Microwave Materials Processing at the Karlsruhe Institute of Technology

G. Link, T. Kayser, A. Melcher[1], D. Prastiyanto[2], S. Soldatov[3], J. Jelonnek[4]
Karlsruhe Institute of Technology, Institute for Pulsed Power and Microwave Technology
guido.link@kit.edu

Abstract—*Karlsruhe Institute of Technology (KIT), Germanyis one of the largestresearch and education institutions worldwide. KIT is an institution pursuingtwo missions, the mission of a universitywithtasks in research and teaching and the mission of a large-scaleresearch institution of the national Helmholtz Association withprogrammatic provident research on behalf of the Federal Republic of Germany. Within this frameworkthe Institute for Pulsed Power and MicrowaveTechnology (IHM) is conducting basic and applied research in the field of pulsed power and high-power microwave technologies. Additional to the research in high power microwave sources (gyrotrons) for electron-cyclotron resonance heating and current drive (ECRH&CD) for magnetically confined plasma experiments, there exists long time experience in application of high power microwaves for materials processing. Applications are for example high temperature processing of ceramics, glasses andmetal powder compacts. Various microwave processes in the field of calcination, sintering and melting have been investigated. Furthermore large scale microwave processing chambers have been developed and successfully transferred to Industry for low temperature processing like drying of various materials and curing of glass andcarbon fiber reinforce polymers (CFRP). A short survey of this field of activities which is accompanied by electromagnetic simulation, system design and dielectric measurements will be given in the following paper.*

Keywords—*ceramics sintering, curing of CFRP, dielectric measurements, high-power microwaves, materials processing*

I. INTRODUCTION

The Institute for Pulsed Power and Microwave Technology (IHM) at Karlsruhe Institute of Technology (KIT); Germany is doing research and development as well as education and technology transfer in the areas of pulsed power and high-power microwave technologies. The activities of the department for pulsed power technologiesare related to applications of pulsed electron beamsas for example for surface remelting or alloying of metal parts to improve wear and corrosion resistance [1]. Another activity of growing interest is the application of pulsed electric fields for electroporation of biomassand the stimulation of biological cells [2].

A key competence of the department for high-power microwave technologies is the design and development of high power microwave sources(gyrotron oscillators) [3]. Gyrotronsprovide RF output powers in the megawatt-range (up to 2 MW in continuous wave (CW)) at millimeter wave frequencies up to 170 GHz. At KIT, gyrotrons are primarily designed for the specific needs as RF sources for electron cyclotron resonance heating and current drive (ECH&CD) of magnetically confined plasmas for nuclear fusion. As a spin off, about 20 years ago an additional focus was directed towards potential applications of high-power microwave and millimeter wave sources for materials processing.

Using microwaves for processing of materials (microwave processing) is motivated by the unique feature of microwaves, the volumetric and selective heating of materials. Conventional thermal processingbases on convection and radiation. Consequently, it results in heating of the product surfacesprimarily, due to the low penetration depth of infrared radiation. The microwave is penetrating deep into dielectric materials, depending on the dielectric constant and loss factor. It results the volumetric heating effect. Based on that,microwave processing can be significantly faster, in particular in case of bulky samples with low thermal conductivity. At the same time, this results in high potential for energy savings because only the material itself is heated but not the whole oven. At last, the possibility for selective heating can be positively exploited in special applications like gluing processes. A selective heating of the glue willresult in increased bonding strength at reduced processing time and lower energy consumption.

Above named benefits of microwave processing motivate investigations in various fields of applications such as processing of functional and structural ceramics for drying, debindering and sintering, melting, tempering or recrystallization of glass and glass ceramics, curing, post curing and tempering of thermoset and thermoplastic resins and composites. In the following, some examples of process specific microwave systems and potential applications will be given.

