University of Stuttgart Institute of Interfacial Process Engineering and Plasma Technology

Annual Report 2015

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Institute of Interfacial Process Engineering and Plasma Technology University of Stuttgart

Preface



Interfaces are constituted where material phases are in contact. Innovation is very often created at the interfaces where different disciplines meet and combine their knowledge and efforts. The Institute of Interfacial Process Engineering and Plasma Technology IGVP at the University of Stuttgart constitutes a highly active meeting space of creative researchers covering a wide range of expertise. The focus of our application-oriented basic research is on materials and their interaction with the living surroundings or on plasma processes for the energy generation of tomorrow.

The following report highlights selected research themes presently in the focus of our research. In 2015, the IGVP was directed by Prof. Dr. Thomas Hirth and its research was organized in the two sections "Interfacial Process Engineering" (directed by Prof. Dr. habil. Günter Tovar) and "Plasma and Microwave Technology" (directed by Prof. Dr. Thomas Hirth).

The section "Interfacial Process Engineering" is dedicated to the design, functionalization and characterization of surfaces and materials of inorganic or organic origin as well as of bio-, nano- and hybrid materials and their interaction. Of special interest are the interactions with biological interfaces as occurring in infection of human cells with viruses, bacteria, or fungi and the formulation of hydrogels and foams as (bio-)inks for additive manufacturing processes. Further activities include the simulation and engineering of interfacially driven processes, e.g. in membrane technology and biotechnology.

The section "Plasma and Microwave Technology" covers a wide field of fundamental and applied research topics in the range from fusion-oriented high-temperature plasma physics to industrial applications of low-temperature plasmas. A focal point is research on these plasmas for surface activation and deposition of new coatings as well as the development of new plasma sources and processes, as fostered by the synergy between microwave and plasma physics know-how. Research in fusion-related plasma physics reaches from fundamental investigations of plasma dynamical processes and simulation of electromagnetic waves to plasma heating with high-power microwaves and development of corresponding transmission systems. Fusion-related research is carried out in co-operation with the Max-Planck-Institute for Plasma Physics (IPP) in Garching and Greifswald and with the Karlsruhe Institute of Technology (KIT). Experimental studies are accompanied by numerical simulations using codes developed at the IGVP.

In both sections many topics are treated in close co-operation with the Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB in Stuttgart. Support is obtained from the German government, foundations, the European Union, and industry.

Since the beginning of 2016 the IGVP is directed by Prof. Dr. habil. Günter Tovar as Acting Director since Prof. Dr. Thomas Hirth accepted a call to the Karlsruhe Institute of Technology (KIT) as Vice President for Innovation und International Affairs. The research topics at the IGVP will continue in their full range.

Günter Tovar



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Institute of Interfacial Process Engineering and Plasma Technology IGVP

The Institute of Interfacial Process Engineering and Plasma Technology IGVP is part of the Faculty of Energy Technology, Process Engineering and Biological Engineering of the University of Stuttgart. In 2015, the research budget accounted for 3.22 million euros. At the end of that year, staff of 92 scientific, technical and administrative employees, among them 41 doctoral students, worked at the three IGVP facilities (Pfaffenwaldring 31, Allmandring 5b, and Nobelstrasse 12) – along with 30 other students researching for their master or bachelor thesis.

Technically, the institute is organized in the department "Interfacial Process Engineering," headed by Prof. Dr. habil. Günter Tovar, and "Plasma and Microwave Technology," which was headed by Director Prof. Dr. Thomas Hirth, and is, since the beginning of 2016, also headed by Tovar. Both departments consist of three research groups each.

Collaboration

Cooperation of the IGVP with Fraunhofer IGB makes it possible to pursue projects from basic research to application. This approach is reflected in the variety of funding received by the IGVP, including German Federal Ministries (e.g. BMBF), the German Research Foundation (DFG), the German Federal Foundation for the Environment (DBU), the EU, the Land of Baden-Württemberg, various foundations and industry. Key partners include the Max Planck Institute for Plasma Physics in Garching, Karlsruhe Institute of Technology, and the Dutch Institute for Fundamental Energy Research in the Netherlands.

Research and teaching

The IGVP focuses on the design, functionalization and characterization of surfaces as well as of bio-, nano- and hybrid materials and their interaction. Special interest lies in the interactions with biological interfaces as, for instance, occurring in infection of human cells with viruses and the formulation of hydrogels and foams to bio-inks for additive manufacturing. Further activities include the simulation and engineering of interfacially driven processes.

On the second part, scientific contributions cover the wide range from fusion oriented high-temperature plasma physics to industrial applications of low-temperature plasmas. A focal point is research on these plasmas for surface activation and deposition of new coatings as well as the development of new plasma sources and processes, as fostered by the synergy between microwave and plasma physics know-how. Research in fusion related plasma physics reaches from fundamental investigations of plasma dynamical processes and simulation of electromagnetic waves to plasma heating with microwaves and development of corresponding transmission systems.

Teaching activities are centered on the subject areas of interfacial process engineering, infection biology, nanotechnology, industrial biotechnology, biomaterials, resource-efficient processes as well as plasma physics and plasma technology.

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Members of the Institute

Director: Prof. Dr. Thomas Hirth (until December 31, 2015)

Deputy Director: Prof. Dr. habil. Günter Tovar

Professors: Prof. Dr. Achim Lunk (retired) Prof. Dr. Uwe Schumacher (retired)

Secretary: Christine Demmler Ingeborg Wagner

Administration:

Dr. Matthias Walker Anja Bluhm Ruth Edelmann-Amrhein

Personnel:

Katja Rösslein M. A. Eva Mühlbauer M. A.

Biological-Medical Interfaces:

PD Dr. Susanne Bailer (Head)

Yannick Bantel (PhD student) Lisa Blaschke (PhD student) Eva Brauchle (PhD student) Daniel Foshag (PhD student) Christina Funk (PhD student) Dr.-Ing. Christina Kohl Tanja Maucher (PhD student) Dominik Rais (PhD student) Katrin Sommer (PhD student) Philip Stevens (PhD student) Debora Teixeira Duarte (PhD student) Débora Marques (PhD student) **Chemical-Physical Interfaces:** Dr. Monika Bach (Head)

Vanessa Lima Albernaz (PhD student) Matthias Bieligmeyer (PhD student) Christiane Claaßen (PhD student) Mathias Eichler (PhD student) Philipp Grimmer (PhD student) Dr. Eva Hoch Birgit Huber (PhD student) Isabel Jesswein (PhD student) Simon Kölking (PhD student) Benjamin Riegger (PhD student) Mara Ruff (PhD student) Fabian Schuster (PhD student) Georg Umlauf (PhD student) Michael Walz (PhD student) Annika Wenz (PhD student)

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Plasma Technology:

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Alexis Argiropoulos (Apprentice) Wolfgang Baur Isabel Dieterle (Apprentice) Manfred Krämer Uwe Männl Matthias Matler Sven Sorichta

Electrical Workshop and Electronics:

Karl-Heinz Schlüter (Dipl.-Ing. FH, Head)

Mostafa Gharibyar (Apprentice) Kay Christopher Hörner (Apprentice) Holger Röhlinger Johannes Sunke



Biological-Medical Interfaces

Research at the department of biological interfacial process engineering BGVT focuses on interactions with biological interfaces as occurring during infections of cells with viruses, bacteria and fungi. Part of the BGVT research is focused on herpesviral infections. Here, conserved and essential processes required for herpesviral propagation are analysed potentially providing novel panherpesviral drug targets. Recombinant virus engineering aims at developing innovative therapies and materials based on viruses. Finally, screening approaches facilitated by high-throughput cell-based assays and 3D tissue models are applied to identify small molecules with therapeutic potential.

Another topic of the department is the development of diagnostic methods and devices. Genome-wide methods are applied to analyze cellular and microbial systems, in particular their interaction with the environment, e.g. in infection processes. The identification of biomarkers will provide diagnostic tools to identify and follow diseases. Furthermore, DNA-microarrays are developed to diagnose diseases by a highly parallel molecular detection of various pathogens.

The Institute is also involved in investigating the potential of microorganisms and enzymes for use in industrial biotechnology. Further activities include the simulation and engineering of interfacially driven processes, e.g. in membrane technology and biotechnology. Additional topics are cell-free protein synthesis as well as synthetic biology.

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Molecular and Functional Characterization of Pathogenesis-Related proteins in Candida albicans Contact: Yannick Bantel

The secreted glycoproteins Rbe1 and Rbt4 are part of the conserved CAP pro- this is of high medical importance. Diftein superfamily and have an important impact on the virulence of the human pathogenic fungus Candida albicans. Fungal cells lacking these two proteins are less virulent in a mammalian host and show increased sensitivity towards host innate immune cells.



Fig. 1: Homology model of the CAP domain of Rbt4 showing its characteristic α-β-α fold

Until now little is known about the molecular function of these two proteins, even though CAP proteins are distributed among all kingdoms of life. Currently a role of CAP proteins in the export of sterols and the formation of amyloid fibrils is discussed. As Rbe1 and Rbt4 might be interesting therapeutic targets the aim of the project is to uncover their molecular contribution to the virulence of Candida albicans. Regarding the alarming mortality rates

of systemic Candida albicans infections ferent techniques like next-generation sequencing, lipidome and protein interaction analysis are being used to characterize the function of Rbe1/Rbt4. Current experiments showed that both Rbe1 and Rbt4 play a potential role in the export of sterols and that there might be a link towards the fungal metabolism. Future research will also be focusing on the effect of these secreted proteins on the human host.

Collaboration: K. Sohn, Fraunhofer Institute of Interfacial Engineering and Biotechnology IGB; B. Helms, Utrecht University, Netherlands; R. Schneiter, University of Fribourg, Switzerland Funding: PhD Scholarship Landesgraduiertenförderung Baden-Württemberg

Providing Oxidoreductases for (Chemo-)Enzymatic Methanol Production from Methane Contact: Lisa Blaschke

Sustainable production technologies of platform chemicals such as methanol have become of high interest during the last years. Only minor attention has attached purification tag and purified been paid to methane from biogas as a carbon source for material use.

In the presented work, oxidoreductases are developed for their application in an exemplary (chemo-)enzymatic cascade converting methane to methanol. In-situ cofactor recycling is crucial for economic process design. Here, this is achieved by combining several oxidoreductases such as methanol dehydrogenase (MDH) and formaldehyde dismutase (FDM).



Fig. 1: Crystal structure of homotetrameric formaldehyde dismutase (FDM)

MDH from *Bacillus methanolicus* belongs to the few alcohol dehydrogenases which show catalytic activity towards C1-compounds. FDM from Pseudomonas putida exhibits an integrated cofactor recycling mechanism

which is very rare amongst oxidoreductases. Both multimeric enzymes were heterologously expressed with to homogeneity resulting in specific activities of ca. 14 U g-1 for MDH and 338 U m^{g-1} for FDM.



Fig. 2: Homology model of homodecameric methanol dehydrogenase (MDH)

During the one-step purification procedure, both enzymes retained their activity and thus showed excellent stability properties. Characterization of their catalytic performance will allow the choice of process parameters where both enzymes exhibit good activity. Based on this data, implementation of the (chemo-)enzymatic cascade is possible.

