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Emerging Microwave-Plasma Technologies for Chemical Processes

Javier F. de la Fuente[1], Anton A. Kiss[2,3], Marilena T. Radoiu[4], Georgios D. Stefanidis[5*]

[1] Process & Energy Laboratory, Delft University of Technology, Leeghwaterstraat 39, 2628 CB, Delft, The Netherlands
[2] AkzoNobel – Supply Chain, Research & Development, Process Technology SRG, Zutphenseweg 10, 7418 AJ Deventer, The Netherlands
[3] Sustainable Process Technology Group, Faculty of Science and Technology, University of Twente, PO Box 217, 7500 AE, Enschede, The Netherlands
[4] Independent consultant
[5] Chemical Engineering Department, Katholieke Universiteit Leuven, Celestijnenlaan 200f, 3001 Leuven (Heverlee), Belgium

Abstract

Microwave plasma (MWP) technology is currently being used in application fields such as semiconductor and material processing, diamond film deposition and waste remediation. Specific advantages of the technology include the enablement of a high energy density source and a highly reactive medium, the operational flexibility, the fast response time to inlet variations and the low maintenance costs. These aspects make MWP a promising alternative technology to conventional thermal chemical reactors provided that certain technical and operational challenges related to scalability are overcome. Herein, an overview of state-of-the-art applications of MWP in chemical processing is presented (e.g. stripping of photo resist, UV-disinfection, waste gas treatment, plasma reforming, methane coupling to olefins, coal/biomass/waste pyrolysis/gasification and CO_2 conversion). In addition, two potential approaches to tackle scalability limitations are described, namely the development of a single unit microwave generator with high output power (> 100 kW), and the coupling of multiple microwave generators with a single reactor chamber. Finally, the fundamental and engineering challenges to enable profitable implementation of the MWP technology at large scale are discussed.

1. Introduction

A sustainable and green economy represents one of the major challenges of contemporary society. It involves mostly the reduction of waste generation, but also the optimization of raw material consumption in order to mitigate current alarming pollution problems, and lower the energy requirements of industrial conversion processes. For the chemical industry to progress towards a sustainable economy, novel waste-to-product approaches need to be developed to reduce the dependency on fossil fuels-based raw materials. Carbon Capture & Utilization (CCU) is an emerging concept, which utilizes waste (e.g. greenhouse gases) as chemical feedstock to produce valuable products[1]. In most cases, however, the required energy input to transform waste into products tends to be rather high, making the re-utilization process unprofitable. Renewable energy sources, such as wind and solar power, are expected to have an increasing share in the future energy scene – that is, a large fraction of the energy needed for chemical conversion processes can be obtained from these sources during peak electricity production periods. This falls within the so-called power-to-chemicals approach[2], whereby greenhouse gases and/or water are converted into hydrocarbons by means of surplus electric power.

In this context, plasma reactors represent a novel alternative technology due to certain processing benefits such as: fast process dynamics, process flexibility, no need for catalyst use in many processes, no need for (bulky and costly) gas-fired furnaces, low maintenance cost, and high quality products (low byproducts formation). In this work, we focus on microwave plasma (MWP) assisted reactors, which appear to be one of the most promising plasma reactor types. The principal advantage of MWP over other discharges is that it does not require electrodes, which must be placed inside the reactor leading to operational issues, such as regular maintenance (replacement) due to erosion[3]. The high frequencies at which MWP systems are operated produce a large fraction of electrons, i.e. high electron densities, and high electron temperature (energy) compared to other plasma sources. This results in high concentrations of active species rendering MWP an ideal highly reactive medium for chemical reactions.

Here we present the state-of-the-art of MWP at laboratory stage, existing industrial chemical applications, current technical and operational limitations, and an overview of the fundamental and engineering challenges for further development of the MWP technology. Finally, some promising potential applications, which mainly concern high temperature processes, such as pyrolysis, gasification and reforming of organic waste, biomass and fossil fuels are discussed. Overall, this paper intends to set up a roadmap describing the main requirements and the next steps needed for the implementation of the MWP technology in the chemical manufacturing industry.

2. MWP technology: State-of-the-art

2.1. Laboratory stage

A remarkably broad range of chemical applications at laboratory-scale have been explored in different MWP systems with the exception of biomass gasification for syngas production, which has been investigated both at laboratory scale[4] and in medium-scale plants[5,6]. Concerning chemical manufacturing applications, several processes are highlighted due to the large number of research studies (see Table 2 in Ref. [7]). These include plasma-assisted reforming, biomass gasification and pyrolysis, H_2 production, and CO_2 utilization. Figure 1 shows an atmospheric air microwave plasma at different values of net input power used for biomass gasification purposes at Delft University of Technology. Herein, certain processes should be acknowledged due to the notably good performance shown in MWP reactors. As reported by Jasinski et al.[8], H_2 generation via plasma-assisted dry reforming of methane can be carried out at a specific energy consumption of ~3 kWh/m^3 H_2, and a H_2 generation cost ~2.3 \$/kg H_2 (assuming 0.06 \$/kWh). This value is close, within a factor 2, to the reported industrial cost of H_2 production (representative value: 1.5 \$/kg H_2)[9] through the mature and highly integrated and optimized process of steam reforming of natural gas in high temperature furnaces. Another example is reported by Uhm et al. (2014)[5], in which the gasification of brown coal in a pilot-scale setup was studied, obtaining cold gas efficiency (CGE) values up to 84%, while conventional gasifiers[10] have CGE values between 70-80%.

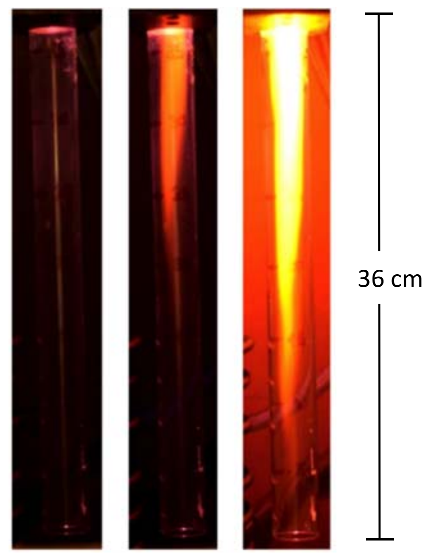


Figure 1. Atmospheric air microwave plasma at 2, 2.5 and 3 kW microwave power, from left to right.

2.2. Industrial applications of MWP technology

The applications of MWP at industrial-scale have been previously discussed in the literature[11]. In summary, the main industrial applications include photoresist stripping in semiconductor manufacturing, deposition of barrier layers in PET bottles, high rate deposition process of quarts on polycarbonate windows, plasma photo curing of paintings applied to the automotive industry, UV disinfection for water treatment, waste gas treatment for decomposition of fluorine-based components such as CF_4, C_2F_6, CHF_3, and SF_6[12] or ammonia, and plasma reforming to increase efficiency in wood gas engines. Table 1 presents the latest industrial applications of MWP together with a brief description of the main features of each process. Most of the future industrial applications of the MWP technology will be relevant to high temperature processes for chemical synthesis and (oxygenated) fuels conversion including pyrolysis, gasification and reforming of organic waste, biomass, and fossil fuels. Other application fields in which MWP will play a role are water and air purification, material synthesis (nano-particle production, diamonds, textiles) and biomedicine (cancer treatment, wound healing, disinfection).

Table 1. Summary of novel MWP processes for chemical applications.

PROCESS (COMPANY)	**DESCRIPTION**	**MAIN FEATURES**
BIOMASS GASIFICATION (Plasma2Energy)[6]	- Medium-scale plant for biomass gasification. - It run for four years. - It exploited the concept of coupling multiple microwave generators to a single gasification chamber. - The plant consumed only 20% of the energy generated.	▪ Input microwave power = 30 kW (plasmatrons) ▪ Pressure = 1 bar ▪ Annual Biomass Capacity = 3.3 kton ▪ Selectivity (H_2) = 50-52% ▪ Annual Production = 1830 m^3 ethanol & 253 m^3 diesel fuel ▪ Maintenance: 5 years ▪ Lifetime: 25 years
CARBON FIBER MANUFACTURING (RMX Technologies)[13]	- Low-pressure microwave plasma enhanced the oxidation and carbonization steps. - Reduction in the residence time and the equipment size by 1/3. - Energy requirements were reduced by 75% and manufacturing costs by 20% compared to conventional one.	▪ Input MW power = 30 kW (915 MHz) ▪ Pressure = 10 mbar ▪ Reactor size: Diameter = 0.05 m, Length = 4 m ▪ Energy efficiency = 17 kWh/kg carbon fiber
PECVD OF Si_3N_4 ON MULTI-CRYSTALLINE SILICON SOLAR CELLS[14]	- Deposition of silicon nitride anti-reflective layers on solar cell wafers by plasma enhanced carbon vapor deposition (PECVD).	▪ Input microwave power = 2 x 4 kW (pulsed) ▪ Pressure = 0.01-1 mbar ▪ Reactor size: Diameter = 0.02 m, Length = 1.5 m ▪ Production = 1500 solar cells wafers per hour
TREATMENT OF CHRONIC WOUNDS (Adtec Europe SteriPlas)[15]	- Wound healing by reduction of microbial load and by modifying the wound microenvironment. - Working gas is Argon, which ensures the reproducibility of generated active agents.	▪ Input microwave power = 200 W ▪ Working gas = Argon, purity 99.95% ▪ Operating temp range = 10-30 °C
PRODUCTION OF SYNTHETIC DIAMOND MPECVD (ASTeX)[16]	- Synthetic diamond growth from the gas phase by microwave-plasma enhanced vapor chemical deposition (MPECVD). - Synthesis diamonds are presented as a much affordable option over naturally mined diamonds.	▪ Frequency = 915 MHz, Power = 90 kW ▪ Pressure = 180 torr ▪ Gas temperature = 4000 K ▪ Working gas = H_2 + 1-5% CH_4 ▪ Deposition rate = 1 g/h ▪ Annual production rate = 214,300 carats (10 reactors) ▪ Diamond production cost = 14 $/carat

3. State of development and outlook

3.1. Current status of the technology

3.1.1. Microwave generator

The largest single-unit continuous wave (CW) microwave generator, namely magnetron, presents a limitation of maximum output power of 15 kW at a frequency of 2450 MHz and 100 kW at 915 MHz. From an economic and regulatory point-of-view, there are two commercially preferable frequencies on the ISM bands, 915 MHz (L-band) and 2450 MHz (S-band) that can be used for MWP reactors. To date, most of the work with MWP has been done at the standard microwave frequency of 2450 MHz. In the case of the 915 MHz frequency, the waveguide components are characterized by larger sizes (about three times larger than those of one at 2450 MHz), which makes these microwave generators more costly compared to 2450 MHz generators.

3.1.2. Plasma ignition, stability and uniformity

Plasma is ignited when the applied electric field strength overcomes the breakdown voltage of the working gas, which is called electric breakdown[17]. When low pressure MWP is considered, the required field strength to ignite and maintain the plasma is less demanding compared to MWP at atmospheric pressure. For large scale chemical applications, uniformity and stability become imperative in the production process, as it is highly important to ensure a constant and reproducible product composition. One of the major challenges for use of the MWP technology is its inherent instability, which has implications in reproducibility of the results. When operating MWP at atmospheric pressure and high power conditions, plasma stability depends mostly on the interplay between input microwave power and flow dynamics (working gas flow rate, feed gas composition, swirl flow). Additionally, operating at a minimum reflected power implies operation very close to or at unstable conditions that can lead to plasma loss or fluctuations (non-uniform). In this regard, there are a number of practical measures to improve plasma stability: (1) addition of a carrier/working gas; argon, helium, nitrogen, air and water are the most commonly used gases, (2) design of a novel reactor configuration such as “Vortex/Tornado-type” or multi-point microwave coupling, and (3) combination of microwave and other fields (e.g. radio-frequency).

3.1.3. Cooling of the plasma reactor

MWP is characterized by high power densities, which enable MWP reactors to achieve energy efficiencies up to 90%. As a result, one of the major technical challenges is the cooling of the reactor due to the high values of power input per unit wall area (W/cm^2), which increases significantly the chance of reactor melting (quartz tube). Hence, MWP reactors require carefully designed cooling systems to ensure a continuous operation. The most common cooling techniques are forced-air/N_2 in combination with cooling water jacket-type[18] surrounding the plasma reactor. When the power input per unit wall area becomes relatively high (> 40 W/cm^2)[3], a common measure of protection of the reactor wall is the use of high-speed tangential gas injection (swirl flow) to confine the plasma at the core of the reactor by creating a tornado/vortex gas motion, which isolates the reactor wall from the plasma column.

3.1.4. Reactor material

As already stated, one of the main features provided by MWP is the high energy density, which also implies high temperatures inside the reactor. In this regard, the material of the reactor is a crucial aspect in MWP operation. At lab-scale, the most commonly used material is quartz, which seems unsuitable for commercial applications due to its fragility. Therefore, other materials such as ceramics (alumina-based), aluminium oxynitride (melting point above 2000 ºC) or silicon carbide can be used to build large size MWP reactors. The latter has already been used in a plasma gasification unit[19]. Moreover, in the pilot-scale setup developed by Uhm et al.[5], alternative materials such as HACT180 (fire-resistance ceramic) and INCT120 (insulating-cement) were used to form the inner and outer layers of the MWP reactor respectively, showing great performance at temperatures up to 1800 ºC.

3.1.5. Process control and safety

MWP-based processes show remarkably fast dynamics, in which most of the events take place in the micro/milli-second range. Such dynamics require demanding continuous process control tools that are capable of adjusting process parameters within a response time of milliseconds. The input power is the most important process variable, as it influences directly the absorbed energy by the plasma and consequently the temperature of the reactor. The gas flow rate largely affects reactor stability as low flow rates can lead to severe increase in the specific energy input (SEI, J/m^3), i.e. the ratio between the input power and the inlet flow rate, causing rupture of the reactor. When the flow rates are excessive, plasma is extinguished due to the drop of SEI. Control of the operating pressure is particularly important when working with low-pressure MWP, as it influences both plasma ignition and sustenance.

With respect to safety, the primary concern is related to exposure of operators and/or fuel to microwave radiation. Furthermore, considering the possible risk of reactor breaking, it is advisable to operate MWP reactors within a properly ventilated area to contain the hazard of a gas leakage.

3.2. Scalability

When evaluating the development of a new process, the production capacity represents the main design guideline, thus dictating the equipment requirements. Bulk chemicals are commonly produced on a very large scale, implying the need to operate at considerably high throughput and therefore demanding high energy input. A pilot-scale MWP gasification unit was developed by Uhm et al.(2014)[5]. In their work, two microwave generators of 75 kW output each were attached to the gasification chamber and enabled inlet flow rates of 2.2 ton coal/day with respective throughput of 1.9 ton syngas/day corresponding to a total calorific value of 0.5 MW_{th}. To our knowledge, there are two possibilities to address the high input microwave power required to sustain the plasma at rather high throughput: (1) coupling multiple microwave generators to a single reactor chamber, or (2) developing single unit microwave sources with > 100 kW output power at lower frequencies (e.g. 433 MHz).

The first approach has already been explored for a medium-scale gasification plant as shown in Sanchez A.L. (2010)[6]. To increase the capacity, multiple 20 kW microwave plasmatrons were arranged around and along a single MWP gasification chamber. A schematic representation is presented in Figure 2. As a final remark, the MWP gasification system reported in Sanchez A.L. (2010)[6] was designed to have maintenance every 5 years and a lifetime of 25 years, which is common practice in the chemical process industry.

Figure 2. Schematic representation of multiple microwave generators attached to a single reactor[6]. Note that each of the black elements represents a 20 kW microwave plasmatron.

