# 4 Energy Efficiency, Materials and Resources (EMR): Energy-Efficient Processes -Multiphases and thermal processes-

Contact: Dr. Guido Link

Besides the activities on development of technologies and systems for the plasma heating in the FUSION Program, IHM is also in charge of research and development in the topic Energy Efficient Processes, part of the EMR Program.

An important part of this research is the dielectric characterization of the processed materials in the parameter range relevant to processes under development. Therefore existing test-sets are continuously improved and new test-sets are developed following the new requirements regarding materials compositions or process parameter range. Meanwhile a very versatile test lab for dielectric characterization exists. This allows temperature dependent dielectric measurements in the frequency range from 10 MHz to 30 GHz for low as well as high loss materials and from room temperature up to 1000°C for solids, liquids and at pressures up to 20 bar.

All this expertise and the existing industrial scale high power microwave infrastructure faces growing interest from industry and research. As a consequence the research group is involved in several national and international joint research projects with objectives in various fields of applications. The H2020 project SYMBIOBTIMA requests the design of an industrial prototype reactor for the microwave assisted depolymerization of PET plastic waste for the purpose of energy efficient recycling. In the frame of the H2020 Marie Curie international training network TOMOCON that started end of 2017 a microwave tomographic sensor will be developed. Within the German-Korean project REINFORCE the potential of microwave dielectric heating as well as microwave sustained plasma heating will be investigated with respect to energy efficient carbon fiber production.

Solid state microwave amplifiers getting more and more competitive compared to magnetron sources with respect to power and costs. Furthermore such amplifiers allow precise control not only of power level but also of frequency and phase and promise significant longer lifetime than magnetrons. Those features might be door openers for novel application that could not be satisfied with magnetron sources. Therefore national funded collaboration projects have been started with HBH microwave GmbH recently to develop affordable high power solid state generators that meet the requirements for novel process control concepts. Furthermore those novel microwave sources might be useful for microwave sustained plasma generators for plasma activation of CO<sub>2</sub> in the frame of research activities like Power to X. Therefore recently a novel lab for plasma chemistry using atmospheric microwave plasma has been established. The status of major projects is briefly introduced in the following chapters.

## 4.1 Materials Processing with Microwaves

## 4.1.1 SYMBIOPTIMA

#### Contact: M.SC. Vasileios Ramopoulos

The KIT task within the European project SYMBIOPTIMA is to support the development of an industrial scale and modular microwave reactor for the recycling of PET plastic waste. In this sense recycling means depolymerization of the PET molecules into monomers by use of microwave assisted alkaline hydrolysis. Further information about this project may be found under http://symbioptima.eu/ Based on the measured dielectric properties and using common 3D software tools, an optimized industrial scale and modular microwave cavity design has been developed. The cavity is designed for use in combination with an Archimedean screw to transport the reaction material within a cylindrical dielectric tube with a diameter of 250 mm. The proposed design is modular and can be easily scaled in length. The developed design offers a well-defined and homogeneous power distribution of a single 2.45 GHz magnetron source within the applicator and the process material. Results of various simulations show that a homogeneous heating of the process material in axial as well as azimuthal direction can be achieved by using the TE<sub>1,0</sub> field mode. The simulated design provides high energy efficiency with a reflected power of less than 10 %, insensitive to even significant variation of materials permittivity. The CAD model and a photo of the manufactured microwave cavity are shown in Fig. 4.1.1.

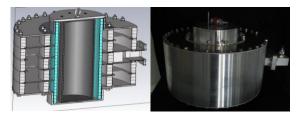


Fig. 4.1.1: Left: CAD model of the microwave cavity; Right: Experimental setup

For testing with a 800 W magnetron, a hollow cylindrical sample with 183 mm outer diameter and 171 mm inner diameter and dielectric parameters of  $\epsilon$ '=15, tan $\delta$ =0.3 was chosen. The measurement of the energy (temperature) distribution is carried out by an infrared camera FLIR type AX5. Fig. 4.1.2 shows the measured temperature distribution in comparison with the simulated absorbed power distribution within the sample. The measured temperature variation is found to be ± 8 °C at a mean temperature level of 90 °C. A proper agreement between simulation and experiment exists.

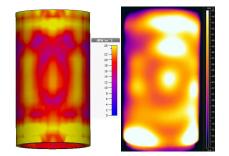


Fig. 4.1.2: Simulated power distribution (left), measured temperature distribution (right).

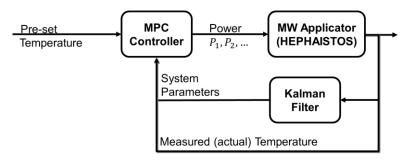
Funding: H2020-SPIRE-2015; grant agreement: 690426

## 4.1.2 InnoConTeMP

#### Contact: M.Sc. Dominik Neumaier

Microwave heating has a great potential to replace classical heating processes (e.g.: hot air). The main advantage of microwaves is the volumetric heating effect, which does not limit the heating process by slow thermal conduction. Compared to a classical heating system both energy consumption and cycle time can be significantly reduced in various applications. However, the application of microwave technology in industrial processes very of suffers from insufficient temperature uniformity of the heated product.

