt DBU (German Federal Environmental Foundation)

Development of Extraction Processes for the Cascade Utilization of Microalgae Biomass in Food, Feed and Cosmetic Applications Felix Derwenskus, Matthias Stier

Microalgae contain a broad range of ingredients which can be used by the food, feed and cosmetic industry. Depending on the specific strain and the cultivation conditions (e.g. nutrient supply, light intensity, pH value and temperature) microalgae are able to produce high amounts of storage lipids (which occur as triacylglycerides), proteins, polyunsaturated omega-3 fatty acids (e.g. eicosapentaenoic acid, EPA) as well as different micronutrients like carotenoids, e.g. lutein and fucoxanthin. The basic idea of this project is to fractionate different types of microalgae biomass as gentle and selective as possible by applying a combination of pressurized liquid extraction (PLE) and supercritical fluid extraction (SFE) depending on the specific product.

Thus, accessory pigments like fucoxanthin as well as fatty acids were successfully extracted from different algae, e.g. *P. tricornutum*. Subsequently, fucoxanthin was purified (Fig. 1). The extraction parameters, evaluated in the lab, can now be used to scale-up the process into technical scale. This is done in cooperation with the project partners from Fraunhofer CBP in Leuna. Currently, the microalgae extracts as well as purified fucoxanthin are investigated concerning their nutritional properties, safety and techno-functional quality, to finally make them applicable for the food and cosmetic industry.

Publications: Derwenskus, F., Metz, F., Gille, A., Schmid-Staiger, U., Briviba, K., Schließmann, U., Hirth, T. (2019) Pressurized extraction of unsaturated fatty acids and carotenoids from wet *Chlorella vulgaris* and *Phaeodactylum tricornutum* biomass using subcritical liquids. GCB Bioenergy 11, 335–344. https://doi.org/10.1111/ gcbb.12563

Neumann, U., Derwenskus, F., Gille, A., Louis, S., Schmid-Staiger, U., Briviba, K., Bischoff, S. C. (2018) Bioavailability and safety of nutrients from the microalgae *Chlorella vulgaris, Nannochloropsis oceanica* and *Phaeodactylum tricornutum* in C57BL/6 mice. Nutrients 10, 16. https://doi. org/doi:10.3390/nu10080965

Neumann, U., Louis, S., Gille, A., Derwenskus, F., Schmid-Staiger, U., Briviba, K., Bischoff, S. C. (2018) Anti-inflammatory effects of *Phaeodactylum tricornutum* extracts on human blood mononuclear cells and murine macrophages. Journal of Applied Phycology, accepted 2018. doi.org/10.1007/s10811-017-1352-7

Collaboration: Ulrike Schmid-Staiger, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; Fraunhofer Center for Chemical-Biotechnological Processes CBP, Leuna; Thomas Hirth, Karlsruhe Institute of Technology KIT; Max Rubner-Insitut Federal Research Institute of Nutrition and Food, Karlsruhe; University of Hohenheim, Institute of Clinical Nutrition, Stuttgart

Funding: Baden-Württemberg Bioeconomy Research Program, reference no. 33-7533-10-5/93), funded by the Baden-Württemberg Stiftung and the Ministry of Science, Research and the Arts



Fig. 1: Sample of fucoxanthin (> 90% w/w), a pigment extracted from the diatom *Phaeodactylum tricornutum*.

Development of a Bioreactor for the Conversion of Biogas Ilka Mühlemeier, Matthias Stier

The project "Development of a bioreactor for the conversion of biogas into chemical/pharmaceutical products" is part of the biogas reseach area of the Bioeconomy Research Program Baden-Württemberg.



Fig. 1: Schematic view of the developed membrane bioreactor.

The aim of the project "Development of a bioreactor for the conversion of biogas into chemical/pharmaceutical products" is to synthesize industrial and pharmaceutical products with aerobic methanotrophic organisms based on a new reactor concept. With regard to the 2010 world market prices of carbon obtained from retail sugar (0.72 euros per kilogram) in comparison with sugar derived from natural gas (0.19 euros per kilogram), methane produced by biogas plants appears to be a location-independent and cheap alternative carbon source for a variety of industrial and pharmaceutical products.

By using methane as a substrate for microorganisms, difficulties arise towards the fermentation process due to the formation of the explosive gas mixture and the substrate limitation caused by the poor water solubility of methane. This requires the development of an aerated membrane reactor, which prevents the generation of potentially explosive bubbles inside the reactor.

Publication: Mühlemeier, I. M., Speight, R., Strong, P. J. (2018) Biogas, bioreactors and bacterial methane oxidation. In M. G. Kalyuzhnaya & X.-H. Xing (Eds.), Methane Biocatalysis: Paving the Way to Sustainability (213–235). https://doi.org/10.1007/978-3-319-74866-5_14

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart; Thomas Hirth, Karlsruhe Institute of Technology KIT

Funding: Baden-Württemberg Bioeconomy Research Program of the Baden-Württemberg Stiftung and the Ministry of Science, Research and the Arts, reference no. 7533-10-5--/103/1

Production of Novel Polyhydroxyalkanoate Variants for Sustainable Packaging Nicole Werner, Thomas Hahn, Susanne Zibek

Plastic accumulation is one of the most visible forms of environmental pollution. The images of plastic polluted oceans caused a global outcry to decrease the application of petrochemical-based plastics and a simultaneous increasing demand for bioplastics.



Fig. 1: Microbial biopolymers PHVB (poly(3-hydroxybutyrate-co-3-hydroxyvalerate), left) and PHB (polyhydroxybutyrate, right).

Polyhydroxyalkanoate (PHA) is a bioplastic raw material consisting of natural polyesters exclusively produced by bacteria as carbon and energy storage from lipids and sugars. PHA-based bioplastics are highly biodegradable not only in composting plants but also in marine environments [1]. Anyway, it demonstrates comparable characteristics to their synthetical analogous with diverse applications including packaging of food or cosmetics [2]. The main challenge is to achieve a cost-efficient production of PHA, thereby making bioplastic economically competitive and appropriate as a drop-in product. Our intention is to meet these challenges and to increase marketability: We already selected suitable bacteria that enable the utilization of inexpensive and sustainable carbon sources from agroindustrial side streams. The combination of different substrates with distinct microorganisms provides a construction kit that enables us to generate PHAs with various compositions and defined properties for its application in packaging materials.

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Collaboration: Dieter Jendrossek, University of Stuttgart, Institute of Microbiology, Stuttgart; Cornelia Stramm, Fraunhofer Institute for Process Engineering and Packaging IVV, Freising; Matthias Harsch, LCS Life Cycle Simulation, Backnang; Uwe Bölz, HPX Polymers GmbH, Tutzing; Kasim Yilginc, Gaplast GmbH, Peiting; Dietmar Kammerer, WALA Heilmittel GmbH, Bad Boll; Murat Haner, WELEDA AG, Schwäbisch Gmünd

Funding: German Federal Ministry of Education and Research (BMBF): Cost-efficient production of biopolymer polyhydroxyalkanoates (PHA) for the manufacturing of tailor-made sustainable packaging concepts for the cosmetic industry, reference no. 031B0371D

Process Engineering for the Production of the Microbial Biosurfactant Mannosylerythritol Lipid (MEL) Alexander Beck, Susanne Zibek, Steffen Rupp

Mannosylerythritol lipids (MEL) produced by yeasts of the Ustilaginaceae family are highly promising microbial glycolipid biosurfactants with possible applications in household detergents, cosmetics or personal care. The aim of this project is to optimize the bioreactor production of MEL for promising microorganisms.



Fig. 1: Different structures of MEL produced from rapeseed oil depending on the employed microorganisms.

Based on a previous screening of different microorganisms and plant oil substrates, we selected three microorganisms showing the largest potential regarding titer and yield. Moreover, the structures of the resulting MEL biosurfactants are considerably different to account for the diverse fields of application. The production process is individually optimized in small scale bioreactors (1-L to 7-L scale) to evaluate the most critical parameters: aeration, mixing and pH control. Foaming, another major challenge to address, could be reduced by mounting mechanical foam brakers onto the agitator shaft. The next steps are to increase the robustness of the fermentation and then scale-up the process into our 40-L pilot reactor. Based on the thus generated data, techno-economic and life cycle assessment will finally measure the feasibility and ecological impact of the MEL production process.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Funding: Baden-Württemberg Bioeconomy Research Program (Reference no. 7533-10-5-85B), funded by the Baden-Württemberg Stiftung and the Ministry of Science, Research and the Arts



Fig. 2: Bioreactor production of MEL is characterized by foaming of the culture broth.

Cellobioselipids: Biosurfactants Produced by Microorganisms from Sugars Amira Oraby, Susanne Zibek, Günter Tovar, Steffen Rupp

Cellobioselipids (CL) are microbial biosurfactants that are produced by microorganisms utilizing sugars. Due to their surface active behavior and antimicrobial activity they have many potential fields of application, such as in detergents or in cosmetics as a potential substitute for petrochemical surfactants.



Fig. 1: Illustration of the CL-foam fractioning method during fermentation.