The second alternative envisages the development of single unit microwave sources with >100 kW output power. Within the microwave ISM frequencies for industrial processing, the frequency band 433.05 – 434.79 MHz (central frequency 433.92 MHz) appears to be the most interesting one for scale-up. Currently, there are no reported industrial applications operating at 433.96 MHz. However, according to magnetron manufacturers, CW magnetrons operated at this frequency can be designed to deliver much higher microwave power levels than the L-band (896

MHz, 915 MHz, 922 MHz and 929 MHz) magnetrons namely, between 0.5 and 1 MW. The design of high power 433.96 MHz equipment should consider the development not solely of the magnetron and the HV DC power supply, but also all the high power rated WR2100 waveguide components (isolators, impedance tuners etc.) required to run industrial applications.

3.3. Potential for chemical applications

At the current technological state of MWP, the concept of modularized production seems to be the most promising approach to respond to: (1) the decentralized electricity generation via renewable energy sources, and (2) the present volatile markets. In this regard, the development of modular MWP units powered by locally generated renewable electricity for distributed manufacturing may, at least partially, change the current model of very large scale centralized industrial processing and also form an attractive strategy to overcome rapid changes in the market demand. Therefore, the production of syngas, hydrogen, acetylene and localized waste treatment represent some of the opportunities that MWP technology can address at present.

Figure 3 presents in the form of a timeline the main scientific and engineering challenges to be addressed before MWP can be extensively used in the bulk chemical manufacturing industry. These challenges mainly concern: (1) development of higher than 100 kW microwave power sources and of effective plasma reactor designs that can be powered by multiple microwave generators to attain wide throughput range, (2) development of suitable reactor materials for MWP operation, (3) improvement of process reliability (controllability, stability and uniformity), and (4) development of chemical kinetic models that can be implemented into multidimensional multiphysics models for process design, optimization, and control.

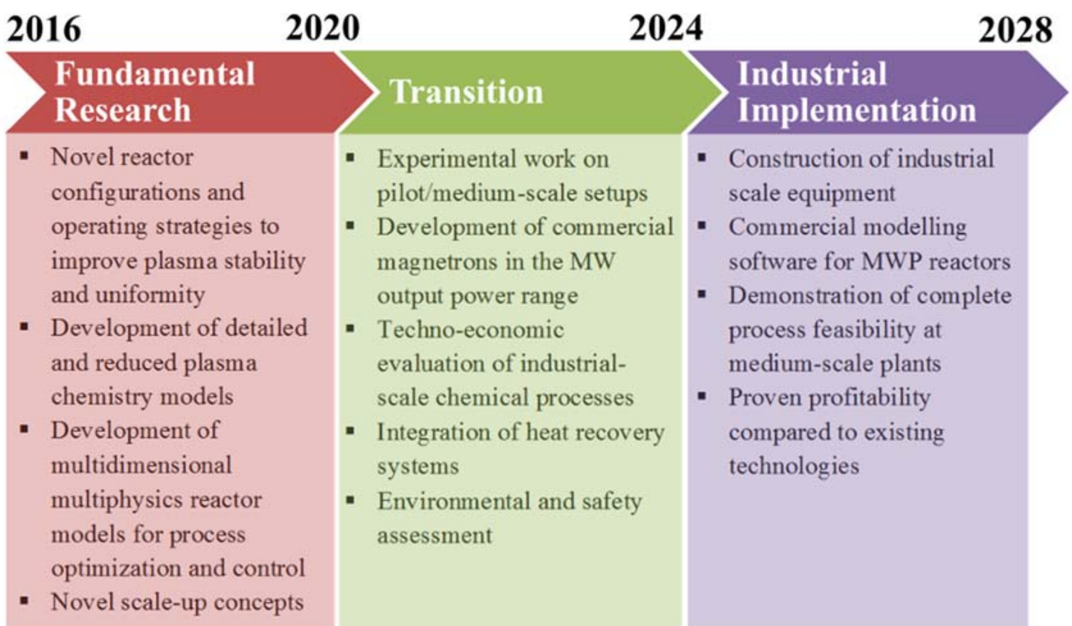


Figure 3. Timeline for the implementation of MWP technology in chemical manufacturing industry.

4. Conclusions

Microwave plasma (MWP) is one of the most promising enabling technologies for electricity-based reactors as regards the future partial electrification of the chemical industry. In this article, we have summarized the extensive research carried out on MWP at laboratory-scale combined with some successfully demonstrated industrial applications. Concerning chemical processing applications, high temperature processes, such as pyrolysis, gasification and reforming of organic waste, biomass and fossil fuels have the highest potential to benefit from MWP. However, it is imperative to perform research with medium-scale setups to quantitatively demonstrate the profitability, reliability and operational benefits of the technology, as already shown for biomass gasification. In parallel, work on development of (a) single-unit microwave sources with >100 kW output power (0.5-1.0 MW), (b) suitable reactor materials that can withstand harsh operating conditions, and (c) reaction kinetic models that can be implemented into multidimensional multi-physics reactor models appear to be key scientific and engineering challenges that need to be addressed to promote wider application of the technology to large scale operations.

For further reading:

1. Styring P, Jansen D. Carbon capture and utilization in the green economy. Centre for Low Carbon Futures (2011).
2. Mennicken L, Janz A, Roth S. The German R&D program for CO2 utilization-innovations for a green economy. Environ Sci Pollut R 23: 11386-11392 (2016).
3. Ferreira CM, Moisan M. Microwave discharges fundamentals and applications. NATO ASI Series (1993).
4. Sturm GSJ, Munoz AN, Aravind PV, Stefanidis GD. Microwave-driven plasma gasification for biomass waste treatment at miniature scale. IEEE Trans. Plasma Sci 44: 670-678 (2016).
5. Uhm HS, Na YH, Hong YC, Shin DH, Cho CH. Production of hydrogen-rich synthetic gas from low-grade coals by microwave steam-plasmas. Int J Hydrogen Energ 39: 4351-4355 (2014).
6. Sanchez AL. Method and apparatus for plasma gasification of carbonic material by means of microwave radiation. US Patent: 2010/0219062 (2010).
7. de la Fuente JF, Kiss AA, Radoiu MT, Stefanidis GD. Microwave plasma emerging technologies for chemical processes. Journal of Chemical Technology & Biotechnology: (2017).
8. Jasinski M, Czylkowski D, Hrycak B, Dors M, Mizeraczyk J. Atmospheric pressure microwave plasma source for hydrogen production. Int J Hydrogen Energ 38: 11473-11483 (2013).
9. Dincer I, Zamfirescu C. Sustainable Hydrogen Production. Elsevier (2016).
10. Grabner M. Industrial Coal Gasification Technologies Covering Baseline and High-Ash Coal. Wiley-VCH (2014).
11. Kaiser M, Baumgartner KM, Mattheus A. Microwave plasma sources - Applications in industry. Contrib Plasm Phys 52: 629-635 (2012).
12. Radoiu M, Hussain S. Microwave plasma removal of sulphur hexafluoride. J Hazard Mater 164: 39-45 (2009).
13. Terry LW. System to continuously produce carbon fiber via microwave assisted plasma processing. US Patent: 8,679,592 (2014).
14. Schlemm H, Mai A, Roth S, Roth D, Baumgartner KM, Muegge H. Industrial large scale silicon nitride deposition on photovoltaic cells with linear microwave plasma sources. Surf Coat Tech 174: 208-211 (2003).
15. Adtec, SteriPlas, http://www.adtecplasma.com/products.html#home. (accessed 25-09-2016).
16. Dischler B, Wild C. Synthetic Diamond: Manufacturing and Applications. Springer-Verlag (1998).
17. Fridman A. Plasma Chemistry. Cambridge: Cambridge University Press (2008).
18. de la Fuente JF, Moreno SH, Stankiewicz AI, Stefanidis GD. Reduction of CO2 with hydrogen in a non-equilibrium microwave plasma reactor. Int J Hydrogen Energ 41: 21067-21077 (2016).
19. Willis KP, Osada S, Willerton KL. Plasma gasification: Lessons learned at EcoValley WTE facility. 18th Annual North American Waste-to-Energy Conference (2010).

About the Authors

Dr. Javier Fernandez de la Fuente will soon receive his Ph.D. at Delft University of Technology in the Netherlands on the application of microwave plasma technology to convert CO_2 into high value chemicals. He completed his M.Sc. degree in Chemical Engineering at University of Valladolid (Spain) in 2012. He has broad experience as a R&D engineer, mostly focused on the production of liquid fuels through alternative technologies. He worked on projects such as the design of a pervaporation unit to separate butanol from acetone-butanol-ethanol (ABE) mixture as an alternative to distillation, and the start-up and optimization of an ammonia fiber expansion pilot plant to pre-treat wheat straw for the production of ethanol. Moreover, he explored a new approach to produce MCM-41 for ibuprofen encapsulation by ultrasound-assisted supercritical CO_2 during his stage at Ruhr University of Technology in Germany.

For more information:
https://www.linkedin.com/in/javierfdelafuente
E-mail: J.FernandezdelaFuente@tudelft.nl

Dr. Anton Alexandru (Tony) KISS is a senior project manager at AkzoNobel - Research, Development & Innovation, where he serves as an expert in fluid separation technologies, reactive-separation processes, process intensification, and integrated sustainable processes, as well as process modeling and simulation of industrial processes. He is also a part-time professor of separation technology at the University of Twente (Sustainable Process Technology) in the Netherlands. He has published several textbooks, book chapters, patents, and more than 100 scientific articles. For his pioneering work and career achievements, he received in 2013 the Hoogewerff Jongerenprijs and the AkzoNobel Innovation Excellence Award. Kiss holds a PhD in chemistry and chemical engineering from the University of Amsterdam, and MS and BS degrees in chemical engineering from Babes-Bolyai University of Cluj-Napoca in Romania, and he was a post-doctoral research fellow at Delft University of Technology (TU Delft) and the University of Amsterdam. He is a member of AIChE, IChemE, Society of Chemical Industry (SCI), European Society of Mathematical Chemistry (ESMC), Process Systems Engineering – The Netherlands (PSE-NL), European Federation of Chemical Engineering (EFCE), and EFCE Working Party on Computer Aided Process Engineering (CAPE-WP).

For more information: http://www.tonykiss.com
E-mail: Tony.Kiss@akzonobel.com

Dr. Marilena Radoiu, Chartered Chemist (CChem) and Member of the Royal Society of Chemistry (MRSC), received her M.Sc. in Organic Technological Chemistry from the Polytechnic University of Bucharest in 1993 and her Ph.D. in Radiochemistry and Nuclear Chemistry from the same University in 1998. She has extensive work experience in different international academic and industrial environments. She has worked for 20 years in Romania, Canada, UK and France in the development of microwave-assisted technologies with applications to chemical synthesis, biomass extraction, plasma etc. Her work has included engineering and development of novel industrial and scientific standard and custom products, such as Zenith Etch and Sirius6000 - microwave plasma reactors for semiconductor gas cleaning, laboratory equipment such as MiniFlow 200, and Minilabotron 2000. Dr. Radoiu is also a member of several professional associations, including the Association for Microwave Power, Education and Research in Europe (AMPERE).

For more information:
https://www.linkedin.com/in/marilenaradoiu
E-mail: marilena.radoiu@btinternet.com

Prof. Georgios Stefanidis (KU Leuven, Belgium) received his chemical engineering education from the National Technical University of Athens (M.Sc.) and University of Gent (Ph.D.). His research interests revolve around process intensification, alternative energy forms and transfer mechanisms (mainly microwaves and plasma), biomass gasification and pyrolysis, catalytic reforming and partial oxidation of hydrocarbons and oxygenated fuels and CO_2 utilization. He is co-author of more than 60 peer-review papers in the field of process intensification.

* Corresponding author, E-mail:
georgios.stefanidis@kuleuven.be

Computer-Aided Analysis and Optimization of Microwave Heating Systems

Vyacheslav V. Komarov

Department of Radio Electronics and Telecommunications, Institute of Electronic and Mechanical Engineering, Yuri Gagarin State Technical University of Saratov, Saratov, 410054, Russia

The application of microwave energy for thermal treatment of different materials and substances is a rapidly growing trend of modern science and engineering. Deep penetration of microwaves in dielectric media improves uniformity and intensifies the heating process. The variety of microwave heating devices like kitchen ovens, industrial plants, laboratory setups, and medical applicators is considerably large.

Design and optimization of microwave heating devices is impossible without theoretical studies of the physical processes of electromagnetic waves interaction with lossy dielectric media. Mathematical modeling and experimental measurements are the main tools for investigation of such processes. The development of applied electromagnetics, including the theory of numerical modeling, computational software and hardware, has led to the appearance of different mathematical models for simulating electromagnetic and thermal fields in microwave heating systems. Aggregate state of irradiated sample, operating temperatures, possible chemical or biological reactions, peculiarities of particular technology realization and some other factors, influence on the formulation of such models. The most well-known among them is the coupled electromagnetic heat transfer problem, which takes into account the influence of temperature on the distribution of microwave power sources in an interaction domain.

Such mathematical models can be built using numerous commercial software. One of them is the package COMSOL[1] on the finite element method (FEM), which is widely used in computer-aided design (CAD) of many microwave devices. This multi-physics software is suitable for the solution of coupled problems, which makes it a useful tool in modeling of microwave heating processes.

Another approach using the finite time-domain method (FDTD) is employed by the QuickWave-3D[2] (QW3D) software. The features of this software, such as conformal mapping of rectangular meshes and a new method to extract the wave impedance and propagation constant directly from time domain simulations, allow saving computer resources. The geometrical model of microwave device can be expressed in symbolic variables employing the so-called User Defined Object language. This makes the optimization process very flexible and efficient. Combined application of both packages for simulation of one object can increase the efficiency and accuracy of the numerical modeling. Here we consider a few examples of such approach.

Purification of polluted soils

Contamination of soils is a very widely spread problem in many countries. The in-situ method of microwave decontamination of soils is an attractive alternative to commonly employed ex-situ technologies because it prevents possible intoxication during excavation and is much cheaper.

A coaxial antenna with an operating frequency of 2.45 GHz. shown in Fig. 1a, is intended for a realization of such an in-situ remediation method[3]. Different contaminants like oils and other chemical substances are evaporated during microwave heating of the soil and can then be exhausted. The antenna design includes a waveguide-coaxial transition, a one-meter coaxial line with 20 slots that is short-circuited at its end. It is equipped with a metal cone to easily insert the antenna into the ground. A standard rectangular waveguide WR340 is utilized as a feeder.

Theoretical and experimental studies of the described air-filled antenna, carried out in

Karlsruhe University[3], have shown quite good coupling for dry soils. However, for moist soils an increased reflection factor was observed. Additional numerical simulations by means of FEM and FDTD techniques have helped to upgrade this device and decrease antenna return loss for moist soils[4] as well.

Preliminary theoretical investigations fulfilled by using a simplified 1D analytical models of stratified dielectric media have demonstrated that the best result is achieved when the slotted antenna is separated from the soil by a 5-mm thick Teflon coating (Figure 1b). Figure 2 illustrates the numerical simulation results for different levels of moisture content in microwave exposed soils. The return loss at 2.45 GHz does not exceed 0.25 for the highest moisture content.