Collaboration: S. Zibek, Fraunhofer Institute for Interfacial Engineering and Biotechnology; M. Pietzsch, Institute of Pharmacy, University Halle-Wittenberg; A. Martin, Heterogeneous Catalytic Processes, Leibniz Institute for Catalysis

Functional analysis of Nucleo-Cytoplasmic Transport of Herpesviral Proteins Contact: Christina Funk, Débora Marques

Context of the Research

The Herpes Simplex Virus-1 (HSV-1) belongs to the herpesvirus family and may cause harmless but also lifethreatening infections. Critical steps of herpesviral propagation occur in the nucleus of infected cells including viral gene transcription, viral DNA replication, capsid morphogenesis and nuclear egress. All of these processes require the presence and activity of herpesviral proteins within the nucleus. We propose that the coordinated nucleo-cytoplasmic exchange is crucial for the successful and efficient herpesviral propagation.

The Aim – A Target for an Anti-Viral Therapy

The aim of these studies is the analysis of nucleo-cytoplasmic trafficking of herpesviral proteins and its functional impact on viral propagation. Detailed analysis will provide novel insights into herpesviral processes and potentially unveil novel targets for anti-herpesvirus therapy.

Strategy

Numerous import and export activities were identified for individual HSV-1 proteins. A range of cell and molecular biology assays is currently used to verify nuclear import and export activities of viral proteins. The functional role that nucleo-cytoplasmic trafficking of herpesviral proteins plays during viral propagation is determined by traceless mutagenesis using the bacterial artificial chromosome technology.

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

Funding: Peter und Traudl Engelhorn Stiftung; Sciences without Borders – CNPq



Fig. 1: Graphic depiction of processes occurring in the host cell nucleus during HSV-1 infection.

Bacterial Enzymes for Lignin Modification Contact: Dominik Rais

Introduction

White-rot fungi attack lignin via secreted laccases, lignin-manganese or versatile peroxidases and accessory enzymes such as certain oxidases, which supply H₂O₂ for the peroxidases. Besides fungi, also some bacterial strains are capable of degrading lignin, but the underlying mechanisms and involved enzymes are hardly known. Bacteria may use similar strategies including laccases, peroxidases and accessory enzymes. However, so far only bacterial laccases are described, while bacterial manganese-, lignin- and versatile peroxidases are unknown. Nevertheless, the so called dyp-type peroxidases could be a substitute for these enzymes in bacteria. For bacterial enzymes are supposed to be produced more easily than fungal lignin modifying enzymes they harbor a great potential as a tool for industrial applications.

Approach and Results

characterized.

Bacterial dyp-type peroxidases DypA

and Dyp-type peroxidases as well as

a bacterial aryl-alcohol oxidase were

heterologously expressed, purified and

We could show that DypA can degrade

several dyes. However, DypC was able

to oxidize aromatic monomers, lignin

indicating a potential for lignin modi-

fication. The aryl-alcohol oxidase was

model substances and manganese,

shown to convert aromatic compounds under O_2 consumption, generating H_2O_2 . In further works DypC will be investigated regarding lignin modification. Furthermore, an enzymatic cascade, including DypC and the aryl-alcohol oxidase for H_2O_2 supply will be established.



Fig. 1: Lignin and lignin derivatives.

Collaboration: S. Zibek, Dr. H. Strittmatter, G.Unkelbach Fraunhofer IGB; Schmiedl, Fraunhofer Institute for Chemical Technology; O. Deppe, Fraunhofer Institute for Wood Research; T. Germer, Robert Kraemer GmbH & Co. KG; C. Terfloth, Jowat AG; M. Wolperdinger, Linde Engineering Dresden GmbH; M. Werth, RAMPF Ecosystems GmbH & Co.KG; K. Schamel, RAMPF Giessharze GmbH & Co. KG; C. Wallenhorst, ASK Chemicals GmbH **Funding**: Deutsche Bundesstiftung Umwelt (DBU); Fachagentur Nachwachsende Rohstoffe (FNR)

Chemical-Physical Interfaces

Chemical-Physical Interfaces

In chemical interface process engineering new functional polymeric materials, in particular polymeric nano- and microparticles, and hydrogels of biobased synthetic and bio-inspired polymers are produced in interfacial processes. Manufacturing processes, structural property relationships and downstream processing, which are constantly experimentally developed, are of particular interest. The focus of our-application oriented basic research is the formulation of biological and chemical drugs or the development of synthetic and natural biomaterials for contact with biological systems (cells). Through *in vitro* research of the fundamental mechanisms that govern cell-biomaterial and cell-cell interactions we develop functional surfaces and interfaces of biomaterials that have the ability to direct cellular responses and regulate the formation and integration of tissue.

By controlled cross-linking of synthetic or natural molecular building blocks we obtain hydrogels with adjustable mechanical and biological properties. A particular focus is the formulation of hydrogels to produce (bio)inks for additive manufacturing processes.

Main research fields of physical interface process engineering are the adaptation of the properties of material surfaces that come into contact with solid/liquid/ gaseous media. At our institute we use mainly plasma technology in which surface changes can be achieved by physical and chemical interactions.

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Surface Active Monomers: Building Blocks for Particle Functionalization Contact: Vanessa Lima Albernaz

Surface active monomers (known as "Surfmers") are polymerizable surfactants that may carry a functional group. Due to the surface-active properties, these molecules are largely on the particle surface and, during surfactantbased emulsion polymerization processes, the surfmers are directly incorporated into the particle's polymeric backbone. This represents an increase in the particle's stability and it also allows the controlled display of the functional groups on the surface of the particles. Thus, particles produced though this route are easily tailored for conjugation with biomolecules. This work aims to produce two surfmer molecules and develop polymeric particles using these surfmers as Comonomers in miniemulsion polymerization

The configured particles with customized functional surfaces are promising candidates for multifunctional platforms suitable for biomedical applications.



Fig. 2: Scanning electron microscopy image of polystyrene nanoparticles using a surfmer molecule as a comonomer.



Fig. 1: Preparation of polymeric dispersions with functionalized surface using surfmer molecules though emulsion polymerization.

systems, in order to obtain nanoparticles of 50 to 300 nm containing a bioreactive surface functionality.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB Funding: Science Without Borders Program (CAPES, Brazil)

Chemical-Physical Interfaces

Reconstitution of Membrane Porin OmpF into Biomimetic Block Copolymer – Phospholipid Hybrid Membranes Contact: Matthias Bieligmeyer

Transmembrane proteins fulfill versatile tasks in all creatures. They are the gatekeepers of cells regarding the exchange of substances with the extracellular space p. ex. for nutrition or signal transduction. Some membrane proteins show selectivities for certain ions or small molecules, which renders them very interesting also for technical applications like water desalination or nanofiltration.

However, the integration of natural lipid bilayers into technical systems is not possible, as these membranes are not stable enough. To overcome this issue, lipids may be replaced by amphiphilic diblock Copolymers to form biomimetic membranes with higher stability.

Additionally, the function of membrane proteins may be limited by the composition of the surrounding membrane. To overcome this problem, membranes were formed from poly(1,4-isopreneblock-ethylene oxide) block copolymers blended with a small amount of 1,2-diphytanoyl-sn-glycero-3-phosphocholine. With that, higher membrane stability is combined with inherent lipid biocompatibility. By reconstituting the membrane porin OmpF from Escherichia coli into these membranes, for the first time, correct protein functionality of this protein in biomimetic lipopolymer membranes was demonstrated [1]. This opens up further perspectives for biomimetic block copolymer-protein hybrid membranes for purification or sensing applications.



Fig. 1: The reconstitution of membrane porin OmpF in biomimetic membranes was monitored by voltage-clamp experiments. Upon insertion of OmpF into the membranes, characteristic conductances increasing stepwise were monitored. With that, the reconstitution of a transmembrane protein in biomimetic lipopolymer-membranes was monitored.

> **Collaboration:** Prof. Dr. S. Nussberger, Biophysics Dept., Institute of Biomaterials and biomolecular Systems, University of Stuttgart

Light-responsive Hydrogels for Controlled Release of Growth Factors in Tissue Engineering Contact: Christiane Claaßen

Presently insufficient oxygen and nutrient supply in tissue engineering grafts due to poor vascularization is a fundamental limitation. Controlled release of pro-angiogenic growth factors is reported to be a promising approach to stimulate vascularization and thereby biointegration. Growth factor concentration and duration of delivery are critical factors. Light-responsive hydrogels are one approach to externally control the release of therapeutics in vitro and *in vivo*.

We investigate the immobilization of growth factors in biopolymer-based hydrogels through photocleavable linkers and their functional light-responsive release into the surrounding tissue matrix. A depiction of the conception for the light responsive hydrogel system can be seen in Fig. 1. So far, the first synthesis steps for preparation of the photocleavable linker as well as the functionalization of gelatin and heparin with cross-linkable groups and determination of their degree of substitution were successful. Hydrogels could be obtained through thermal-redox initiated crosslinking. Currently optimal hydrogel compositions for immobilization and controlled release of VEGF as well as characterzation of the release kinetics via ELISA are investigated.

Collaboration: K. Borchers, Fraunhofer IGB; B. Stanzel, Department of Ophthalmology, University of Bonn 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: Schematic structure of light responsive release system. It consists of two biopolymers and a growth factor loaded, photocleavable linker. All release system components are functionalized with cross-linkable groups.

Mixing Enthalpies of Binary Solutions Measured by Isothermal Flow Calorimetry Contact: Mathias Eichler

Binary solutions (e.g. ethanol/water, electrolyte/water) may experience a change in enthalpy when the mass fraction of their constituents is changed. The subsequent reduction or increase in temperature can be quantified by calorimetric measurements. However, only the data of solutions produced in industrial scale like NaOH_(aq) are well recorded over a wide mass fraction range due to their corrosive properties. temperature equilibrium between the mixed solution, the reactor chamber and the inlet fluids the thermal power needed to maintain this steady state can be obtained and together with the precisely regulated mass flow the specific mixing enthalpy of the system can be derived. The achieved measurement precision is 6 J/g.



Fig. 1: Measurement principle of isothermal flow calorimetry. The calibration heater is used to evaluate measurement precision. In case of temperature equilibrium $T_{in} = T_{out}$ and steady state in power consumption the specific mixing enthalpy can be derived.

The present work contributes to the exploration of enthalpy diagrams by setting up a corrosive resistant isothermal flow calorimeter. The measurement principle is illustrated in Fig. 1: Two liquid flows of temperature T_{in} are mixed in a corrosion resistant reactor. A thermoelectric temperature control unit consisting of resistance heaters and a Peltier cooler allows temperature adjustment of the reactor and the mixed liquid at T_{out} . In case of

With this setup the knowledge of the enthalpy values for highly concentrated corrosive binary solutions can be expanded.

Funding: Graduate and Research School Efficient use of Energy Stuttgart (GREES)

Biomimetic Micro- and Nanostructured Anti-Ice Polyurethane Films by Hot Embossing and PECVD-Coating Contact: Philipp Grimmer

Functional, superhydrophobic anti-ice surfaces can reduce cost, energy and use of chemicals compared to stateof-the-art de-icing methods for the transportation or industry sectors. Polyurethane films are used as substrates, being suitable for outdoor use because of their high stability.



Fig. 1: Microstructured/PECVD-coated, superhydrophobic polyurethane surface: a) scanning electron microscopy, b) confocal laser scanning microscopy, c) light microscopy, d) confocal fluorescence microscopy.

Hot embossing is done with metal stamps, which are differently microstructured by laser ablation. Then a PECVD process creates a hydrophobic fluorocarbon-like or silicone-like polymer thin film. The thermal behavior of the PU was analyzed by DSC and IRspectroscopy. The microstructures were characterized by several microscopy methods (incl. SEM), and the plasma polymer films by ellipsometry, AFM and ESCA. The water wetting behavior was analyzed by dynamic contact angle measurements. 25 μ L water drops were frozen on the surfaces with a constant cooling rate. The temperature of the drop was continuously recorded by an IR-camera. The nucleation/crystallization temperatures and duration times were determined automatically with ImageJ. Very low nucleation temperatures were measured on the best "anti-ice" surfaces.