In order to broaden the field of application, we examine microorganisms, that produce different CL structures. We furthermore optimize the fermentation and purification processes, in order to obtain highly pure biosurfactants with high product titers. Recent research resulted in the production of different CL structural compositions produced by different microorganism strains. Furthermore we managed to establish a foam fractioning method, that directly separates the product enriched foam fraction from the bioreactor. The sediment of this fraction contains up to 98.7% crude cellobioselipids. By extracting the CLs from this fraction, we obtain a highly pure product, continuously separated during the fermentation.

To guarantee the sustainability and feasibility of our developed process, we continuously evaluate our fermentation process techno-economically and ecologically via life cycle assessment.

Funding: PhD scholarship of the German Federal Environmental Foundation (DBU)



Fig. 2: Purified cellobioselipid crystals.

Innovation Alliance Biosurfactants Susanne Zibek

The Innovation Alliance Biosurfactants is the first strategic alliance in Germany between renowned companies and research institutions to produce functionally optimized biotechnologically produced surfactants starting from domestic renewable raw materials and residual materials in an economical way. By systematic investigation of the performance profile, production and purification of these biosurfactants are to be optimized in such a way that they can be used in application areas such as detergents and cleaners, cosmetics, bioremediation, crop protection and food as an alternative to chemically synthesized surfactants.



Fig. 1: The production and purification of biosurfactants are to be optimized to enable their use in the application areas detergents and cleaning agents, cosmetics, bioremediation, crop protection and food.

Within the Innovation Alliance, various surfactants are to be produced by fermentation or enzymatic reaction. By using various microorganisms, enzymes and raw materials, such as different sugars and fatty acids of variable chain lengths, the surface active properties of these biosurfactants can be varied over a wide range, thus optimizing their functional properties. This structure-based development enables the definition and production of biosurfactant molecules with functional properties adapted to the application.

The companies in the consortium investigate the specific properties and the performance profile of the new biosurfactant molecules already at an early stage. The developed prioritized biosurfactants will be produced biotechnologically, the processes optimized for a high space-time yield and efficient purification processes will be developed.

Throughout the entire process chain, sustainability analysis and evaluation of the renewable raw materials used, the processes and obtained products, a techno-economic evaluation, an assessment of the technological maturity of the processes and an estimation of the market relevance of the developed products are carried out in parallel to the development.

Collaboration: https://www.allianz-biotenside.de/ index.php/uber-uns/

Funding: German Federal Ministry of Education and Research (BMBF)



Plasma and Microwave Technology

Plasma Technology

Microwave Plasmas – Fundamentals and Applications

Dr.-Ing. Matthias Walker, matthias.walker@igvp.uni-stuttgart.de

The research group on plasma technology works on the development of new microwave-generated plasma sources at low pressure and atmospheric pressure, the characterization of these plasmas, and the development of a variety of resulting applications. An example of a plasma source developed at IGVP is the so called Duo-Plasmaline which is operating at low pressure. The Duo-Plasmaline is a linearly extended plasma source and can be expanded by combining several of them to a homogeneous two dimensional plasma array. Other at the IGVP utilized low pressure plasma sources are the Planartron and an ECR-plasma. These plasma sources are well suited for plasma assisted surface treatment like surface activation, etching, and thin film deposition. The coating and various characterizations of insulating layers, O_2 - and H_2O -permeation barriers, or scratch protection layers, and the sterilization of food packaging materials and herbs are our main research topics.

A current major issue is the development of plasma sources for the coating of large areas. This is desired equally from the ecological and economical view. The energy-efficient and resource-friendly material consumption of the plasma process reduces costs, but it also must keep pace with the cycle times of modern production technology.

In recent years the basis for large-scale and high-rate deposition of dielectric layers were investigated with a microwave plasma process and demonstrated in a semi-industrial scale at the plasma technology division of the Institute. The focus of the large area plasma process was on the coating of plastic panels such as polycarbonate (PC) with substrate sizes of 6000 mm length and 3000 mm width. The basic research and layer development were performed on small substrates, typically 150 x 100 mm², and then transferred to the semi-industrial system with a length of 1000 mm and a width of 600 mm.

Plasma-Based Chemical Products by Renewable Energy Charge Andreas Schulz, Irina Kistner, Matthias Walker

The temporarily limited availability of renewable energy sources, such as wind and photovoltaics, will in times of great overcapacity increase storage technologies and alternative uses of electrical energy. The conversion of electrical energy into chemical energy has many advantages. On the one hand, a high energy density comes along with safe and simple storage, and on the other hand, fossil resources are preserved by the generated basic chemicals.

Providing basic chemicals goes hand in hand with the competition of different processes based on different resources. As examples, the extraction of acetylene from crude oil in competition with the syntheses in an arc process or the ammonia production in the Haber-Bosch process compared to the plasma synthesis from hydrogen and nitrogen [1].



Fig. 1: Microwave plasma torch with an air plasma at 1 kW power and 20 slm gas flow.

The IGVP is working on the synthesis of nitric acid from an air plasma [2]. According to the reaction equation: $N_2 + O_2 + plasma \rightarrow 2 NO$, nitrogen monoxide NO is formed in a microwave plasma torch, which reacts downstream with oxygen to form nitrogen dioxide NO₂: $2 NO + O_2 \rightarrow 2 NO_2$. In contact with water, the nitrogen dioxide is protonated to nitric acid HNO₃. $3 NO_2 + H_2O \rightarrow 2 HNO_3 + NO$.

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Plasma-Assisted Ignition of Coal Powders Stefan Merli, Andreas Schulz, Matthias Walker

The growing trend of renewable sources of energy forces coal-fired power plants to increase their flexibility. Therefore, coal power plant operators need to reduce their minimum load and when required shut down the plant. This necessitates an economic and energy-efficient start-up method. Conventional methods with oil or gas do not meet these expectations. An alternative are electrical ignition systems, i.e. DC arcjets. In this work arcjets are investigated to directly ignite the coal in a cold start-up scenario and to sustain a stable flame under minimum load conditions.

The ignition tests were carried out in a pulverized fuel combustion rig at the Institute of Combustion and Power Plant Technology (IFK) with different coal types. The experiments were diagnosed with a high speed camera, which was used as a monitor for the ignition behavior by plotting the average light intensity of each frame over the time. The data was further analyzed by correlating the measured intensities with an "ideal ignition behavior", which was expressed as a square function. The so obtained correlation coefficient gives then a number ranging from 1 (perfect correlation, successful ignition) over 0 (no correlation, unseccessful ignition) to -1 (inverse correlation, does not occur in the experiments). In this way, the correlation

coefficient can be conveniently used to characterize the ignition experiments.

Collaboration: Institute of Combustion and Power Plant Technology IFK, University of Stuttgart; PlasmaAir AG; Mitsubishi Hitachi Power Systems Europe GmbH

Funding: Federal Ministry for Economic Affairs and Energy (BMWi)



Fig. 1: Examples of a successful and an unsuccessful ignition with corresponding correlation coefficients.

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FEM Simulation of the Coaxial Resonator of a Microwave Plasma Torch Irina Kistner, Andreas Schulz, Matthias Walker, Günter Tovar

On the basis of preliminary tests and technological requirements for a microwave plasma unit for CO_2 conversion a modular plasma torch has been constructed, which enables a self-ignition and stable operation of a CO_2 plasma over a wide range of parameters. However, after some experimental trials a heating and subsequent destruction of the NBR sealing rings placed in the area of the coaxial resonator was observed and in order to resolve this issue a FEM-simulation model has been developed.



Fig. 1: Electric field distribution in the coaxial resonator at the ignition position without (top) and with (bottom) cooling and sealing rings.



Fig. 2: Total power dissipation for sealing rings consisting of NBR (top) and teflon (bottom).

In the first step a simplified simulation model of the plasma torch has been compiled and the electric field distribution inside the plasma torch has been investigated. Thereafter more details like cooling and sealing rings were included (Fig. 1), the material of the sealing rings was varied and the total power dissipation in the system was investigated. As shown in Fig. 2 the total power dissipated in the sealing rings consisting of NBR is much higher than for teflon. On the basis of these findings material of the sealing rings used in the experimental setup was switched to teflon and ever since no damage of the sealing rings was observed.

Funding: Federal Ministry of Education and Research (BMBF)

Investigation of Two Different Perovskite Hollow Fibers in Air and CO₂ Plasma Irina Kistner, Andreas Schulz, Matthias Walker, Günter Tovar

In order to produce syngas, which is a mixture of e.g. carbon monoxide and hydrogen, the oxygen contained in a CO₂ plasma has to be removed. For this purpose a tubular reactor, which can be connected to the microwave plasma torch, has been constructed, such that a ceramic hollow fiber can be placed in a gland sealing inside the reactor (see inset picture in Fig. 1). Hollow fibers consisting of two different materials (BaCo_{0.4}Fe_{0.4}Zr_{0.2} (BCFZ) and La_{0.6}Ca_{0.4}Co_{0.8}Fe_{0.2} (LCCF)) have been tested in air and CO₂ plasma. The oxygen concentration was measured as a function of time for microwave powers of 0.6, 0.8 and 1 kW.



