

MICROWAVE TECHNOLOGIES FOR COMPOSITE PROCESSING

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Summary

The application of microwave heating is examined for the production of carbon fiber-reinforced polymer components. Three methods for the thermal treatment of the composite polymers are identified. The preconditioning of the resin at the laboratory level in preparation for an ensuing injection is examined as well as the preforming of bonded, semi-finished materials and the curing of components. The suitability of the microwave process is demonstrated by representative material parameters. These input data are used to implement the technology with industrial standards, which is demonstrated by a resin heating device and a microwave autoclave.

Microwave Technology for the CFRP Production

In view of increasing industrial production, the optimization of current processes is necessary for the manufacture of carbon fiber-reinforced plastics. In addition to shortening cycle times, a quality-assured production process is of primary importance in aerospace applications.

The use of electromagnetic radiation, particularly microwaves, to heat up products has been known for a long time. The heating of products using microwaves is characterized by a selctivly efficient introduction of heat into the product, which shortens the entire process and reduces the necessary energy expenditure.

Heating carbon-fiber reinforced plastics with microwaves is not yet a very common process. Because of its many thermal process steps, the process chain for the manufacture of CFRP components is suitable for examining the application of microwaves. Due to the material's low thermal conductivity, the thermal process steps are a bottle neck in the manufacture of components. An optimization of the process, particularly in aerospace-related applications, is necessary due to the use of high-temperature duroplasts. In addition too the curing of structures, additional areas of application are also the preforming of bonded, semifinished materials and the preconditioning of resins.

The use of a universal microwave oven with a working frequency of 2.45GHz gave rise to a process technology that has advantages compared to the state of the art.

The prospect arises of applying the microwave heating process of CFRP materials also to fiberreinforced thermoplastics and low-temperature thermosets. The focus of the work is on the development of the processes used in the field of aerospace. In a first step in the microwave heating process for the preconditioning of resin, the preforming and component curing processes are developed using already qualified molds and processing tools. Pre-gualified processing tools are used for the identification of a possible influence of the microwaves on the material. In order to examine this more closely, material samples are made and parameters are examined that are strongly dependent on the temperature of the material. Mechanical as well as thermal tests are carried out.

Heating up materials using microwaves is also called dielectric heating [1]. Compared to conventional heating methods by means of forced convection, a supporting medium like air, as used in many industrial processes, is not necessary to heat products. The microwaves interact directly with the product to be heated, making the heating a selective process and keeping the surrounding area cold. Carbon fiber reinforced-plastics are heated by the induction of eddy currents via the so-called skin effect [10] in which is heating occurs via the fiber and the orientation polarization of the matrix. The microwaves penetrate the material, which is why the process is called volumetric heating.

Microwave heating has already found its way in the manufacture of CFRP and fiber glass-reinforced structures at the laboratory level [7-9]. Statements on this method describe an influence of microwave heating on the material properties of the matrix and composite. However, it must be noted that the method described here was carried out under nonconstant circumstances, which is why these results remain inconclusive. What all methods have in common is the application of microwaves to speed up the process. Processes with constant boundary conditions seem to particularly advantageous for the application of microwave heating. This is the reason why the continuous flow-heating used during infiltration [11] to decrease the viscosity was examined. Further tests are based on a comparison of conventional with microwave-based methods in which, among other things, the material properties dominated by the matrix are examined more closely (2-5). The results presented there come from an unadapted technology. For this reason, the material samples produced by microwaves have a lower material performance than their conventional counterparts. When manufacturing CFRP components with microwaves, the boundary conditions that result from the physics of microwaves must be taken into consideration. The potential advantages of microwave heating can only be achieved with an adapted technology.

Particular advantages come about within the process due to this particular form of heat. The introduction of the temperature at the component is directly influenced by the emission of microwaves, which enables a very precise tempering of the component. Direct heating of the material via microwaves eliminates dead times and inertia during the process that were previously unavoidable due to the heating of molds and atmospheres. The introduction of temperature directly at the component has advantages regarding guality assurance since the reaction time in the process regulation is low due to the cold oven atmosphere. Minimizing the inertia in the process can lead to a maximizing of heating gradients within the manufacturing process, which saves time.

In addition to the skin effect, the polarization properties of a material, which are characterized by a complex relative permittivity, are particularly decisive for with microwave heating [2].