1. Microwave systems and applications

Microwaves cover a rather broad frequency band from 300 MHz up to 300 GHz with corresponding wavelengths in free space starting from 1m and ending at 1 mm. Within this frequency range there are specific narrow frequency bands reserved for industrial, scientific and medical application, the so called ISM bands. The ISM band at 2.45 GHz is the most used one. It is exploited for domestic microwave systems as well as for many other industrial microwave applications. Other ISM bands exist at 915 MHz, 5.8 GHz or 24.1 GHz.

1.1. High power millimeter-wave system for high temperature processing

For some materials heating applications the use of frequencies even above 24 GHz, i.e. wavelengths smaller than 12.5 mm is beneficial. First of all,in applicators with limited volume it is easier to achieve a homogeneous field distribution due to the shorter wavelength and thus a more homogeneous materials heating, as compared to microwaves at 2.45 GHz or below. Additionally, for low loss materials like high-purity ceramics, heating at frequencies above 24 GHz isenergy efficient, as the energy absorbed in the material is increasing proportionally to the frequency. Furthermore,for such ceramics the dielectric loss factor is increasing with the frequency usually as well. Thus, the use of microwave susceptors for indirect pre-heating of low-loss materials is not necessary.
A compact, 30 GHz, 15 kW CW gyrotron system [4] has been installed in 1994already (see Figure 1).It has been used for numerous investigations on sintering of functional and structural ceramics materials.

Fig. 1: Photography of the compact technological gyrotron system.

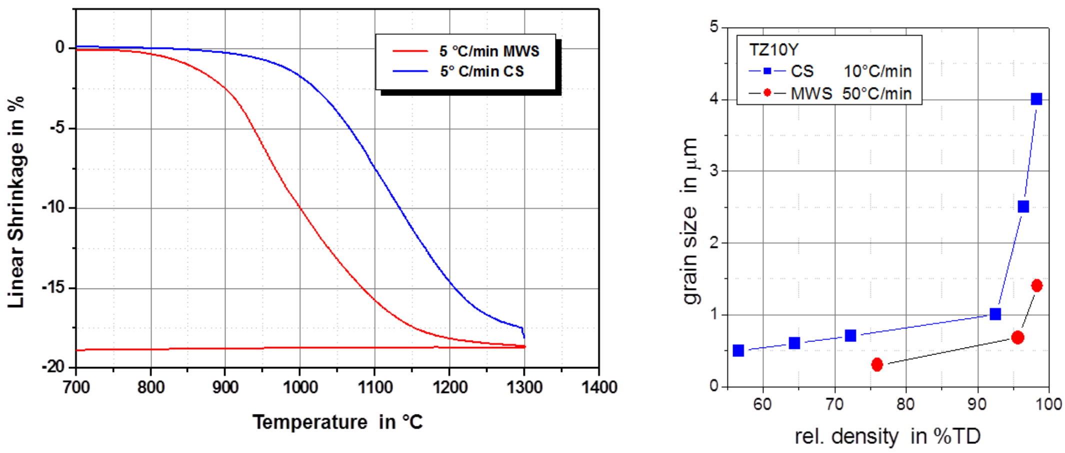

Fig. 2: Comparative studies on mm-wave sintering of yttria-stabilized zirconia. Results of dilatometer experiments (left) [5] and grain size evolution (right) [6].

Additional to the benefit of fast and volumetric heating, a reduction of the necessary process temperaturehas been reported, if comparing microwave sintering of ceramics with conventional sintering methods. An exemplary result is shown in Figure 2. The left graph presents a comparison of the linear shrinkagesof ayttria-stabilized zirconia measured by a dilatometer in a microwave system (MWS) and measured in a conventional furnace (CS). Thezirconia ceramicswas stabilized in the tetragonal phase by 3mol% yttria. As can been seen from this graph,usingmicrowave heating, the sintering process starts at lower temperatures as compared to conventional sintering. That has been reported in several papers already. Due to this effect, it is believed that electromagnetic energy can supply additional driving forcesto the sintering process which result in enhanced materials diffusion during sintering. A further indication of such a microwave effect is the fact that during enhanced densification with microwaves the grain growth is inhibited, as shown in the right graph of Figures 2 for a zirconia ceramic stabilized in the cubic phase by 10 mol% yttria.For two samples of comparable density, the microwave sintered sample reveals a significantly smaller average grain size as compared to conventionally sintered samples.