Fig. 1: Oxygen concentration over time for a BaCo_{0.4}Fe_{0.4}Zr_{0.2} hollow fiber in air (red dots) and CO₂ (blue squares) plasma. The inset picture shows the fiber inside the reactor.

Since the temperature of the plasma and accordingly of the capillary rises with higher microwave power and hence the activation energy for diffu-

sion is decreasing, the oxygen permeation and concentration is increasing with increasing microwave power. A comparison of the two fiber materials shows that the BCFZ fibers (Fig. 1) have in general a higher oxygen permeation. However, the oxygen permeation in CO₂ plasma decreases strongly after one hour of exposure from 2.9 to 1.5 ml/(min·cm²). This decrease is attributed to the formation of barium carbonates. In contrast the oxygen permeation of the LCCF fibers (Fig. 2) remains almost constant at 2.3 ml/ (min·cm²) after one hour in CO₂ plasma.

Collaboration: Frederic Buck, Thomas Schiestel, Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB, Stuttgart

Funding: Federal Ministry of Education and Research (BMBF)



Fig.2: Oxygen concentration over time for a $La_{0.6}Ca_{0.4}Co_{0.8}Fe_{0.2}$ hollow fiber in air (red dots) and CO_2 (blue squares) plasma.

Simulation of the Cold Gas Flow of a Microwave Plasma Torch for CO₂ Gas Conversion Irina Kistner, Andreas Schulz, Matthias Walker, Günter Tovar

In order to establish the most suitable operation conditions of the microwave plasma torch for CO_2 conversion the gas feed had to be optimized and hence the gas flow characteristics were determined. Therefore, a model for the cold gas flow has been developed via a FEM-simulation program. The model includes four tangential and one axial gas inlet (through the nozzle tip). For the simulation a k- ω turbulence model was chosen, since this model gives more accurate solutions near walls and for strongly bent flow characteristics.

The simulations show that due to the tangential gas inlets with a velocity $v_r = 7$ m/s a rotational flow along the whole length of the quartz glass tube (20 cm) arises. This rotational flow is essential for enclosing the plasma in the center of the quartz tube and hence preventing the contact of the hot plasma with the tube. However, as depicted in Fig. 1 a backflow in the center of the tube is generated without an axial inflow $v_a = 0$ m/s. By adding an axial gas flow of $v_a = 1$ m/s (Fig. 2) the backflow of the gas can be prevented while the rotational flow is maintained.

Collaboration: Ante Hecimovic, Emile Carbone, Federico Antonio D'Isa, Ursel Fantz, Max Planck Institute for Plasma Physics (IPP), Garching







Fig. 2: Streamlines and velocity profile for a gas inflow of v_i = 7 m/s through four tangential inlets and of v_a = 1 m/s through an axial inlet.

Eigenfrequency Analysis of a Microwave Plasma Torch for CO₂ Gas Conversion Irina Kistner, Andreas Schulz, Matthias Walker, Günter Tovar

On the basis of preliminary tests and technological requirements for a microwave plasma unit for CO_2 conversion a modular plasma torch has been constructed, which enables a "self-ignition" and stable operation of a CO_2 plasma over a wide range of parameters. The "self-ignition" takes place if the length of the coaxial resonator equals the wavelength of the microwave and this resonance frequency can be measured via a network analyzer as a function of the coaxial resonator tor length.



Fig. 1: Electric field distribution in the coaxial resonator at the ignition position without (top) and with (bottom) cooling and sealing rings.



Fig. 2: Resonance frequency in dependance of the coaxial resonator length determined by measurement (blue dots), simple (red rectangles) and detailed (green triangles) simulation.

Via a FEM-simulation program a model of this plasma torch has been developed. In the first step a simplified simulation model of the plasma torch has been compiled, the electric field distribution inside the plasma torch has been investigated and an eigenfrequency analysis has been conducted. Thereafter more details like cooling and sealing rings were included (Fig. 1). The resonance frequency in dependence of the coaxial resonator length determined experimentally and via the two different models is shown in Fig. 2. As can be seen here including more details in the model leads to a much better agreement with the experimental results.

Funding: Federal Ministry of Education and Research (BMBF)

Study of the Optical Emissions of a CO₂ Microwave Plasma Discharge at Atmospheric Pressure Kerstin Kiefer, Irina Kistner, Andreas Schulz, Matthias Walker, Günter Tovar

Since electricity from renewable sources of energy is subject to fluctuations, energy storage on demand plays a crucial role to create a reliable grid system. The CO₂ conversion into syngas via a plasma assisted gas conversion powered by renewable energy is one promising approach towards energy storage.

In order to optimize the process the CO_2 microwave plasma source has been examined via high resolution spectrometer. This instrument was used to detect molecular and atomic components such as atomic carbon, atomic oxygen, carbon monoxide (CO) and diatomic carbon (C₂). Although using pure CO_2 the nitrogen molecular component cyanide (CN) were also detected, due to air diffusion from the environment.

In order to investigate the influence of microwave power on the reactions in the plasma, the optical emission spectra of different plasmas were normalized to their total intensity. Fig. 1 shows the emission spectra for three different plasmas powered with 0.4, 0.7 and 1.0 kW microwave power.

Increasing the microwave power supports the dissociation of the CO_2 molecules. Thus, in comparison of the intensity-normalized spectra, an increase of the atomic constituents is noted and a reduction of the CO molecules. Similarly, an increase of the C_2 (swan band) and CN molecules can be seen.





Fig. 1: CO_2 plasma emission spectra normalized to their total intensity at constant gas flow (6 slm) and by varying microwave power of 0.4 kW (black), 0.7 kW (blue) and 1.0 kW (black).

Apparent Activation Energies of PECVD Thin Film Barriers as Detectors of Bulk Sub-Micro Defects Mariagrazia Troia, Andreas Schulz, Matthias Walker

Assuming a defect-free solid film permeated by a gas, a purely Fickian permeation mechanism shows a dependence on the temperature that follows an Arrhenius-like trend. Consequently, measurements of the oxygen transmission rates of PECVD thin films acting as barriers on top of PET foils, carried out in a wide range of temperatures, should provide their relative activation energy. The obtained results, for an organic layer with poor barrier properties and two inorganic barriers deposited via a microwave plasma with HMDSN as monomer and different oxygen feeds, show however no increase in the activation energy and even an apparent reduction in their values. Such results directly demonstrate the presence of defects in the barriers bulk which invalidate the Fickian approach. For the 70/1 barrier, the diffusion seems to take place almost exclusively through micro- and nano-defects in its bulk, while the 40/1 barrier shows an extremely complex behavior to which at least three different diffusion pathways contribute, and with a discrete threshold among them corresponding to a temperature of around 37.5°C. In spite of the poorer bulk performances when compared to the 70/1 samples, the 40/1 films exhibit better overall performances, making them the best suited for barrier applications.



Fig. 1: Arrhenius plot for the oxygen transmission rates for three different plasma-deposited thin films and uncoated PET substrate for reference in the $20-60^{\circ}$ C range, with the apparent activation energies for the resulting diffusion processes calculated through a linear fit. **Investigation on the Deposition of Polymer-like Layers for Applications in Medical Engineering** Deepa Chirakkal, Mariagrazia Troia, Andreas Schulz, Matthias Walker, Günter Tovar

Gas permeation through polymers is a major problem regarding industrial packaging of food or drugs. Oxygen permeation leads to oxidation processes which decrease the durability and the effectivity of drugs. In order to reduce gas permeation through polymers, plasma enhanced chemical vapor deposited (PECVD) films are obtained from gaseous hexamethyldisilazane and oxygen. The deposition of SiO_x-barriers on polyethylene terephthalate (PET) was carried out in an electron cyclotron resonance (ECR) reactor. The variation of the gas feed composition, which affects the barrier's chemical composition, and operating power, which influences reactivity and degree of dissociation in plasma, was then investigated. Figures 1 and 2 show that the effect of such parameters on chemical composition, i.e. the ratio of -CH₂ and Si-O infrared absorption bands around 1260 and 1100 cm⁻¹ is in good agreement with the transmission rates of oxygen through the layers. Higher operating power and oxygen feed gas content lead to deposition of inorganic thin films, which effectively prevent oxygen permeation.



Fig. 1: Intensity ratio of the $\mbox{CH}_{3}/\mbox{Si-O}$ infrared absorption bands.

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Fig. 2: Oxygen transmission rates for the coated PET-foils.

Numerical Simulation of a Linear Microwave Plasma Source Stefan Merli, Andreas Schulz, Matthias Walker

Electrical power from controlled nuclear fusion is expected to be an important source of energy in the future. For upcoming fusion power plants, the deuterium-tritium reaction is considered to be the most efficient. Here, the atomic nuclei merge into helium in a hot, magnetically confined plasma and thereby emit energy-rich neutrons. One disadvantage of this method is the use of tritium, which is very rare on earth due to its short half-life of 12.3 years. Therefore, the used tritium amount has to be reduced by the direct internal recycling of the unburnt tritium from the waste gas. This works with a metal foil pump, which separates hydrogen isotopes from other gases by superpermeability. For this, the hydrogen must be present as atoms or ions, so that the waste gas has to be transferred back to the plasma state. This is supposed to be accomplished with the Duo-Plasmaline, which is a linear microwave plasma source developed at the IGVP. It is highly scalable linearly or as an array and therefore well suited to process large amounts of gas.