 $\varepsilon = \varepsilon' + i\varepsilon'' \quad (1)$

- ϵ := complex relative permittivity
- ε ' := real part (stored share)
- ε " := imaginary part (share of loss)

Molecules with a dipolar character are aligned in the alternating electric fields of the microwaves. During this so-called polarization, heat occurs due to loss mechanisms in the material. A permanent dipole is present as long as the centers of charging of the negatively loaded electrons of the molecules do not coincide with the positively loaded center of charging of the atomic core. The dipoles in an alternating field are aligned out of place. This phase displacement is described with the loss angle and has the effect of a thermal loss. This effect is exploited for heating with microwaves. The dielectric loss factor $tan\delta$ is the measure for this phase displacement and the argument for the complex dielectricity number [3].

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'}$$
 (2)

The dielectric loss factor is the operating parameter for polymers to test their suitability for microwave heating.

The properties necessary to heat the material depend on influencing factors. Viscositv. temperature and degree of polymerization are some of the factors for duromeres. Alignment of the molecules in the alternating field is better feasible with declining viscosity than when in a solid condition so that, in the end, the entire molecule is aligned in the direction of the alternating field since the dipoles are locally set on the molecule. The viscosity of the matrix system therefore also influences the dielectric loss and, with that, the complex relative permittivity. The effect described here is called a relaxation. The relaxation was described for the first time through the Debye model. In this model relaxation times of the dipoles τ_d occur that describe the alignment time frames of the individual dipoles. Therefore, a complete relaxation is possible as long as the imaginary share of the dielectric number reaches a maximum and the loss angle is maximized as well [1]. The following applies:

$$\varepsilon' = \varepsilon_U + \frac{\varepsilon_R - \varepsilon_U}{1 + (\omega \tau_d)^2}$$
(3)

$$\varepsilon'' = \frac{\sigma}{\omega\varepsilon_0} + \frac{(\varepsilon_R - \varepsilon_U)\omega\tau_d}{1 + (\omega\tau_d)^2}$$
(4)

 ε_0 := electrical field constant

ε_r := relaxed dielectric number

 ϵ_u := unrelaxed dielectric number

 ϵ_{d} := relaxation time of the dipoles

 σ := conductivity

The frequency of the alternating field is an additional factor that influences the relaxation. The viscosity primarily has an influence on the mobility of the load carrier but if the frequency of the alternating fields is located in the area of the rotation frequency of the dipoles, the polarization declines since the dipoles no longer reach the orientation distribution.



If a lossy dielectricum is exposed to an alternating field, the electromagnetic wave penetrates the dielectricum but is damped in the process. Energy is released and a term is defined for the performance difference ΔP [5,6].

$$\Delta \mathbf{P} = \mathbf{P}(1 - \mathbf{e}^{-2a\mathbf{x}}) \tag{5}$$

a := damping constants

x := path in the field direction of the dielectricum

The damping constant a is given by:

$$a = \omega \sqrt{\frac{\mu' \varepsilon'}{2} \sqrt{(1 + \frac{\chi^2}{\omega^2 \varepsilon'^2}) - 1}}$$
(6)

 $\begin{array}{ll} \mu' & := \mbox{ real part of the complex permeability} \\ \chi & := \mbox{ conductivity of the dielectric} \end{array}$

The conductivity of the dielectric results from the imaginary part of the complex dielectric constants [4].

$$\chi = \omega \varepsilon_0 \cdot \varepsilon'' \tag{7}$$

The depth of penetration in a dielectricum, however, is relevant for the processing. The penetration depth is defined as the depth with which the performance difference of 1/e was realized. It is also dependent on the wave length of the alternating electrical field and shows that the penetration depth increases with decreasing frequency but also that the released heat decreases in the dielectricum. The wave length of the microwave of 2.45 GHz is a good compromise.

$$\upsilon = \frac{\lambda_0}{2\pi\sqrt{\varepsilon''}\tan\delta} \tag{8}$$

The penetration depth of the microwave in the product to be heated is a measure for an even distribution of the heat in relation to the wall thickness of the component. If a component has a greater wall thickness than the penetration depth of the microwave in the component material, the influence of the inner heat conduction in the component increases again.

Here it becomes clear that the process and methods can only be successfully carried out if certain dielectric boundary conditions are taken into consideration. The dielectrically constant surface, i.e. a surface that is evenly heated in dependence on the field distribution is particularly necessary for CFRP with its very dielectrically different output components. Excess temperatures that cannot be measured are critical in a microwave process. These excess temperatures are caused by "hotspots" and "arcings".