The aim of the technology transfer project InnoConTeMP (Innovative Control of Temperature Distributions for Microwave heating Processes) is to improve the temperature distribution of a workpiece in HEPHAISTOS systems of the industrial partner Vötsch Industrietechnik GmbH. A model predictive controller (MPC), which uses a thermodynamic model and the results of a Kalman filter, is used for controlling the temperature level as well as the temperature distribution in a workpiece. This is achieved by individual control of the power levels of the distributed microwave sources. The Kalman filter permanently observes the influence of the electric field and the material properties. The structure of the whole control algorithm is presented in Fig. 4.1.3.



**Fig. 4.1.3:** Structure of the control algorithm.

Thereafter, an architecture was developed to integrate this algorithm in the existing SIMPAC controller of our partner Vötsch. The control structure is shown in Fig. 4.1.4. Alternatively, in case of limited CPU power of standard SIIMPAC controllers a more powerful industrial PC can be connected over TCP/IP. Thus the MPC and Kalman filter can be computed on this powerful PC and the results are sent to the SIMPAC. In addition to that, the new MPC controller can also be used and tested with the Labview control system, which was developed at KIT.

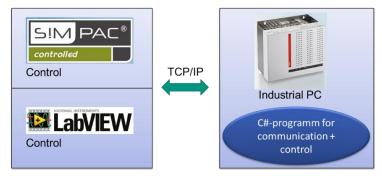


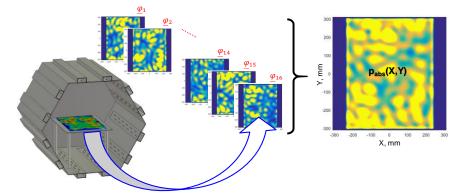
Fig. 4.1.4: Architecture of the whole control system.

Funding: KIT technology transfer project; project no.: N041

### 4.1.3 High power solid-state microwave generators

#### Contact: Dr. Sergey Soldatov

For industrial scale microwave processing, major issues are precise and stable process control as well as homogeneous heating of the products. This depends on the homogeneity of the absorbed power in the dielectric load, which, in its turn, depends on the electric field pattern as well as on the dielectric properties of the processed materials. The field patterns result from the superposition of the excited eigenmodes in the microwave oven. The use of solid state high power amplifiers instead of commonly used magnetron oscillators promises to get significantly more influence to the process control and temperature uniformity within the product. Those will not only allow control of the power amplitude like in magnetrons but also the precise control of frequency and phase correlation in-between different sources. A fast superposition of different specific field patterns, resulting from well-defined amplitude, frequency and phase variation, will allow to further improve temperature uniformity as compared to existing methods. This approach is illustrated in Fig. 4.1.5. This approach motivated a collaboration project with HBH microwaves GmbH, supported by the Federal Ministry for Economic Affairs and Energy (BMWi) that started in March 2017. A major objective of the project is the development of novel high power (~ 1 kW) solid-state microwave sources with fast computer control. Those sources will enable the variation of frequency within the ISM band from 2.4 GHz to 2.5 GHz, the variation of phase from 0 to  $2\pi$  and the variation of power from 10 W to 1000 W at a time scale of 100 ms. To get the maximum use of that novel microwave sources, with respect to temperature uniformity in large scale applicators with distributed microwave sources, the MPC control concepts developed in InnoConTemp (see previous chapter) will be extended accordingly.



**Fig. 4.1.5:** Schematic pathway from non-controlled heating dominated by standing wave pattern towards homogeneous heating based on the intelligent control of launched power.

Another promising application of fast controlled microwave sources is the development of advanced microwave sustained plasma reactors, which is strongly motivated by the establishment of the "Closed Carbon Cycle Economy" and conversion of CO<sub>2</sub> into synthetic fuels. As it was proven in many experiments non-thermal CO<sub>2</sub> plasmas provide maximal conversion efficiency. Microwave sustained atmospheric plasmas can be operated far from the thermal equilibrium if the microwave energy is fed in short nanosecond pulses. Such pulsed operation prevents the thermalization of ions which happens at µs time scales. High power microwave pulsed in this timescale are not feasible with commercial vacuum electronic devices (magnetron). By use of high power solid-state amplifiers this timescale can be reached. First solid-state generators and experimental results will be expected in 2018.