Fig. 1: Radial profile of the electron density for an argon plasma at 50 Pa and different microwave powers.

To better predict the yield of the atoms and ions of the Duo-Plasmaline over a wide parameter range, a self-consistent simulation model was set up using Comsol Multiphysics. Here, the field equations of the electromagnetic waves are coupled with the transport equations of the plasma using the fluid approximation. The densities of the electrons, atoms and ions are then calculated on a 2D-axisymmetric geometry. First simulations of argon plasmas show a good agreement with the expectations.

Collaboration: Institute for Technical Physics (ITEP), Karlsruhe Institute of Technology; Muegge GmbH, Reichelsheim

Modeling and Study of the Electric Field **Distribution of a Remote Plasma Source (RPS)** Steffen Pauly, Andreas Schulz, Matthias Walker, Günter Tovar

Photoresists are used in industry for lithographic processes to produce surface structures in the sub-micrometer range. In the final step of the manufacturing process, the cured polymer layer acting as shaping die for the microstructures grown by electroplating must be removed. Etching of the cured resist pattern poses an extreme challenge, as the microstructures must not be damaged. Dry plasma chemical etching by means of radicals generated in the plasma chamber of a remote plasma source (RPS) is a suitable means avoiding damage to the microstructures made of metals like nickel, copper or gold.

Using FEM, a model of the RPS has been developed to investigate the microwave distribution and the microwave coupling into the plasma chamber. The E-field distribution is experimentally measured by heating up substrates in the plasma chamber of the RPS and visualized by liquid-crystal sheets and thermal camera pictures. The picture shows from right to the left the calculated electrical field distribution, the heated-up substrate with a sheet of liquid-crystals on it and the heated-up substrate photographed with a thermo camera.

Collaboration: Monika Balk, Markus Endermann, Klaus



Baumgärtner, Muegge GmbH, Reichelsheim

Fig. 1: Different methods showing the electric field distribution in the plasma chamber of the RPS. From right to left: Heated up substrate photographed with a thermo camera, heated up substrate with a liquid crystal sheet on it and FEM simulation.

Implementation of a Drude Model into the RPS Steffen Pauly, Andreas Schulz, Matthias Walker, Günter Tovar

The aim of the study is to optimize an existing remote plasma source (RPS) with regard to its etching rate and gas temperature and to simplify its setup in order to save production costs. Using FEM, a model of the RPS has been developed to investigate the microwave distribution and the microwave coupling into the plasma chamber. The E-field distribution is experimentally measured by heating up substrates and visualized by liquid-crystal sheets and thermal camera pictures, see also "Modeling and Study of the Electric Field Distribution of a Remote Plasma Source".

If a plasma is ignited, the electron density and thus the permittivity and the conductivity increase, which changes the electric field distribution in the plasma chamber. For this purpose, the model has been extended in a first step by a Drude model. To validate the calculated E-field distribution with the Drude model, images of the plasma were made and compared with the simulations which is shown in the picture. There are three cross-sections of the plasma chamber with the calculated E-field distribution by different electron densities. On the right is a picture of the plasma, which shows eight regions where the plasma shines brighter, which corresponds to the eight E-filed maxima of the simulation c).



Fig. 1: Cross-section of the RPS plasma chamber which shows the calculated E-field distribution by different electron densities n_a . From left to right: a) $n_c = 1 \cdot 10^{15} \text{ m}^{-3}$, b) $n_c = 5 \cdot 10^{17} \text{ m}^{-3}$, c) $n_c = 1 \cdot 10^{18} \text{ m}^{-3}$ and d) a view into the plasma chamber with eight bright shining regions of the plasma.

Microwave Technology

Microwaves for Plasma Heating and Diagnostics

Dr. Carsten Lechte, carsten.lechte@igvp.uni-stuttgart.de

In present-day fusion experiments, Electron Cyclotron Resonance Heating (ECRH) with microwaves in the range of 28–170 GHz at Megawatt power levels are routinely used to heat the plasma, to drive plasma currents, and to control magneto-hydrodynamic instabilities by tailored current drive. The Microwave Technology (MT) research group at IGVP contributes to the development of advanced millimeter-wave heating and diagnostic components, the experimental application of the devices, and the interpretation of experimental results.

For the transmission of high-power millimeter waves, oversized (smooth and corrugated) waveguides as well as quasi-optical transmission lines are used. MT designs, simulates and tests novel microwave components as well as complete transmission systems for various fusion experiments as e.g. Wendelstein 7-X, ASDEX Upgrade, or ITER. At present, emphasis is on the development of remote-steering antennas, holographic reflectors at the inner wall of fusion experiments to redirect the non-absorbed fraction of an ECRH beam into the plasma in a controlled way, calorimeters for power levels in the range of 1–2 MW CW, matching optics to couple the output beams from the generators (gyrotrons) into the transmission systems, as well as in-situ power and mode monitoring devices. MT develops frequency keyed power combiners and switches, which could enhance the performance of the fusion test reactor ITER.

The MT group is also involved in the experiments relying on the microwave devices. The institute contributes to the stabilization of neoclassical tearing modes in ASDEX Upgrade by synchronous switching of millimeter waves to follow the rotating magnetic islands, and supports technologies for steering of the launchers. In support of various fusion experiments, full-wave simulations are carried out to study wave propagation and mode conversion in plasmas. Millimeter waves are not only used for heating of high-temperature fusion plasmas, but also for diagnosis of the plasma. The MT group contributes to Doppler reflectometry for turbulence and flow investigations through simulations of experimental data and the design of components like frequency-steered array antennas. For experiments are designed.

Microwave Technology for ECRH on ASDEX Upgrade, Wendelstein 7-X, and ITER Carsten Lechte, Walter Kasparek, Burkhard Plaum

Electron cyclotron resonant heating (ECRH) systems require matching optics units (MOUs) between the gyrotron source and the transmission line and power and alignment monitoring along it. MOUs usually consist of two beam forming mirrors, and two polarizing mirrors. Power monitoring is achieved by coupling a small fraction of the 1 MW beam power into a diagnostic antenna. The coupling is done either with gratings on selected transmission line mirrors, or with mirror-integrated array antennas. Additional requirements are multi-frequency and dual-polarization capabilities. IGVP has contributed several such designs to the ECRH systems at ASDEX Upgrade, Wendelstein 7-X, and at the gyrotron test facility FULGOR at KIT. For FULGOR, an MOU with an integrated broadband (170-236 GHz) dual-channel coupler for power monitoring and mode-jump detection was designed and fabricated (Fig. 1).

The development of more sophisticated mode-analyzing couplers is discussed in the section "Multiport Coupler".

The design of a dual-frequency, high-power beam splitter is described in section "Design of a 2 frequency beam splitter using a square corrugated waveguide". Design and fabrication of holographic in-vessel reflectors to optimize ECRH scenarios with incomplete absorption are described in the sections "Development of reflection gratings for advanced ECRH scenarios" and "Fullwave simulations of high power microwave components".

Collaboration: J. Stober, D. Wagner, M. Schubert, F. Monaco, H. Schütz, and B. Petzold, Max Planck Institute for Plasma Physics (IPP), Garching; H. Laqua, T. Stange, Max Planck Institute for Plasma Physics (IPP), Greifswald; J. Jelonnek, G. Gantenbein, Karlsruhe Institute of Technology (KIT), Karlsruhe

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Fig. 1: Finished mirror assembly for the M1 gyrotron mirror for the FULGOR test stand at KIT. The two rows of coupling holes for the array antennas are feeding a subsurface waveguide with connectors at the left top and bottom edge of the mirror.

Frequency-Comb Reflectometer Hardware Walter Kasparek, Carsten Lechte

When several frequencies are launched simultaneously into the plasma by a Doppler reflectometer, the radial distribution of the fluctuation spectrum can be obtained in real time. In addition, if a frequency-scanned antenna is used, the wavenumber spectrum of the fluctuations at a given radius can be recorded. For applications at ASDEX Upgrade, the transmitter-receiver electronics of such a comb-reflectometer for millimeter waves in the W-Band (75–110 GHz) is in development.



Fig. 1: Output spectrum of a VDI Integrated Amplifier/ Multiplier Chain with power control, without (blue) and with (red) a SAGE 25 dB output amplifier in saturation, when fed with two input frequencies of 16.0 GHz and 16.2 GHz. For the reflectometer hardware, several concepts are being studied, and a modular design is envisaged, to allow "classical" operation of the reflectometer with one (tunable) frequency only as well as multi-frequency operation.

