

PROCESSING OF (GLASS) FIBER REINFORCED PLASTICS WITH 2.45 GHz MICROWAVES: CURRENT STATE, CHALLENGES, AND PERSPECTIVES

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Abstract

This paper handles four points. First, a very brief introduction into the microwave technologies background is given. This will help understand the challenges when changing from conventional heating to microwave heating. Second, a view on the current state of development in form of a literature sum-up is presented. For this, the literature will be analyzed in respect to the type of publications, equipment used, and the materials investigated. Third, some practical challenges will be described that were faced in microwave process development by the author and how they were overcome. Last, an outlook with potential applications for microwave processing and further needs of development is given.

1. Introduction

The heating of materials with microwave irradiation is common all over the world. In most kitchens microwaves are used to reheat food faster compared to hotplates or ovens. For this, the electromagnetic waves penetrate the material and lead to internal oscillations and friction. Main advantage of this in depth heating is a reduced temperature gradient over the thickness. This direct interaction and in depth heating can be used for composite heating. An especially high potential can be seen for the cure and post cure process of glass fiber reinforced plastics (GFRP): Since the glass fibers are virtually transparent to microwaves, the irradiation can directly heat up the resin over some centimeter thickness. However, only 9 of the 45 publications in the last 28 years known to the author focus on microwaves and GFRP; of this nine only two publications use equipment that allows for industrial-scale manufacturing. When looking at the publications on carbon fiber reinforced plastics (CFRP) the picture looks similar; most extensive investigations were done with laboratory equipment. So, why is it a problem for microwave processing that there is less research on industrial scale? Why would this be important and what are the challenges when going into microwave process development? And, what is the perspective and why should it be done? To answer these questions, this paper will go into four points. First, a very brief introduction into the technologies peculiarities is given. This will help to understand the challenges when changing from conventional heating to microwave heating. Second, a view on the current state of development in form of a literature sum-up is presented. Third, some practical challenges will be described that were faced in microwave process development by the author and how they were overcome. Finally, an outlook on the potential of microwave processing and further needs of development are given.

2. Technical Peculiarities of Microwave Heating

The most important thing to know about microwave heating is its mode of operation. Microwaves are electromagnetic waves that are reflected by metals and conducting materials. The waves penetrate other materials and interact with their molecules. Therefore, microwave heating is not the transport of heat into a material, but rather the conversion of electromagnetic energy to heat. So, what important microwave characteristics do you have to keep in mind when going from known classical heat transfer phenomena (oven) to microwave's heating by interaction?

Microwaves have a "Size"

"Microwaves" is a common name for electromagnetic radiation between 300 MHz and 300 GHz. This frequency-band results in electromagnetic waves of certain length. The wavelength λ_0 in air is defined by

$$\lambda_0 = \frac{c}{f}, \quad (1)$$

where c is the speed of light in vacuum here and f the frequency. The most common frequency, used for kitchen appliances and WiFi, is the 2.4 GHz band. Since this frequency can be used without license and thus without restrictions it is widely distributed. Furthermore, the use in domestic microwaves results in a good and cheap availability of 2.45 GHz microwave sources in form of magnetrons. With equation 1, this commonly used microwaves have a wavelength or "size" of 12.2 cm. In equipment like domestic or industrial microwaves and in normal applications, the electromagnetic waves are constantly or at least regularly fed over the input antenna, reflected at the walls, and absorbed by the load. Since the waves are reflected randomly and not all the energy is instantly absorbed, the waves interfere with each other and a field pattern forms. The distance between high and low energy areas of the field pattern has a similar order of magnitude than the electromagnetic waves. The field is, most likely, not static and changes slightly due to environmental variations. Summarizing, it can be said that microwaves have a certain size and thus resonance effects can occur; the size of a microwave applicator or part may influence the system's behavior.