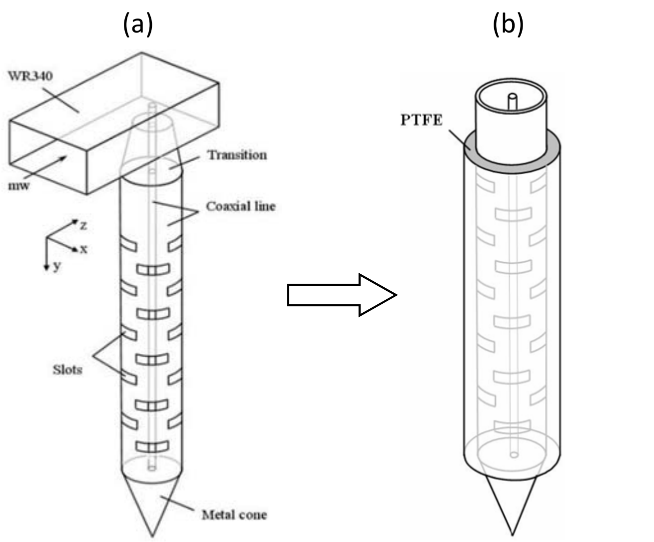


Figure 1. Initial (a) and upgraded (b) designs of the microwave antenna.

Chemistry reactors

Electromagnetic (EM) energy has been proven to be a useful and helpful tool of scientific research nowadays. EM waves are widely used in experimental studies in physical chemistry, food science, medicine, biology, material science, and so on. It is known that microwaves accelerate many chemical reactions. Today microwave chemistry is one of the most rapidly developing trends of science and engineering. Early studies in this field employed conventional domestic microwave ovens. The special equipment required in analytical chemistry resulted in commercial multi-mode microwave ovens designed for these purposes.

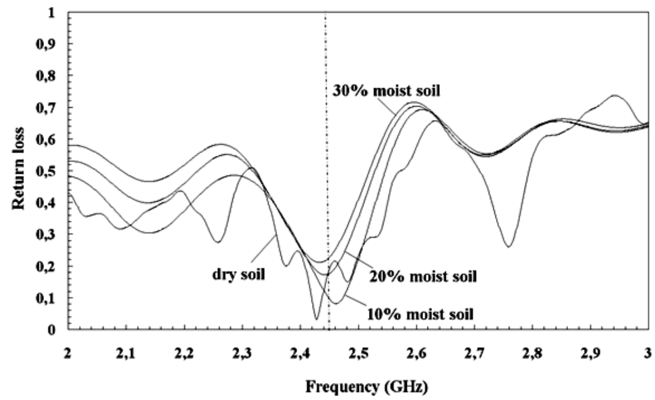


Figure 2. Reflection properties of microwave antenna

Different single-mode and multi-mode microwave heating systems find wide practical application in analytical chemistry[5]. The repeatability of chemical reactions is one of the main requirements for such systems. That is, quite uniform distribution of power density and temperature in an interaction domain must be achieved. Single-mode waveguide and resonator cavities, intended for heating of only one sample, usually satisfy this requirement. In multi-mode cavities, where several samples are heated, the problem of non uniform distribution of power sources is solved by rotating the samples.

Almost all microwave chemistry applicators are designed on a basis of rectangular or cylindrical waveguides and cavities. The so-called reentrant cavity with extended capacitance gap has been proposed[6] as a basic unit of microwave chemistry applicator, as shown in Fig. 3. This resonator has higher values of resonance wavelengths of the dominant mode than simple reentrant cavity well known in microwave electronics. The last feature allows us to select the operating frequency 915 MHz, and, consequently, to increase EM field penetration depth in lossy dielectric.

The dominant mode in the cavity is excited by a coaxial probe on a central axis. The glass test tubes with liquid samples (water, protein and pyrrolidin) are arranged around the probe in capacitance gap in a special ring-shaped Teflon holder.

The coupled problem was solved in the present study by using the FEM and commercial software COMSOL. One more numerical technique, the FDTD method implemented in another commercial code QW3D was employed to find cavity sizes, which provide the best coupling at operating frequency.

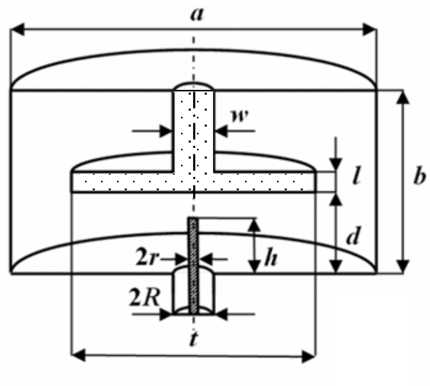


Figure 3. A microwave chemistry reactor design, $a = 210$ mm, $b = 105$ mm at 915 MHz.

Simulations have demonstrated that the best coupling is observed for water at 60°C. It is interesting to note, that the reflection coefficient values are the same for temperatures 40°C and 80°C at 915 MHz. Slightly higher reflection has been achieved for protein. Simulations of the microwave applicator with 8 pyrrolidin samples have shown an almost complete reflection of EM power. But, as it has been proven numerically, coupling can be improved in this case by changing the capacitance gap sizes.

Figure 4 illustrates temperature field pattern in four water samples. Given solution of a coupled problem did not include convection processes, and in reality microwave heating of liquid samples with low viscosity will be much more uniform.

Tumor ablation

As it is known microwave energy is widely used in medicine. For example, it can be a very promising tool for tumor ablation. Malignant biological tissues during microwave ablation (MA) are heated up to relatively high temperatures (60…100°C) in order to achieve coagulative necrosis of tumor cells. This minimally invasive procedure provides less bleeding, possibility of using local anaesthesia and other advantages in comparison with conventional approaches. Cancerous tissues are heated up to high temperatures using, for example, a coaxial antenna[7] as presented in Figure 5.

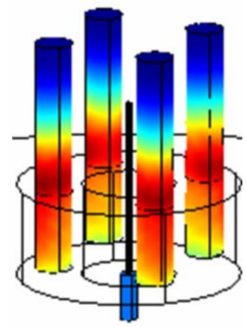


Figure 4. Temperature distribution in water samples heated in a cavity such as shown in Fig. 3. The tube diameter and height are 10 mm and 50 mm, respectively. The temperature varies along the tube from 20°C (Dark Blue) to 81°C (Red).

The radiating part of this antenna is inserted in the middle of tumor zone. Antenna diameter is less than 2 mm, and the operating frequency is 2.45 GHz. The space between coaxial conductors is filled with Teflon. Cone shaped component made of ceramics protects the thin probe from damage, and provides low level of reflected power during microwave ablation. The necessary temperature in the biological tissue is controlled by special remote system.

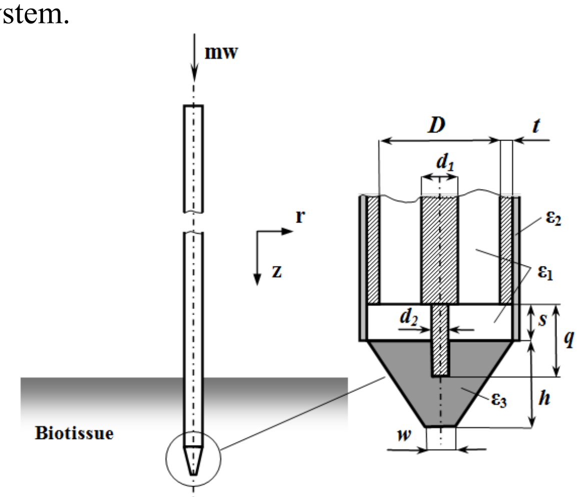


Figure 5. Microwave tumor ablation antenna (of less than 2-mm diameter)

Preliminary experimental studies on phantom models of biological tissues, such as muscle, liver and brain, have shown relatively low level of reflected power for this antenna (less than 3%) at operating frequency 2.45 GHz. Then, temperature patterns have been simulated by FEM in the ablation zone. The coupled EM-bioheat problem was solved employing COMSOL software, and besides, temperature dependencies of the tissues dielectric properties were taken into account.

Reflection characteristics of the antenna have been simulated and optimized by means of the QW3D software.

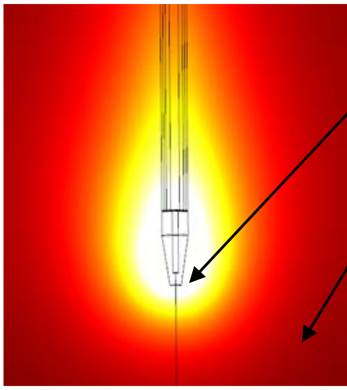


Figure 6. Temperature distribution in liver.

The computed results allow to estimate the electromagnetic and thermal characteristics of the interstitial coaxial antenna of 2.45 GHz at different temperatures during the MA procedure. Such antenna shows relatively low values of return loss at body temperature for various biological tissues, such as muscle, liver, kidney and brain, and increasing the reflected power up to 9 – 25 % at high temperatures in the range $80 \le T$ [ºC] ≤ 100 for liver. But, even in the worst case (T = 100ºC), the antenna delivers 75% of microwave energy to the tissue. Figure 6 shows the temperature patterns after 6 minutes of heating at 10 W. The lesion domain radius and sphericity are R_l = 7.6 mm and W_s = 0.34, respectively.

Summary

The three examples presented above illustrate powerful capabilities of the commercial codes COMSOL and QW3D for comprehensive analysis and optimization of industrial, scientific and medical microwave systems (as shown in Figs. 1, 3 and 5, respectively). The application of two numerical approaches in parallel allows increasing the efficiency and flexibility of the mathematical modeling of microwave heating processes, when the disadvantages of one method are compensated by the advantages of the other, and vise versa.

For further reading:

1. COMSOL Multiphysics V.5, 2016. Available online at http//:www.comsol.com.
2. QuickWave-3D, QWED, Warsaw, Poland, 2015. Available online at: http//:www.qwed.com.pl.
3. M. Pauli, T. Kayser, W. Wiesbeck. A coaxial antenna for microwave assisted soil decontamination. *IEEE MTT-S Proc., Int'l Microwave Symposium*, June 2006, San Francisco, CA, pp. 2027-2030.
4. M. Pauli, T. Kayser, W. Wiesbeck, V.V. Komarov Impedance matching of coaxial antenna for microwave in-situ processing of polluted soils. *Int'l Journal of Microwave Power and Electromagnetic Energy*, 2011, Vol. 45, No. 2, pp. 70-78.
5. C.O. Kappe , A. Standler and D. Dallinger, *Microwaves in Organic and Medical Chemistry*, Weinheim: Wiley-VCH Verlag GmbH, 2013.
6. V.V. Komarov. Single-mode microwave chemistry applicator at 915 MHz. *COMPEL - The Int'l Jour. Computation and Mathematics in Electrical and Electronic Engineering*, 2017 (submitted for publication).
7. V. V. Komarov. Numerical study and optimization of interstitial antennas for microwave ablation therapy. *The European Physical Journal. Applied Physics*. 2014, Vol. 68, No. 1, Art. No. 10901 (8 pages).

About the Author

Vyacheslav Komarov is a Professor in the Department of Radio Electronics and Telecommunications, Institute of Electronic and Mechanical Engineering, Yuri Gagarin State Technical University of Saratov, Saratov, Russia. Dr. Komarov received the Ph.D. degree in Radio Physics from the Saratov State University in 1994, and the D.Sc. degree in Antennas and Microwave Devices from the Yuri Gagarin State Technical University of Saratov in 2007. He was a Visiting Scientist at the Montena EMC, Baden, Switzerland (1997), the Chalmers University of Technology, Göteborg, Sweden (1999), the Washington State University, Pullman, WA (2003), the Karlsruhe Institute of Technology (KIT), Karlsruhe, Germany (2006 and 2009), and the Kokushikan University, Tokyo, Japan (2013). Dr. Komarov's interests include mathematical modeling of electromagnetic and thermal fields in microwave systems, propagation of electromagnetic waves in complex media, CAD and optimization of microwave and terahertz devices.

E-mail: vyacheslav.komarov@gmail.com

Green-Tech Microwave Studies at Tohoku University

N. Yoshikawa[1], C. C. Lee[1,2], M. Sunako[1], K. Kawahira[1], S. Taniguchi[1]

[1] Graduate School of Environmental Studies, Tohoku University, Aramaki, Aoba-ku, Sendai, Japan, 980-8579
[2] School of Manufacturing Engineering, Perlis University, Kampus Tetap Pauh Putra 02600 Arau, Perlis, Malaysia

1. Introduction

Microwave heating applications for environmental technologies have been studied since the discovery of microwave heating in 1940's[1]. They were applied for industrial waste treatments, regeneration of active carbon catalysis[2], detoxification of asbestos fibers[3], and recycling of metals[4]. In these applications, the characteristic features of microwave heating are important to be taken into consideration, as a means for rapid, internal and selective heating. This article is intended to introduce some of our recent research topics on microwave applications for environmental technologies. The selected projects deal with the following two subjects:

Diesel Particulate Filter (DPF) - Rapid heating by microwave irradiation (Project A)

The use of diesel engine has been revived recently, because of its high thermal efficiency, less CO_2 emission, and the possibility to scale up to the large driving capacity. However, it emits particulate matter (PM), which is harmful to human bodies[5]. Therefore, PM is captured from the exhaust gas with a DPF before emission to the ambient atmosphere[6]. Currently, the PM choked by the DPF is combusted with blowing air and fuels after predetermined periods or mirages. For the small PM filtration such as PM2.5, fine meshed filter is required, however, it leads to much easier choking. Therefore, instantaneous PM combustion, especially upon ignition (cold start phase) is demanded. Microwave rapid heating has been taken into consideration. In this study, we attempted to fabricate DPF material for the microwave rapid heating.

Vapor de-phosphorization from Tri-calcium phosphate (Project B)

Phosphorus is a valuable element which is essential for human body, growing plants and indispensable as a raw material for various industrial chemical products[7]. On the other hand, phosphorous natural resource is running short, recently. Therefore, it is required to recycle the phosphorous from used materials or waste. Large amount of slag is emitted as by-products in ion and steel making industry, which contains large amount of phosphorous in the form of oxides. It is intended to reduce it with carbon.

In this study, carbon reduction by microwave heating thermal process is applied, because it enables to acquire phosphorous in vapor phase. Rapid vapor removal from the heated body is esential[8], because the slag usually contains ion oxides, which is also reduced to (liquid) metal iron and absorbs phosphorous. This paper reports on the carbon reduction kinetics of slag related materials (TCP: TriCalcium Phosphate, $3CaO{\cdot}P_2O_5$ with and without iron oxides), as a fundamental research study on the phosphorous recycling process.

2. Materials and Methods

In Project A, glass (SiO_2 – RO, R: alkaline earth metal) and stainless steel (SUS303) powder mixture was sintered at 950°C for 1 hour. Porous cylindrical metal/glass compacts (of 15-mm diameter, 5-mm length) were obtained for DPF. In this process, an aqueous slurry of powder mixture was infiltrated into polyurethane sponge preform, then it was fired to burn out the preform. Porosity was determined by Hg porosimeter. They were placed at a position of maximum magnetic field in a single mode microwave applicator operating at 2.45-GHz. Carbon black powder (PM simulants) was contained in the DPF in advance, as a simulating PM. Their combustion kinetics were examined by measuring the CO/CO_2 concentration in the flowing gas.

In Project B, powder mixtures of graphite and TCP, with and/or without ion oxide (Fe_3O_4) were contained in a silica cell tube of a 10-mm diameter

(~25-mm tapped height). Single Fe_3O_4 reduction kinetics was also studied. They were also heated at maximum magnetic field in a single mode 2.45-GHz microwave applicator. The degree of carbon reduction of single TCP was determined measuring the phosphorous concentration by ICP (Inductively Coupled Plasma) analysis of the residue (phosphorous containing calcium oxide). On the other hand, however, the reduction ratio of TCP with iron oxide mixture was difficult to evaluate, because of the problem in separating small iron particles (hence phosphorous concentration distribution in the reduced iron and TCP was not accomplished).