For the transmitter, two different frequency multipliers (Hextuplers) were investigated with the aim to generate a spectrum of equidistant frequencies. It was found, that upon injection of two frequencies, both multipliers produce spectra of 5-7 lines within a useful power range of 10 dB. It was tried to flatten the spectrum by a SAGE 25-dB amplifier operating in saturation, however, without remarkable success. An example is given in Fig. 1, where the spectrum of a VDI Hextupler for input frequencies of 16.0 and 16.2 GHz is shown with and without amplification. The total output power of typ. 100 mW is sufficient to measure Doppler spectra at typically six frequencies. At present, the detailed characterization of the transmitter is ongoing, and components for a prototype receiver channel have been identified and are on order.

Collaboration: T. Happel, K. Höfler, G. Conway, Max Planck Institute for Plasma Physics (IPP), Garching; P. Hennequin, LPP Palaiseau

Funding: This work is performed within the collaboration with the Max Planck Institute for Plasma Physics (IPP), Garching and Greifswald

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Fullwave Reflectometry Simulations for ASDEX Upgrade Carsten Lechte

Doppler reflectometry is an important microwave diagnostic for turbulent fusion plasmas. Probing beams are scattered at density fluctuations and measure a density fluctuation wavenumber spectrum. However, the amount of scattered microwave power is strongly non-linear and is investigated using fullwave simulations, with density fluctuations supplied by a separate plasma turbulence code. In general, low-wavenumber fluctuations are strongest, and non-linearly saturate the backscattered power.

Earlier simulations suffered from the low number of data points in the "interesting" part of the spectrum (Fig. 1), where a power law was fitted to just 4 or 5 points. For meaningful comparison to experimental spectra and turbulence simulations, the spectral index n needs to be known by ± 0.2 . Fig. 2 shows the same plasma turbulence, but now probed with many more beams, made possible by aggressive code optimisation. The knee in the spectrum is clearly defined, and the power law fit has much better confidence. The value compares very well to the experimental n=-3.6. This in turn supports the validity of the plasma turbulence simulations.

Publication: Happel, T., Görler, T., Lechte, C., Bernert, M., Conway, G., Freethy, S. J., Hennequin, P., Honoré, C., Pinzón, J., Stroth, U., and the ASDEX Upgrade Team (2017) Comparison of detailed experimental wavenumber spectra with gyrokinetic simulation aided by two-dimensional full-wave simulations. Plasma Phys. Contr. Fusion, 59 (5) 054009. doi:10.1088/1361-6587/aa645b

Collaboration: G. Conway, T. Görler, T. Happel, S. Freethy, Max Planck Institute for Plasma Physics (IPP), Garching

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Fig. 2: High resolution wavenumber spectrum with highly confident power law fit.

Fullwave Simulations of High Power Microwave Components Carsten Lechte

The reflecting grating tiles discussed in section "Development of reflection gratings for advanced ECRH scenarios" are designed in an extensive optimization process that only considers local properties. While it is possible to characterize the overall performance of the finished grating tile by low power laboratory measurements, this rarely is comprehensive and can only be performed after fabrication.



Fig. 1: Beam power distribution of the input and scattered beams. Clearly visible are the four mounting holes and the edges of the rectangular tile.

Using the 3D version of the IPF-FD3D code, the grating (including mounting holes) is illuminated by the nominal input beam, and the outgoing wave is decomposed into 1000s of plane waves for analysis of all scattering orders. The setup and beams are shown in Fig. 1. The output beams have been isolated from each other by spectral filtering. Less than 1% of power is lost in the mounting holes. Fig. 2 shows the spectral analysis. The scattering orders are at the expected locations. The spurious orders from -1 to +2 contain about 14.4% of the scattered power. About 3.6% of power is lost by beam truncation. Gaussian parameter analysis of the output beam shows good agreement with the design values. This kind of analysis would not have been possible in experiment. It was also found that if a misaligned input beam has a more shallow incidence, a substantial fraction of power is lost, whereas the opposite tilt causes little power loss.

Collaboration: J. Stober, D. Wagner, Max Planck Institute for Plasma Physics (IPP), Garching

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Fig.2: Plane wave decomposition of the outgoing beam. The various grating orders are displaced by a constant vector. The main output order (+3) contains 81.9% of the incident power.

PROFUSION Code Development Burkhard Plaum

The PROFUSION code (Programs for multimode analysis, simulation and optimization) is constantly extended and enhanced as requested by other projects. In 2018, the focus was on the optimization of reflecting gratings. The optimizer module was upgraded such, that two parameters now define the number of iterations. In addition to the maximum number of iterations. the number of subsequent idle iterations, i.e. iterations which don't result in an improvement, can be limited. If the maximum number is set very high (e.g. 50,000), difficult optimizations have enough iterations to converge, while fast converging problems finish early. In experiments, the average optimization time decreased significantly, Fig. 1 shows the development of the cost function for ten optimizations, with different seeds for the random generator. The maximum number of iterations was 50,000, the maximum number of idle iterations was 5,000. The average iteration count was 19,607.



Fig. 1: Development of the cost function during ten optimizations.

Another development was a calculator for Gaussian beam parameters. A Gaussian beam can be defined either by the waist radius and the location of the waist. Another way is to define a reference plane on the beam axis and give the beam radius and the curvature radius of the phase front, which is infinite at the waist. The conversion is done via the complex beam parameter and this tool allows the calculation from the command line or from scripts.

Development of Reflection Gratings for Advanced ECRH Scenarios Burkhard Plaum, Walter Kasparek, Achim Zeitler

The development of reflecting gratings for advanced ECRH scenarios was continued. They are installed in the vacuum vessel of fusion experiments, where they redirect the microwave beam back into the plasma, if the power was not fully absorbed.

One request was the limitation of the minimum curvature radius of the grooves. A smaller value allows finer structures of the groove profile and can therefore improve the reflection characteristics. On the other hand, the subsequent process of coating does not allow sharp edges and imposes a minimum limit for the curvature radius. Therefore the calculation of the minimum curvature radius was added to the PROFUSION module and the optimizing frontend was extended to optionally define a minimum radius. Fig. 1 shows the reflection efficiency for a 140 GHz grating as a function of the minimum convex curvature radius. One can see that the effect becomes noticeable for values larger than approximately 0.1 wavelengths. The effect is, however, quite strong and for the target value of 0.4 mm, the efficiency is already decreased by more than 10%. Fig. 2 shows the groove profiles for some values for the radius.

Collaboration: J. Stober, M. Schubert, Max Planck Institute for Plasma Physics (IPP), Garching

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Fig. 1: Reflection efficiency as a function of the minimum curvature radius.

Fig. 2: Normalized profiles of gratings with different values for the minimum curvature radius.

Design of a 2 Frequency Beam Splitter Using a Square Corrugated Waveguide Burkhard Plaum, Hiroshi Idei

For the design of a diplexer, which works in two distinct frequency bands. a beam splitter is needed. It is realized as a square corrugated wavequide, which is fed by a Gaussian beam under an angle. A simple beam splitter for one frequency band can easily be designed using the Talbot principle, which predicts a good beam splitting performance, if the wavequide length L is $2a^2/\lambda$, where a is the width of the wavequide and λ is the wavelength. If the beam splitter should work in two frequency bands (110 GHz and 138 GHz), we need a more sophisticated approach to find the optimum dimensions. First, we define a figure of merit (FOM), which specifies the overall performance of the beam splitter. It is composed of four quantities, which are the beam powers for the two output beams, calculated for two frequencies. Since all of these numbers need to be as high as possible, the FOM essentially corresponds to the lowest of all beam powers. Then, the parameters (e.g. the length and the incident angle) can be scanned in order to find good operating regimes.



Fig. 1: Figure of merit (FOM) for a beam splitter as a function of the angle and the length.

Fig. 1 shows the calculated FOM for the example of a 60 mm wide waveguide. Higher numbers correspond to better performance. The optimization with the FOM works very well and will be used to find a final set of parameters for manufacturing a prototype.
Multiport Coupler Alexander Zach

For electron cyclotron resonance heating (ECRH), gyrotron beams with about 1 MW power at 140 GHz are guided by oversized waveguide transmission lines into the plasma vessel to be absorbed by the plasma. Coupling from the gyrotron source to the waveguide is done via matching mirrors in free space. Small alignment errors of the system lead to excitation of higher order modes inside the waveguide beside the main transmission mode (HE₁₁). Those modes have comparably higher losses and can in worst case result in local fields exceeding the breakdown limit of the medium inside the waveguide. For alignment control, a set of hole-array couplers located in a miter bend mirror probes the field inside the waveguide in order to detect the tracer modes for beam offset and tilt (LP₁₁), beam waist mismatch (LP₀₂), and astigmatism (LP₂₁). In addition, the power in the main mode is monitored.



Fig. 1: 2D scan from low power calibration for a field with a large LP_{11} , odd content (left: linear amplitude, right: phase).

A waveguide based pre-processing unit for down-mixing of the 140 GHz information to kHz range was tested. The complete prototype diagnostic system was assembled and calibrated at low power and high power tests at the IPP in Garching were performed with up to 0.95 MW.