Hotspots are the result of an inhomogeneous field distribution in the process area and also as the result of materials with different dielectric properties. Based on the carbon fiber and a matrix, the matrix is more strongly heated in the microwave field than the fiber. This is the reason why the accumulations of resin, so-called resin-rich volumes, have to be avoided at the component since these areas cause a momentum that can damage the component.

Arcings are electric arcs caused by high local potential differences.

Basic Laboratory Research

Basic research takes place in a 1m³ universal microwave oven, as seen in Fig. 1. The microwave oven has an overall performance of 8kW and is a device that is controlled by the temperature. Three pyrometers and four fiber-optical thermocouples are available for temperature measurement. Microwave-specific process parameters are recorded by a directional coupler that measures the emitted and reflected microwave performance and therefore enabling an assessment of the effectiveness of the microwave process.



Fig. 1 Universal microwave oven 2.45GHz 8kW

The use of microwave heating is applicable for the process steps of the preforming, the preconditioning of the resin and for the curing of components. Components cured with microwave



heating takes place for prepregs as well as in the injection process. Because of the laboratory furnishing with a multi-mode-atmosphere microwave oven, the injection method without using an autoclave is first looked at here.

The injection method for fiber-reinforced composite components is the state of the art for many components. The injection of a matrix system depends on its viscosity. The lower the viscosity, the easier it is to carry out an injection process and the duration of the injection is shorter as well. The viscosity of a matrix system depends on the temperature and degree of polymerization whereby the degree of polymerization, in turn, depends on the temperature and time.

For this reason, the resin is heated to a pre-defined temperature in preparation for an enusing injection in order to obtain optimum properties with regard to the flow characteristics and processing duration. The heating of individual resin containers greatly depends on the shape of the container and takes a lot of time with today's level of technology. Batches ranging from 10 to 20 kg are common in aerospace applications.

Due to the low inner heat conduction of resin system the containers are stored in a preheated oven for up to four hours. It takes this long for the entire container to be adequately and evenly heated. It is hardly possible to shorten the process with today's technology. A too high temperature of the oven in which the container is heated results in an inhomogeneous heating of the container. Such high temperatures on the container walls could inevitably result in an exothermal reaction of the system. This excessive temperature cannot be compensated by inner heat conduction of the container. The temperature curves depicted in Fig. 2 are a reference point measurement that was carried out in a standard 10kg container. Heating took place in a convection oven that was pre-heated to 80°C. An even temperature can be attained in the entire container only after 8 hours at a constant temperature, which is not acceptable for industrial application.

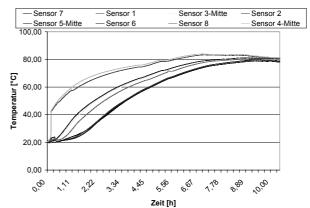
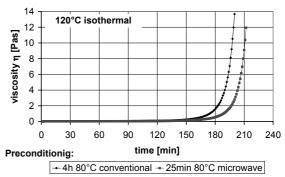


Fig. 2 Conventional heating of a resin container

Still, the heating process needs to be shortened in order to guarantee the availability of injectable resin and to provide a degree of flexibility in the production process. The disadvantage of heating with today's technology, in addition to the availability, is the reduced processing time for the resin system.

One way to shorten the heating process is by using microwaves. The dielectric properties of epoxy resins make them particularly suitable for being heated with microwaves.

In order to verify the suitability of microwave heating for the preconditioning of epoxy resin, RTM6 from Hexcel in this case, a test based on a conventional processing method was carried out. Since in this case only the matrix did not undergo a qualified process, the material parameters qualified by the matrix have to be determined. The remaining enthalpy, viscosity, shear strength and shear module are selected. Material samples are made for this purpose that are cured under identical conditions. The only difference between the samples is the pre-treatment of the matrix. One is conventionally heated in a convection oven for 4 hours and the other by microwaves for 25 minutes. The temperature of both matrix systems is 80°C after being heated up.

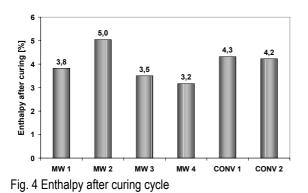




The rheological analysis in Fig. 3 clearly shows an uncritical influence of the microwaves on the

viscosity. The viscosity of the matrix system should be reduced as much as possible for an injection following the heating of the resin. Due to the low rise in viscosity, RTM6 heated with microwaves is just as suited for the injection process as conventionally heated RTM6. The time frame necessary to carry out an injection is increased with microwave-heated resin.