Fig. 2: Testing set-up with freezing chamber and IR-camera for characterization of freezing behavior on anti-ice surfaces.

Funding: Graduate and Research School Efficient use of Energy Stuttgart (GREES)

Surface Modification of Hollow Fiber Membranes by a Continuous Dip-coating Process Contact: Isabel Jesswein

For the separation of substances membranes will play an increasingly important role due to their inherent energy efficiency. In contrast to other techniques the separation of materials by membranes is generally carried out without a phase transition.



Fig. 1: Continous dip-coated polyethersulfone (PES) hollow fiber membrane with a hydrophilic coating (membrane: blue; coating: green).

To enhance the potential of membranes one method is to modify the membrane surface with a functional coating material. Prevailingly, these coatings influence the separation performance positively and additionally improve further properties, i.e. antifouling behavior or chemical resistance.

Surface modification of hollow fiber membranes can be performed through a dip-coating procedure. This coating technique can be implemented into the manufacturing process of the membrane itself as an additional treatment directly after the production of the hollow fiber.

So far only simple empirical models exist, to describe the dip coating pocedure of fibers. Therefore further investigations regarding additional influencing factors and the extension of these theories are in process. With this knowledge coating-systems for humidification / dehumidification membranes and adsorber membranes will be developed and the performance of these systems will be evaluated.

Funding: Scholarship of the Landesgraduiertenförderung Baden-Württemberg, University of Stuttgart

Preparation and Characterization of Surfaces for a Coating with Click-Modified Extracellular Matrix Contact: Silke Keller

Biomaterials which are commonly used in medical applications have to meet certain mechanical requirements and have to be compatible with the human body.



Fig. 1: Generation of azide-modified extracellular matrix (ECM) via metabolic oligosaccharide engineering (MOE) and the subsequent coating of either cell culture surfaces or medical implant surfaces via click chemistry ([3+2] Huisgen cycloaddition)

To enhance this biocompatibility, biomaterials are often coated with biomolecules found in the extracellular matrix (ECM). The ECM can be considered as the natural environment of cells in a natural tissue. Due to its high biological activity, the ECM is able to promote cell adhesion, proliferation, and differentiation. These properties make the ECM exceedingly interesting as a biocompatible and bioactive surface coating. However, a major limitation of conventional FCM coatings is the lack of mechanical stability since the adhesion of the biomolecules is only based on physisorption. To overcome this limitation, Mara Ruff, a former PhD-student, developed a "clickable" ECM (clickECM) by performing metabolic oligosaccharide engineering on fibroblasts to introduce azide groups into the ECM (Fig.1). This clickECM can be covalently immobilized on likewise click-functionalized surfaces via copper-free click chemistry. The functionalization with click groups requires an activation of the surfaces which was done using wet chemistry. In my work I developed an alternative protocol using plasma technology and was able to show that this even more practical technology is also well suited to generate stable ECM coatings.

Collaboration: V. Wittmann and D. Wieland, University of Konstanz

Funding: The "clickECM" – project is supported by contract research (Glycobiology/Glycomics, Grant number: P-BWS-Glyko/09) of the Baden-Württemberg Stiftung as well as Fraunhofer Internal Programs (Discover, Grant number: Discover 828 355). Furthermore, I kindly thank the Peter and Traudl Engelhorn Foundation for rewarding me with their PhD-Scholarship.

Chitosan-based Nanoparticles for Wastewater Remediation Contact: Benjamin Riegger

To sustain the water quality at a high level there are different techniques available to eliminate pollutants. Next to membrane processes, sedimentation and oxidation treatment there is waste/ water treatment by adsorption techniques. Due to the easy design and the simple handling adsorption techniques especially nanoparticle-based adsorbers are eminently suitable for the removal of the stated pollutants. It is evident that there is a need for cheap, easy to manufacture, long - term stable and recyclable adsorbents. Hence this work shows the effort - not only to achieve the mentioned challenges - but also to prepare the desired nano-particular adsorbent based on the renewable polysaccharide chitosan.

and emulsified in oil using a suitable surfactant (Fig. 1). Different crosslinkers have been used to synthesize Chitosan nanoparticles in the range of 50–200 nm (Fig. 2)



Fig. 2: SEM-picture of prepared chitosan nanoparticles. Lower left corner: raw low molecular weight Chitosan powder.



Fig. 1: Example of miniemulsion preparation via ultrasonication

To ensure longterm stability of the material the biopolymer is cross-linked. Chitosan nanoparticles were synthesized via miniemulsion crosslinking technique. To achieve a stable emulsion chitosan is dissolved in aqueous media

*click*ECM – A New Approach to Covalently Immobilize Human ECM on Implant Surfaces Contact: Mara Ruff

Click chemistry provides extremely specific reactions which proceed rapidly with high efficiency, high selectivity, and high reaction yields. Many applications have evolved in a broad range of scientific fields. As part of a project a new strategy was developed to covalently immobilize human extracellular matrix (ECM) on artificial surfaces using copper-free click chemistry. In a first step, a click-modified ECM (named clickECM) was generated by adding azide-functionalized sugar analogues to the media of human cell cultures. These sugar analogues were subsequently incorporated into the extracellular glycans of the cells via the natural metabolic pathway (metabolic oligosaccharide engineering). After isolating the extracellular structures, this matrix was covalently immobilized onto substrates, which have been equipped with the complementary function.

This resulting *click*ECM-coating was significantly more stable compared to a conventional physisorbed coating because of the covalent bonding beetwen the artificial surface and the biological material. Therefore, we assume that *click*ECM could be a promising material for stable coatings of artificial materials in order to enhance cell adhesion and growth. In the future, this technology could be used for example on implant materials like titanium to speed up implant ingrowth.

Collaboration: Fraunhofer IGB; V. Wittmann, Universität Konstanz

Funding: The "*click*ECM" – project is supported by contract research (Glycobiology/Glycomics, Grant number: P-BWS-Glyko/09) of the Baden-Württemberg Stiftung as well as Fraunhofer Internal Programs (Discover, Grant number: Discover 828 335)



Fig. 1: *click*ECM was produced by incorporating azide groups (stained in green) into the extracellular structures of fibroblast cell cultures (nuclei stained in blue). After isolating this extracellular matrix, a distinct network of azide-modified fibers can be detected. The absence of green staining in fibroblast cultures without azide-modification proves the specifity of the staining.

Processes that are dominated by interfacial interactions often require large surface areas. Porous polymers as a subgroup of porous materials, provide the advantage of easy manufacturing and handling compared to crosslinked particle layers. Open cell foams are interesting materials for a variety of applications such as adsorber materials, support materials for chemo- and biocatalsis or as 3-D-carriers for cells and microorganisms.



Fig. 1: Scanning electron microscopic picture of the formed porous structure by placing two drops of the reactive compounds ontop of each other.

Furthermore, porous polymers are of interest in the general industry used as foams, e.g. car roofs, matresses or as light weight materials. In many of the above mentioned applications spatially resolved structures are of high interest as most porous materials are simply manufactured in bulk processes. Hence, micro-scale polyurethane foams could lead as a first step into the direction of digital fabrication of printed foams. It was shown that the approach of using full reactive inkjet printing is promising towards production of micro-scale PU-foams. As seen in Fig. 1 two drops of the reactive components form a porous structure after being deposited ontop of each other. Yet, it is still under investigation how the conditions have to be to assure complete intermixing of the layered drops.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB Funding: PhD-Scholarship by the Friedrich-Ebert-Stiftung e.V.

Tribology System for Cold Forming Process Based on Volatile Lubricants and Laser Structured Surfaces Contact: Georg Umlauf

In the light of technical, economic and ecological developments the usage on lubricants during the metal forming should reduce. This basic research project aims to realize a system for deformation of metal sheet without mineral oil based lubricant. The ambitious approach is to integrate volatile liquid CO_2 as an interfacial medium, with a laser-structured surface. The liquid medium is injected directly into the clearance during metal forming process. Several arrays of micro-holes ought to transfer the liquid medium into the clearance.



Fig. 1: A 60 bar CO_2 spout through a microhole (diameter d = 140 μ m) againt atmosphere pressure.

Into the contact area between workpiece and tool surface the liquid CO_2 can expands and remodels into solid dry ice and the gas phase.

During the forming operation under high pressure CO₂ acts as lubricant. After the metal forming CO, evaporates completely into the gas phase and a dry formed metal sheet without any clearing steps is obtained. Further treatments can start immediately. An autoclave is used to observe the wetting behaviour of liquid CO, on metal surfaces with different additional layers and microstructures. For the optimization of the surface energy we used low-pressure plasma modified layers made of fluorocarbon, SiC⁻ or AlO_a. All this improve the wetting of metal surface and reduce the friction coefficient during the metal forming.

Collaboration: E. Zahedi; Institut für Strahlwerkzeuge (IFSW), University of Stuttgart; C. Wörz, Christoph; Institute for Metal Forming Technology, University of Stuttgart

Funding: Deutsche Forschungsgemeinschaft (DFG) SPP 1676: Dry Metal Forming - Sustainable Production through Dry Processing in Metal Forming

Carbohydrate-based Polymers for the Encapsulation by Spray-Drying Contact: Michael Walz

Microparticles in the environment origi-
nating from industry and consumer
products, e.g. cosmetic products made
of non-degradable polymers such as
polyethylene are a pressing issue.of the particles should be degradable.
We investigate the usage of carbo-
hydrates from renewable resources
as encapsulation material for the
spray-drying technology, to show pos-





Fig. 1: Comparison of spray-dried particles from inulin containing dexpanthenol after incubation in aqueous solution – (A) native inulin, (B) modified inulin.

The organisms in the aquatic environment are able to ingest and incorporate those non-degradable particles containing pollutants adsorbed on their surface. In order to prevent bioaccumulations in the food chain, the material

of the particles should be degradable. We investigate the usage of carbohydrates from renewable resources as encapsulation material for the spray-drying technology, to show possibilities and applications to substitute non-degradable polymers as capsules material for cosmetic, agriculture and food industry. Therefore industrial established carbohydrates such as inulin, xanthan, cellulose and derivatives are investigated for the spray-drying method by using design of experiment.

In order to adjust specific properties, the biopolymers are chemically modified, e.g. to prevent a burst release of the encapsulated substance or to create a functional surface for further functionalization. Representative hydrophilic and hydrophobic low molecular substances are used for the encapsulation and investigation of release behavior like dexpanthenol, ascorbic acid or limonene.

Collaboration: Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB Funding: PhD Scholarship of the Konrad Adenauer Foundation

Development of Bioinks for Bioprinting of Vascularized Bone Tissue Equivalents Contact: Annika Wenz

Tissue engineering via bioprinting is a promising tool for the fabrication of tissue equivalents using living cells and biomaterials – not only for the use as implants, but also as test systems in the pharmacological and chemical industry.



Fig. 1: Osteogenically differentiated stem cells in a hydrogel containing hydroxyapatite. The bone-specic protein osteopontin (red) and the cells' DNA (blue) are stained.

This project aims to develop a printing process as well as suitable bioinks on the basis of methacrylated gelatin (GM) for the printing of vascularized bone tissue equivalents.

The introduction of components like glycosaminoglycans and hydroxyapatite provides cues that not only support, but also induce the osteogenic differentiation of stem cells encapsulated in GM-hydrogels, what can be shown by the analysis of the formed bone matrix,

e.g. the expression of the bone-associated protein osteopontin (Fig. 1).