Interaction has "no" Temperature Limit

Classical heating processes like convection or conduction heating have a build-in temperature limit. For an oven or a hot press you set a temperature. A system with a set temperature of 120 °C heats up the part close to that temperature. For example, the convective heat-up in an oven can be described by

$$\dot{q}_{wall} = \alpha (T_{wall} - T_{\infty}), \quad (2)$$

where q is the area-specific heat flux per time, α is the object specific heat transfer coefficient, and T_{wall} respectively T_{∞} are the temperature of the wall and environment. As can be seen, excluding exothermic reactions, the set oven temperature or T_{∞} defines the target temperature; no further heat-up is possible. For comparison, the possible power (p) that is dissipated per volume (Ad) in a microwave workload is defined by the dielectric heating equation

$$p = 2\pi f \varepsilon_0 \varepsilon'' E_i^2 \cdot [1] \quad (3)$$

Here, π and the permittivity ε_0 are constant, the loss factor ε'' or complex part of the relative permittivity is a material property, and E_i is the inner electric field. The inner electric field depends on the outer electric field i.e. applied microwave power and the real part of the relative permittivity ε' —that is again a material property. In comparison to the convective heat-up, the temperature limit is not a set temperature, but the equilibrium between dissipated power and temperature losses by conduction, convection, and heat radiation. The maximum temperature in microwave heating cannot be directly correlated to a equipment parameter. Summarizing, while the air temperature of an oven sets a Temperature limit such a direct limit does not exist for microwave heating.

Microwave Matter Interaction is Material Sensitive

As was described above, the heat-up under microwave irradiation according to equation 3 depends on the dielectric properties. This dielectric properties are the real part ϵ' and the imaginary part ϵ'' of the relative permittivity

$$\epsilon^* = \epsilon' + i\epsilon'' \quad (4)$$

Both values are material specific, compare Figure 1. Thus, a change in matrix or fiber material would change the heat-up behavior of a part.

Dielectric Properties are Temperature and State Sensitive

In addition to the fact that different materials have different dielectric-properties, the dielectric properties of a material change with its temperature and state. During microwave-matter interaction the oscillation of molecules is encouraged and—simply spoken—the induced friction results in a temperature rise. This induced oscillation strongly depends on the mobility and form of dipoles inside the material. Thus, when the material heats up, the mobility changes and the losses inside the material may rise or fall depending on different mechanisms at work, see Figure 1. Similarly, when a resin undergoes cure and the cross-linkage increases, the mobility is reduced and the dielectric properties—in most circumstances— will decrease.