Collaboration: J. Stober, F. Monaco, and H. Schütz, Max Planck Institute for Plasma Physics (IPP), Garching; H. Idei, Research Institute for Applied Mechanics, Kyushu University, Japan

Funding: This work is supported by the Max Planck Institute for Plasma Physics (IPP), Garching and Greifswald



Fig. 2: Assembled multiport coupler prototype diagnostic system placed in a miter bend for low power calibration.

Receiver Antenna and Transmission Line for Collective Thomson Scattering on W7-X Carsten Lechte, Peshawa Husain, Walter Kasparek, Burkhard Plaum

Collective Thomson scattering (CTS) is a plasma diagnostic that uses the scattering of electromagnetic waves on the electron cloud around the plasma ions to get information about the ion velocity distribution. On the stellarator Wendelstein 7-X (W7-X), it uses gyrotrons and various antennas from the electron cyclotron resonance heating (ECRH) system. The receiver employs a free channel of the ECRH multi-beam waveguide.

An optical remotely steered receiver antenna (ORSA) has been developed and built. It is based on the multibeam concept, where a beam pivot point located at the edge of the plasma is imaged to a point located in the vacuum window. A movable mirror outside the vacuum provides the beam steering around the point in the vacuum window and thus in the plasma. A schematic of the antenna is shown in Fig. 1.



Fig. 1: Schematic of the optical remote-steering antenna (ORSA) for W7-X.



Fig. 2: Beam paths in the confocal multi-beam waveguide if paraboloid mirrors were used (which are, however, not optimal for Gaussian beams).

Inherent to multibeam waveguides is the distortion of all non-central beams, which is more pronounced at large steering angles or displacements. Fig. 2 shows a mirror configuration ("zig-zag") that minimizes this. The beam distortion for various arrangements and mirror surface shapes was analyzed with a physical optics code. A certain type of ellipsoidal shape was found to be optimal. The results were verified experimentally on a low power test setup.

Publication: Husain, P. (2018) Simulation of a 4-mirror imaging antenna for plasma diagnostics. Master thesis, Universität Stuttgart and Universiteit Gent

Collaboration: D. Moseev, H. Laqua, and M. Weißgerber, Max Planck Institute for Plasma Physics (IPP), Garching and Greifswald; S. K. Nielsen, F. Leipold, DTU Risoe

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Plasma Dynamics and Diagnostics

Plasma Dynamical Processes in Generation and Confinement

Dr. Mirko Ramisch, mirko.ramisch@igvp.uni-stuttgart.de

Key issues of fusion research are to find conditions under which hot (beyond 10 million Kelvin) and dense fusion plasmas can be sustained and confined for a sufficiently long time as to achieve a positive net energy outcome from fusion reactions for utilization in future power plants. Conceptually, these plasmas are confined in toroidal magnetic field configurations allowing for central peak energy densities. Steep density gradients in the plasma edge region, however, can prevent electromagnetic waves from reaching absorption layers for efficient heating. Moreover, plasma fluctuations arising from these gradients can cause turbulent cross-field transport of particles and heat out of the confinement region and, thus, affect thermal isolation of the plasma.

At IGVP, the stellarator experiment TJ-K is operated with low-temperature plasmas for the purpose of conducting fundamental research in the fields of plasma/microwave interactions and plasma turbulence. To capture the non-linear spatio-temporal plasma dynamics, specifically developed diagnostics, e.g. probe arrays, are employed. Studies on wave-conversion processes aim at a detailed understanding of efficient heating scenarios incorporating wave scattering processes at turbulence-distorted boundary layers. The microscopic turbulent dynamics across the interface between confined plasma and scrape-off layer determines the global confinement. The mechanisms of self-generated flows and flow/turbulence interactions are studied in dependence on the magnetic field geometry in view of possible transport control or optimization options.

Experimental investigations are supported by complementary simulations using high-level codes developed on-site or at the Max Planck Institute for Plasma Physics (IPP).

The Effect of Magnetic Curvature on Intermittency Stephen Garland, Mirko Ramisch

Drift-wave (DW) turbulence at the edge region of fusion reactors is a major contributor to particle and heat transport towards the reactor wall. The role of reactor magnetic field geometry in triggering intermittent ("bursty") plasma density fluctuations in DW turbulence has been investigated using a 2D extended Hasegawa-Wakatani (EHW) model. The EHW model consists of coupled equations for the plasma density, n, and the vorticity, Ω :

$$\partial_t n + {\phi, n} + L_n^{-1} \partial_y \phi = C^{-1} (\phi - n) - 2{ln B, \phi - n}$$

 $\partial_{1}\Omega + \{\phi,\Omega\} = C^{-1}(\phi - n) - 2\{\ln B, -n\},\$ where ϕ is the plasma potential, related to the vorticity by $\Omega = \nabla^2 \phi$, $\{a, b\} =$ $\partial_x a \partial_y b - \partial_x b \partial_y a$ is the Poisson bracket, L_{u} is the gradient length scale, C is the collisionality, and B is the magnetic field strength. By choosing suitable values of the magnetic field gradients, expressed by the Poisson brackets, the poloidal curvature profile of TJ-K can be approximated. The intermittency level of fluctuations can be quantified by the "intermittency parameter", µ, deduced through a structure function analysis of the turbulent fields. To compare with the simulations, experimental measurements of density fluctuations were performed at the stellarator TJ-K using a poloidal 64-probe array.



Fig. 1: Simulated (\blacktriangleright) and experimentally obtained (\bullet) intermittency level, μ , as a function of the poloidal angle, θ , for fluctuations in the plasma density. The grey shaded region indicates positive geodesic and negative normal curvature.

From the figure a clear peak in μ is evident, coinciding in both the simulated and experimental data. This peak occurs in the region where the normal curvature is negative and the geodesic curvature is positive (shaded grey), indicating a link between intermittency and magnetic curvature that can be interpreted in terms of previous work as due to a curvature-driven decoupling of the density and potential.

Collaboration: Peter Manz, Max Planck Institute for Plasma Physics (IPP), Garching

Microwave Beam Broadening due to Turbulent Plasma Density Fluctuations Alf Köhn, Eberhard Holzhauer

In magnetic confinement experiments, electromagnetic waves in the microwave frequency range are commonly used for diagnostic purposes as well as heating and current drive applications. ITER, the next generation tokamak currently under construction, relies on high power microwave injection in the megawatt regime to heat the plasma core and drive localized currents to control and mitigate magnetohydrodynamic instabilities. Especially the successful implementation of the latter application is inevitable; failing to stabilize those instabilities can lead to a loss of the confinement. The quality of the injected microwave beam is, however, deteriorated by plasma edge density fluctuations, which leads to a reduced efficiency of the stabilization mechanism. The parameters in ITER in terms of microwave wavelength and propagation length, and thus plasmamicrowave interaction length, cannot be investigated in today's experiments but requires numerical modeling. Due to the typical size of the perturbing density structures being similar to the wavelength of the injected microwave, geometrical optics tools cannot be applied. In a broad international collaboration, we have used two full-wave codes, IPF-FDMC & EMIT-3D, and a wave-kinetic equation solver, WKBeam, which treats the effects of fluctuations in the limit of the Born approximation. Benchmark studies of the codes against each other were performed in scenarios with substantial average broadening of a traversing microwave beam due to the plasma density fluctuations.

For the first time, we could demonstrate how turbulent plasma density fluctuations in the edge of the DIII-D tokamak broaden the injected microwave beam. The experimental results were successfully verified by full-wave simulations of the respective scenarios. Full-wave simulations are computationally expensive due to the requirement of large ensembles in order to get statistically relevant results. One simplifying approach is to use guasi-analytical models or homogenization techniques, where the layer of turbulent density fluctuations is approximated by a layer with an effective constant permittivity. The range of validity of such simplifications is explored in the context of an Enabling Research project.

Publication: Köhn, A. et al. (2018) Plasma Phys. Control. Fusion 60, 075006. doi:10.1088/1361-6587/aac000

Collaboration: Omar Maj, Emanuele Poli, Max Planck Institute for Plasma Physics (IPP), Garching; Roddy Vann, University of York; Michael Brookman, General Atomics, San Diego; Christos Tsironis, University of Athens

Parametric Decay Instabilities during Microwave Heating in TJ-K Alf Köhn, Eberhard Holzhauer

Microwave heating in magnetic confinement experiments can be accompanied by locally large wave electric fields occurring, for example, in the vicinity of the so-called upper-hybrid resonance, which is a resonance for a wave in X-mode polarization. These large fields can give rise to non-linear phenomena like parametric instabilities. The simplest case of these instabilities is the three wave coupling where a pump wave with a freguency fo excites two waves with the frequency f_1 and f_2 . This process can have consequences for plasma heating applications as one of the excited waves oscillates often a few orders of magnitude below the pump wave and can thus transfer energy to the plasma ions.

Previously, a parametric decay instability (PDI) has been identified in TJ-K in scenarios with the 2.45 GHz microwave heating system, where the heating occurs at the upper-hybrid resonance. Recent measurements with the 8 GHz microwave heating system indicate the occurrence of an additional PDI. The plasma heating in this scenario was identified to be due to electrostatic electron Bernstein waves. The high-frequency component of the PDI was successfully detected with a directional coupler installed in the transmission line of the microwave heating system. As a next step, it is planned to detect and identify the low-frequency component. The experiments at TJ-K will be the basis for a collaboration with the DTU Copenhagen, where similar experiments are planned.