In order to examine the influence on the degree of polymerization and remaining enthalpy, RTM6 preheated with microwaves and conventionally preheated RTM6 are cured at 180°C and then evaluated by means of a DSC analysis. The curing of individual samples takes place under the same conditions so that the only difference in the process is the pre-heating of the matrix. As seen in Fig. 4, there is no significant influence of the microwave heating on the RTM6 resin system on the enthalpy remaining in the system. The measured remaining enthalpy is at the same level as the reference samples (conv1, conv2).



A possible influence of the chemical structure of the resin system, which has an effect on the boundary layer between the fiber and matrix, is examined on the basis of the shear strength and shear module of a conventionally cured composite. In order to research the chemical bonding properties of the matrix on the carbon fiber, the shear properties of a composite component are examined according to an internal norm from Airbus: AITM 1.0007. The component is manufactured by using the microwave resin heating (MW) and a reference (REF) heated with the currently used conventional heating method. The individual material samples are cured under identical conditions. The test is carried out with a HTA 6K fiber that is weaved to a fabric. The results shown in Fig. 5 show no direct influence of the microwave heating neither on the shear strength nor on the shear module, which is not shown here. Neither can a change be determined in the structure

of the matrix using an infrared spectroscopy, which is not shown here either.

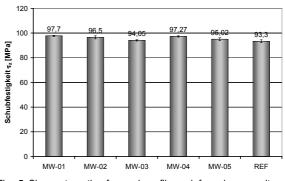


Fig. 5 Shear strength of a carbon fiber-reinforced composite with a 57% fiber volume content

This is the evidence that heating RTM6 with microwaves is a suitable method. This method saves a considerable amount of processing time, which is necessary for the preparation of individual resin batches.

The amount of process time saved in the preforming process is also examined.

Preforming using the binding technology is also possible with the microwave heating process in order to minimize the effect of material-specific properties like poor heat conduction in the dry fiber preform. In the binding technology, the fiber plies and weaves are partially impregnated with a thermoset or thermoplastic polymer. Impregnation takes place with polymer non-woven materials, polymer powder or filaments. The binder melts or connects by means of a thermal activation. The matrix system binds the binder as a component in the chemical structure or the elements physically remain in the composite.

Today, fiber plies are thermally activated with conventional heating methods. The most simple method is the use of heating plates or irons with which the semi-finished fiber is treated and draped in a mold. Other methods make use of convection ovens and infrared facilities in which flexible membranes are used. The membranes press the semifinished fiber into the desired contour using a vacuum and activation takes places with an additional heating device by means of convection and heat radiation. This way it is possible to lay down single or several fiber plies at the same time. However, the introduction of heat into the product is not very efficient. The heating is directly related to the temperature of the convection oven, which passively heats the fibers and binder. It is necessary to heat the oven atmosphere and the mold, which is why only a fraction of the energy can



be used to heat the product. This is where the microwave technology provides a solution since the carbon fibers are selectively heated. Heating takes place solely in the preform and not in the periphery. However, integrating microwave heating into the preform process is not a trivial matter since the requirements of the process including the precise molding of the fiber preform and the application of the vacuum deformation must be taken into consideration. An at least one-sided transparency of the mold must be guaranteed in microwave heating so that the microwaves can penetrate the product.

In addition to the process technology requirements, the influence of the microwave heating on the fibers themselves needs to be researched. The fiber being researched here is a HTA 6K fiber that is woven to a Cramer 445 fabric. A possible effect of the microwave heating on the boundary layer between the matrix and fiber needs to be researched that, among other things, is influenced by the size of the fibers. In addition, the tensile strength of the fiber is examined in order to eliminate any structural changes in the fiber.

The basic suitability of microwave heating for the preform process is shown in the following work. The process is reproduced with a simple setup. The setup consists of 8 plies of carbon fiber-reinforced fabric and is symmetrically laid down on an aluminium sheet. The fibers are consolidated over a vacuum with the aid of a membrane and then heated with microwaves. The preform process takes 20 minutes. Fig. 6 shows the influence of the microwave heating on the stiffness of the fiber or the structure of the sizing. The values derived from microwave preforming are compared with those derived from conventional methods such as the ironing of individual fiber plies and a conventional convection oven. Microwave heating was carried out at 2 temperature levels. The fibers were heated up to 90°C in the first test and to 120°C in the second. This, however, only had an influence on the haptics of the of the dry preform that, after treatment at 120°C, showed greater stiffness than the other preforms. But it did not have a severe influence on the mechanical parameters. The results provided here were determined according to an AITM and statistically secured. Microwave heating is appropriate for the preforming. The advantages of this process are savings in time and energy. The reference process took 30 to 50 minutes in order to achieve the same degree of strength as with dry fiber preforms.