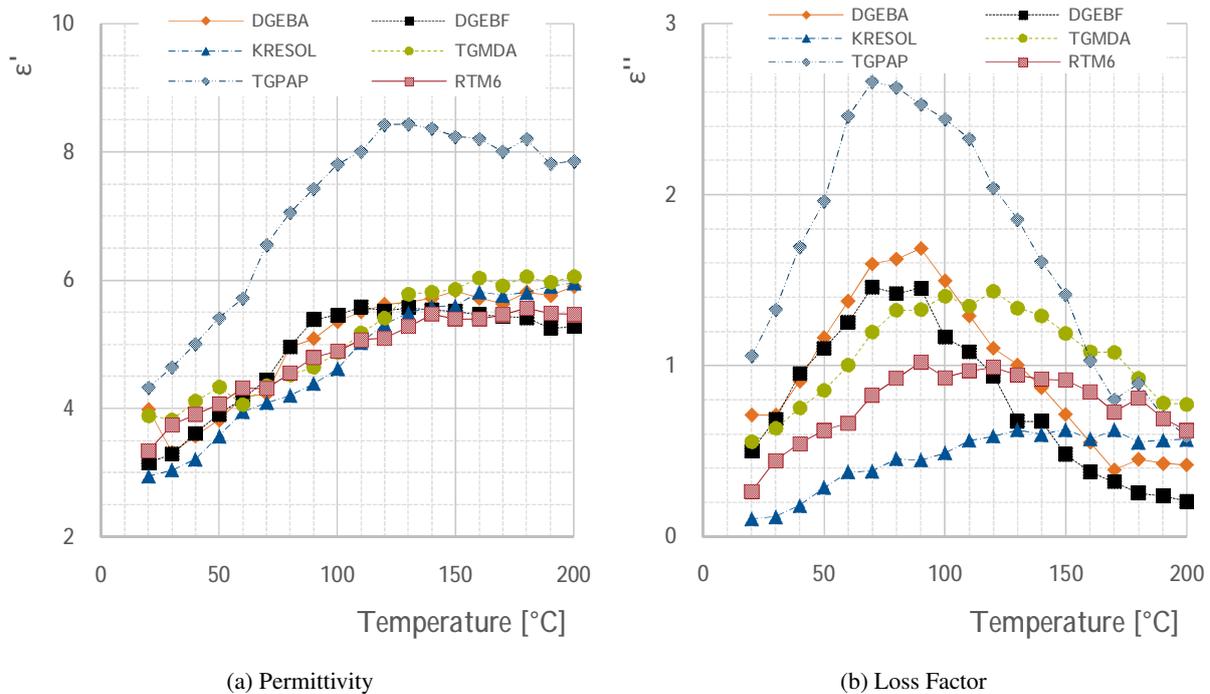


Figure 1: Material and temperature dependency of ϵ' and ϵ'' . (Data from [2])

Microwaves are “Attracted” to Absorbers

Last, high absorbing materials do heat up more than low absorbing materials. To explain the concept and its consequence, we look at three examples. First, we irradiate identical objects sequentially under comparable conditions—position, field strength, environment temperature. They heat up in the same manner since the time of day does not have an influence. Second, we simultaneously introduce two of the objects in our microwave and apply the same conditions; the heat-up changes. Providing we do not have boundary effects, they will both heat-up in an identical way but will heat up slower than a single object. The energy available in the electromagnetic field is “split”. Last, we introduce one of the previous objects and a second one of a similar shape but with high absorption material. The new, high absorption object heats up more than the previous object from earlier trials. Figuratively spoken, it takes up the

bigger part of the energy stored in the electromagnetic-field since it has the higher absorption.

Detailed Microwave Matter Interaction

Detailed descriptions on microwave matter interaction can for example be found in Meredith's book "Engineers' handbook of industrial microwave heating" [1], in Thostenson's Paper "Microwave processing: fundamentals and applications" [3], or—very fundamental—in Metaxas' book "Foundations of electroheat" [4].

3. Literature Sum-Up

The following literature sum-up aims to provide an overview over existing publications in the area of composites and microwaves. The collection presented is comprehensive but—of course—not necessarily complete; it is a list of publications collected by the author over his close to 7 years of work. To get an overview over the existing literature and where we stand in the research of microwave heating of composites, the publications will be divided into different categories. But, where to start and what categories to choose?

As stated above, heating with microwave interaction has some peculiarities. 2.45 GHz microwaves have a wavelength of 12.2 cm and thus field patterns with varying energy input over an area may build-up. The temperature may not easily be controlled and the microwave matter interaction changes over the temperature. For these reasons, measures should be taken to equalize field patterns and an adequate temperature monitoring should be used. Furthermore, since scaling has an effect, the equipment should have a certain size to begin with—also this induces other problems¹. Consequently, the existing literature is exemplary first divided in two; this gives a rough overview over the current state of research. The first category are publications that are theoretical or use small equipment of about 300 mm edge length [5–32]. The second category uses equipment that is much bigger and would be capable to manufacture "industrial sized" parts [2, 33–48], compare Figure 2.

When comparing these categories, 28 of 45 publications—or 62%—were done with small "Lab" equipment; when looking at the reviewed publications, 82% were done with small equipment. The differences in the number of publications and reviewed papers foremost expresses that the research done with "Lab" equipment is more extensive at the moment. This is not surprising for several reasons. Foremost, the first letter on microwave curing of composites was published in 1990 by Boey and Lee [17]. This was 19 years before Gaille presented the first conference paper utilizing an industrial sized microwave for composite manufacturing 9 years ago [43]. Additionally, several reasons can be thought of: bigger equipment is harder to come by, only promising results should be scaled up, and trials on a bigger scale will often be more expensive and time consuming.

More interesting than the amount of publications are the investigated topics. For small "Lab" equipment all but 3 publications focus on materials. About $\frac{1}{3}$ of the works examine epoxies and about $\frac{1}{4}$ of the works examine each GFRP and CFRP. The publications of big "industrial" equipment, on the other hand, do not investigate pure epoxies. While $\frac{1}{2}$ of these investigate materials, mostly CFRP is investigated. The other 50% of the "industrial" publications discuss the process itself and tooling, compare Figure 3 and Table 1.