3. Result and Discussion

Microwave DPF heating (Project A)

An example of the obtained DPF is shown in Fig. 1(a), having porosity of 60~70%. The pore size has a range of several nanometers to several hundred micron meters. Stainless steel particles (white color particles) are distributed in the glass matrix (gray color area) as shown in Fig. 1(b). An example of heating curves is demonstrated in Fig. 2 for the cases of 400-W input power[9]. An increase of the metal volume fraction leads to a faster heating rate, thus the time to reach a temperature of 600°C (the PM combustion temperature) becomes shorter. However, as indicated in the Fig.2, a tendency of unstable heating occurs in the case of 40% by volume (40vol%) at 60 s, due to local arcing. Therefore, we have selected the optimal metal fraction as 30vol%, and continued further tests.

The time variation of the exhaust gas composition was detected, as plotted in Fig. 3. CO and CO_2 gases by PM combustion were detected after about 25 seconds of microwave heating. Combustion of carbon black was confirmed to occur under the air flowing condition (500 ml/min) in the laboratory scale experimental system. In the present study, over 10 seconds were required to reach 600°C for PM combustion.

For DPF material having rapid heating capability, it was decided to use a metal/glass composite body for the following reasons: (a) The metal particle has a rapid response to microwave irradiation, hence it is rapidly heated by an induction heating mechanism. The volumetric rapid heating of DPF body can be accomplished by rapid heating of metal particles embedded in non-absorbing matrix, such as a glass, through which the microwave penetrates to the interior.

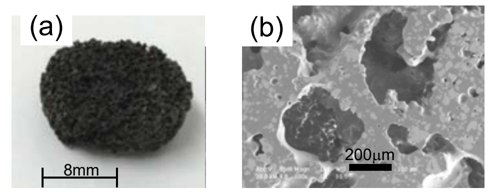


Figure 1. (a) The fabricated DPF, and (b) its microstructure observed with SEM. The volume fraction of stainless steel particles is 30%.

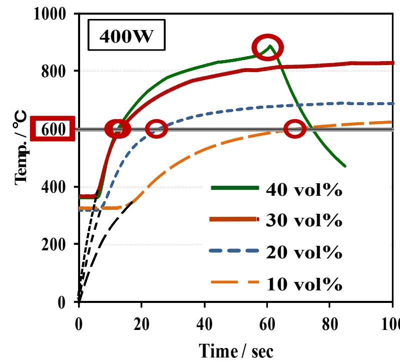


Figure 2. Heating curves of DPF having various metal volume fractions at 400-W power input[9].

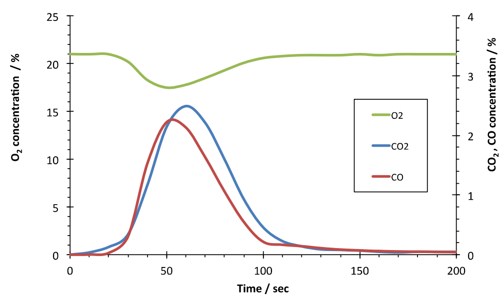


Figure 3. Temporal variations of the exhaust gas composition from DPF.

Otherwise, microwave absorbing ceramics are the other candidates. In Practice, SiC has been taken into consideration for DFP. Currently, microwave heating has not been adopted in practice, though the possibility of microwave heating of SiC-DPF was reported[10]. However, it still takes a longer heating time than our present cases. In our study, it is still required that the heating time has to be shortened by modifying the DPF microstructure, and by developing the materials by considering the durability against the cyclic heating of the composite body.

De-phosphorization by microwave heating (Project B)

The chemical reaction of reduction is given by:

$$3CaO\cdot P_2O_5(s)+5C(s)= P_2(g)+3CaO(s)+5CO(g) \quad (1)$$

The predicted equilibrium phase constitution (thermodynamic analysis by HSC Chemistry, Ver. 7.0) is illustrated in Fig. 4. The reduction of TCP occurs, and P_2 and CO gas generation is expected to occur, above 1300ºC. As the temperature increases, the partial pressure of these gases becomes higher. The reduction experiments by microwave heating were conducted for the specimen powder mixtures contained in a silica cell, placed in a silica tube of 40 mm in diameter. The vaporized phosphorous was deposited on the inner surface of the silica tube, as shown in Fig. 5. The yellow-brown colored deposits were chemically analyzed and confirmed to be phosphorous.

The reduction ratio of TCP is presented in Fig. 6 at various heating rates and target temperatures. It is shown that the lower heating rate and the higher target temperature resulted in the larger reduction ratio. This is as expected, because of the principle that the total residence time at high temperature causes the higher degree of reduction reaction. Therefore, the reduction ratio was calculated under the assumption of the additive law (the heating process is discretized by steps k (k=1,2,3…n) with increments of time and temperature, Δt and ΔT, respectively, as follows:

$$\alpha_n=\sum_{k=1}^{n}\left[A\exp\left(-\frac{E}{RT_{k-1}}\right)\right](1-\alpha_{k-1})^m\Delta t\ , \quad (2)$$

where A is a constant, R is the gas constant, and E is an apparent activation energy of the reaction (E = 220 kJ/mol for the reaction in Eq. 1) [11]. In this equation, the reaction rate constant is expressed in an exponential form of temperature dependence. The calculated reduction ratio (α) is also presented in Fig. 6 by solid lines. In this study, the reaction order index m was best fitted to be 3. The drawn lines in Fig. 6 were obtained under this assumption of reaction kinetics.

It is shown that the reduction occurs at 1100ºC, a temperature lower than estimated by the thermodynamic calculation presented in Fig. 4. One of the possible reasons is that the surface temperature recorded by the optical method was lower than expected. However, we obtained the apparent third order (m = 3) reaction kinetics by calculation, which is not generally common. What is needed is to consider some other special effects in microwave heating on the reaction mechanism.

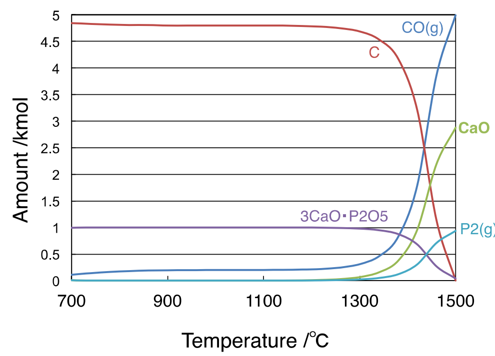


Figure 4. Calculated phase constitution at elevated temperature.

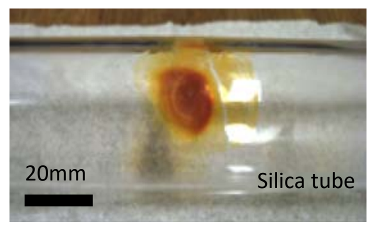


Figure 5. Photograph of phosphorous deposits on silica tube.

The slower heating rate to higher target temperature is favorable for the higher reduction ratio of TCP. However, reduction of Fe_3O_4 occurs much easier as shown in Fig. 7, and mostly reduced above 97% by the range of temperature and heating rates shown in the plot. This fact suggests that the iron oxide is readily reduced to molten metal iron (above 1200ºC, considering the carbon content present). Phosphorous vapor is much easier to be absorbed in molten iron than its solid form.

Considering the results shown in Fig. 6, the degree of reduction becomes large above 1200ºC. Therefore, the operating temperature shall be set above it. The phosphorous has to be vaporized out by rapid heating to 1200ºC before it is absorbed in the molten iron. This requirement is important for the phosphorous recycling process.

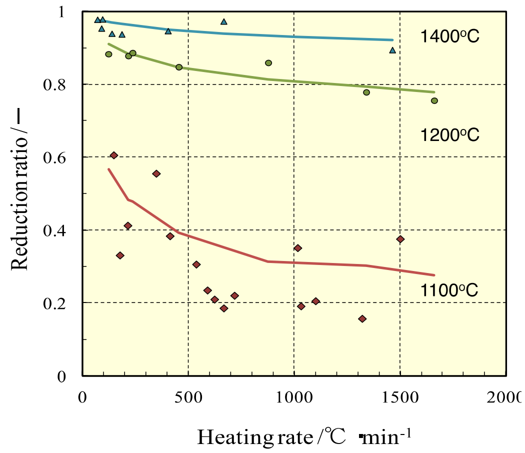


Figure 6. Dependence of experimental reduction ratio of TCP on heating rate for three target temperatures (1100, 1200 and 1400ºC); the calculated data are shown by lines.

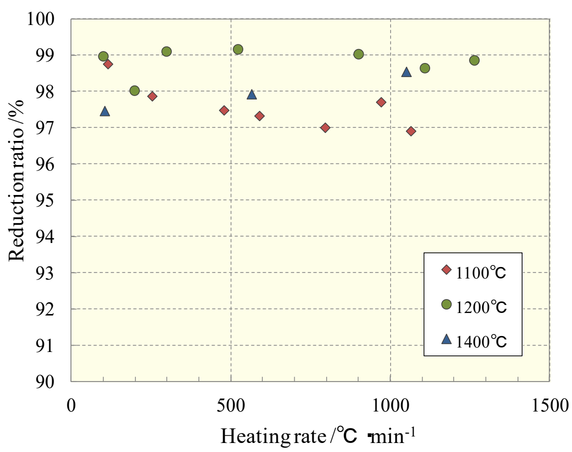


Figure 7. Dependence of experimental reduction ratio of Fe_3O_4 on the heating rate and target temperature (1100, 1200 and 1400ºC).

Selected research projects of Green-Tech microwave studies in the authors' group are briefly introduced. First is the fabrication of DPF materials, consisting of metal particle dispersed in glass, which can be rapidly heated, over 10 s to 600ºC. Combustion tests of PM simulants were demonstrated. Second, for the purpose of phosphorous recycling, microwave excited carbon reduction kinetics of TCP was studied with and/or without Fe_3O_4. Criterion of heating rate was discussed for phosphorous removal in vapor phase, without being absorbed in the reduced molten iron.

For further reading:

1. H. Sobel, K. Tomiyasu, "Milestones of microwaves," IEEE Trans. Microwave Theory & Tech., Vol. 50, pp. 594-611, 2002.
2. J.A. Menendez, E.M. Menendez, M.J. Iglesias, A. Garcia, JJ. Pis, "Modification of the surface chemistry of active carbons by means of microwave induced treatments," Carbon, Vol. 37, pp.1115–1121, 1999.
3. N. Yoshikawa, K. Kashimura, M. Hashiguchi, M. Sato, S. Horikoshi, T. Mitani, N. Shinohara, "Detoxification mechanism of asbestos materials by microwave treatment.", J. Hazardous Mater., Vol. 284, pp. 201-206, 2015.
4. C.A. Pickles, "Microwaves in extractive metallurgy: Part 1 – Review of fundamentals", Miner. Eng., Vol. 22, pp.1102-1111, 2009.
5. C.A. Pope, M.J. Thun, M.M. Namboodiri, D.W. Dockery, J.S. Evans, F.E. Speizer, C.W. Health, "Particulate air pollution as a predictor of mortality in a prospective study of U.S. adults," Amer. J. Respir. Crit. Car. Med., Vol. 151, pp. 669-674, 1995.
6. K.S. Martirosyan, K. Chen, D. Luss, "Behavior features of soot combustion in diesel particulate filter", Chem. Eng. Sci., Vol. 65, pp. 42-46, 2010.
7. USGS Website - http://minerals.usgs.gov/minerals/ (browsed in May 2016).
8. S. Takeuchi, N. Sano, Tetsu-to-Hagane, Vol. 66, pp. 2050-2057, 1980 (in Japanese).
9. Lee Chang Chuan, Fabrication of Porous Composite Material for Diesel Particulate Filter and the Regeneration by Microwave Heating, Ph.D Thesis at Tohoku Univ., March 2013.
10. S.Pallavkar, T.H. Kim, D. Rutman, J.Lin, and T. Ho, "Active Regeneration of Diesel Particulate Filter Employing Microwave Heating", Ind. Eng. Chem. Res. Vol. 48, 2009, pp. 69–79.
11. M. K. Hussein, G.A. Kolta, A.E. Saba, A.M. El Roudi, "Kinetics of calcium phosphate reduction by carbon", Thermochim. Acta, Vol. 10 (1974), 177–186.

About the Corresponding Author

Dr. Noboru Yoshikawa has started research work in various fields of materials science and materials processing since his graduation from the department of Materials Science of Engineering (MSE) at Tohoku Univeristy (BSc, MSc and PhD). He is now an associate professor at Graduate School of Environmental Studies at Tohoku Univeristy (combined with the MSE department). He has been in the field of microwave engineering for about twenty years. His main interests is microwave application for materials processing and green technology. He has been involved in many projects and performed many collaborative researches. Currently, he is an administrative member of JEMEA (Japan Society of ElectroMagnetic Energy Application) and JSPS No. 188 University-Industry Cooperative Research Committee.

E-mail: yoshin@tohoku.ac.jp

Rendering of Waste by Application of Microwave Energy

Ryszard Parosa

Prometeus Ltd. (PROMIS-TECH Sp. z o.o.), ul. Na Grobli 6, 50-421 Wrocław, Poland

1. Introduction

Waste utilization is one of the most important problems of civilization. Continuously growing stream of waste from industrial processes and communal waste, and at the same time emission of polluted gases into atmosphere, needs new efficient solutions. Prometeus Ltd. has developed several new innovative methods for waste treatment with the use of high power microwaves. The technologies developed have been applied in industrial scale, and they seem to be very promising alternatives for conventional methods.

Development of the high-power microwave systems create a new technical possibility to apply microwaves in industrial scale with reduced utilization costs. The main advantages of the developed microwave reactors are:

- Possibility of bringing up any dielectric material to the temperature range of 1000-1200ºC (rousting).
- Possible precise control of the temperature within the reactor (temperature stabilization).
- Shorter starting times of the installation (a 1000ºC temperature is reachable after 20 minutes).
- Rousting process could be operated in a controlled atmosphere, for example in a reactor filled with air (in waste burning) or with inert gases (in heat treatment without oxidation).
- Rotational operating mode (provides a continuous mixing of the reactor contents, which increases the possible contact with oxides from the air, and uniformly distributes the heat).
- Possibility of nesting active centers in the catalyst carrier in the form of granulated ceramics (depending on wastes qualities, using the appropriate catalyst could accelerate a temperature reduction or even enable reducing the problem of toxic compounds created in undesirable reactions of thermal condensation).
- Ability to meet customer needs in terms of capacity along the technical line. It is possible to build a mobile unit mounted in a standard 20-foot container with a capacity of about 200kg/h, or to build a plant with a capacity of several tons per hour.
- Research of the hazardous wastes utilization shows 99% loss of harmful organic compounds.

The system can be used in many fields thanks to the flexibility and ease of control of the three basic parameters of the process, namely: temperature, quantity and quality of the provided atmosphere, as well as the time spent in the reaction zone.

2. HR-series microwave reactors

The most important element of the installation is the microwave reactor made in the form of a cylindrical drum situated within a metal chamber equipped with microwave radiators. The microwave energy emitted from the microwave radiators is transmitted inside the ceramic drum, which contains the treated waste (sometimes mixed with a special additive enhancing the absorption of microwaves). Depending on the process, the content is heated up with microwave energy to a temperature in the range 900-1300°C in its entire volume. The system guarantees a uniform heating with microwaves thanks to the mixing of the waste in the rotating drum and the application of a special system of microwave radiators deployed along the process chamber. The reactor applies several horn antennas, which excite the TE_{01} and TE_{02} modes. The antennas (radiators) are positioned in such a way that neighboring antennas emit waves of mutually-perpendicular polarization. This minimizes the transmission of microwave energy between neighboring microwave lines. The construction of the HR reactor is shown in Fig. 1.