1.2. 2.45 GHz technology for fiber composite curing

The curing of glass or carbon fibre-reinforced plastics (GFRP or CFRP), which are seen as the materials of the future in avionics and automotive industries for strong and light-weight non-structural and structural components , requires large furnaces with excellent elektromagnetic field homogeneities and a curing in well defined temperature limits. So far, both, the requirement on accuratetemperature control and the requirement on homogeneous electromagnetic field distribution were the major limiting factors for the breakthrough of microwaves in this field. More generally, generating highly uniform fields in large-scale ovens at 2.45 GHz has been the core issue of industrial microwaves technologies.

To overcome the inhomogeneous field distribution the modular microwave system technology, named HEPHAISTOS has been developedat KIT [7]. It represents a radical innovation in the field of microwave heating systems. The hexagonal design of the chamber geometry results in outstanding field distributions. And, due to the modularity of the HEPHAISTOS concept, it can be easily scaled up to the size required for nearly any desiredproduct based on fibre-reinforcedplastics.

So far,different HEPHAISTOS microwave systems with process volumes up to 7.5 m^3 are available for industrial process developments. In meantime, the technology is licensed to and is further under development with theindustrial partner VötschIndustrietechnik, Reiskirchen, Germany (see Figure 3).

Fig. 3: Photography of the HEPHAISTOS Experimental Center and set-up for a VAP process for CFRP curing.

Within a joint research project, funded by Federal Ministry of Education and Science (support code 01RI05133), in collaboration with several industrial partners, various tooling concepts have been developed for successful adaption of the EADS patented, vacuum assisted process VAP®to the described microwave technology. In comparison to conventional convection ovens, a significant reduction in energy consumption of up to 75% and in processing time of up to 40% have been demonstrated for curing of CFRP composites.

Beside the development of highly overmoded and unturned batch kilns, compact single mode applicators have been developed for endless pultrusion of CFRP profiles. For this process, the dry carbon fibres are pulled through a resin bathto be impregnated by the thermoset resin. Afterwards, the fibres are pulled through a special tool, in which the resin is formed and, finally, cured (see Fig. 4). Endless production of lightweight CFRP profiles can be realized by this technique. Again, the motivation using microwave in this field of application is the potential of direct volumetric heating that may result in increased productivity by increase of pulling speed. Due to volumetric heating there is no need to relay on thermal conductivity, which is rather low for such type of composites materials. Thus, the overall dwell time within the tool can be reduced. A prototype of such microwave assisted pultrusion tool has been successfully designed and tested already. An important prerequisite to succeed in this process is the precise knowledge of the dielectric properties of the materials processed.

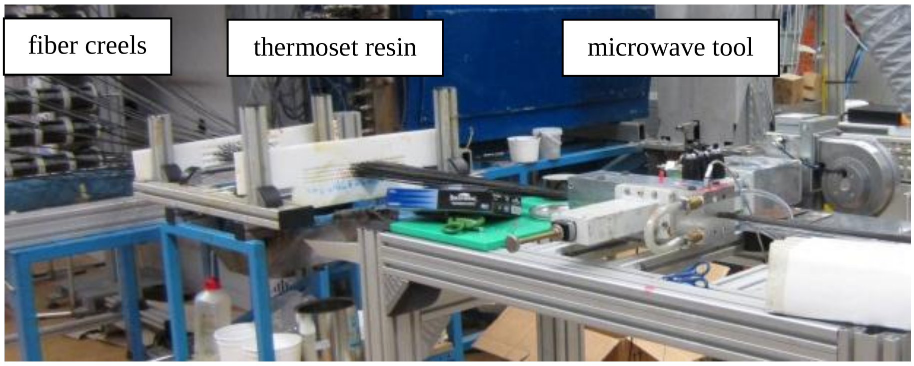

Fig. 4: Picture of a microwave assisted pultrusion experiment.