¹compare Q-factor [1]

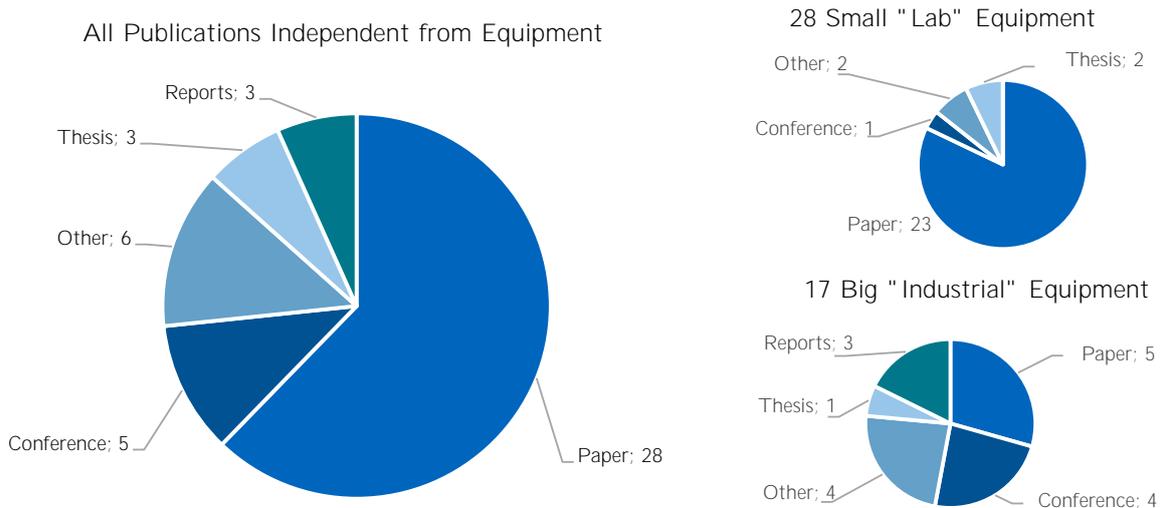


Figure 2: Publications in the area of microwave processing of materials divided into the publication type and split into manufacturing equipment

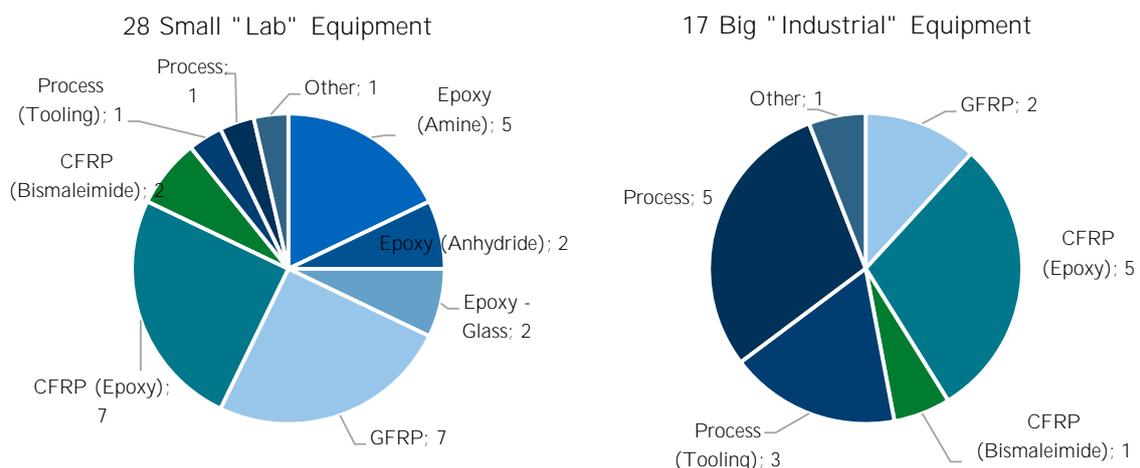


Figure 3: Publications split by the manufacturing equipment used and divided into main topics investigated

4. Practical Challenges

In the following, some practical challenges during microwave processing and the solutions used will be stated. This is meant to give an overview. More detailed descriptions of the process used and the steps taken are available in the authors—open access—publication [40] and will be available in his PhD-thesis [49].