The reactor with a capacity of 200 kg/h is powered by 14 microwave generators operating at

2,450-MHz and 3-kW CW power each. Ferrite circulators with water loads are installed in the microwave system. The reflection coefficient, measured during the utilization of asbestos waste, varied in the range $|\Gamma| \approx 0.08 - 0.1$ (in conditions in which the asbestos waste introduced into the reactor was preheated to ~800°C).

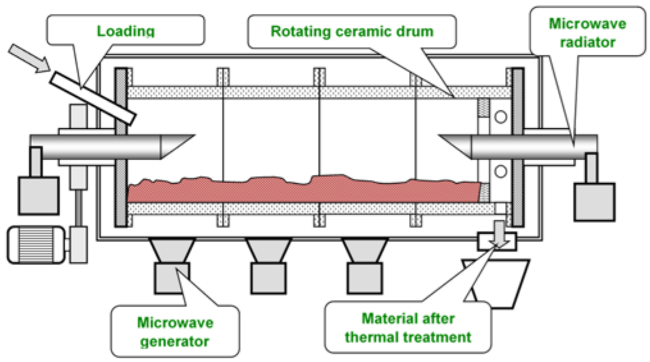


Figure 1. A schematic of the HR reactor.

Figure 2. The HR-200 reactor installed inside a standard 20-feet container.

3. Microwave oxidation system (MOS)

One important element of the installation is the device which purifies exhaust gases leaving the HR reactor. It is also used for afterburning of exhaust gases from furnaces, incinerators, thermal processing equipment for certain substances, from paint shops, arduous odour-emitting plants, and other sources that emit polluted exhaust gases.

In case of utilizing asbestos waste, a stream of exhaust gases may contain asbestos fibers and other harmful substances released athigh temperature (e.g. hydrocarbon emitted due to degradation and burning of varnished coatings and other contaminants). For effective purification of hot gases, a unique structure of the microwave generator has been designed, in the form of an insulated chamber filled with special crystals made of ceramics which absorbs microwaves. These elements are heated up to ~900-1100°C by microwaves emitted from radiators deployed on the metal casing of the chamber. In turn, gaseous contaminants become oxidized; however, this process is catalyzed through contact with the special ceramics heated to a very high temperature. This process is illustrated in Fig. 3 by a simplified scheme of the MOS reactor structure.

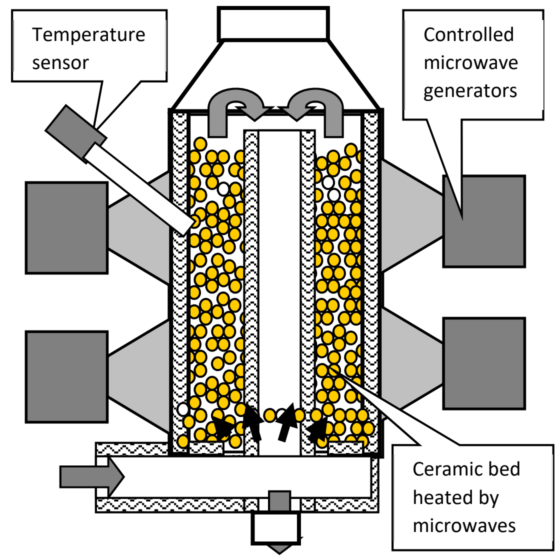


Figure 3. MOS construction scheme.

The temperature of the ceramic insert inside the MOS-reactor chamber is measured with a system of thermocouples deployed in each section. The MOS reactor with a capacity of 700 – 1000 Nm^3 is equipped with 12 microwave generators (2,450 MHz, 3-kW CW each). The microwave generators are connected with the radiators through water-loaded ferrite circulators. Microwave energy is emitted into the MOS reactor through horn antennas deployed on the metal casing. Further in this case, the antennas emit polarized waves hence their proper deployment reduces the coupling between individual generators and ensures the uniform heating of the ceramic inserts within the chamber. Measurements of the temperature distribution inside the chamber have shown that the heating up of ceramic inserts is sufficiently uniform (the temperature differences do not exceed 60-90 °C at ~1000 °C average temperature of the ceramics). The effectiveness of gases purification in the MOS reactor has been confirmed numerous times in studies conducted by certified laboratories. This effectiveness is illustrated, among others, by the data shown in Fig. 4. Two MOS reactors of capacities of 600 m^3/h and 4500 m^3/h are shown in Fig. 5.

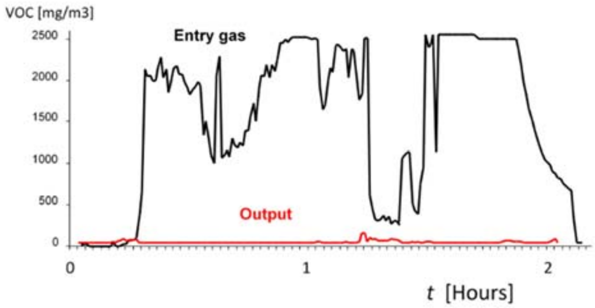


Figure 4. Measurements of volatile organic compounds (VOC) before and after MOS reactor.

(a)

(b)

Figure 5. MOS reactors installed in 20-feet containers, with capacities of 600 m^3/h (a) and 4500 m^3/h (b).

4. Municipal waste utilization and energy recovery

One of the most significant applications of the HR reactor is in the processing of municipal waste. Its storage is currently being reduced through legal regulations in many countries, which makes it necessary to use utilization technologies, i.e. by way of burning or gasification. The described microwave technology provides some unique opportunities in this respect because the process of thermal treatment in HR reactors can be conducted in a controlled atmosphere, e.g. in high shortage of oxygen. Therefore, the process of gasification of waste in the HR microwave reactor provides an advantage over conventional methods thanks to heating the whole volume of waste and without the transmission of heat energy through the walls of the chamber. No slag or other coke substances settle on the walls of the HR reactor and the material is heated volumetrically, and is gassed effectively when mixed in the ceramic drum. At the same time, through a controlled selection of the amount of air fed into the reactor, waste can be partially burned, thus creating only carbon monoxide (CO), which, together with other generated gases such as methane, hydrogen, butane, propane and other light hydrocarbons, are then used for powering the engine coupled with the power generator. Figure 6 presents a diagram of an installation for utilization of municipal and other waste with recovery of electricity.

The most important element of the installation is the HR 5000 microwave reactor, which allows the gasification of 3-4 tonnes of waste per hour and is equipped with two microwave generators with capacity of 75 kW or 100 kW (CW) each and operating at 915 MHz. Gases from this reactor are directed to the unit of the separator of oily and tar fractions, where they are pre-cooled and purified. The separated substances are returned to the HR 5000 reactor, in which, due to heating up with microwaves to a high temperature, they are decomposed into light hydrocarbons and then fed again to the separator. The stream of gases from the separator is then directed to the scrubber, where chlorine, fluorine, and sulphur (elements harmful to the operation of the motor) are separated. The purified gases power the engine coupled with the power generator. Such a system allows to generate e.g. ~2 MW of electric power when utilizing around 3-4 tonnes of selected waste with calorific value of around 12-14 MJ/kg.

The system with HR5000 reactor is shown in Fig 7. The estimated energy balance for the system with HR5000 reactor used for gasification of waste is shown in Fig. 8. The method described here of municipal waste utilization by microwave energy is protected by a patent.

5. Treatment of Asbestos Containing Materials

With its outstanding physical and chemical characteristics, asbestos has been used on a mass scale for many years. For its thermal resistance and chemical stability, it has been applied as a component in insulation materials, roof covering (Eternit panels), construction elements, insulation filling in ships, laboratory elements, and many others. This material has turned out though to be highly harmful to people, and to cause the so-called asbestosis (fatal lung disease) among others. In Poland, approximately 14m tonnes of asbestos waste have been removed, mostly in Eternit panels, which were once commonly used for roof covering. Also in other countries it has been necessary to neutralize millions of tonnes of this hazardous material.

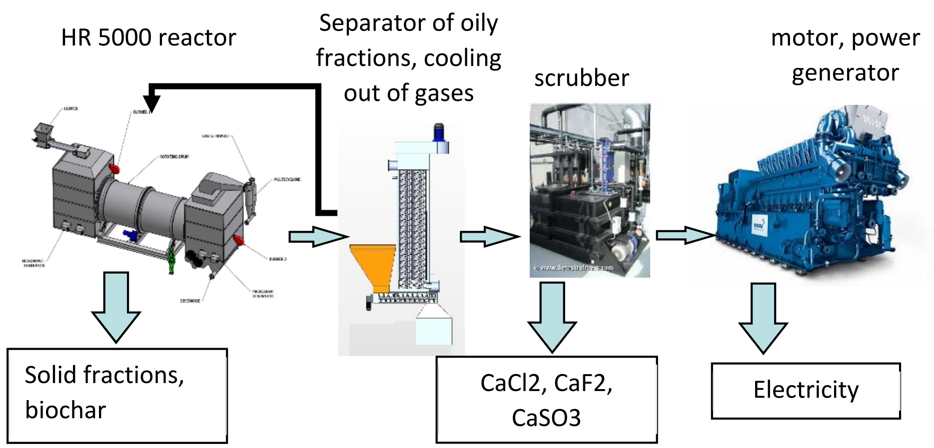


Figure 6. Installation for utilization of municipal waste with electricity production.

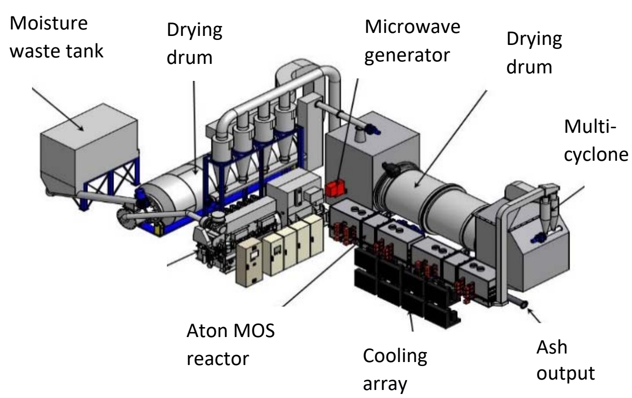


Figure 7. A system with HR 5000 reactor.

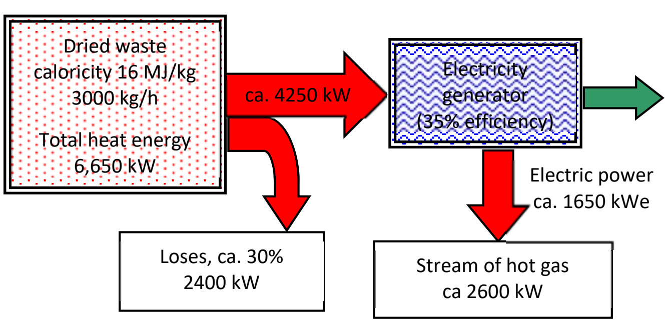


Figure 8. Energy balance for the system of microwave gasification of waste with caloricity of ~16 MJ/kg.

The scale of the problem associated with the removal and neutralization of asbestos waste is huge, and, like in other countries with large amounts of such waste, it is necessary to come up with, and to implement, new effective methods of its neutralization. There are unique opportunities in many developed countries (e.g. in Switzerland and the UK) to adopt such methods, which previously deposited asbestos waste being uncovered in order to neutralize it with physical and chemical methods. Also Poland currently has technical and economic capacities allowing to implement procedures for neutralizing asbestos with the new microwave thermal treatment (MTT) method, developed in the country and implemented at a technical scale.

The system of microwave reactors described in this article has also been successfully applied, following proper adjustments, in other processes of waste utilization. The applications include purification of ground contaminated with oil derivatives; utilization of municipal waste with energy recovery; utilization of medical waste, and so on. A separate scope of application is provided by the MOS gas purification system, described further in the article.

The essence of the technical solution developed and implemented by ATON-HT SA and developed by PROMETEUS Ltd. consists in thermal destruction of hazardous asbestos fibers by heating these with microwaves. In this method, which is protected by patent applications (P-209165 in

Poland and in EP16461505.6 in Europe), Eternit or other waste containing asbestos, after preliminary shredding (in a shredder with a specially pressurized structure), is mixed with small amounts of enhancers and fed to the chamber of the microwave reactor. As a result of heating this mixture up to a temperature of ~1200ºC, the crystalline structure of asbestos fibres transforms into an amorphous one. The transformation of the physical structure of asbestos as a result of thermal processing enhanced by microwaves (MTT method) is presented in Fig. 9.

(a) (b) (c)

Figure 9. (a) Chrysotile asbestos, (b) asbestos fibers in shredded Eternit, and (c) ATONIT, a product of Eternit processing in the microwave reactor by the MTT technology.

One characteristic of the method is the "contact-free" heating up of hazardous waste with a high concentrated microwave energy to the required temperature and in a controlled gas atmosphere, optimized for the process. Other existing conventional methods do not provide such capabilities. Importantly, the method in question - thanks to applying appropriate enhancing substances - involves an improved process of absorption of microwaves by the shredded waste virtually regardless of their composition, and moreover the temperature in which the whole transformation (destruction) of hazardous asbestos fibers takes place is reduced. It is significant for obtaining complete effectiveness of the transformation of all asbestos fibers into a safe material and for the improvement of energy-efficiency of the whole process.

The portable set consists of both reactors together with auxiliary devices, such as a system for loading waste, systems for cooling microwave generators, cabinets with chargers of microwave generators and control sets, all installed in two containers, as shown in Fig. 10. A similar system of a microwave reactor for utilization of waste has also been designed which has a capacity of 3-5 tones per hour. It is a stationary structure with two microwave generators of 75/100 kW CW power, each operating at 915 MHz. The choice of this frequency allows to heat thicker layers of waste moved inside the drum due to a deeper penetration of the electromagnetic field. Thus, it is possible to transport higher amounts of material to be thermally processed and heated up.

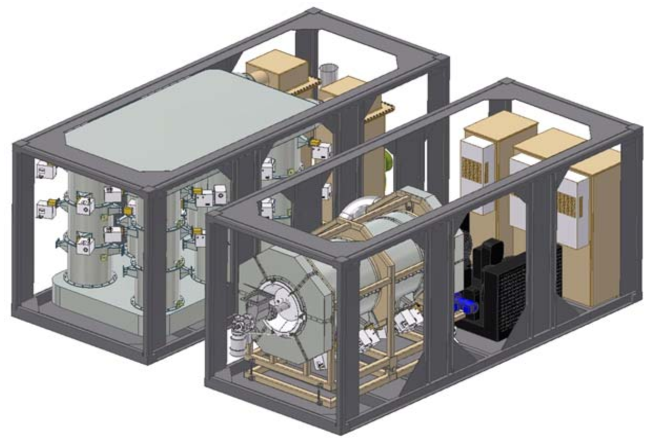


Figure 10. System for asbestos treatment installed within two standard containers.