For the vasculogenesis in the hydrogels, a process in which blood vessel-like structures are formed via self-assembly of endothelial cells, a GM-based bioink was developed which possesses suitable mechanical properties for supporting the cells. The resulting vessel structures in the gels were detected by specific immunofluorescence stainings (Fig. 2).



Fig. 2: Vessel structures in GM-based bioinks. The EC-surface marker CD31 (white) and the DNA (blue) are stained.

Funding: Doctoral scholarship of the Carl Zeiss Stiftung



Interfacial Processes

Interfaces often play an important 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 look at the respective process in its entirety. 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 the standard fermenter but in addition also self-developed reactor systems as flat-panel airlift reactor (developed at Fraunhofer IGB) and 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 bio-based future are developed. For new products an effective downstream processing is also necessary. For example microalgae contain a broad range of ingredients that could 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 to get the products efficiently out of the biomass.

Contact

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Investigations and Technical Development of "Adsorption Thermal Energy Storage Systems" with Simulation and Different Control Strategies Contact: Mazen Abou Elfadil

Thermal energy storage (TES) has been receiving an increasing worldwide attention, especially with the growing concerns about environmental problems caused by an inefficient utilization of energy.

system components and simulating the overall system will be carried out, by taking into consideration the possibility of combining different reactors, heat pumps or heat storage units.

In this investigation, a thermo-chemical method with a reversible adsorption/ desorption process will be used as a key component for a fully controlled integrated system. The principle of the adsorption storage system is based on a gaseous working fluid (e.g. water) which gets adsorbed by a highly porous material (e.g. zeolite). This adsorption process is an exothermal one, thus heat is being released and can be extracted and used. In order to recharge the heat storage system, desorption of the working fluid is done by heating the porous material.

However the technology of the adsorption TES is still under development. therefore more investigations are still required in the following fields: increasing the storage efficiency, reducing the recharging temperature and releasing the stored heat at a constant temperature. Hence the aim of this thesis is to enable these improvements by providing operation algorithms, by which the system will be able to achieve an optimal operational status in terms of energy utilization and time efficiency. In addition modeling of the

Development of a Controllable Fermentation Process for Biosurfactant Production by Pseudozyma Strains Based on Lignocellulose Substrates Contact: Paula Carrillo-Riveros

Biosurfactants are amphiphilic activesurface biomolecules that have unique properties such as low toxicity, biodegradability and structural diversity. One aphidis for the synthesis of microbial of the most promising glycolipid biosurfactants currently known are manno- substrates that can be transferred to svlervthritol lipids. Mannosvlervthritol lipids (MELs) are mainly synthetized in high amounts by *Pseudozyma* sp. They can be produced on a variety of renewable substrates and be tailored in their surfactant performance. The production of MEL at higher scale has not yet been fully investigated and an optimization of the production process is necessary to achieve market penetration. Thus, to compete with large scale synthetic surfactants, efforts need to be centered on the use of cheap substrates such as renewable resources, optimal media and operation conditions.

The present project has as main objective to develop a fermentation process using the strain Pseudozyma biosurfactants based on lignocellulose pilot and production scale. To achieve this, the process will be enhanced by using different fermentation strategies, evaluating the expression level of a targeted gene responsible for MEL production and developing a model that best fits the experimental data.

Collaboration: S. Zibek, Fraunhofer IGB: C. Svldatk, Karlsruhe Institute of Technology (KIT), BBW ForWerts Graduate Program, promotional reference 85 A Funding: BBW ForWerts Graduate Program promotional reference 7533-10-5-85 B



Fig. 1: Fermentation process of mannosylerythritol lipids (MELs) based on renewable substrates.

Extraction Processes for the Cascade Utilization of Microalgae Biomass for Food and Feed Applications Contact: Felix Derwenskus

Microalgae contain a broad range of ingredients that could potentially be used in the food and feed sector. 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 (like eicosapentaenoic acid, EPA) as well as different micronutrients like carotenoids and phytosterols. Due to the broad range of valuable products and specific cell wall properties of different microalgae species, elaborated down- stream processing is necessary to disrupt and fractionate high quality nutrients.

Within the frame of the project three different microalgae species will be investigated. The basic idea is to fractionate different types of microalgae biomass as gentle and selective as possible. By cascaded extraction of residual biomass, up to three different fractions containing proteins, lipids, polar membrane-lipids and carotenoids will be obtained. Subsequently, the nutritional quality and techno-functional properties of each fraction will be analyzed by our project partners within the Bioeconomy Research Program Baden-Württemberg.

Collaboration: Fraunhofer IGB Funding: Ministry of Science, Research and the Arts Baden-Württemberg, promotional reference Az. 33-7533-10-5/93



Fig. 1: Fatty acid content and protein content of Chlorella vulgaris, Nannochloropsis oceanica and Phaeodactylum tricornutum

Characterization of Microbial Dynamics in Anaerobic Biogas Production Systems Contact: Anja Grohmann

The anaerobic digestion process during biogas production is very complex and therefore often a "black box" for operators. Many biogas plants do not run in an optimal way, as interferences at an early stage are currently not well defined and controllable.

There is little knowledge about the dynamics of microbial populations during the digestion at different process stages and between varying operating conditions. However, the identification of potential key players in the process dynamics and a better understanding of the role of different groups of microorganisms represent the basis for a targeted intervention. Therefore the diversity of sludge samples from diverse biogas plants will be investigated using next generation sequencing and are correlated with analytical and operating parameters.

The bacterial class Clostridia is already known to be highly abundant in anaerobic sludge and to have an exceptional substrate and metabolite diversity. As Clostridia could play a key role in anaerobic digestion, the focus of this PhD thesis will be on these microorganisms. Species with a defined substrate spectrum will be isolated from sludge of biogas plants and compared with respect to their metabolic potential by growth characteristics and investigations on different molecular levels. In addition of an improved knowledge about biogas production, species of this class could offer further applications in biotechnological processes.

Collaboration: R. Rabus, Institute for Chemistry and Biology of the Marine Environment (ICBM), Carl von Ossietzky Universität Oldenburg

Process Development for the Chemo-enzymatic Epoxidation of Plant Oils and Fatty Acid Derivatives Contact: Fabian Haitz

Epoxidized triacylglycerides (ETAG) are valuable precursors in oleochemistry and can be utilized as polymer building blocks or plasticizers. ETAG are produced via the Prileschajew epoxidation reaction, in which a peracid is used for the oxygen transfer to the double bonds. The peracid is formed in situ from hydrogen peroxide and e.g. acetic acid using a strong mineral acid as catalyst. The strong acid is considered to cause undesired ring opening. Biotechnology facilitates novel and more selective approaches for ETAG synthesis. One opportunity is the use of result we selected suitable criteria for a enzymes that catalyze the formation of peracids (Fig. 1).

Starting with different lipases we investigated their potential to catalyze the epoxidation and selected suitable enzymes.

We optimized the process for different plant oils using an immobilized lipase and obtained nearly complete conversion. The obtained epoxidized products were analyzed with regard to their chemical and physical properties. An important aspect that has been addressed during our work was the process stability of the biocatalyst. An adapted protocol was evaluated maintaining the enzyme activity over consecutive reaction cycles. One step further we focused on scale-up of the three-phasic reaction system. As a scale-up.

Collaboration: S. Zibek, Eraunhofer IGB



m = 5 - 9; n = 0 - 3; o = 0 - 4; R = un-/saturated alkyl chain R' = saturated/epoxidized alkyl chain

Fig. 1: Chemo-enzymatic epoxidation of fatty acid derivatives (Rüsch gen. Klaas & Warwel, 1999).

Influence of Different Light Conditions on the Growth of Chlorella sorokiniana Contact: Claudia Holdmann

Microalgae are a future source of high value products and platform chemicals. Advantages compared to terrestrial plants are a higher biomass productivity, the use of non-arable land and a lower water consumption.

Light availability is the most important parameter for the growth rate of microalgae. The flat-panel airlift reactor (developed at Fraunhofer IGB) has a unique shape which allows to distribute the light on the surface among all cells in the culture. In laboratory tests usually 24 h illumination is used, but for cost effectiveness of the production outdoor cultivation is the best choice. The big challenges during outdoor cultivation are the changing light and temperature conditions between the seasons, but also during one day.

The influence of different light conditions on the cultivation of the microalgae Chlorella sorokiniana is investigated, in outdoor cultivation and in laboratory with different periods of illumination. Thus the optimal growth conditions for biomass production can be determined and a contribution to the future of the cost effective algae cultivation can be made.





Collaboration: Fraunhofer IGB Funding: Fraunhofer IGB, Fachagentur Nachwachsende Rohstoffe e.V. (FNR) promotional reference 22403211 and 11EKF032: Subitec GmbH; Südzucker AG



Fig. 1: Laboratory plants with 28-liter flat panel airlift reactors

Interfacial Processes

Study on the Ion Mobility of Rare Earth Metals in the Electric Field of the Free Flow Electrophoresis Contact: Lea König

Recycling of metals is getting more and more important. Within recycling and beneficiation processes the separation of metal ions is one of the most challenging process steps as metals for high-tech applications are often present as components of complex mixtures. With the state of the art methods separation of rare earths in high purities is very complex and cost intensive. Free flow electrophoresis using different mobility of ions in an electric field is a promising alternative to established separation processes.



Fig. 1: Free flow electrophoresis prototype.

The separation of the dissolved rare earth metals by an electric field in the free flow electrophoresis is based on the difference in the elements' electrophoretic mobility. This difference increases with the complexation of the metals. Our working group has developed a model linking the net charge of complexed rare metals, physical and chemical test parameters and the mobility. With this model it is possible to predict the mobility of dissolved ions, to untersand the separation process and optimize the down-stream process. High yields of separating metal ions have been obtained by free flow electrophoresis separation and purity has been improved resulting in up to 100% pure metal fractions by using organic acids as complexing agents.

Collaboration: Fraunhofer IGB

Development of a Bioreactor for the Conversion of Biogas Contact: Ilka Mühlemeier

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.



methane produced by biogas plants appear to be a location-independent and cheap alternative carbon source for variety of industrial and pharmaceuticals 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.

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 Euro per kilogram) in comparison with sugar derived from natural gas (0.19 Euro per kilogram),

Fig. 1: Aerobic, methanotrophic bacteria with the first oxidation step of methane to methanol.

Interfacial Processes

Organosolv Fractionation of Beech Wood at Pilot Scale Contact: Marlen Verges

In order to achieve a complete material use and high valorization of lignocellulosic feedstock following the biorefinery priciple, a fractionation process into its main polymer components cellulose, lignin and hemicellulose is required. At the Fraunhofer Center for Chemical-Biotechnological Processes CBP in Leuna a pilot plant designed for the ethanol-water organosolv fractionation of beech wood was succesfully established. 70 kg of industrial beech wood chips can be fractionated in a 460 L batch digester using temperatures up to 200° C (Fig.1). Delignification and hemicellulose extraction kinetics are investigated for different fractionation temperatures the first time

at this scale using and charges of sulfuric acid as a catalyst.

Products obtained under various process conditions are characterized and used by project partners for the development of new appilcations. Enzymatic hydrolysis of the fiber fraction in order to generate monomeric sugars for fermentation processes has been investigated but also cellulose applications are promising. The overall aim is to optimize the process with regard to product quantities and qualities and to obtain a deeper understanding of organosoly fractionation mechanisms.