Equipment Configuration

The microwave used for all trials is a *Vötsch Hephaisots VHM 180/200* microwave applicator, see Figure 4. The applicator consists of two 1 m long modules, each of which is equipped with 12 magnetrons of nominal 1 kW each. The magnetrons can be activated individually or in any combination. This provides a nominal power output of 1 to 24 kW. In addition, the power-output can be limited to a range of 10 to 100%. This is arranged by a pulsed operation of the used magnetrons. Consequently, when only one module and thus magnetron is used the minimum power available is 100 Watts. We ideally want to

Table 1: Publications split by the main topics investigated

Material	Publications*	Process	Publication*
Epoxy - Glass	[15, 16]	Process (General)	[2, 33, 36, 41, 42]
Epoxy (Amine)	[5, 10, 17–20]	Process (Setup)	[2, 10, 14, 38, 43, 44]
Epoxy (Anhydride)	[21, 22]		[13, 47, 48]
GFRP (Epoxy)	[23–29, 39, 40]	Process (Simulation)	[10, 31, 36]
CFRP (Epoxy)	[8–14, 34–38, 44, 48]	Process (Tooling)	[2, 13, 32, 36, 38, 45–47]
CFRP (Bismaleimide)	[6, 7, 33]	Process (Winding)	[36, 48]
Phenolic Foam	[38]		

*Small “Lab” Equipment [5–32], Big “Industrial” Equipment [2, 33–48]

use more than one microwave source, however, to get a more chaotic and thus homogeneous field pattern. This leaves us with at least two magnetrons and 200 Watts. For the 300x480x3 mm³ GFRP samples that were manufactured by the author, this is too much power to get a stable temperature of 85 °C; the microwave controller works at his lower limit and the process gets unstable. This divergence between available and needed power was solved by increasing the needed power by an additional water-load. For this, a tube with was introduced into the back of the microwave chamber and connected to a faucet.