6. Hospital waste management

A microwave system with HR reactors and MOS reactors can also be effectively applied for treatment of hospital waste. In this case, the waste material can be loaded into the shredder in standard plastic bags or left in loose form. The size of the bags should not exceed 120 *l*. The waste is crushed to a fraction of ~25 mm in an airtight chamber of the shredding machine, and then it is fed to the microwave process chamber using a worm heater. The purpose of the worm heater is to (*i*) make the water content in the waste evaporate, and (*ii*) preheat the waste. Optionally, the device does not need to be equipped with a shredder. Should this be the case, the shredded waste can be directly loaded into the worm heater. As a result of microwave heating to the temperature of ~800-850°C, the waste material is thermally cracked in the absence or deficiency of oxygen into smaller molecules, and thereby disposed of. The following processes, initiated by electromagnetic energy, occur in the reactor: decomposition, reduction and gasification. Their products include post-process gases and small amounts of solid fractions consisting of carbon and mineral substances. The released post-process gases are purified in the MOS microwave reactor. Its interior is made of a cylinder filled with special microwave-absorbing ceramic balls; this is where complex physical and chemical reactions take place at the temperature of ~1000°C influenced by electromagnetic waves generated by microwave emitters. The purification process occurs between the microwave-absorbing ceramics and the gas phase and it is initiated by electromagnetic excitation. Its key elements include separation of carbon particulates and mineral substances, breakdown (decomposition) of complex chemicals in strong magnetic fields, reduction, oxidation, and chemical reactions as a result of which chlorine combines with other elements.

In this novel process of gas purification, compound gaseous and solid substances (such as particulates) break down and contaminants in the pseudo-catalytic system are removed under the influence of strong magnetic fields. Both the microwave field and the surface of ceramic parts increase the rate of chemical reactions. The purification process might be exothermic (i.e. the heat is released with the products) but it always requires additional supply of microwave energy in order to (*i*) initiate some physical and chemical reactions, and (*ii*) maintain (stabilize) optimal process conditions. Once the gases pass through the MOS and the recuperator (heat exchanger), they are cooled down in the gas cooler before they reach the exhaust fan. Energy is recovered in the gas cooler (heat exchanger) from heating utility water to be used, for instance, back in the hospital.

7. Summary

Microwave energy has been applied in a number of significant processes for the neutralization of hazardous wastes, such as waste containing asbestos, as well as in utilization of municipal and other wastes. The most important advantages of this microwave method, which have already been verified on industrial scales, include the following capabilities:

- to heat up the waste volumetrically to very high temperatures;
- to conduct the process in a controlled atmosphere, e.g. lack of oxygen; and
- to control precisely the process parameters through a quick and precise stabilization of the temperature of the waste in the process chamber.

Conventional methods do not provide such capabilities, and the use of microwaves is currently a very beneficial and often the only way of effective neutralization of a number of wastes.

The practical experience gained in this work allows one to design and produce safe and long-lasting installations on an industrial scale, and to create new and often unique technological opportunities. So far, such installations are built and operated at a test stage. However, currently an installation is being prepared for utilization of municipal waste through gasification by microwaves with a capacity of over 3 tonnes per hour and which will generate electricity of ~2 MW.

For a few years now, odor-removal installations (MOS reactors of varying capacities) have been in operation. Their characteristics, including their high efficiency in burning out contaminants in the air and high reliability, provide a positive outlook onto a growing number of uses of installations based on the microwave technology described in this paper. The obstacle, however, is the high cost of building high-power microwave installations. Yet, the popularization of such microwave technologies will ultimately decrease the costs, and make these more competitive.

About the Author

Ryszard Parosa, researcher, president of PROMIS-TECH LTd (www.promistech.pl) and urrently scientific consultant in PROMETEUS LTd - was born in 1948 in Wroclaw, Poland. Graduated at the Technical University of Wroclaw, where received an M.Sc and a PhD in Electronic Science. In 1972–1983 he worked at the Technical University of Wroclaw as a university lecturer, and the next two years he was engaged in research at the Institute of Plasma Physic Nagoya University, in Japan. In 1985 he established a small high-tech company PLAZMATRONIKA LTd specializing in the application of microwave power for laboratories and industry. In his research he has concentrated on microwave plasma generation and the application of microwaves for drying of fruits and vegetables and for microwave pasteurization of fruits. Latterly he has concentrated in developing high power microwave equipment for processing many different forms of wastes. He is the author and co-author of several international patents and scientific publications.

E-mail: ryszard.parosa@promistech.pl

Microwave Flash Sintering

Yu. V. Bykov, A.G. Eremeev, S.V. Egorov, V.V. Kholoptsev, I.V. Plotnikov, K.I. Rybakov*, A.A. Sorokin

Institute of Applied Physics, Russian Academy of Sciences, Nizhny Novgorod, Russia

In recent years, significant interest has been attracted to the studies of very rapid ("flash") sintering of ceramics under a DC or low-frequency AC voltage applied to the samples undergoing heating[1]. The common feature attributed to flash sintering of various materials is fast densification (within several seconds to minutes) occurring at a certain threshold temperature of the furnace that depends on the product of the applied voltage and current. The densification is accompanied by a sharp increase in electrical conductivity of the samples undergoing sintering.

To date, it is almost generally agreed that a primary role in the initiation of flash sintering is played by the thermal instability associated with the Joule heating by the electric current flowing through the sample. The development of an instability in dielectrics heated volumetrically by internal thermal sources was described as early as in 1928 by V.A. Fock[2]. An increase in the electrical conductivity with temperature, inherent in most dielectric materials, can lead to a disbalance between the power deposited in the bulk of a sample and the heat loss from its surface.

The thermal instability also known as thermal runaway is a widely discussed issue in microwave processing of materials. As a rule, thermal runaway is viewed as one of the main shortcomings of the use of microwave heating for high-temperature processing of materials. However, we have recently demonstrated[3, 4] how the controlled development of the thermal instability is used advantageously for ultra-rapid microwave sintering of oxide ceramic materials (Al_2O_3, Y_2O_3, $MgAl_2O_4$, and Yb:$(LaY)_2O_3$). In the experiments, the samples were sintered in the applicator of a gyrotron system with a microwave power of up to 6 kW at a frequency of 24 GHz equipped with a computerized feedback power-control circuit[5]. The samples were heated at various constant ramp-up rates up to the preset temperatures T_{max}. The microwave power switched off automatically upon achieving T_{max}, and the sample cooled down along with the thermal insulation surrounding it.

Detailed results of the experimental studies on flash microwave sintering of oxide ceramics have been previously published [3,4,6], and only a brief account is presented here. The results of the flash microwave sintering of various ceramic materials are presented in Table 1. Ceramic samples in densities of 98-99 % have been obtained in the processes with a zero hold time, and the total duration of the high-temperature stage of the sintering process not exceeding several minutes.

Table 1. Results of flash microwave sintering experiments

Ceramic	Relative density [% th. d.]	T_{max} [C]	Heating rate [°C/min]	P_{abs} [W/cm^3]
Yb: $(La_{0.1}Y_{0.9})_2O_3$	98.5	1500	50	15
	98.5	1500	150	22
	98.0	1300	1350	120
Al_2O_3	98.6	1600	200	100
Y_2O_3	98.3	1600	100	40
3YSZ	95.8	1100	50	80
	81.3	1300	50	7 (< 10)
	98.8	1300	50	87
$MgAl_2O_4$	98.5	1780	150	25
$MgAl_2O_4$+1wt.% Y_2O_3	99.1	1780	150	27

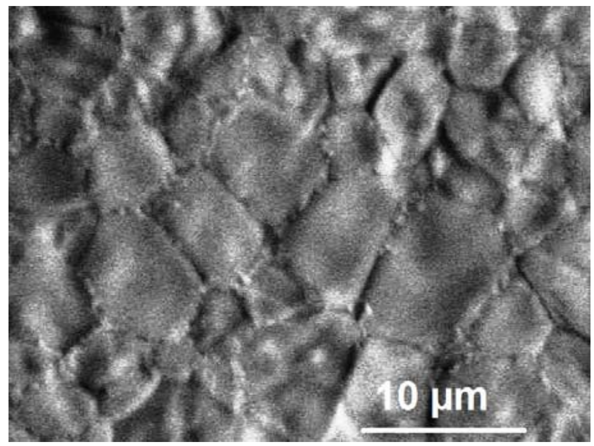
The microwave power absorbed in the sample undergoing heating increases its temperature and compensates the heat losses from it. The microwave power absorbed per unit volume of the sample, P_{abs}, can be estimated using the energy balance equations[3]. We have demonstrated that, similar to the DC/AC flash sintering studies, P_{abs} is a critical parameter that determines the onset of the flash sintering effect under microwave heating[4]. Interestingly, the critical value of P_{abs} for microwave flash sintering happens to be of the same order of magnitude (10-40 W/cm^3) as that determining the onset of flash sintering under the applied DC/AC electrical voltage[7].

Despite the growing number of publications on the DC/AC flash sintering, the mechanism responsible for the effect of fast densification remains a subject for discussion. As follows from estimates[8], the increase in the temperature in the bulk of the samples during the development of the thermal instability may reach 800°C relative to the temperature before the onset of flash sintering. However, such an increase is insufficient to explain the observed ultra-rapid sintering by the enhanced rates of thermally activated solid-state diffusion mass transport processes. The exponential growth of the conductivity *per-se* does not explain the accelerated sintering[8].

Our results of the microstructure characterization of the samples of various composition, microwave flash sintered in different heating regimes, revealed the features that are characteristic of the process of ceramic sintering in the presence of the liquid phase. The SEM studies of microwave sintered Yb: $(La_{0.1}Y_{0.9})_2O_3$ samples revealed traces of the (quasi-) liquid phase in the microstructure. At relatively low temperatures and/or heating rates, spherical tiny droplets were seen at grain boundaries of the sintered samples (Fig. 1a). With an increase in the heating rate (and hence in the microwave power absorbed in the sample), liquid phase surrounded the grains which acquired a concave shape (Fig. 1 b).

The kinetics of grain growth under microwave heating of Yb:$(La_{0.1}Y_{0.9})_2O_3$ samples is typical of liquid-phase sintering, and the average grain size is several times higher than the grain size obtained under conventional heating[3]. Fast segregation of dopants (La and Y, respectively) to the grain boundaries is observed in Yb:$(La_{0.1}Y_{0.9})_2O_3$ and $MgAl_2O_4$+1wt.% Y_2O_3 samples. The microstructure of the sintered samples depends strongly on the material composition. The Yb:$(La_{0.1}Y_{0.9})_2O_3$ sintered samples have uniform density and grain size profile across the sample diameter, despite strong temperature gradients existed in the samples during sintering[3]. On the other hand, a pronounced bimodal grain size distribution and abnormal grain growth are characteristic for $MgAl_2O_4$+1wt.% Y_2O_3 samples sintered to high density (Fig. 2).

(a)



(b)

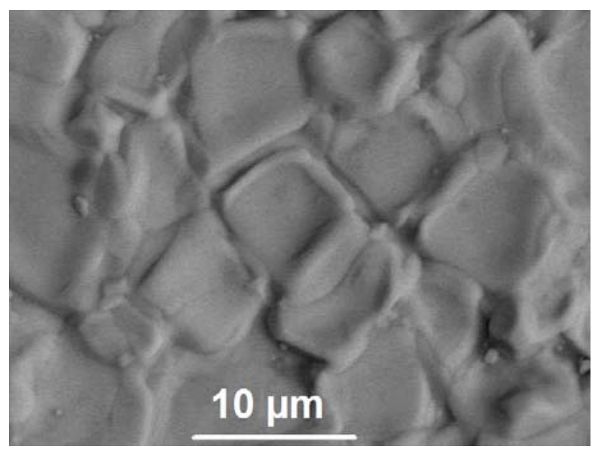


Figure 1. SEM images of the surfaces of flash microwave sintered Yb: $(La_{0.1}Y_{0.9})_2O_3$ samples: (a) heating rate 50 °C/min, maximum temperature 1500 °C; (b) heating rate 200 °C/min, maximum temperature 1500 °C.

Based on the analysis of the results of experiments with various materials, the following mechanism of flash sintering has been suggested[4]. Due to the abundance of impurities and defects in the near-boundary regions of particles, the melting temperature of the surface / boundary can differ noticeably from the melting point of pure solid material. Provided that the temperature and the density of deposited power are high enough, the particle surface pre-melting occurs well below the melting point of the bulk of grains, and a (quasi-) liquid phase with a low viscosity surrounds the grains. The liquid phase wets the particles completely due to the affinity of their chemical compositions. The capillary pressure attracts the adjacent particles together, and causes their rearrangement via rotation and sliding. The resulting densification is enhanced by particle shape accommodation due to fast diffusion mass transport through the quasi-liquid phase.

During volumetric heating, the highest temperature arises in the core of the sample and the process of particle surface melting starts from that position. In the course of densification, the liquid phase is extruded out into the more porous peripheral structure and contributes to its densification. Melting of particle surfaces results in a very sharp growth of absorption because the conductivity of the liquid phase is much higher than the conductivity of the solid material. In effect, a densification front, coinciding with the region of the maximum deposition of the microwave power, propagates from the core of the sample to its periphery, producing a fully dense ceramic material within a very short time.

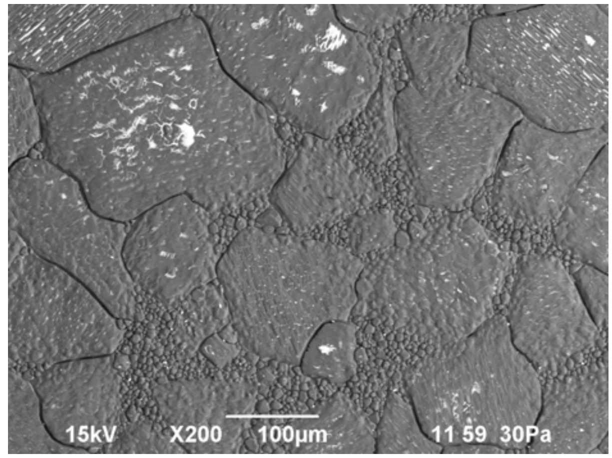


Figure 2. SEM image of the surface of $MgAl_2O_4$+1wt.% Y_2O_3 sample microwave sintered at 1780 °C with zero hold time.

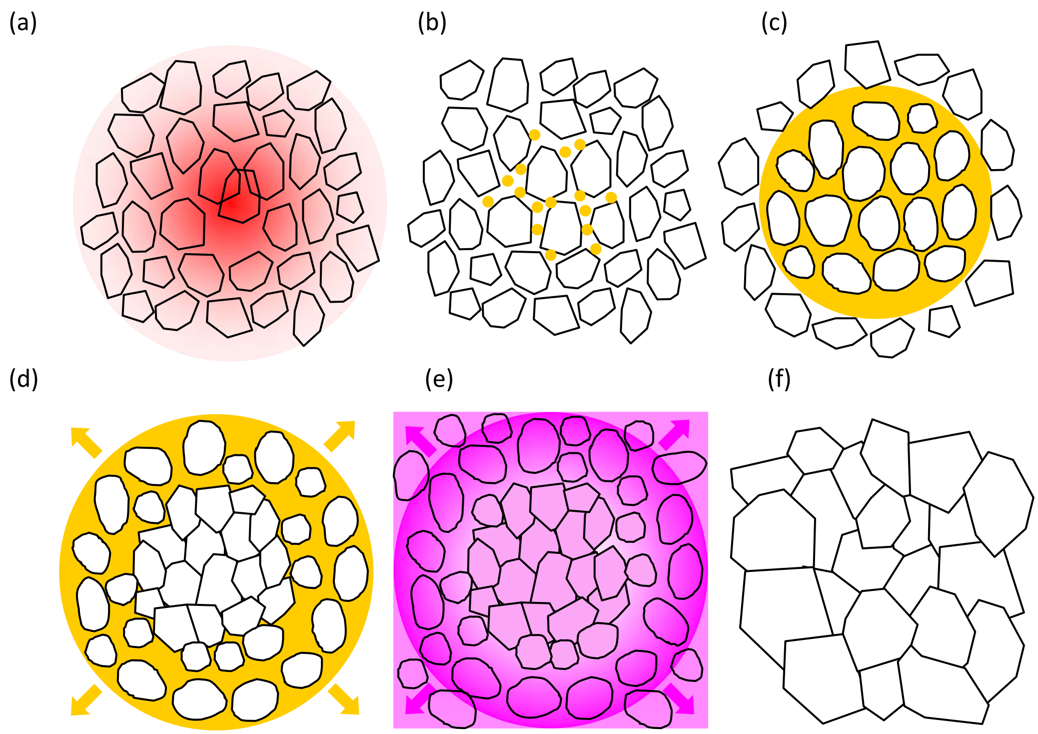


Figure 3. Illustration of the mechanism of flash sintering under volumetric heating: (a) volumetric heating creates a non-uniform temperature distribution with the core of the sample being the hottest; (b) droplets of liquid phase begin to form at particle surfaces; (c) liquid phase surrounds particles, the particle shape becomes rounded, particles rotate and slide relative to each other; (d) after rapid local densification liquid phase is squeezed from the core region towards the periphery of the sample; (e) the region of maximum power absorption propagates along with the liquid phase due to the elevated effective conductivity of the latter; (f) the propagation of the densification front results in achieving full density throughout the sample with a concurrent grain growth.