1.3. Measurements of dielectric properties at microwave frequencies

Any microwave process is determined by the dielectrics properties of the material processed.Therefore,the base for any optimization of microwave assisted processes is theexactknowledge of the dielectric properties of all materials involved in the process. Of course, those microwave properties may significantly change with changing frequency and temperature as well as with ongoing phase changes during the process. To cover the wide spectrum of materials and dielectric properties various test-sets for dielectric characterization are needed and are under development at IHM. The development of such test-sets is a continuous process, as with changing applications the specific needs are changing as well. All of those measurements techniques are based on transmission and/or reflection methods in non-resonant or resonant systems.

At IHM, for reflection measurements a commercial coaxial probe from Agilent is used. This is a rather simple technique that allows broadband dielectric measurements at frequencies up to 20 GHz. It is suitable to characterize high-loss material with loss tangenttan$\delta \geq 0.05$. Best results have been obtained with liquid or powder samples. Any air-gap in-between the probe end and the sample surface gives rise to measurement errors. Thus,in combination with solid samples one has to make sure that the sample surface is sufficiently flat [8].

Beside the reflection method using a coaxial probe, a transmission-reflection method based on the WR340 waveguide has been developed (see Fig. 5). Thatsystem allows temperature dependent dielectric measurements up to 200°C for dielectric materials with medium and high-loss factor [9].

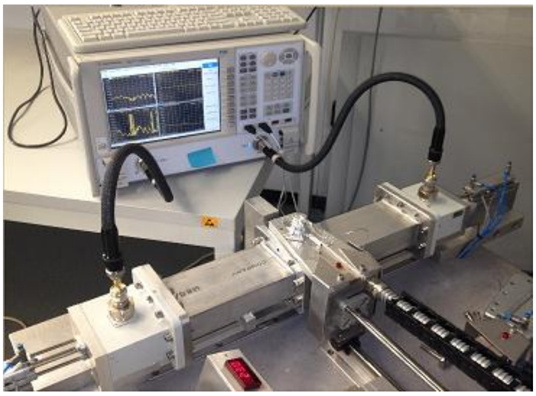
Fig. 5: Set-up for temperature dependent dielectric measurements using transmission-reflection method.

Samples that fully or partially cover the waveguide cross section can be used. The temperature of the material under test can be set to predefined values by controlled resistive heating of the sample holder.Heating elements are installed in the waveguide sample holder. The scattering parameters measured by the vector network analyzer can be acquired in parallel to the temperature information given by the thermocouple. Both, liquid and solid materials can be measured using this setup. Experimental results for two different polymers are shown in the following graph.

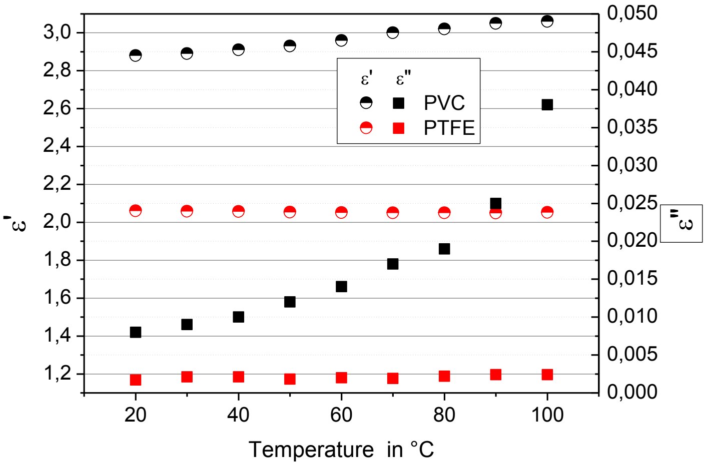

Fig. 6: Complex permittivity measurement for PTFE and PVC as a function of temperature at 2.45 GHz.