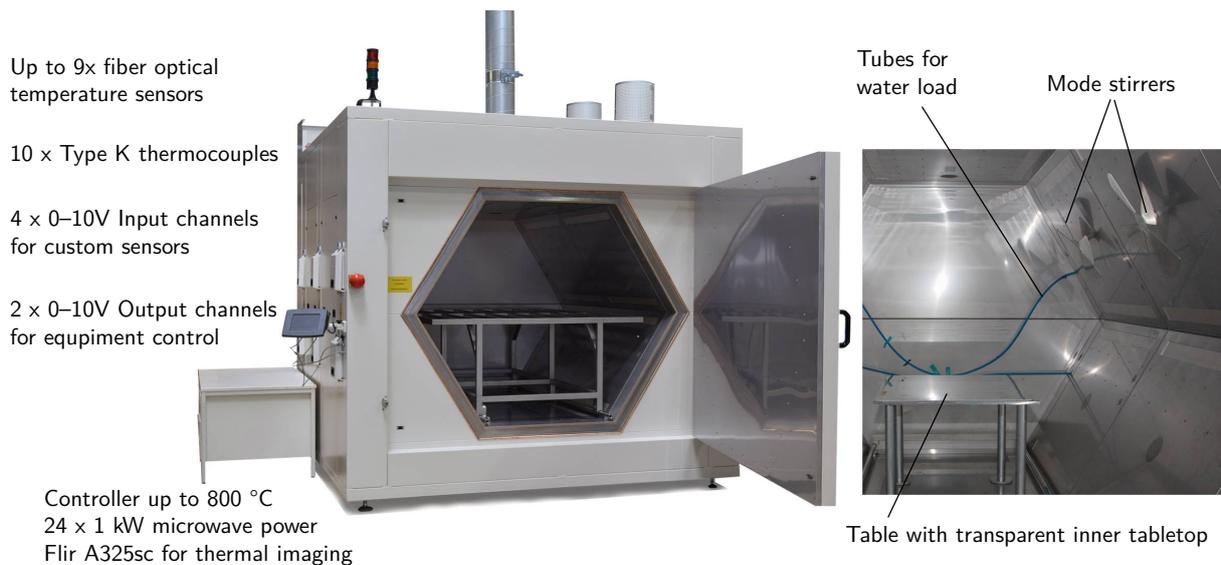


Figure 4: *Vötsch Hephaistos 180/200* with its capabilities and additions. The hexagon has a outer diameter of 1.8 m and a depth of 2 m.

Field Homogeneity

According to the developers of the Hephaistos system, its hexagonal shape improves the field homogeneity [50, 51]. The homogenization by the geometry alone, however, was not good enough for the processing of small GFRP plates on a transparent tool. Trials with a black natural rubber sheet on a transparent tool show that three measures significantly increase the temperature homogeneity. A regular change of the active magnetrons, the use of more magnetrons on a lower%-power level, and the addition of mode stirrers all increase the temperature homogeneity in a constant test setup, compare Figure 5. The complete homogeneity study will soon be published in the authors PhD-thesis [49].

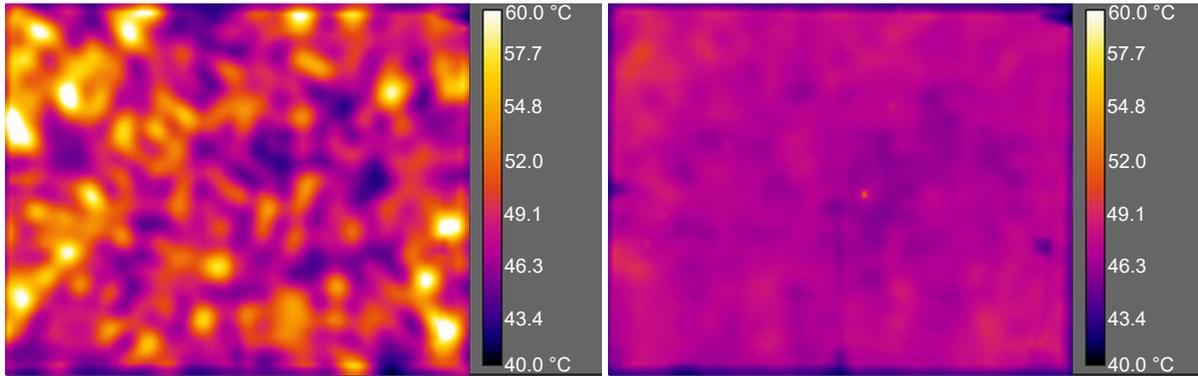


Figure 5: Thermal camera images of different system configurations taken during an homogeneity study.

Process Control

Due to the technical peculiarities of microwave heating, the process control is complex and sensitive to the set-up. An absorbing material heats up directly while the environment stays cold; if you stop the energy input, the material will cool down fast since the environment is cold. Consequently, the microwave power is constantly controlled to achieve a stable ratio between energy-input through microwave irradiation and cool-down due to heat transfer effects. However, the material properties and energy input change with temperature and degree of cure and the heat transfer effects increase with temperature. To reach a stable process, the control-parameters have to be adapted to the set-up. The influence of PID-control parameter optimizations can be seen in 6.