The tension and shear properties are partially shown in Fig. 7 and 8. Even in additional tests no

influence of microwave heating on the mechanical properties of the composite was determined.

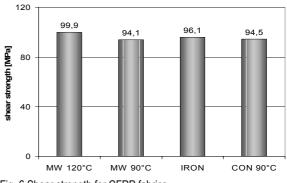


Fig. 6 Shear strength for CFRP fabrics

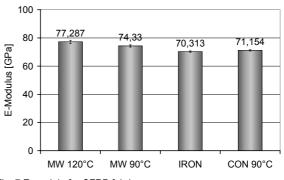


Fig. 7 E-module for CFRP fabrics

This proves that the microwave heating of individual carbon fiber-reinforced polymer components is a suitable process. These results now need to be applied to the manufacture of CFRP components. The curing of structures via microwave heating remains to be tested at the end of the thermal manufacturing chain of CFRP structures.

The effect of selective and volumetric heating is also exploited in the curing of composite structures. Injected components as well as prepreg structures can be cured in a microwave field. However, only the curing of injected components is explained in the following since the basic laboratory equipment consists of an atmospheric pressure microwave oven. Both the dielectric properties of prepregs and an injected fiber setup are similar.

The process chain for the manufacture of a carbon fiber-reinforced composite structure ends with the curing of structures. The curing is the most demanding process since the highest temperatures are generated at low tolerances in connection with the vacuum strength of the production setup. Knowledge of the distribution of the temperature over the component is necessary to guarantee the quality of the component later on. The maximum temperature generated in the process is 180°C. The height of the temperature generated by microwaves depends on the power density of a microwave chamber that



chamber that occurs from the quotient of maximum microwave performance and chamber volume. The power density was 8 kW/m³ in the following tests. In addition to the maximum temperature, the homogenous distribution of the temperature over the component surface and in the component volume is decisive. The temperature distribution is dependent on an appropriate coupling of the microwaves in the system, i.e. the number of microwave sources and the antenna form. The facility used for the tests is equipped with slotted wave guide antenna. A homogenous temperature field is also necessary for the process control since the microwave process is one controlled by the temperature. The maximum measured temperature is compared with the setpoint value and the microwave performance is accordingly emitted. The temperature measurement technology is integrated into the processing tool in accordance with a series process.

Figure 8 shows an example of a temperature curve. The temperature is recorded at two measurement areas. Fluctuations in temperature are caused by the inertia of the temperature measurement in the tool and not by the microwave facility. The temperature distribution in the entire tool is known from pretests. The critical areas are selected as measurement points for the process control in order to achieve a sufficient temperature distribution.

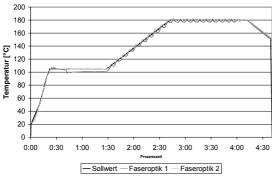


Fig. 8 Temperature distribution in a microwave process

Knowledge of a possible influence of the microwave heating on the material is necessary in order to acheive a qualified curing of carbon fiber-reinforced polymers. After the processing setup is adapted to a secure processing in the microwave field, a standard curing cycle for the RTM6 resin system from Hexcel is carried out first. The influence of shortening the curing time and of faster processing by increasing the heating gradients are researched within the processing parameters prescribed within the resin system. The parameters to examine the microwave process are the curing times and the heating rate. Curing at 180°C is shortened from 90min to 60min for the MW sample (1hour at 180°C) and the heating rate of 1°C/min is maintained. The MW sample (3°C per min) is heated to 3°C/min and curing is maintained at 180°C/90min. Despite the faster processing, there are no considerable changes in the shear properties compared to the microwave reference process and autoclave reference (Fig. 10). The finished samples are different only in the manufacturing process and identical with regard to fiber orientation and fiber volume content.