Another test set, currently under development is based on the cavity perturbation method [10]. It will allow dielectric measurements at temperatures up to more than 1000°C at 2.45 GHz for low-loss and medium-loss materials. Here, a small dielectric perturbation (sample) is placed into the cavity at a position with maximum electric field. The cavity resonance frequency and quality factor is changing according to the dielectric constant ε' and loss factor ε'' of the sample, respectively. The experimental set-up is shown in Fig. 7. It bases on a standard WR-340 waveguide.On both ends, the waveguide is closed by parallel plates with small irises, forming a resonator (cavity). The maximum transmission coefficient for the TE_{104} mode at resonance and without perturbation is -36 dB and the quality (Q)-factor is about 12000. Two coaxial waveguide adapters are used to connect the vector network analyzer (VNA) Agilent E5071C to the cavity. To provide an accurate positioning of the material under test (MUT) within the cavity a quartz glass tube with an inner width of 8 mm and 1 mm wall thickness is used as the sample holder. This sample holder can be moved between the cavity and a resistive heated tubular furnace that provides the desired temperature to the sample. A pyrometer is installed at the opposite side of the applicator to monitor the MUT temperature during the dielectric measurements. Both VNA and pyrometer are connected to a personal computer for remote controlled acquisition of the temperature and the S_{21} data.

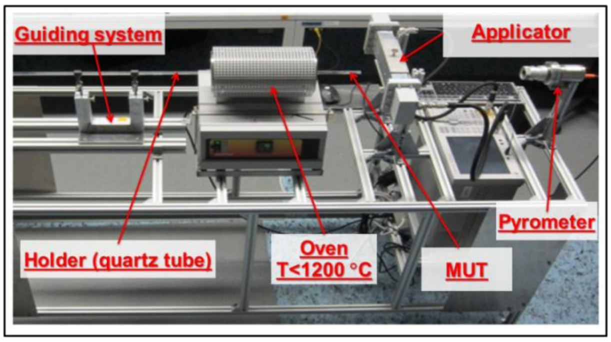

Fig. 7: Set-up for temperature dependent dielectric measurement using the cavity perturbation method.

The graph in Fig. 8 shows an example of measurement results obtained for MACOR glass ceramic at temperatures up to 550°C. It gives a rather good agreement to published data that was measured at ambient temperatures. At this point, the measurements results might be considered as preliminary results, which will be repeated with increased accuracy for the system.

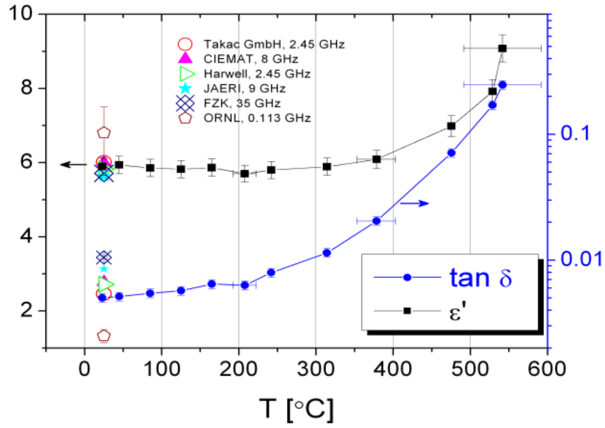

Fig. 8: Dielectric properties for MACOR material. Some previously reported data at roomtemperature are presented for comparison.

II. CONCLUSIONS

The mission of the Institute for Pulsed Power and Microwave Technology (IHM) which is part of the Karlsruhe Institute of Technology (KIT) has been introduced. The presented research and development activities, in the field microwave processing, demonstrate the large potential of this technique for industrial applications. The benefits of selective and volumetric heating with respect to time and energy saving motivates research in various fields of microwave processing. In particular this is true for thermal processing of dielectric materials with low thermal conductivity, such as powders, glasses, polymers, composites.Additionally to this, the application of microwave can have a positive influence on materials properties. It has been shown for the case of microwave sintering of ceramics like zirconia.Mandatory for successful microwave processing is the knowledge about the dielectric properties of the materials involved. Accordingly, different measurement test-sets using transmission line method and cavity perturbation method are currently in use and under permanent development at IHM.

III. REFERENCES

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