Collaboration: Stefan Kragh Nielsen, DTU Copenhagen

Influence of Plasma Density Fluctuations on Mode Conversion Processes in Plasmas Alf Köhn, Eberhard Holzhauer

Microwave heating plays an important role in high- and low-temperature plasmas. If the plasma density, however, exceeds the cut-off density of the injected microwave, the wave can no longer penetrate the plasma and thus not heat it. One way to overcome this barrier is to use microwaves at harmonics of the resonance frequency which requires high electron temperatures for the heating process to be efficient.



Fig. 1: Full-wave simulations of the O-X mode conversion with plasma density fluctuations.

Another method, which also works for low electron temperatures, is to couple to the electrostatic electron Bernstein wave which is very well absorbed at the electron cyclotron resonance and its harmonics. To achieve high coupling efficiency, the microwave beam has to be injected at the correct angle with respect to the background magnetic field.

In collaboration with colleagues from the IAP in Nizhny Novgorod we have studied the influence of plasma density fluctuations on this mode coupling process. Comparisons between fullwave simulations and analytical models have been performed, quantifying the degrading effect of the fluctuations. The strongest degradation was observed when placing the fluctuation layer well ahead of the conversion layer. The degradation then is due to the corresponding stronger scattering of the O-mode resulting in non-optimal angles for the conversion process. The figure shows an example from full-wave simulations, where the perturbing effect on the wave electric field can be clearly seen.

Collaboration: E. Gospodchikov, A. Shalashov, Institute of Applied Physics (IAP), Nizhny Novgorod

Causality Study of Zonal Flow Drive Bernhard Schmid, Mirko Ramisch

Like in a self-organizsation process zonal flows (ZFs) are generated by the ambient turbulence itself. Turbulent eddies are tilted and drive the shear flow $\langle v_{\theta} \rangle$, which suggests a self-amplification of the ZF. For tilted vortices the so-called Reynolds stress (RS), $R = \langle \tilde{v}, \tilde{v}_a \rangle$, is unequal zero and the radial gradient of this flux surface averaged quantity, as indicated by the brackets, drives the ZF, i.e. $\partial_t \langle v_{\theta} \rangle = -\partial_r R$. The dynamics of this system has the character of a predator prey relationship. However, in time series the causal relation is difficult to reveal as a correlation, and even a lack of it. does not imply causation and vice versa.

A method to overcome this problem is convergent cross mapping [1], where the estimate of the evolution of one variable by the other gets more precise with longer time series. Data measured with a poloidal probe array, which consists of 128 Langmuir probes with 32 probes on each of four neighbouring flux surfaces, is used to estimate the RS drive and the ZF response, simultaneously. Applying a conditional averaging technique results in the time evolution of both quantities around the ZF occurrence.



Fig. 1: Convergent cross mapping for zonal flow (ZF) response and Reynolds stress (RS) drive. The correlation coefficent (ρ) converges with the length (L) of the time series, where the difference indicates an unidirectional coupling.

As shown in the figure, for longer time series, the correlation coefficient converges but shows a difference depending on the direction of the cross mapping. Thus, the prediction of the ZF response by the RS drive is better, which implies that the radial RS gradient causes the growth of the ZF.

[1] G. Sugihara, R. May, H. Ye, C.-h. Hsieh, E. Deyle, M. Fogarty, and S. Munch, Science 338, 496 (2012)

Collaboration: Peter Manz, Ulrich Stroth, Max Planck Institute for Plasma Physics (IPP), Garching

Diagnostics for Direct Temperature Fluctuation Measurements Bernhard Schmid, Jens Meyer, Mirko Ramisch

For plasma analyses, information on temperature *T*, density *n* and plasma potential ϕ are required. Simultaneous time resolved measurements of these quantities are important to capture the non-linear dynamics of turbulent particle and heat transport. At TJ-K, fluctuations \tilde{n} and $\tilde{\phi}$ are routinely measured with standard Langmuir probes.



Fig. 1: Double probe characteristic measured in an argon plasma at the TJ-K stellarator. The slope around the zeropoint is anti-proportional to the electron temperature.

A new double probe system is tested for comparatively easy access to the electron temperature T_e . To this end, two close electrostatic probes are biased against each other. With U the bias voltage and $I_{i,sat}$ the ion-saturation current, the ideal characteristic of a double probe is described by $I = I_{i,sat} \tanh \frac{eU}{2T}$. For small U, T_e can be obtained from the slope of the linearized characteristic. An example of a symmetric characteristic in an argon plasma is shown in the figure, together with a linear fit to a small region around the origin. For measuring equilibrium T_e , the double probe system has proven useful.

In a next step, two double probe systems are planned to be tested for usability for measuring electron temperature fluctuations \tilde{T}_{a} on small time scales. Arrangements of either three or four probes come into consideration. In the first case the temperature is estimated from the voltage difference of a double probe and a floating probe. Alternatively, the temperature can be deduced from measurements at two operation points in the linear regime of the characteristic and the simultaneous recording of *I*_{i sat}. With the temperature dynamics available energy confinement times and turbulent heat transport are going to be addressed.

Progress of the Electron Cyclotron Emission Diagnostic System for TJ-K Gabriel Sichardt, Eberhard Holzhauer, Alf Köhn, Mirko Ramisch

The electron cyclotron emission (ECE) from TJ-K was further investigated and the diagnostic was improved. 3D full-wave simulations of the ECE propagation in the vacuum vessel were refined to reflect the situation in the experiment and the antenna realistically. An improvement of the measurement localization by a factor of about three due to the installation of a mirror was shown. However, the received signal is still dominated by ECE being reflected around the torus and passing the plasma multiple times.



Fig. 1: Comparison of thermal electron energies from Langmuir probe diagnostics to the ECE diagnostic using a linear and square evaluation model. A good agreement exists for the T_e^{2} -evaluation.

A tunable resonator setup was designed and installed in TJ-K to enhance the localization of the measurements beyond that obtained by the installation of the mirror only. With this setup no dominant polarization could be detected but nevertheless the evaluation of electron temperatures from the hot-cold calibrated ECE diagnostic shows a good agreement with Langmuir probe measurements in the standard parameter range of TJ-K operation. The integration of the radiation transport equation for TJ-K indicated a square dependence of the emission power on the electron temperature T_e whereas the classical linear dependence shows less accurate results (see figure).

In measurements with neutral gas pressures below the normal range. hints of super-thermal electrons were seen in the ECE diagnostic signals. Particle tracing simulations for Maxwellian and mono-energetic velocity distributions in the TJ-K geometry were performed to estimate the toroidal net currents that can arise from the drift orbits of super-thermal electrons. Using a 3D density model for confinement region and scrape-off layer toroidal net currents were evaluated showing that the experimental results of former measurements have the same net current orientation. The collisionless simulations predict an upper limit for toroidal currents of thermal plasmas far above the experimental values and show that already low fractions of super-thermal electrons significantly increase the currents' strength.

Shearing Rate Dependence of Energy Transfer into the Zonal Flow Til Ullmann, Bernhard Schmid, Mirko Ramisch, Günter Tovar

In toroidal fusion devices the development of and the interplay between shear flows in the edge of the plasma, such as zonal flows (ZF) and the equilibrium shear flow, are of interest, because both are supposed to be involved in the evolution of turbulent transport and, hence, in spontaneous transitions into high confinement. ZFs have their origin in the gradient of the Reynolds stress (RS), where the RS describes the tilt of vortices. Equilibrium shear flows are considered as seed flows for initially tilting vortices and stimulating the ZF drive. Drift-wave turbulence constitutes a direct energy source for ZFs, whereas the RS is responsible for the energy distribution. Here the non-linear energy transfer into the ZF is analyzed and interrelated with the background shear.

In order to vary the shear a biased, ring-shaped electrode is placed into the plasma. The current drawn from the plasma imposes localized strong $E \times B$ – flows ensuring a change in the background shear. The spectral energy transfer is deduced with the "Kim method" from plasma potential fluctuations, measured with a 128-pin, poloidal Langmuir probe array and analyzed with respect to the imposed flow shear.



Fig. 1: The behavior of the energy transfer (black) and ZF (blue/dashed) in dependence of the shearing rate. Both behave the same and have a local minimum at zero background shear. For higher shearing rate the turbulence is reduced and so the energy transfer into the ZF.

In Fig. 1 the energy transfer (black) is plotted versus the shearing rate, in comparison with the ZF power (blue/ dashed). Both show a similar behavior. With the energy transfer, the ZF power decreases. Both assume locally minimal values where the shearing rate vanishes. Here, the equilibrium flow does not provide support for vortex tilting, which is reflected in a reduced coupling of drift-waves with the ZF. For higher shearing rates ($\Omega > 8 \ kHz$) the turbulence is reduced together with the energy transfer into the ZF.