Funding: ZIM cooperation project, support code ZF4204602PR6

### 4.1.4 REINFORCE

#### Contact: M.Sc. Julia Hofele

Due to the high tensile strength to weight ration of carbon fibers compared to steel and their lightweight potential, carbon fibers find increasing applications in the automotive and avionic industry. In 2014 the industry required a carbon fiber volume of 53 kt worldwide which is expected to increase to 100 kt in 2020. Until now, the carbon fiber production is rather expensive and energy intensive compared to the production of aluminum and steel.

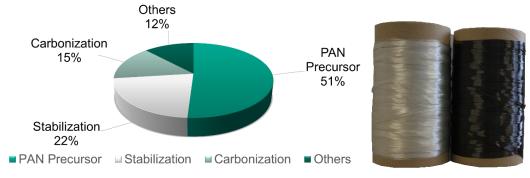


Fig. 4.1.6: Manufacturing costs of carbon fibers

The project REINFORCE, which started officially in September 2017, has the objective to improve the carbon fiber production. The three most time and energy consuming production steps, precursor creation, stabilization and carbonization, are to be optimized. A new PAN-precursor, which can be used for melt-spinning, will be developed by the Korean partners KCTECH, DongMyung Technologies and Yeungnam University. In the frame of REINFORCE, KIT is involved in the replacement of conventional heating in the stabilization and carbonization process by dielectric heating or microwave sustained plasma heating. It will be evaluated which method is more efficient and which has a better influence on the fiber properties compared to conventional heating. The development of an appropriate microwave applicator and the experimental investigation of potential advantages from dielectric and plasma heating are performed at IHM. In a first step, different PAN fiber samples and a stabilized fiber have been characterized dielectrically. Ideally, the information of the dielectric properties can be used to evaluate the end of the stabilization process. The next steps include first experiments in the cavity that was also used for the dielectric measurements in order to get a better understanding of the process. Finally, microwave applicators will be built for dielectric heating and plasma heating in order to stabilize the fiber. Final results of the project are expected in 2020.

Sample	Fiber Tow	Dielectric Constant $oldsymbol{arepsilon_r}'$	Dielectric Loss Factor $oldsymbol{arepsilon_r}''$
PAN Company A	12k	3.9	0.005
PANOX Company A	12k	5.8	0.012
PAN Company B	3k	3.8	0.004

 Table 4.1.1: Dielectric Properties at 2.45 GHz measured for virgin and stabilized PAN fibers

Funding: ZIM cooperation project; support code ZF4204603SY7

### 4.1.5 TOMOCON

#### Contact: Dr. Guido Link

The European Marie Skłodowska-Curie Training Network "Smart tomographic sensors for advanced industrial process control (TOMOCON)" joins 12 international academic institutions and 15 industry partners, who work together in the emerging field of industrial process control using smart tomographic sensors. The network shall lay the scientific and technological fundamentals of integrating imaging sensors into industrial processes and will demonstrate its functional feasibility on lab and pilot-scale applications.

15 doctoral researchers are being trained in the fields of process tomography hardware, software and algorithms, control systems theory and design, industrial process design, multi-physics modelling and simulation, human-computer interaction, and massive parallel data processing. Together with their supervisors and industry partners they are engaged in multi-disciplinary research on various tomographic imaging modalities, tomographic image processing as well as advanced multi-physics modelling of processes, sensors and actuators. Proof-of-principle demonstrations of tomography-based process control are being foreseen for important industrial processes, such as inline fluid separation, microwave drying of porous materials, continuous steel casting and ultrasound-controlled crystallization. The doctoral researcher at KIT will be involved in the development of a microwave tomograph to detect the moisture distribution in a porous material and to use that information to get a feedback for the process control. The technology finally is planned to be demonstrated on a conveyor belt system for microwave assisted drying of polymer foams.



<

Fig. 4.1.7: Hybrid HEPHAISTOS system for continuous processing of materials

Funding: H2020-MSCA-ITN-2017; Grant agreement 764902

## **Involved Staff**

L. Baureis, Frau J. Frank, J. Hofele, Prof. J. Jelonnek, S. Layer, **Dr. G. Link**, D. Neumaier, V. Nuss, Ramopoulos, T. Seitz, S. Soldatov, Frau S. Wadle

## **Journal Publications**

Link, G.; Ramopoulos, V. (2017). Simple analytical approach for industrial microwave applicator design. Chemical engineering and processing, 125, 334-342.

Sanz-Moral, L. M.; Navarrete, A.; Rueda, M.; Martín, Á.; Sturm, G.; Stefanidis, G.; Link, G.; Stefanidis, G. (2017). Release of hydrogen from nanoconfined hydrides by application of microwaves. Journal of power sources, vol. 353, 131–137.

Li, N.; Li, Y.; Jelonnek, J.; Link, G.; Gao, J. (2017). A new process control method for microwave curing of carbon fibre reinforced composites in aerospace applications. Composites / B, vol. 122, 61–70.