Fig. 1: Process scheme of the organosolv pilot plant in Leuna



NanoBioMater

The Team Functional Polymers and Hydrogels, headed by Dr. Alexander Southan, links the Institute of Interfacial Process Engineering and Plasma Technology IGVP and the Institute of Organic Chemistry. In the team, the entire development process leading to synthetic functional polymers and spatially defined hydrogels is covered. The development process comprises development of functional polymers and cross-linkers, the development of new cross-linking chemistry, the formulation of hydrogels for 3-D printing and the processing of spatially defined hydrogels by 3-D printing.

The goal of functional polymer and cross-linker development is to obtain biocompatible and biofunctional components for hydrogel formulations. Due to their excellent biocompatibility, we focus on the development of polymers which have a poly(ethylene glycol) backbone and which carry functional groups suitable for cross-linking. The polymer development deals with the synthesis and characterization of monomers as well as the resulting polymers. Additionally, we develop cross-linkers with functional groups which are complementary to the polymer-bound functional groups. The reaction of the cross-linkers and polymers results in hydrogels. Polymer and cross-linker development also is aimed at the development of systems suitable for processing of hydrogels by 3-D printing.

Contact

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NanoBioMaster

Surface Functionalization of Superporous Hydrogels for Additive Manufacturing Contact: Karishma Adatia

The aim of this work is to develop surface functionalized, microstructured hydrogels for additive manufacturing via polymer engineering. The research focuses on the surface functionalization of hydrogel pores with defined molecular anchor points.

Furthermore, a hydrogel microstructuring procedure which is compatible with additive manufacturing processes is developed. Additive manufacturing opens the possibility to print spatially resolved 3-D materials. The combination of the two work packages should lead to a surface functionalized 3-D microstructured hydrogel 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.



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

In order to be able to form a 3-D microstructured hydrogel, 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.

Hydrogels with Specific Charge Densities Contact: Tobias Götz

Hydrogels with tailor-made properties are intersting materials for different applications. Starting material for this special type of hydrogel is polyethylene glycol, which is modified with different moieties of allylic side chains. Via thiol-ene click reactions the charge carriers and the functional groups for crosslinking are coupled to the polymer side chains. To prepare hydrogels one possibility for crosslinking is given by introducing thiols to prepare hydrogel networks with michael-addtion type crosslinker. For future applications – like 3-D printing – the thiols are equipped with photoprotection groups to trigger the crosslinking with UV radiation. To investigate the mechanical properties and the biological activity of the hydrogel due to the charge density the adjustment of the charge density can be controlled by the amount of functional side chains attached to the polymer backbone.



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

Funding: Baden-Württemberg Stiftung



Plasma Technology

The division plasma technology works on the development of new microwavegenerated plasma sources at low pressure and atmospheric pressure, the characterisation 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 utilised 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 characterisations of insulating layers, O_2 - and H_2O -permeation barriers, or scratch protection layers, and the sterilisation 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. It 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.

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Numerical Modelling of a Microwave Plasma Torch Contact: Sandra Gaiser, Andreas Schulz, Matthias Walker

To study a microwave plasma torch at atmospheric pressure simulations were carried out by using the simulation software COMSOL Multiphysics[®].

In a first step a model was set up to describe the electromagnetic properties of the plasma by the Drude theory. It showed the influence of the electron density on the electric field. Next, fluid equations and a set of reaction mechanisms were added to the Drude model in order to describe an argon plasma.



Fig. 1: Electron density profile at 1 mbar (left) and at atmospheric pressure (right).

At a pressure of 1 mbar diffusion of particles through the volume and a recombination only at the walls were enabled. This resulted in an electron density distribution (Fig. 1, left side) with a maximum of 1.5.1019 m-3 in the center of the plasma and a decreasing density towards the walls. Compared to that the plasma at atmospheric conditions (Fig. 1, right side) with densities in the range of 10²⁰ m⁻³ sticks to the wall on the left which is facing the incoming microwave. The reason for this is that for atmospheric pressure also recombination processes via collisions of three particles in the volume had to be considered. This leads to a decrease of the electron density in the center. Since the microwave cannot enter the plasma area due to the increased density on the left wall, a further ionization is prevented.

Collaboration: Eight project partners from Germany in the AiF/DFG cluster project OGAPLAS Funding: German Research Foundation (DFG)

High Rate PECVD of Silicon Oxide and Zinc Oxide Coatings on Polycarbonate Contact: Stefan Merli

In this work, a plasma enhanced chemical vapor deposition (PECVD) process of silicon oxide (SiO_x) and zinc oxide (ZnO) coatings on polycarbonate (PC) was investigated. The aim was to protect the PC surface against mechanical damage and photodegradation due to UV-light during outdoor exposure with a weathering-resistant layer system. The SiO_x-coating acts as a scratch protection and the ZnO-coating as a UV-absorber.



Fig. 1: SEM-image of the combined SiOx and ZnO multilayer system

The coatings were deposited with the help of a plasma from a mixture of O_2 and hexamethyldisiloxane and diethylzinc, respectively. The plasma was generated with a microwave plasma source, the so called Duo-Plasmaline, which provides a high conversion efficiency of the used gases and which

is, due to its high scalability, also applicable on large scales.

High deposition rates of up to 60 μ m/ min for SiO_x and 700 nm/min for ZnO could be achieved, which is important for a low processing time and a high profitability. A combined layer system with SiO_x and ZnO (Fig.1) was developed, showing a good adhesion on the PC, a high transparency and a high absorption in the UV region. With taber-abrasion- and accelerated weathering tests it could be shown, that the coating system had a higher scratch resistance and an improved UVprotection, compared to commercial lacquers.

Collaboration: Bayer MaterialScience (Covestro); Muegge GmbH; ReuterTechnologie GmbH Funding: German Federation Ministry of Education and Research (BMBF) promotional/reference : 13N10523 **Plasma Technology**

Spectroscopic Study of a DBD-Discharge for Waste Gas Treatment

Contact: Andreas Schulz, Stefan Merli, Matthias Walker

Biological sources of air pollution like animal farms, food processing, sewage and waste handling industries are of increasing concern as the risks posed by the pollutants from these sources are understood more profoundly. The current method investigates the possibility of using a low temperature plasma to treat pollutant gases. Multiple parallel powered steel electrodes are separated with ceramic plates to create a dielectric barrier discharge, a picture detail is shown in (Fig.1).



Fig. 1: View into the discharge gap of the DBD

Spectroscopic investigations show that the blue-violet light emission origins only from one electronic transition of the N₂ molecule. In Fig. 2 the relative calibrated spectrum is shown. A view into literature reveals the 2nd. positive system as the rotational-vibrational band from the state $C^3\pi_u$ to $B^3\pi_g^{[1]}$. Mainly the vibrational transition from $\Delta\nu$ =+3 produces the plasma light. In Fig. 2 the band head ν '=0 to ν "=3 is indicated. The more intensive bands are in the UV region.

The reason why only the 2nd positive system can be observed is that due to the very short lifetime of 3.8·10⁻⁸ s the C³π_u state has simply the change to relax radiatively^[2]. All other excited states relax by collisions with other molecules, for example to generate oxygen radicals by the reaction N₂(B³π_g) + O₂ \longrightarrow N₂(X¹Σ⁺_g) + 2 O· which is then followed by the ozone reaction O· + O₂ + M \longrightarrow O₃* + M \longrightarrow O₃ + M.



Fig. 2: Spectrum of a DBD air discharge.

 ^[1]R.W.B. Pearse and A.G. Gaydon, London, New York, 1976.
 ^[2]Alf Lofthus and Paul H. Krupenie, J. Phys. Chern. Ref. Dato, Vol. 6, No. 1, 1977

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

Sterilisation with a DBD Plasma in Complex Structures Contact: Andreas Schulz, Stefan Merli, Matthias Walker

The primary causes for the spoilage of food are microorganisms, which are inserted by raw materials or by contamination in the handling process into the product. At present, different possibilities exist to reduce the germ load of spices effectively. However, these procedures are connected with substantial disadvantages like the loss of colour and ethereal oil ingredient.



Fig. 1: SEM of a cellulose test stripe

The dielectric barrier discharge (DBD) has already prooved a sufficient sterilisation at atmospheric pressure and at a low process temperature on smooth and closed surfaces. However, spices, herbs and ingredients show highly complex surface structures. An adequate test system for standard sterilisation processes are cellulose test stripes inoculated with a test germ combination of *Bacillus atrophaeus* (*Bacillus subtilis* var. *niger*, ATCC 9372) with an average of 10⁶ species and *Bacillus stearothermophilus* (ATCC 7953) with an average of 10⁵ species. Due to the complex structure shown in (Fig.1) they also represent a test for plasma sterilisation processes.

In Fig. 2 the sterilisation kinetics of the DBD in air is shown. Within 120 s the detection limit of 10 spores is reached. Although the major part of the germ is inside of the cellulose mesh shown in (Fig.1) the total inactivation can be reached without any indication on damages of the fibres. This indicates on a good penetration capability of the DBD plasma sterilisation mechanisms.



Fig. 2: Sterilisation kinetics with the DBD.

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

Plasma Technology

CO₂-Based Test for the Detection of Defects in Oxygen Barrier Layers

permeates at first through the defects

in the barrier layer, causing the follow-

 $Ca(OH)_{2} + CO_{2}(\uparrow) \longrightarrow CaCO_{3}(\downarrow) + H_{2}O;$

Calcium carbonate forms tiny crystals

found and investigated at the scanning

electron microscope by means of mark-

Preliminary tests show how good bar-

rier layers (O_2 /HMDSN = 40:1) possess average defect densities more than 30

times lower than poor barrier layers

 $(O_{A}/HMDSN = 10:1)$; such result is in

agreement with their previously calculated barrier improvement factor (wrt the uncoated substrate), equal respectively to 40.9 and 1.1. Likewise, the later, slower appearance of defects in good barrier layers mirrors the longer span of their oxygen permeation curves.

on the defects, which can later be

ers placed on the sample itself.

ing reaction to take place:

Contact: Mariagrazia Troia, Andreas Schulz, Matthias Walker

Punctual micro- and sub-micro-metrical defects in barrier layers for OLEDs encapsulation worsen their performances, leading to a reduction of lifetime and eventual failure.



Fig. 1: Barrier defects at the optical microscope (top left) and at the scanning electron microscope (top right and bottom)

In order to determine the density of defects in barrier layers, their origin and effect on the overall oxygen transmission rate, a new test for their detection has been developed and optimized: a polyethylene terephthalate foil, coated with the barrier to be tested, is interposed between a pure CO_2 atmosphere and a saturated limewater solution: CO_2 **Plasma Technology**

Printing of Light Emitting Devices on Conducting Textiles (POLEOT) Contact: Mariagrazia Troia, Andreas Schulz, Matthias Walker

Active layers and electrodes in OLEDs are quickly degraded by oxygen and water vapor, thus requiring the highest degree of protection amongst electronic devices. Depositions of interposed barrier and organic layers were performed by means of an Electron Cyclotron Resonance (ECR) lowpressure plasma. Hexamethyldisilazane (HMDSN) was employed as precursor, and mixed with O_2 and H_2 . Gas feed and film thickness have been studied and optimized.

H₂/HMDSN feed resulted in fairly organic films with high retention of the original chemical composition, ensuring a good protection for the OLEDs against the oxygen and radicals of subsequent depositions. Both feeds resulted in transparent and colorless films for all the investigated ratios. Preliminary tests on prototypes showed the OLEDs to survive the process with an increase in lifetime of more than 2 orders of magnitude (from less than few minutes up to 40 h).



Fig. 1: OxygenTransmission Rate for O_z/HMDSN-based films (left) and OLED prototypes before and after encapsulation (right).