The volumetric energy deposition and surface thermal loss are characteristic of both the DC/AC and microwave flash sintering processes. However, it should be emphasized that from the potential applications standpoint the latter process has an obvious advantage as it requires no electrodes to supply the power to the articles undergoing sintering. Further research is necessary in order to optimize such factors as the properties of powder materials, materials composition, geometrical limitations imposed on the configuration of the sintered products, structural, functional properties, and performance of the materials obtained by flash sintering.

For further reading:

1. M. Yu, S. Grasso, R. McKinnon, Th. Saunders, M. J. Reece, "Review of flash sintering: materials, mechanisms and modeling," *Advances in Applied Ceramics: Structural, Functional and Bioceramics*, **116**, 24 (2017).
2. V.A. Fock, "On the theory of thermal breakdown," *Proc. Leningrad Institute for Physics and Technology*, **5**, 52 (1928), in Russian.
3. Yu.V. Bykov, S.V. Egorov, A.G. Eremeev, V.V. Kholoptsev, K.I. Rybakov, A.A. Sorokin, "Flash microwave sintering of transparent Yb:$(LaY)_2O_3$ ceramics," *J. Am. Ceram. Soc.* **96**, 3518–3524 (2015).
4. Yu.V. Bykov, S.V. Egorov, A.G. Eremeev, V.V. Kholoptsev, I.V. Plotnikov, K.I. Rybakov, A.A. Sorokin, "On the mechanism of microwave flash sintering of ceramics," *Materials* **9,** 684 (2016).
5. Yu. Bykov, A. Eremeev, M, Glyavin, V. Kholoptsev, A. Luchinin, I. Plotnikov, G. Denisov, A. Bogdashev, G. Kalynova, V. Semenov, N. Zharova, "24-84-GHz gyrotron systems for technological microwave applications," *IEEE Trans. Plasma Sci.* **32**, 67–72 (2004).
6. Yu. V. Bykov, S. V. Egorov, A. G. Eremeev, V. V. Kholoptsev, I. V. Plotnikov, K. I. Rybakov, A. A. Sorokin, "Sintering of Oxide Ceramics under Rapid Microwave Heating," in *Processing, Properties and Design of Advanced Ceramics and Composites* (*Ceramic Transactions,* Vol. 259), Eds. G. Singh, A. Bhalla, M.M. Mahmoud *et al*. Wiley, 2016, pp. 233–242.
7. R. Raj, "Analysis of the power density at the onset of flash sintering," *J. Am. Ceram. Soc.*, **99**, 3226–3232 (2016).
8. R. Raj, "Joule heating during flash-sintering," *J. Eur. Ceram. Soc*. **32,** 2293–2301 (2012).

About the Authors

The team of Laboratory for Microwave Processing of Materials at the Institute of Applied Physics (IAP), Russian Academy of Sciences, has been active in the field of microwave processing research for more than 25 years. The two main lines of the team's efforts are (1) design and development of systems for high-temperature processing of materials based on gyrotron millimeter-wave sources, and (2) investigation of the physical mechanisms underlying the interaction of microwave fields with materials. The gyrotron systems developed at IAP are currently in operation at a number of laboratories in Germany, Japan, China, and the United States.

Over the past decades, the team accomplished experimental studies of microwave sintering of a wide range of ceramic and composite materials. In recent years, the research carried out at the laboratory was focused on metal-ceramic functionally graded materials and transparent ceramics for laser applications. A theoretical study of the ponderomotive effect of the microwave electromagnetic field on the mass transport in solids has been accomplished, and novel models of effective microwave properties of conductive powder materials have been developed. An important recent development was the world's first demonstration of an ultra-rapid ("flash") sintering of oxide ceramic materials when heated by high-power microwave energy.

* Corresponding authors, E-mail: rybakov@appl.sci-nnov.ru

The research team (left to right):
A.A. Sorokin, I.V. Plotnikov, Yu.V. Bykov, A.G. Eremeev, K.I. Rybakov, S.V. Egorov, V.V. Kholoptsev

Microwave Drying of Seeds of Agricultural Interest for Ecuador

Ángel H. Moreno, Rafael Hernández, Isabel Ballesteros

Universidad Técnica de Cotopaxi, Av. Simón Rodríguez s/n, Barrio El Ejido, Sector San Felipe, Latacunga, Ecuador

Seed conservation in Ecuador

Agriculture is a predominant activity in Ecuador, especially in the province of Cotopaxi, where this sector is one of the principal economic activities. The main crops grown in Cotopaxi are sweet maize (chulpi), soft maize (choclo), corn, cocoa, sugar cane, potatoes, barley, bananas including plantain which is one of its variety, and dried beans[1]. For most of these crops, seed conservation is an important issue within agricultural practices that requires a drying process for maintaining seed viability during storage. Moreover, seed conservation in Ecuador is not only important from an agricultural point of view, but also as a strategy for the conservation of the country's wild plant biodiversity.

The main method of drying seeds in Ecuador is by exposing them to natural air and the sun (see Figure 1). This method is widely employed because this form of energy is free and abundant in all parts of the world, especially in tropical countries. Moreover, it does not require expertise. However, it has several disadvantages since it is extremely weather dependent, it involves very long drying times, and it requires large spaces for drying. In addition, the total control of the drying process is not possible, and a non-uniform drying of the products may be the result. Besides, the seeds are exposed to climatic variations, which diminishes their viability and quality. They are also susceptible to threats such as environmental pollution, pests, diseases, and contamination with soil and dust.

Since agricultural activity and biodiversity conservation are priorities for the Ecuadorian State[2], research into new technologies to improve the drying process would be of great interest. Accordingly, the main objective of this study is to assess the feasibility of applying microwave drying to seed conservation for its potential use in Ecuador.

Figure 1. Solar drying of quinoa seeds in Latacunga, province of Cotopaxi, Ecuador.

Microwave drying of seeds

The microwave drying of seeds has attracted considerable attention in the literature over the past decade. However, only a few works have focused on the drying of seeds for conservation purposes. The aim of most of these studies has been to assess the quality of the seeds, mainly, through four parameters: germination rates, seedling vigour, physico-chemical properties and pest control. An increase in the germination rate (G.r) of the species analyzed depends on the power density (P.d), expressed in Watt/gram, and the microwave drying procedure employed, as can be seen in Table 1.

Table 1. Germination rate by species and the power density employed (best results obtained to date)

Scientific name (common name)	P.d. [W/g]	G.r. [%]	Ref.
Cicer arietinum L. (bengal gram)	6	100	[3]
Glycine max (L) Merr. (soybean)	0.13	95	[4]
Triticum aestivum L. (wheat)	0.3	96	[5]
Vigna radiata (L.) R. Wilczek (Green gram)	6	80	[3]
Zea mays L. (corn)	2.8	97	[6]

In some cases, such as rapeseed, no germination occurs over the power ranges studied[7]. However, in some species, such as green gram, moth bean, and Bengal gram, it has been proven that the best germination rates are achieved using a low power density and a shorter exposure time[3]. A wide range of physico-chemical properties has been analyzed after microwave drying. The most common properties considered have been the bulk density of the seeds, stress cracking, and the percentage of chemical elements (amino acids, nucleic acids, magnesium, phenolic compounds, etc.). It has been found that physical properties are affected by microwave assisted drying. The results indicate that an increase in drying rate at higher power levels reduces the bulk density of corn[6, 8-10]. Besides, in rapeseed[7] more than 60% of broken seeds were observed in the range 400-800 W.

Microwave irradiation has also been used to reduce the contagion by different insects with promising results. Reddy et al.[11] found that the percentage of wheat seeds infected by Fusarium graminearum could be reduced to below 7% (from 36% for the controls), while retaining a seed germination level as high as 85%. Similar results were obtained by Warchalewski et al.[12] with a significant level of insect pest elimination, with exposure times of up to 90 seconds and grain temperatures not exceeding 64°C. Moreover, Pande et al.[13] achieved a 99.5% insect mortality rate, in green gram seeds, without reducing quality parameters such as the green color of seeds at high power levels (808 W for 80 seconds). However, no germination tests were performed to evaluate the effect of high power energy on seed viability.

The studies analyzed employ a wide range of methods. For example, different amounts of seeds, a broad range of power levels, and different physico-chemical parameters to assess quality criteria have all been used in these seed studies. For these reasons, it is difficult to draw general conclusions. The common pattern in the cases studied, was for microwave assisted drying to greatly reduce the drying time compared to other techniques[4, 6, 14]. In general, with microwave techniques, the drying rate increases as the microwave power and temperature increase[15-18]. In addition, it is generally agreed that energy efficiency is enhanced when microwave technology is applied for drying purposes. However, the number of studies that examine this aspect in seeds is limited[19, 20-24].

For the reasons mentioned above, microwave drying is highly recommended for drying seeds because essentially it reduces the drying time and energy consumption. However, in the case of biological materials it is necessary that the quality parameters of the final dried product be carefully analyzed. Therefore, to establish the feasibility of microwave technology for the drying process, various parameters such as drying time, energy efficiency and quality, must be considered simultaneously. Most of the studies are based on drying rate and quality criteria, with energy consumption being evaluated only in a few studies[16,19,25].

In most cases, tests have revealed that microwave drying increases the drying rate without impairing the quality of the final product. Nonetheless, the methodology employed, and the power level need to be carefully chosen in order to avoid damage to the samples, (e.g. the germination rate and the quality of the final product decrease at higher power levels or under overexposure to microwave radiation)[6,12,26]. It should also be pointed out that the time required for drying the seeds depends not only on the power or temperature employed but also on the initial moisture content[6]. A higher initial moisture content requires a longer exposure time, which is a matter for concern, especially in relation to tropical or subtropical species. One of the problems that may cause a significant decrease in the germination level is the presence of hot spots during the drying process. This subject has been discussed by Manickavasagan et al.[27, 28] where they observed that temperature peaks above 65°C prevent the process of germination from taking place.

Most of the studies analyzed have been performed in order to investigate the effects of different microwave power levels on the quality parameters mentioned above. Fixed microwave power levels have been applied during the entire process, but no temperature control has been included in the studies. The absence of a temperature control causes the presence of hot spots. Moreover, during the drying process, the mass of the product decreases, due to the loss of the initial moisture and, therefore, the power density increases during the last part of the drying stage, leading to high temperatures, which cause charring of the sample and, consequently, a decrease in seed viability and a deterioration of the quality of the final product. According to Li et al.[29], to achieve the ideal drying effect over the entire microwave drying process, the sample temperature must be controlled and the microwave power must be adjusted accordingly, especially in the final stage of the drying process.

Temperature control during the microwave drying process has been performed only in a few studies[29, 30, 31, 32]. However, in these cases the drying effects in terms of time, energy efficiency and product quality were improved. The sample temperature in microwave drying is more difficult to control than in hot-air drying, where the product temperature never rises above the air temperature. Therefore, the best method of temperature control is to adjust the power taking into account the power–moisture content relationship and feedback temperature control[32]. Furthermore, adequate temperature and power adjustment will avoid the need to use combined techniques, which are more complex and expensive to implement.

Our contribution to microwave seed drying

Quinoa and amaranth, crops indigenous to the Andean region, have acquired great importance worldwide due to their nutritional properties and their potential to adapt to different agroclimatic conditions. Owing to the morphological characteristics of quinoa seed, the drying of this material is very susceptible to climatic variations, especially to any increase in environmental humidity, which can cause the seed to germinate during the air-drying process. Moreover, the tiny size of amaranth seeds also makes them very susceptible to climatic variations. For example, they can easily be dispersed by the wind with the consequent loss of product. In short, an improvement of the drying process based on techniques that allow a better control and make it more energy efficient is necessary.

Previous studies on corn seed drying[6] have shown that the use of microwave technology reduces the drying time and energy consumption without affecting the viability of the seeds. However, hardly any data are available on the quality of the seeds. Furthermore, in respect of quinoa and amaranth, there are no data at all relating to the use of this technology in the drying process.

In the project titled "Evaluation of the microwave drying process of seeds of agricultural interest for the Cotopaxi Province", which is being currently developed by our research group[33], the drying process of quinoa and amaranth seeds in a microwave oven and in a conventional electric dryer at three different temperatures is being evaluated. The drying curves of the seeds studied will be obtained for the three temperatures in two types of furnaces. This will allow us to determine the drying time required for each technology and temperature in order to attain the optimal conditions of humidity for the conservation of the seeds. Moreover, the energy consumption will be measured in order to evaluate the energy efficiency of each of the experiments.

To assess the quality of the dried seeds achieved by the different technologies employed, four parameters will be evaluated: grain surface deterioration or contamination, germination rates, seedling vigour and physico-chemical properties. A comparison of the results obtained, together with the energy efficiency data, will make it possible to determine whether the use of microwave technology, at controlled temperature, reduces the drying time and energy consumption without impairing the viability and quality of the seeds. If the results expected of this project are achieved, they will contribute to the development of an efficient and competitive technology for the drying of seeds of agricultural interest for implementation on a commercial scale in the province of Cotopaxi.

Acknowledgements

Financial support from Universidad Técnica de Cotopaxi (Ref. UTC-2017-PI-01) is greatly acknowledged.