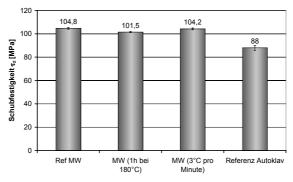


Fig. 9 Shear strength with material samples cured by microwaves

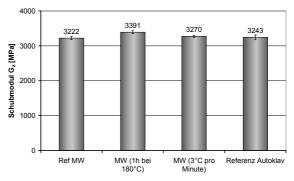


Fig. 10 Shear module with material samples cured by microwaves

The enthalpy remaining in the system that is characteristic for the degree of curing, Fig. 11, is measured by means of a differential scanning calorimetry (DSC). The enthalpy remaining in the system is lower for all microwave processes than for conventional references. Accordingly, the degree of cross-linkage of the samples cured with microwaves is higher than the conventional reference. The glass transitional temperature for the samples cured with microwaves is correspondingly higher than the conventional reference.



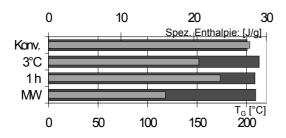


Fig. 11 DSC Test for cured structures; specific enthalpy in the foreground; glass transition temperature $T_{\rm G}$ in the background

This, however, is not a specific effect that can be ascribed to the use of microwaves but rather the direct temperature control at the component itself is responsible for the improved material performance. If the microwave process shown in Fig. 8 is compared with the temperature control in Fig. 12, it becomes clear that the conventional process is limited by the thermal inertia.

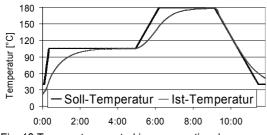


Fig. 12 Temperature control in a conventional process

The processing of carbon fiber-reinforced polymer components using microwave heating is possible for the process presented here. Microwave heating does not negatively influence the material properties of the composite later on. The process is shortened in either case with a precise control of the temperature and a direct heating of the material. The potential of the microwave process has not been exhausted since the curing of the composite fiber components and the preforming have not yet been implemented in the industry. However, the primary goals of saving processing time and energy are being met.

Implementation in the industry

Exhaustion of the full potential microwave heating has to offer is only possible with an adapted facility technology. The effect described above can even be increased with the use of a special facility. The facility depicted in Fig. 13 was especially developed for the heating of RTM6.

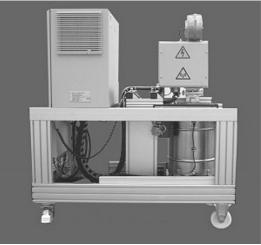


Fig. 13 Microwave resin warming facility

The facility warms 10kg of RTM6 from room temperature to 80°C within 12 minutes. Compared to the temperature distribution shown in Fig. 2, the temperature distribution in Fig. 14 clearly demonstrates the performance spectrum of the facility. The temperature distribution within the container is sufficiently homogenous so that it can be assumed that the viscosity of the entire container is constant.

The temperature in the container was maintained for over 8 hours to demonstrate the stability and precision of the process in the facility.

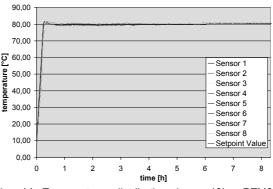


Fig. 14 Temperature distribution in a 10kg RTM6 container on several levels

The application of the component curing is looked at more closely in the following. In view of CFRP structures that are constantly increasing in size, the processing facilities also have to be adapted to the requirements of the components. The state-of-theart autoclave technology for the manufacture of prepreg components will most likely not be dismissed in the next few years. The energy expenditure required for the operation of an autoclave is enormous. The facilities used today have a loading diameter of more than 8 meters and a length of over 30 meters.



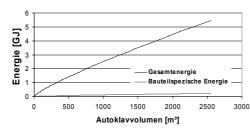


Fig. 15 Energy results of a convection oven

Looking at Fig. 15 it becomes clear that only a minimum amount of energy expended in the process is actually used for the component itself. Due to selective heating, microwave heating has the advantage of saving energy and therefore operating costs. For this reason, Scholz Maschinenbau and the German Aerospace Center DLR / Institute of Composite Structures and Adaptive Systems have entered into a cooperation agreement for the development of a microwave autoclave at the industrial level.

This comprises launching the microwaves in the pressure area of the autoclave as well as the development of a cost-effective temperature measurement system. The design of a microwave autoclave is depicted in Fig. 16. A microwave system can be upgraded for an existing facility but is not discussed in more detail here.

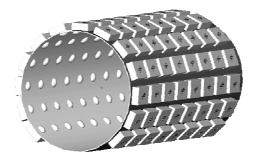


Fig. 16 View of a microwave autoclave

At present, the plant technology, Fig. 17, is being run-in in a first step and typical patterns of the facility are being tested.



Fig. 17 Microwave autoclave at DLR/FA in the test phase

Acknowledgements

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