Collaboration: Peter Manz, Max Planck Institute for Plasma Physics (IPP), Garching



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- 54. Altukhov, A., Gurchenko, A., Gusakov, E., Irzak, M., Krutkin, O., Esipov, L., Niskala, P., Kiviniemi, T. P., Leerink, S., Lechte, C. & Heuraux, S. (2018) Benchmarking of full-f global gyrokinetic modeling results against the FT-2 tokamak doppler reflectometry data using synthetic diagnostics
- 55. Chellai, O., Alberti, S., Baquero-Ruiz, M., Furno, I., Goodman, T., Manke, F., Plyushchev, G., Guidi, L., Köhn, A., Maj, O., Poli, E., Hizanidis, K., Figini, L., Ricci, D. & TCV Team (2018) Direct measurements of mm-wave beam scattering by field-aligned blobs in magnetically-confined plasmas. Proceeding of the 45th EPS Conference on Plasma Physics, Prague, Czech Republic 42A, 14.406
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Conference contributions – Posters

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- Frick, K., Aponte Silva, J., Derwenskus, F., Schmid-Staiger, U., Schließmann, U. & Tovar, G. (2018) MIATEST – Future application of microalgae in plant protection. Europäische Tagung zu Kupfer als Pflanzenschutzmittel – 3. European Conference on Copper in Plant Protection, November 15–16, 2018, Berlin, Germany
- Goodman, T. P., Chellai, O., Alberti, S., Baquero-Ruiz, M., Furno, I., Fontana, M., Plyushchev, G., Porte, L., Manke, F., Farina, D., Figini, L., Ricci, D., Guidi, L., Köhn, A., Maj, O., Poli, E., Hizanidis, K. & TCV Team (2018) Experimental study of high power mm-waves scattering by plasma turbulence in TCV and TORPEX. 20th Joint Workshop on Electron Cyclotron Emission and Electron Cyclotron Resonance Heating, May 14–17, 2018, Greifswald, Germany
- Guidi, L., Maj, O., Snicker, A., Köhn, A., Weber, H., Lasser, C. & Poli, E. (2018) Microwave beams in plasmas: numerical study of scattering and reflections in the semiclassical limit. P26.23, DPG-Frühjahrstagung, March 4–9, 2018, Erlangen, Germany
- Kistner, I., Schiestel, T., Schulz, A., Walker, M. & Tovar, G. (2018) Microwave plasma reactor for CO₂ decomposition. 16th International Conference on Plasma Surface Engineering (PSE 2018), September 17–21, 2018, Garmisch-Partenkirchen, Germany
- Kistner, I., Schulz, A., Walker, M. & Tovar, G. (2018). Investigation of a microwave plasma torch for CO₂ gas conversion. DPG-Tagungen, March 4–9, 2018 Erlangen, Germany
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- Merli, S., Youssefi, R., Maier, J., Schulz, A. & Walker, M. (2018) Investigation of DC-arcjets for the ignition of coal powder. 16th International Conference on Plasma Surface Engineering (PSE 2018), September 17–21, 2018, Garmisch-Partenkirchen, Germany
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- Pauly, S., A. Schulz, Walker, M., Tovar, G., Dünnbier, M. & Baumgärtner, K.-M. (2018) Investigation of a low pressure microwave plasma source for high rate etching. DPG-Tagung, March 4–9 2018, Erlangen, Germany
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- 77. Sewald, L., Claaßen, C., Götz, T., Claaßen, M. H., Truffault, V., Tovar, G., Southan, A. & Borchers, K. (2018) Impact of conditioning process related properties of gelatin on material properties of methacryloylated type A and type B gelatin. 29th Annual Conference of the European Society for Biomaterials (ESB), September 9-13, 2018, Maastricht, Netherlands
- Sewald, L., Granse, T., Tovar, G. & Borchers, K. (2018) Process of physical gelling influences the mechanical characteristics chemically cross-linked gelatin methacryloyl hydrogels. 29th Annual Conference of the European Society for Biomaterials (ESB), September 9–13, 2018, Maastricht, Netherlands
- Troia, M., Schulz, A., Walker, M. & Hirth, T. (2018) CO₂-based test for the detection of defects in oxygen barrier layers. 16th International Conference on Plasma Surface Engineering (PSE 2018), September 17–21, 2018, Garmisch-Partenkirchen, Germany
- Ullmann, T., Schmid, B., Ramisch, M. & Manz, P. (2018) Shearing rate dependence of Reynolds-Stress, P12.14, DPG-Frühjahrstagung, March 4–9, 2018, Erlangen, Germany
- Vann, R. G. L., Brookman, M. W., Köhn, A., Leddy, J. & Thomas, M. B. (2018) First principles simulations of microwave beam propagation through edge turbulence. Workshop: Theory of Fusion Plasmas, August 27–31, 2018, Varenna, Italy

Doctoral theses

- 82. Funk, C. (2018) Funktionelle Analyse der essentiellen Protease des Herpes Simplex Virus 1 und ihres nukleären Exports. University of Stuttgart
- Haitz, F. (2018) Entwicklung von Verfahren zur enzymatischen Epoxidierung von Pflanzenölen und Fettsäurederivaten. University of Stuttgart. University of Stuttgart
- 84. Holdmann, C. (2018) Vergleichende Analyse des Wachstums von *Chlorella* sorokiniana im FPA-Reaktor bei verschiedenen Photoperioden im Labor und im Freiland. University of Stuttgart

- 85. Schmid, B. (2018) Experimental study of the zonal-flow dynamics in the magnetised plasmas of the stellarator experiment TJ-K. University of Stuttgart
- Zadvitskiy, G. (2018) Experimental and numerical study of turbulence in fusion plasmas using reflectometry synthetic diagnostics. University of Stuttgart, IJL Université de Lorraine

Master and bachelor theses, seminar papers

- Bartz, A. (2018) Untersuchung zu gepulsten ECR-Mikrowellenplasma-Abscheidung von Dünnschichtbarrieren für die Anwendung in der Medizintechnik. University of Stuttgart, master thesis
- Bolivar Paypay, V. (2018) Technical feasibility and parameterization of nutrients recovery processes from four different substrates. University of Stuttgart, master thesis
- Brebeck, G. (2018) Entwicklung und Charakterisierung eines Membranreaktors zur blasenfreien Begasung methanotropher Bakterien. University of Stuttgart, master thesis
- 90. Canga, A.-B. (2018) Optimierung des Prozessschritts Hydrolyse bei der Herstellung der Hämoperfusions-Kartusche Adsorba und Methodenentwicklung zur Freigabemessung. University of Stuttgart, master thesis
- 91. Chirakkal, D. (2018) Untersuchung zur Abscheidung polymerartiger Schichten für die Anwendung in der Medizintechnik. University of Stuttgart, bachelor thesis
- 92. Gehring, M. (2018) Title protected. University of Stuttgart, master thesis
- 93. Gopakumar, V. (2018) Image mapping the temporal evolution of edge plasma characteristics in tokamaks using neural networks. University of Stuttgart and Ghent University, master thesis
- 94. Granse, T. (2018) Konstruktion und Etablierung eines Messaufbaus für Druckprüfungen an gelatine-basierten Hydrogelen unter physiologischen Bedingungen. University of Stuttgart, master thesis
- 95. Grunwald, S. (2018) Title protected. University of Stuttgart, master thesis

- 96. Husain, P. (2018) Simulation of a 4-mirror imaging antenna for plasma diagnostics. University of Stuttgart and Ghent University, master thesis
- 97. Ji, R. (2018) Title protected. University of Stuttgart, master thesis
- 98. Kahlert, M. (2018) Einfluss von Small molecule inhibitors auf die Morphologie und Entwicklungsdynamik von niederen Eukaryoten. University of Stuttgart, master thesis
- 99. Kalinin, A. (2018) Herstellung und Charakterisierung von polymeren Nanopartikeln sowie deren Einsatz in Mixed-Matrix-Membranen zur selektiven Adsorption von Tocopherol. University of Stuttgart, master thesis
- 100. Kiefer, K. (2018) Untersuchung der optischen Emissionen einer CO₂-Mikrowellen-Plasmaentladung bei Atmosphärendruck. University of Stuttgart, bachelor thesis
- 101. Kim, J. (2018) Rainwater harvesting for domestic use and groundwater recharge in Coimbatore, India. University of Stuttgart, master thesis
- 102. Kolinovic, I. (2018) Title protected. University of Stuttgart, master thesis
- 103. Kroll, C. (2018) Funktionalisierung eines Herpes Simplex Virus 1 zur Tumorimmuntherapie. University of Stuttgart, master thesis
- 104. Leonberger, E. (2018) Title protected. University of Stuttgart, master thesis
- 105. Libuda, L. (2018) Extrusionsbasierte, additive Herstellung von fragilen Hydrogelstrukturen mithilfe von Photopolymerisation und additiv gefertigten, thermoplastischen Stützstrukturen. University of Stuttgart, master thesis
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- 107. Luft, D. (2018) Preparation and characterization of acitve ester group surface functionalized particles, conjugated with horseradish peroxidase. University of Stuttgart, seminar paper
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