Good barrier properties are obtained for O_2 /HMDSN ≥ 25 , which results in inorganic, silica-like films with an improvement factor (with regard to the uncoated PET foil acting as substrate) up to \cong 50 for a mixture ratio of 40:1. Transmission rate vs. thickness showed good film stability and bendability in an extended range (from \cong 40 to 370 nm), superior to previous O_2 /HMDSObased layers.

Collaboration: Five project partners from Germany and Belgium

Funding: German Federation of Industrial Research Associations (AiF) / project Collective Research Networking (CORNET)

Plasma Dynamics and Diagnostics

Plasma Dynamics and Diagnostics

Fundamental studies of waves in and confinement of magnetized plasmas are carried out at the torsatron TJ-K and the tokamak ASDEX Upgrade at the Max-Planck-Institut für Plasmaphysik (Max Planck Institute for Plasma Physics) in Garching. Furthermore, plasma diagnostics such as laser-induced fluorescence and probes are developed for the application in fusion and low-temperature plasmas.

The torsatron TJ-K at IGVP is a small fusion experiment operated with a low-temperature plasma. The reduced temperature enables probe measurements inside the core of a toroidally confined plasma with high temporal and spatial resolution. At the same time, the dimensionless parameters, which govern plasma turbulence, are similar to those in the edge region of fusion plasmas. Therefore, the experiments conducted at IGVP are relevant for fusion plasmas, too.

Turbulent transport and confinement are key issues in fusion research. Confinement studies at IGVP concentrate on understanding the microscopic properties of turbulent transport. Multi-probe arrays have been developed to measure the fluctuations of the plasma parameter in two-dimensional planes with high temporal resolution. The experimental results are compared with high-level turbulence codes, where the probe arrays are realistically simulated.

The investigation of plasma waves is focused on the propagation and the absorption of the electron-cyclotron wave as well as on Alfvén waves, which are either externally excited or detected as an element of the parallel dynamics of drift-wave turbulence. Electron-cyclotron-resonance heating is one of the major heating schemes for fusion plasmas (see Microwave Technology).

Contact

Dr. Mirko Ramisch Phone +49 711 685-62194 mirko.ramisch@igvp.uni-stuttgart.de

The 3-D Structure of Blob Filaments Contact: Stephen Garland, Mirko Ramisch

The 3-D structure of blob filaments has been investigated in TJ-K using simultaneous Langmuir probe measurements at two toroidal positions, separated by 60°, using a 2-D-scanning probe and a 64-probe matrix. The ion saturation current ($I_{i,sat}$) measurements were conditionally averaged with a common reference probe signal in order to obtain the 2-D blob dynamics at the two different toroidal locations.

The figure shows a frame from the conditionally averaged $I_{i,sat}$ measurement at port O6, where the blob has been fitted with an ellipse (black). The coordinates of the conditionally averaged blob measured using the probe matrix at port O1 were traced back along the magnetic field lines to port O6 and also fitted with an ellipse (white dashed). The degree to which the two ellipses are aligned indicates the degree to which the blob filament is field-aligned. A detailed analysis of the orientation of the ellipses at 3 positions along the blob trajectory has shown that the filaments are field-aligned close to the last closed flux surface (LCFS) but become misaligned further into the scrape-off layer. In fact, as they propagate the filaments do not deform due to the magnetic shear, rather they retain their original form and propagate in a rigid manner.

Collaboration: G. Fuchert, Max-Planck-Institut für Plasmaphysik, Garching



Fig. 1: Conditionally averaged I_{i,sat} measurements using the 2-D-scanning probe at port O6. The black ellipse is fitted to the blob structure at port O6, whilst the white dashed ellipse is obtained by tracing the coordinates of the blob structure at port O1 along the magnetic field lines to port O6.

Full-Wave Simulation of Electron Cyclotron Emission at TJ-K Contact: Eberhard Holzhauer, Gabriel Sichardt

Microwave radiation emitted from the plasma in the TJ-K stellarator is used to obtain information about thermal and non-thermal electron velocity distributions. To model the radiation with a FDTD code, the radial electron density profile is assumed to be symmetric with Gaussian shape and the strength of the confining magnetic field to be decreasing linearly with major radius R. Here, the diagnostic uses radiation emitted by the gyration of electrons (ECE) at the second harmonic.



Fig. 1: Geometry of poloidal torus cross-section and receiver antenna. The vertical line indicates the position of the emitting sources.

By choosing the appropriate frequency of the receiver the position of emitted radiation over r can be determined. For the example shown, the receiver frequency is 16 GHz, corresponding to a vacuum wavelength of 1.875 cm, which is small compared to the diameter of 35 cm of the poloidal vessel cross section.

In the FDTD code, radiation from gyrating electrons is simulated by oscillators with random phases and amplitudes. Close to the resonant surface the evanescent near-field dominates as can be seen in the figure. The far-field distribution arriving at the receiver antenna is in part determined by reflection of the waves from the metal wall. In the next step, density fluctuations will be included in the code to study their influence on the propagation of the microwaves in the plasma.

Collaboration: A. Köhn, Max-Planck-Institut für Plasmaphysik, Garching

Scattering of Microwaves by Density Structures Contact: Alf Köhn

Electromagnetic waves in the microwave regime are widely used for heating and diagnostic purposes in plasmas. Density fluctuations, in fusion plasmas for example present at the plasma boundary, can lead to a widening of the beam and thereby spoiling heating efficiencies or leading to ambiguous diagnostics results.

in a series of parameter scans. To be statistically relevant, averaging over an ensemble of density profiles and thus full-wave runs is required. Depending on the parameters used, the ensemble size can be as large as 20,000. Excellent agreement is found for the two full-wave codes, yielding the strongest perturbation for density



Fig. 1: Scattering of a microwave beam propagating through a layer of fluctuating density obtained from one full-wave simulation. Solid and dashed white lines correspond to positive and negative density contours with respect to the background density.

Within this collaboration, the perturbing effect of plasma density fluctuations on a propagating microwave beam has been investigated by means of two full-wave codes. The fluctuations were created by a Hasegawa-Wakatani drift-wave turbulence model. The average size of the density structures (the correlation length), the amplitude of the fluctuations and the size of the interaction zone have been varied

structures having a size of approximately half the vacuum wavelength of the injected microwave. When the fluctuation level is increased, an approximately quadratic increase of the beam size is found.

Collaboration: T. Matthew, R. Vann, York Plasma Institute, University of York, Vereinigtes Königreich

Equilibrium Studies with 14 GHz Power Upgrade Contact: Ovalle Muñoz, Alejandro Omar

The 14 GHz heating system installed at TJ-K has been upgraded. In addition to two klystrons that were already installed, a third klystron with a separate transmission line has been installed, upgrading the total output power to approximately 6 kW. A phased array antenna is used, installed at an outer port of TJ-K. The antenna allows to vary the injection angle by sweeping the heating frequency. A variation of the frequency from 13.75 GHz to 14.5 GHz results in an angle variation of -42° to 0°. also increases. Hints for a reduced col-

Parameter studies have been performed to assess the effect of the increased heating power.

The plasma density was found to increase with increasing heating power as shown in the figure. The electron temperature was found to only slightly increase with increasing heating power. Spectra in the visible range have been obtained from a wide-range spectrometer showing an increase in the fraction of secondary ionized particles. Due to the strong increase in plasma density, as compared to the temperature increase, the resulting collisionality lisionality, observed at low neutral gas pressures, will be further investigated.

Collaboration: A. Köhn, Max-Planck-Institut für Plasmaphysik, Garching



Fig. 1: Line-averaged density obtained from interferometer measurements as a function of the injected microwave power for different gases.

Confinement Time Studies Contact: Ahmed Omar Abdelaziz Ali, Eberhard Holzhauer

One method to study confinement times is to modulate the plasma heating power and measure the decay time of an appropriate quantity. To obtain the particle confinement time in TJ-K, the decay time of the microwave interferometer measuring the line-averaged plasma density has been used. As shown in the figure, the confinement time is found to decrease with increasing heating power. This has been confirmed by a particle and energy balance model.

To estimate the energy confinement time, the potential of two diagnostics measuring radiation emitted by the plasma have been explored. On one hand, an optical diode detecting radiation in the visible range has been used. The intensity of the radiation in this range depends both on density and temperature. For the parameters used here, however, agreement with values obtained from the particle and energy balance model could be achieved.

On the other hand, a commercial satellite receiver (LNB) has been used. It detects radiation in the range 11.7–12.75 GHz. Decay times down to 20 μ s have been measured being below the expected energy confinement time (which is in the range of 50–200 μ s). The origin of this radiation is subject to further studies. Possible candidates are cyclotron radiation or bremsstrahlung.

Collaboration: A. Köhn, Max-Planck-Institut für Plasmaphysik, Garching



Fig. 1: Decay time of the line-averaged density indicating the particle confinement time as a function of the injected microwave heating power for varying gases.

The poloidal and radial blob velocity

components of detected blobs were

plotted as a function of ρ_{a} in Fig. 2.

determined and averaged. The result is

A decreasing trend is seen in the V and

V velocities. Whilst the trend is similar,

the absolute values of these velocities

differ from experimental values and as

well as those predicted by an analytical blob model. Understanding of this

discrepancy will be the focus of future

Vy

- Vx

Vy-Vd

Blob Studies using GEMR Simulations of TJ-K Plasmas Contact: Allah Rakha, Stephen Garland, Mirko Ramisch

Simulations of turbulence in TJ-K plasmas have been carried out with the gyrofluid code GEMR. The simulation code includes a scrape-off layer (SOL), which allows for the study of blob dynamics. Global simulations were carried out for three ion species, H, D and He at typical TJ-K densities and temperatures. Blob structures were detected propagating through the SOL with a preferred radial and poloidal direction. Blob velocities were determined from center of mass (CoM) coordinates for simulations approximately 20 ms in length. A snapshot of the simulated density fluctuations is shown in Fig. 1.



 ρ_{s} [Cm] Fig. 2: Radial and poloidal blob velocities (V_x and V_y respectively), as well as the poloidal blob velocity with the ExB flow subtracted (V_y-V_d), as a function of the drift scale, ρ_{x}

work.

800

600

400

200

0

0.4

Blob velocities [ms⁻¹]

Collaboration: P. Manz, Technische Universität München; B. Scott, Max-Planck-Institut für Plasmaphysik, Garching

0.5 0.6 0.7 0.8 0.9 1.0

Fig. 1: A snapshot of denstiy fluctuations for a hydrogen plasma from the GEMR code.

Dependence of Intermittent Density Fluctuations on Collisionality in TJ-K

Contact: Kyle Reuther, Stephen Garland, Mirko Ramisch

Particle and heat transport losses due to edge turbulence are well known phenomena commonly seen in toroidal magnetic confinement devices. Furthermore, in the scrape-off layer (SOL), intermittent events are observed in the turbulent density fluctuations and found to strongly contribute to particle transport to the vessel walls. In the adiabatic limit (small collisionality) of the two-field Hasegawa-Wakatani model, simulated turbulent density fluctuations are observed to couple to potential fluctuations and exhibit Gaussian behavior.

The relationship between collisionality and intermittency is investigated experimentally at the stellarator TJ-K. To vary the plasma collisionality, which is related to electron density and temperature, parameters such as gas type, neutral gas pressure, magnetic field, and heating power are varied. Radial profiles of plasma density (n), temperature (T_i) , floating potential (φ) , and vorticity

 $(\Omega = \partial v - \partial v \text{ where } v = (E \times B)/B^2)$ are recorded via a scanning 7-tip Langmuir probe array (Fig.1).