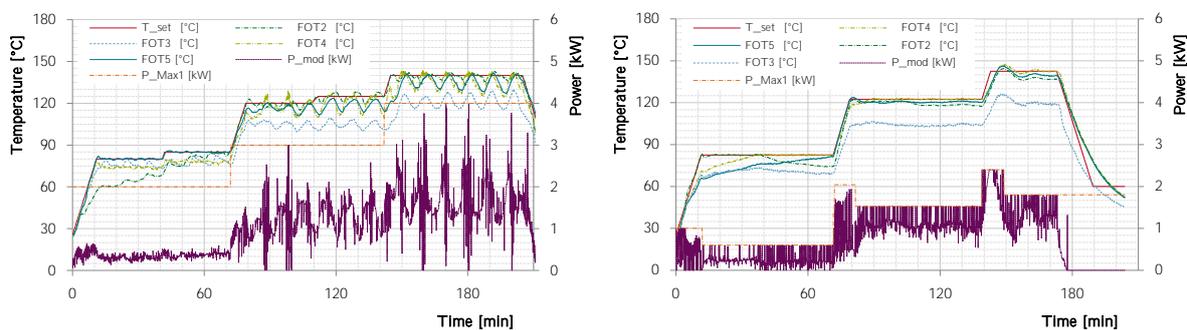


Figure 6: **Left:** early stage of process with water load but without mode stirrers i.e. slowly turning metallic “fans” that reflect the microwaves or adapted control parameters. Strong control oscillations occur. **Right:** adapted process with water load, mode stirrers and adapted control parameters. Control oscillations are minimized.

Environmental Influence

A carbon-fiber-free environment is necessary to manufacture GFRP samples on a transparent tool with no additional absorbers; the environment used by the author—compare Figure 7—was not always clean enough to achieve this. Samples up to 120 °C were manufactured without problems. However, at higher temperatures more power is needed. Through this, some burnings of GFRP plates occurred that were traced back by their appearance to entrapped carbon-fibers. The introduction of the water-load and the optimization of the PID-control parameters seemed to be beneficial; less burnings occurred. It is very reasonable that the water-load has a positive influence; it behaves like a damper. The influence of the optimized PID-control parameters, however, could be arbitrary. No extensive study was undertaken to investigate this observation. It must be said, that the set-up used so far is very sensitive to carbon-fiber contaminations. Larger or thicker samples or an absorbing tool would reduce the risk of carbon-fiber

burning. Both variations increase the energy-to-heat conversion. Therefore, a lower electromagnetic field would reduce the carbon-fiber heat-up.



Figure 7: **Left and middle:** carbon-fiber-free environment used for GFRP preforming.
Right: burning at high temperature trials due to an entrapped carbon fiber

5. Outlook

As we have seen, microwave heating has some peculiarities. Microwaves have a specific wave length, you cannot set a temperature limit, the microwave–matter interaction is material sensitive, and the material properties change over the temperature. These peculiarities make microwave processing much more complex than conventional processing. So, what benefits can be achieved? Is it worth the effort? And what has to be done?

The possible benefits of microwave heating are manifold. Most of them are a direct consequence of microwave's peculiarities. Here are some Examples:

- Microwave heating is a direct interaction. Microwaves do penetrate GFRP and heat it over its depth. This way, you have no need of heat-through or dwell times and can work with ramps only. This can drastically reduce cycle-time and even improve material properties [40].
- Microwave heating is material sensitive and there is no temperature limit. Therefore, one could heat a part or even only a specific area of a part to a high temperatures—say above 400 °C—without the need to equip the microwave itself to withstand this temperatures. The environment remains "cold".
- Microwave–matter interaction is material sensitive. Through this, different temperatures are achieved when different materials are processed at the same time. What about processing a flexible elastomer with a very low T_g and curing temperature with a high T_g epoxy material to form an integrated impact or exhaust area?
- Microwave–matter interaction is material sensitive and the material properties change over the temperature. This could be used to tailor a material with a shut-off temperature. For example, through the use of ferromagnetic material with a low curie temperature. Combine this with a snap-curing resin and $>20\text{ }^\circ\text{C}/\text{min}$ heating rates and you get extremely short variable curing processes.

If the above-mentioned advantages of microwave heating can be put into practice, it is highly likely that there will be applications for this kind of technology in the future. However, a very stable process must be achieved first. For this, tools must be available to "counteract" the microwave peculiarities where they are obstructive. For example, to heat a complex part with different materials homogeneously to

a set temperature. Furthermore, a knowledge base and rules must be available so that process design gets easier and faster. For this, simulation models that take microwave-matter interaction into account could be used. Through the complexity and the current state of development, microwave processing of composites is very limited at the moment. However, if the technology readiness level of microwave curing can be increased, there will be applications such as multi-temperature curing, snap curing, or local pre-curing to maintain an uncured bonding area.

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