Collaboration: P. Manz, Technische Universität München



Collisionality Scaling of Zonal Flows Contact: Bernhard Schmid, Mirko Ramisch, **Ulrich Stroth**

In fusion devices, an increased zonal flow activity is found when the bifurcation to high confinement (H-mode) is reached. With their m=n=0 topology and finite radial wave number k, the zonal flow is intrinsically connected to a poloidal shear flow. Drift-wave eddies are tilted and drive the shear flow, which leads to a self-amplification of the zonal flow. A key parameter in the drift wave – zonal flow system is the collisionality. It determines the coupling strength between the density and potential. Using a poloidal probe array various measurements were carried out in the low-temperature plasmas of the stellarator TJ-K, to investigate the collisionality scaling of zonal flows.

1.00

ZF / P total

0.01

0.01

0.10

In the figure, the relative zonal flow power P_{zE} / P_{total} as a function of the collisionality is shown. In the limit of the adiabatic case $C \rightarrow 0$, the zonal flow contribution to the complete spectrum strongly increases. For low collisionality, density and potential are strongly coupled and act similar. Since the zonal flow is a pure potential structure. but the drift waves are sheared in the density, the drive is more efficient for higher coupling. In future, collisionality scaling of energy sources and sinks of the zonal flow will be adressed.

Collaboration: P. Manz, Technische Universität München



Fig. 1: 7-tip probe constructed in-house. Together, the 7 tungesten wires (isolated from one another via ceramic tubes) measure plasma density, temperature, floating potential, and vorticity.

However, in the hydrodynamic limit (large collisionality) the density and potential decouple. As a result, the density becomes passively advected, evolves towards the vorticity, and exhibits intermittent behavior.



• H - He Ne Ar Kr

1000

100



1.00

10

Electron Cyclotron Emission Measurements at TJ-K Contact: Gabriel Sichardt, Eberhard Holzhauer, Mirko Ramisch

A diagnostic analysing electromagnetic radiation generated by the gyration of electrons (electron cyclotron emission, ECE) is being set up at TJ-K. This diagnostic promises to deliver electron temperature profiles along the line of sight, time-resolved electron temperatures and detection of non-thermal electrons. The setup will be optimized and the temperatures evaluated.Since the E spectrum changes dramatically thro non-thermal electrons, the ECE will used to investigate their occurrence Fast electrons moving parallelly to t magnetic field move on drift orbits t are larger than the flux surface when antiparallel movement leads to sma

The ECE around the second harmonic of the 8.256 GHz heating frequency is captured by an antenna, amplified and its frequency spectrum is analyzed. First signals in this range have been recorded with levels clearly above the noise level. The figure shows a sample spectrum of the microwave heating compared to the ECE spectrum.

temperatures evaluated. Since the ECE spectrum changes dramatically through non-thermal electrons, the ECE will be used to investigate their occurrence. Fast electrons moving parallelly to the magnetic field move on drift orbits that are larger than the flux surface whereas antiparallel movement leads to smaller orbits. Therefore, a toroidal net current can be caused by parallelly moving electrons leaving the plasma. Numerical investigations look into the behavior of drift orbits in TJ-K and show that noticeable displacements from flux surfaces occur at relativistic velocities. Investigations on the generated current range are planned.

Collaboration: A. Köhn, Max-Planck-Institut für Plasmaphysik, Garching



Fig. 1: Emission with and without plasma around the second harmonic of the heating frequency in a 8 mPa Ar plasma heated with 900 W at 8.256 GHz.

Zonal Flow Behavior under Plasma Biasing in TJ-K Contact: Til Ullmann, Bernhard Schmid, Mirko Ramisch

Zonal flows play an important role in the development of the high confinement regime in fusion experiments. These shear flows are driven by the ambient turbulence itself. This drive is considered to be depending on the plasma background flow. With plasma biasing it is possible to induce a stationary flow. Therefore, a positive potential was applied to two differently shaped electrodes, which were in two scenarios in contact with the plasma: firstly, a ring-shaped electrode inside the confined plasma and, secondly, two poloidal limiter plates at the separatrix. For the investigation of the background flow, radial profiles of the floating potential, temperature and density were measured.

Turbulence studies were carried out with an array of 128 Langmuir probes - distributed on four different flux surfaces measuring the floating potential. With the ring-electrode strong gradients in the plasma potential were detected, generating dominant ExB shear flows. The zonal flow level was strongly increased as confirmed by spectral analysis. With limiter biasing the plasma potential can be equalized (Fig.1). In this case the nonlinear mode coupling of the turbulence becomes more broadband, which constitutes the reverse of manifold shrinking in the presence of ExB shear flows.

Collaboration: P. Manz, Technische Universität München



Fig. 1: Radial profiles of the plasma potential under Limiter biasing. The plasma potential increases with higher bias voltage. At $U_{n_{tor}} = 15$ V the gradient in the plasma edge region is equalized.



Microwave Technology

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 plasma, to drive plasma currents and to control magneto-hydrodynamic instabilities. The Microwave Technology (MT) group at IGVP contributes to the development of 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 remotesteering launchers for ECRH on W7-X, 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 1MW-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 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 AS-DEX 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 on collective Thomson scattering, dedicated transmission and antenna components are designed.

Contact

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Microwave Technology

Development of High-Power Millimeter-wave Calorimetric Loads

Contact: Walter Kasparek, Bernhard Roth, Achim Zeitler

For the test of gyrotrons in ECRH systems, high-power, long-pulse, calorimetric loads are required. The loads presently available at IPP Greifswald and IPP Garching feature high internal field strength, with significant probability of arcing. Therefore, loads based on an alternative concept – high absorption and only few internal reflection – are developed and built. These loads use a meshwork of water-filled Teflon hoses as absorber, where a highly turbulent flow suppresses the boiling of the water in the tubes.



Fig. 1: Design drawing of the 2-MW absorber load.

For the ECRH-3 system, a 1-MW, CW load was designed and built. A typical design drawing is shown in Fig. 1, and a photograph of the inner surface is displayed in Fig. 2. The design features a beam concentrator (modified Winston cone) at the beam input, an optimized conical reflector and 240 scattering elements to distribute the input beam power evenly on the walls of the load chamber. 24 Teflon hoses form the absorbing meshwork.

The load has been tested in the ECRH system on W7-X up to 880 kW and 3 min; no arcing was detected. It is now prepared for installation in the ECRH-3 system on ASDEX Upgrade.In parallel, the design of a 2 MW, CW load to be used at W7-X was started.



Fig. 2: Photograph of the inner surface of the 1-MW load showing (part of the) distribution reflector and the arrangement of the teflon hoses.

Collaboration: J. Stober, D. Wagner, Max-Planck-Institut für Plasmaphysik, Garching; H. Laqua, T. Stange, Max-Planck-Institut für Plasmaphysik, Greifswald Funding: Max-Planck-Institut für Plasmaphysik, Garching; Max-Planck-Institut für Plasmaphysik, Greifswald

Matching Optics and Diagnostics for ECRH on ITER Contact: Walter Kasparek, Carsten Lechte, Burkhard Plaum

In collaboration with the European gyrotron consortium, and especially in collaboration with the Karlsruhe Institute of Technology (KIT), IGVP participates in the development of the ITER gyrotron. The work includes the beam characterization of prototype gyrotrons at 170 GHz, the design of a cooled matching optics unit (MOU) including power and mode monitors.



Fig. 1: Sequence of beam profiles of the pre-prototype gyrotron recorded with thermal imaging at various distances from the gyrotron output window. The dotted line shows the target geometry.

In 2015, the beam of the pre-prototype gyrotron was characterized by thermal imaging, and the beam parameters were deduced by phase reconstruction. Fig. 1 shows the evolution of the gyrotron beam. Based on the measurements, two matching mirrors for the gyrotron test stand in KIT have been designed, and are machined at IGVP.

A conceptual design of the MOU with two compact reflectors for (the European part of) ITER ECRH system was proposed, and is continuously improved to match the constraints and requirements for ITER. The MOU includes an evacuated box made directly from a solid aluminium block, where all openings and mounts can be directly machined into the thick walls. The two reflectors feature 4-axes alignment, and efficient water-cooling using the cooling concept which was developed for the W7-X ECRH.

Directional couplers are being developed, which are integrated into the surface of the first matching reflector. A 2-channel design allows the monitoring of the power transmitted over the mirrors, and will provide an error signals in case of unwanted mode jumps of the gyrotron.

Collaboration: G. Gantenbein, J. Jin, T.Kobarg, Karlsruhe Institute of Technology, Karlsruhe Funding: Fusion for Energy (F4E) promotional reference GRT-553

Microwave Technology for ECRH on ASDEX Upgrade Contact: Walter Kasparek, Carsten Lechte, Burkhard Plaum

At IPP Garching, a new plasma heating system "ECRH-3" is built, which consists of 4 gyrotrons (2 frequencies 105 / 140 GHz, power 1MW, pulse length 10 s), individual matching optics, corrugated HE11 waveguide for transmission to the plasma, and steerable reflector antennas. IGVP contributes in the design and construction of components for transmission and related diagnostics.



Fig. 1: Design of the power monitor integrated in a mitre bend, with the 140-GHz antenna pattern of the coupling hole array. For 105 GHz, the main lobe points to the direction of the incoming HE11 mode.

In 2015, the design of the surfaces of the mirrors in the matching optics box has been finalized, and specifications for the machining of the surfaces where provided. The surfaces of the polarizers were machined, and tested in a 3-mirror resonator. From these tests, rectangular-groove parameters were derived which can be used as input for polarization settings at ASDEX Upgrade.

Novel two-frequency directional couplers, which are integrated in the surfaces of mitre-bend mirrors where developed and specified. These couplers employ leaky wave antennas, basically designed for 105 GHz; in addition, an amplitude grating overlaid on the hole coupling structure produces a receiver lobe for 140 GHz (Fig. 1).

A 1-MW, 10-s calorimetric load to be used for power measurement of the four gyrotrons via switching mirrors was designed and constructed (see chapter "Development of High-Power Millimeter-wave Calorimetric Loads"). The design for compact 1-s absorbers, individual for the gyrotrons was started.

The MC III diplexer, which is optimized for experiments on in-line ECE, is ready for integration into a transmission line; the in-waveguide polarizers are presently equipped with motor drives.

Collaboration: J. Stober, D. Wagner, M. Schubert, F. Monaco, H. Schütz, B. Petzold, Max-Planck-Institut für Plasmaphysik, Garching

Funding: Max-Planck-Institut für Plasmaphysik, Garching; Max-Planck-Institut für Plasmaphysik, Greifswald

Fullwave Doppler Reflectometry Simulations for ASDEX Upgrade Contact: Carsten Lechte

Doppler reflectometry is an Important microwave diagnostic for turbulent fusion plasma. The incoming wave is scattered at certain density fluctuation wavenumbers, depending on frequency and angle between beam and density gradient. This way, a wavenumber resolved density fluctation spectrum can be measured. However, the correspondence between fluctuation power and scattered microwave power is strongly non-linear. input. Fig. 1 shows the microwave field propagating in a turbulent plasma. The resultant spectra are shown in Fig. 2. Turbulence spectra are characterized by the power law decay at large k. Both the experimental and the simulated (A=1) spectra have very similar exponents. If the input turbulence amplitude is decreased by a factor of 10 (A=0.1), the spectral shape changes significantly. This shows the non-linear saturation that affects the A=1 spectrum.



Fig. 1: Wave field of the microwave with overlaid density contours of the plasma. The solid blue line is the cutoff density. The antenna is in the upper right corner.

The role of simulations is two-fold: the plasma turbulence code GENE is used to simulate the plasma fluctuations, and the fullwave code IPF-FD3D is used to simulate the Doppler reflectometer with the turbulence field given as



Fig. 2: Simulated wavenumber spectra and comparison to exerimental measurements. A is the scaling factor for the density fluctuation strength.

Collaboration: G. Conway, T. Görler, C. Tröster-Schmid, Max-Planck-Institut für Plasmaphysik, Garching Funding: Helmholtz Virtual Institute "Plasma Dynamical Processes and Turbulence Studies using Advanced Microwave Diagnostics"; High Performance Computing Centre Stuttgart (HLRS)

Remote-Steering Launchers for ECRH on W7-X Contact: Carsten Lechte, Walter Kasparek, Burkhard Plaum

The stellarator Wendelstein 7-X, recently gone into operation at IPP Greifswald, has a multi-megawatt microwave heating system at 140 GHz. In addition to the front steering launchers, which have steerable mirrors on the front near the plasma, there are two remote steering lauchers (RSL1 and RSL5) where the beam is created away from the device and transmitted to the plasma by an oversized imaging waveguide.



Fig. 1: Far field antenna patterns of the beam at various steering angles. The sidelobes are at -30dB.

The inside corrugation of the waveguide and its cross section have been optimized to improve the imaging properties and ensure high power operations. Safe positions for vacuum breaks and miterbends were found. Together with the adaptable input coupling (pictured in Fig. 2), a steering range of $\pm 15^{\circ}$ was achieved. After the galvano-plastic fabrication at the industrial partners, the beam quality for all angles was measured at IGVP. Fig. 1 shows the resultant beam pattern In general, the imaging properties conform to the specifications.

With this, the project FORMIK³ has been brought to a successful conclusion. The RSLs will be incorporated into the stellarator in the next campaign break.



Fig. 2: Input side with movable input mirror (copper), vacuum window assembly (stainless steel), and waveguide start (lower right corner).

Collaboration: V. Erckmann, H. Laqua, Max-Planck-Institut für Plasmaphysik, Garching; M. Weißgerber, Max-Planck-Institut für Plasmaphysik, Greifswald; A. Bechtold, NTG NeueTechnologien GmbH, Gelnhausen; B. Szepaniak, Galvano-T GmbH, Windeck/Rosbach Funding: German Federal Ministry of Education and Research (BMBF) promotional reference 03FUS0017B

In Vessel Reflectors for Multi-Pass ECRH Heating Contact: Burkhard Plaum

ECRH heating at higher harmonics (e.g. 2nd harmonic ordinary mode, O2, or 3rd harmonic extraordinary mode, X3) extends the operating range at the expense of a reduced absorption by the plasma. As a result, a beam with a considerable power hits the inner wall of the vacuum vessel, where is can cause a damage due to a high heat load.

A solution is to place reflectors at the inner wall, which redirect the beam for a second pass through the plasma. The angles and the beam parameters of the indicent and reflected beams are found with beamtracing codes, which simulate the propagation in the plasma.



Fig. 1:TRAVIS calculation of a beam propagation through the plasma.

An optimization algorithm is then used to find a grating profile with the required reflection characteristics. The code was developed for the ECRH-2 system with the beam parameters calculated by TORBEAM code. Such reflectors are installed and tested in ASDEX Upgrade.

For W7-X, a new interface was programmed to handle beam trajectories from the TRAVIS code. Furthermore, several ASDEX Upgrade parameters inside the code were replaced and a completely generalized optimization framework was built. The beams can be redirected for a second pass through the plasma, where more power is absorbed. Finally they hit the wall at a location, where no sensitive parts are installed. The next step is to finalize the parameters and optimize and manufacture the reflectors.

Collaboration: H. Laqua, T. Stange, N. Marushchenko, Max-Planck-Institut für Plasmaphysik, Garching; M. Schubert, Max-Planck-Institut für Plasmaphysik, Greifswald

Funding: Max-Planck-Institut für Plasmaphysik, Garching; Max-Planck-Institut für Plasmaphysik, Greifswald

Investigation of a Remote Steering Antenna for ECEI Applications

Contact: Burkhard Plaum, Daniel Iglesias

The remote-steering principle, which was investigated in detail for ECRH applications, can also be used for setting up an ECE imaging system.

The advantage of this antenna for ECEI is, that radiation can be detected under different angles and frequencies simultaneously while requiring just a small port area. Since the frequency of the ECE radiation depends on the magnetic field, which increases further inside the plasma, this setup allows a 2D imaging. The actual receiver antennas are placed several meters away from the plasma, where space limitations are less critical.



Fig. 1: Gaussian efficiency of a remote steering antenna as a function of the frequency and the steering angle.

In an initial study, the Gaussian efficiency of a remote steering antenna was calculated for a wide range of angles and frequencies. The result is a large number of operating points. For each operating point, the beam propagation in a W7-X plasma was calculated. The location of the maximum absorption of a hypothetical ECRH beam corresponds to the point whose electron temperature will be detected. One can see, that it's possible to cover almost the entire poloidal cross section with just a single antenna.



Collaboration: M. Hirsch, T. Windisch, Max-Planck-Institut für Plasmaphysik, Garching Funding: Helmholtz Virtual Institute "Plasma Dynamical Processes and Turbulence Studies using Advanced Microwave Diagnostics" and Erasmus Mundus Master program "European Master of Science in Nuclear Fusion and Engineering Physics"

PROFUSION Code Development Contact: Burkhard Plaum, Sander Coene

The PROFUSION code (Programs for multimode analysis, simulation and optimization) is constantly extended and enhanced as requested by other projects.

An addition in 2015 was the implementation of the scattering matrix algorithm for rectangular waveguide cross sections. As a result, rectangular horns and tapers can be calculated. A comparison showed a good agreement with another implementation.

Simulated Field (k=22.8711) 0.6 0.8 0.4 0.6 0.4 0.2 0.2 k [m] -0.2 -0.2 -0.4 -0.6 -0.4 -0.8 -0.6 -0.2 0 0.2 0.4 0.6 -0.6 -04 x [m]

methods (like point matching method) can produce reasonable results for cross sections, where Eigenmodes and – frequencies are known analytically (see figures). Problems arise, however, when degenerate solutions i.e. different Eigenfunctions with identical Eigenvalues exist. The current focus is therefore mainly on the detection of these degeneracies. Once a robust, general purpose Eigenmode solver exists, it can be used for designing a large variety of new oversized microwave components.



Fig. 1: Numerically calculated field (Ez) of aTM2,7 mode in a rectangular waveguide

An ongoing project is the development of an Eigenmode solver for waveguides with irregular cross sections in the frame of a master thesis. Different methods are compared with respect to speed and accuracy. Even simple

Fig. 1: Numerical error compared to the analytical solution

Funding: Max-Planck-Institut für Plasmaphysik, Garching; Erasmus Mundus Master program "European Master of Science in Nuclear Fusion and Engineering Physics"

Electron Bernstein Waves Propagation in the Linear Device FLiPS

Contact: Kirill Rumiantsev, Walter Kasparek, Eberhard Holzhauer, Alf Köhn

Electron Bernstein Waves are electrostatic waves which can propagate in overdense plasmas. Over the last two years an experiment that would allow to test general EBW properties against theory and simulations has been designed and built.

FLiPS is a cylindrical plasma vessel with an axially symmetric magnetic field with various configurations; the plasma is created by electron cyclotron resonance heating at 2.45 GHz. For a magnetic trap configuration, interferometric measurements have shown that the plasma is moderately overdense.



Fig. 1: EBW propagation in the FLiPS plasma showing trapping effects.

However, due to problems like X-radiation due to non-thermal electrons and high thermal load on the main heating system's vacuum window (which are being solved now), most experiments were performed in a plasma with homogenous linear field. The O- and X- Waves were injected into the plasma to couple to EBW with open-ended waveguide antennas. Due to the antennas' broad beam and the unstable plasma no definitive conclusions about the EBW's role in the heating could be drawn up to now.

A ray-tracing code with the hot-plasma dielectric tensor has been developed. The simulations for FLIP's parameters have shown that EBW are confined within a narrow wave channel. This channel modifies the EBW absorption and the parametric decays threshold. An example of the trapping geometry is shown in Fig. 1. In parallel, full-wave simulations have been started to visualize the field pattern in the channel.

Collaboration: N. Maruschchenko, Max-Planck-Institut für Plasmaphysik, Greifswald

Funding: Max-Planck-Institut für Plasmaphysik, Garching; Max-Planck-Institut für Plasmaphysik Greifswald In a toroidal plasma, Doppler reflectometry (DR) allows investigating electron density fluctuations with finite wavenumber $k \perp$. The injected microwave beam's frequency determines the cut-off electron density (i.e. the radial position) of the probed region. The beam's tilt angle selects the wavenumber that satisfies the Bragg condition for backscattering. The rotation velocity can be calculated from the Doppler shift of the backscattered signal's frequency. By varying the injected frequency, radial profiles can be reconstructed. By varying the tilt angle, $k \perp$ -spectra of the fluctuations are resolved.



Fig. 1: Left: Feed network with waveguide connectors mounted on horn array. Right: Feed network.

For DR, a pair of series-fed 32-element phased array antennas (PAAs) has been designed, built, and installed in the ASDEX Upgrade tokamak. Beam steering is done by slightly changing the injected frequency, thus, the PAAs do not need any movable parts or electronics inside the vacuum vessel. Between 75 and 105 GHz, the PAAs feature 13 frequency bands, each with an angular scan range of –20 to +20°. So, for each angle, 13 radial positions can be probed. First tests have verified the PAAs' functionality.



Fig. 2: Aperture and flange view of the 32-element horn arrays.

Collaboration: G. Conway, Max-Planck-Institut für Plasmaphysik, Garching; T. Windisch, Max-Planck-Institut für Plasmaphysik, Greifswald Funding: Helmholtz Virtual Institute, Plasma Dynamical Processes and Turbulence Studies using Advanced Microwave Diagnostics

Microwave Technology

Multiport Coupler for *In-situ* Waveguide Mode Analysis Contact: Alexander Zach, Walter Kasparek, Burkhard Plaum

For electron cyclotron resonance heating (ECRH) of ASDEX Upgrade, gyrotron beams with about 1 MW power at 140 GHz are guided by waveguide transmission lines into the plasma vessel to be absorbed by the plasma. Due to the high power these transmission lines need to have a diameter of multiple wavelengths (r > 10 ℓ). This however allows for the propagation of unwanted higher-order modes with comparably high losses. The preferred hybrid mode LP₀₁ (or HE₁₁) should be almost solely excited. Therefore, alignment of the gyrotron beam with respect to the waveguide entrance as well as the wavequide run itself is a crucial point.



Fig. 1: Coupler output for an interferometric setup as a result of tilting the alignment mirror M3.

A set of directional couplers located in a miter bend mirror in the transmission

line allows for *in-situ* real-time detection of the modes most likely to be excited by small misalignments ($LP_{11,even}$ and $LP_{11,odd}$), and beam mismatch (LP_{02}), as well as the main transmission mode (LP_{o1}).

The basic working principle and high power capability could be proven in experiments at IPP Garching with a simplified interferometric setup, as seen in the figure.

An advanced mirror with modematched hole array couplers utilizing 0th and 1st order hole coupling is developed. Both an interferometric setup and complex amplitude measurements are planned.

Collaboration: J. Stober, F. Monaco, H. Schütz, Max-Planck-Institut für Plasmaphysik, Garching; H. Idei, Kyushu University, Japan Funding: Max-Planck-Institut für Plasmaphysik, Garching

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