For further reading:

1. GAD-Cotopaxi (2015). Actualización del Plan de Desarrollo y Ordenamiento Territorial de Cotopaxi 2025. Gobierno Autónomo Descentralizado (GAD) de la Provincia de Cotopaxi, Latacunga, Ecuador.
2. SENPLADES (2013). Plan Nacional de Desarrollo /Plan Nacional para el Buen Vivir 2013-2017. Secretaría Nacional de Planificación y Desarrollo (SENPLADES), Quito, Ecuador.
3. Ragha, L. (2011) Effects of low-power microwave fields on seed germination and growth rate, Jour. Electromag. Analysis and Applications. 3 (5) : 165–171.
4. Shivhare, U.S., Raghavan, G.S.V., Bosisio, R.G., Giroux, M. (1993) Microwave drying of soybean at 2.45 GHz. J Microw Power Electromagn Energy. 28 : 11–17.
5. Soproni, V.D., Vicas, S.M., Leuca, T., Arion, M.N., Hathazi, F.I., Molnar, C.O. (2012). High frequency electromagnetic field modeling and experimental validation of the microwave drying of wheat seeds, Progress In Electromagnetics Research B. 41 : 419–439.
6. Gursoy, S., Choudhary, R., Watson, D.G. (2013). Microwave drying kinetics and quality characteristics of corn. International Journal of Agricultural and Biological Engineering. 6 (1) : 90–99.
7. Łupińska, A., Kozioł, A., Araszkiewicz, M., Łupiński, M. (2009). The changes of quality in rapeseeds during microwave drying. Drying Technol. 27 (7-8) : 857–862.
8. Shivhare, U.S., Raghavan, G.S.V., Bosisio, R.G. (1991). Drying of corn using variable microwave power with a surface wave applicator. Journal of Microwave Power and Electromagnetic Energy.26 (1) : 38–44.
9. Shivhare, U.S., Raghavan, G.S.V., Bosisio, R.G. (1992). Microwave drying of corn II: constant power, constant operation, Transaction of The ASAE. 35 (3) : 951–957.
10. Shivhare, U.S., Raghavan, G.S.V., Bosisio, R.G., Mujumdar, A.S. (1992). Microwave drying of corn III: constant power, intermittent operation. Transactions of the ASAE. 35 (3) : 959–962.
11. Reddy, M.V.B, Raghavan, G.S.V., Kushalappa, A.C., Paulitz, T.C. (1998). Effect of microwave treatment on quality of wheat seeds infected with Fusarium graminearum: J. Agric. Eng. Res. 71 : 113–117.
12. Warchalewski, J., Gralik, J. (2011). Changes in microwave-treated wheat grain properties, In: Advances in Induction and Microwave Heating of Mineral and Organic Materials, Chapter 22, pp. 503–530.
13. Pande, R., Mishra, H.N., Singh, M.N. (2012). Microwave drying for safe storage and improved nutritional quality. Jour. Agricult. & Food Chemistry. 60 (14) : 3809–3816.
14. Vadivambal, R., Jayas, D.S. (2007). Changes in quality of microwave-treated agricultural products-a review. Biosystems Engineering. 98 (1) : 1–16.
15. Chua, K.J., Chou, S.K. (2005). A comparative study between intermittent microwave and infrared drying of bioproducts. International Journal of Food Science and Technology. 40 (1) : 23–39.
16. Ozkan, I.A., Akbudak, B., Akbudak, N. (2007). Microwave drying characteristics of spinach. Journal of Food Engineering. 78 (2) :577–583.
17. An, K., Zhao, D., Wang, Z., Jijun, W., Yujuan, X., Gengsheng, X. (2015). Comparison of different drying methods on chinese ginger (Zingiberofficinale Roscoe): changes in volatiles, chemical profile, antioxidant properties, and microstructure. Food Chemistry. Part B. 197 : 1292–1300.
18. Rayaguru, K., Routray, W. (2011). Microwave drying kinetics and quality characteristics of aromatic pandanus amaryllifolius leaves, International Food Research Journal. 18 (3) : 1035–1042.
19. Sharma, G.P., Prasad, S. (2006). Optimization of process parameters for microwave drying of garlic cloves. Journal of Food Engineering. 75 (4) : 441–446.
20. Sharma, G.P., Prasad, S. (2006). Specific energy consumption in microwave drying of garlic cloves. Energy. 31 (12) : 1921–1926.
21. Holtz, E., Ahrné, L., Rittenauer, M., Rasmuson, A. (2010). Influence of dielectric and sorption properties on drying behaviour and energy efficiency during microwave convective drying of selected food and non-food inorganic materials. Jour. Food Engin. 97 (2) : 144–153.
22. Motevali, A., Minaei, S., Khoshtagaza, M.H. (2011). Evaluation of energy consumption in different drying methods", Energy Conversion and Management. 52 (2) : 1192–1199.
23. Motevali, A., Minaei, S., Khoshtaghaza, M.H., Amirnejat, H. (2011). Comparison of energy consumption and specific energy requirements of different methods for drying mushroom slices. Energy. 36 (11) : 6433–6441.
24. Kassem, A.S., Shokr, A.Z., El-Mahdy, A.R., Aboukarima, A.M.,. Hamed, E.Y. (2011). Comparison of drying characteristics of Thompson seedless grapes using combined microwave oven and hot air drying. Journal of the Saudi Society of Agricultural Sciences. 10 (1) : 33–40.
25. Jiang, H., Zhang, M., Liu, Y., Mujumdar, S.A., Liu, H. (2013). The energy consumption and color analysis of freeze/microwave freeze banana chips. Food and Bioproducts Processing. 91 (4) : 464–472.
26. Wesley, R.A., Lyons, D.W., Garner, T.H., Garner, W.E. (1974). Some effects of microwave drying on cottonseed. Journal of Microwave Power. 9 (4) : 329–340.
27. Manickavasagan, A., Jayas, D.S., White, N.D.G. (2007). Germination of wheat grains from uneven microwave heating in an industrial microwave dryer, Canadian Biosystems Engineering.49 : 23–27.
28. Manickavasagan, A., Jayas, D.S., White, N.D.G. (2006). Non-uniformity of surface temperatures of grain after microwave treatment in an industrial microwave dryer. Drying Technology. 24 (12) : 1559–1567.

29. Li, Z., Raghavan, G.S.V., Orsat, V. (2010). Temperature and power control in microwave drying. Journal of Food Engineering. 97 (4) : 478–483.
30. Nair, G.R., Li, Z., Gariepy, Y., Raghavan, G.S.V. (2011). Microwave drying of corn (Zea mays L. ssp.) for the seed industry. Drying Technology. 29 (11) : 1291–1296.
31. Li, Z., Raghavan, G.S.V., Wang, N., Gariepy, Y. (2009). Real-time, volatile-detection-assisted control for microwave drying. Computers and Electronics in Agriculture. 69 (2) : 177–184.
32. Li, Z.,. Raghavan, G.S.V., and Wang, N. (2010). Carrot volatiles monitoring and control in microwave drying. LWT - Food Science and Technology. 43 (2) : 291–297.
33. Moreno, A.H., Hernández, R., Ballesteros, I. (2016). Evaluación del proceso de secado en Horno Microondas de Semillas de Interés Agrícola de la Provincia de Cotopaxi. Proyecto de Investigación. Dirección de Investigación. Universidad Técnica de Cotopaxi, Latacunga, Ecuador.

About the Authors

Dr. Ángel H. Moreno was born in Cienfuegos, Cuba. He graduated from the University of Cienfuegos in 1989, where he received the title of *"Thermal Engineer"*. He obtained the title of "*Doctor of Industrial Engineering*" from the University of Oviedo, Spain, in 1999, within the Doctoral Program of "*Technology and Energy Saving*". In 2001, he was awarded "*Extraordinary Doctorate Award*" by the Doctorate Commission and the Governing Board of the University of Oviedo. He is currently working as a professor and researcher at the Faculty of Engineering Sciences, in the Technical University of Cotopaxi, where he is a member of its Scientific Committee. His research activity is mainly focused on the fields of increasing the energy efficiency of industrial equipment and processes, the use of renewable energy in rural areas and the reduction of pollutant emissions from the combustion of fossil fuels.

E-mail: angel.hernandez@utc.edu.ec

Dr. Rafael Hernández Maqueda was born in Madrid, Spain. He graduated in 2001 at the Universidad Autónoma de Madrid, where he obtained a degree in *Biology*. After graduation, he worked at the Royal Botanical Garden, CSIC in Madrid, Spain, where he was awarded his PhD within the Doctoral Program of "*Evolutionary Biology and Biodiversity*". Currently, he is working as a professor and researcher at the Facultad de Ciencias Agropecuarias y Recursos Naturales, (Universidad Técnica de Cotopaxi, Ecuador). In addition, he is the coordinator of its Scientific Committee. His research activity is mainly focused on analyzing biodiversity for agronomical or conservation purposes.

E-mail: rafael.hernandez@utc.edu.ec

Dra. Isabel Ballesteros Redondo was born in Toledo, Spain. She graduated in 1998 at the Universidad Complutense de Madrid, where she obtained a degree in *Biology*. After graduation, she obtained her PhD from the same University within the Doctoral Program of *"Genetics"*. She has held two postdoctoral position in the field of Plant Genetics. In 2012, she obtained a Master in *Organic Farming*. At present, she is a professor and researcher at the Faculty of Farming and Natural Resource Sciences, in the Technical University of Cotopaxi, where she is a member of its Scientific Committee. Her research activity is mainly focused on analyzing the local biodiversity and promoting sustainable local development in rural areas.

E-mail: maria.ballesteros@utc.edu.ec

Emerging Applications for Microwave Technology in Chemistry, Polymers and Waste

Tunjar Asgarli, Markus Reichmann, Niko Voit

MUEGGE GmbH, Hochstrasse 4 – 6, 64385 Reichelsheim, Germany

Applications and Discussion

It is obvious that as the microwave generation and transmission get more affordable, the number of its applications is increasing. The straightforward example for the integration of microwave is the food industry (Fig. 1). Besides the general household oven consuming microwave, the technology has found use for other applications in the food industry. The possibility of electronic re-arrangement of the power in microwave technology, thus supplying a different range of temperatures in the defined space enables microwave to be used in different fields of food industry such as (re)heating, baking, tempering, dehydration, pasteurization and even popping.

In contrast to classical variants of applications, there is a constant demand for new dietary varieties in the modern food industry that can be implemented quickly and efficiently by means of microwave energy. Amongst these one may mention the popping up of cereals and cheese products, the preparation of dried meat chips, as well as the preservation of fish and meat products by microwave assisted brine injections.

Furthermore, heating ability soon was utilized in production fields, namely rope, rubber and wood. In contrast with conventional ovens, microwave resonators can be adapted to increase the efficiency in the production line.

The latest production facilities with a microwave heating are so called conti-presses, with microwave warming integrated into the pressing area, for the production of lightweight building boards for doors and cupboard systems or even robot-guided microwave generators for the selective drying of moisture nests in multilayer sanitary ceramic parts (Fig. 2).

It is only logical that this claim has spread beyond and is further adapted to bio and pharmaceutical industry. In these industry sectors, the microwave technology is predominantly used for drying and disinfection applications in batch processes. The possibility for sterilization with microwave technology by proper refinement has created a large market as an alternative technology in latter fields.

Figure 1: Microwave fruit dryer

Microwave technology, which is still sometimes not preferred because of safety cautions and radiation effects, is evolving to supply a healthier environment to mankind. Promising microwave applications in the future are related to the chemical industry, namely pyrolysis and recycling. Microwave pyrolysis application, which is already well understood and has the current status of being optimized, opens a possibility for production of many products from inputs such as bio/plastic waste (Fig. 3).

After a process enhancement in the area of batch systems, continuously working facilities for pyrolysis enabling future-proof and continuous provisioning with “green energy” in industrial scale are now being developed. One of the challenges here is the infeed of microwave energy into the process chamber, that has to be carried out via dirt-proof, atmosphere-separating window system within the waveguides. In other areas of the chemical industry, relevant for climate and environment, highly efficient microwave systems are used for heating processes during the production of extruded, bio-soluble plastic films of metal-free polylactic-acids and for the foaming and stabilization of polyurethane and melamine foams with a very low density that are used as heat and sound insulation.

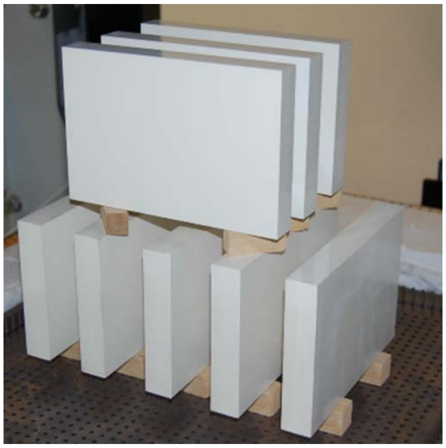


Figure 2: Sputter targets fabricated by MW debinding

Another future application is that of microwave recycling. In such systems, production of expensive fluorine species and new PTFE/PEEK materials is intended from unmixed recyclate of PTFE and plastics. Several high power microwave generators are used in fluidized bed reactors that are enriched with a susceptor material in a chemically high reactive steam atmospheric pressure to achieve a swift warming and reprocessing of the fluorine species.

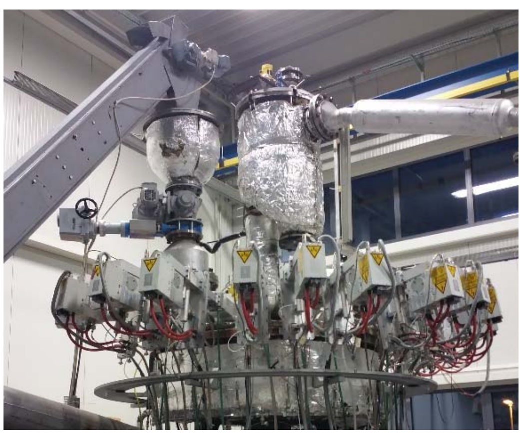


Figure 3: Microwave pyrolysis reactor (installed at Bionic Laboratories BLG GmbH, Germany, www.bionic-world.eu)

A further new area of application is the drying and hardening of fiber-reinforced composites directly during the production of fiber-reinforced composite profiles in the process of pultrusion, not in a batch process as hitherto (Fig. 4). The microwave located in the mold is utilized for a preheating and hardening of resins and coatings. Microwave technology is also the basis for integration with emerging plasma technology applications such as diamond growth. The microwave generator technology is expected to supply a preeminent consistency concerning the stability of frequency and performance over a long period of time that is essential for growing of crystals. A partitioned geometry of the resonator and an application of a specific wave propagation serves as an excellent plasma guidance and therefore offers outstanding results.

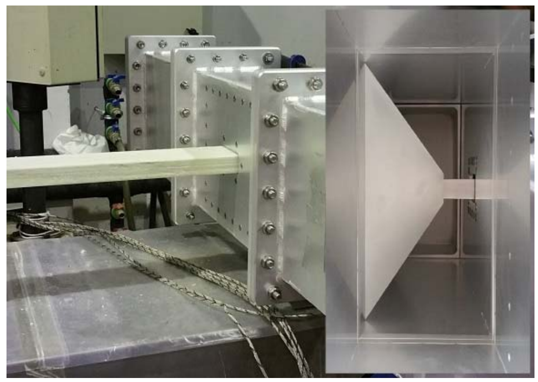


Figure 4: Microwave pultrusion die of fiber-reinforced profiles

Rephrasing the initial idea, miniaturized units are gaining acceptance in the market. Adapting to the concept, shrinking large standard components in microwave technology into small dimensions for size optimization and cost effectiveness has reently attracted much attention Furthermore, there future developments related to solid-state microwave components, establishing small-size and inexpensive solutions to all the previously discussed microwave applications. The main reason for the implementation of the solid-state technology is a fundamental minimizing of service costs thanks to omitting the magnetron tubes working with limited service life, in the power generators (Fig. 5). Interestingly, power ranges of 400 W can be achieved with 1-2 chip usage, which can be further amplified to 2-3 kW application with multi-chip arrays.

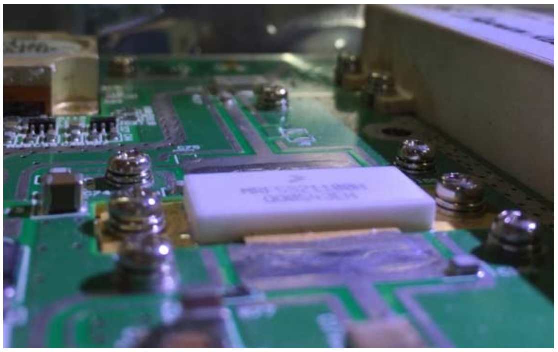


Figure 5: Microwave solid-state-amplifier

Conclusion

Industrial heating using microwaves is required for processing an emerging number of novel materials in a large variety of application areas. Different demands and specifications call for individual and customized solutions concerning coupling of the microwave energy to the material, necessary power level, and frequency to be applied. As the avoidance of downtime is more and more relevant, recent developments in solid state components will lead to an enormous booming of solid-state technology in microwave power supplies. Consequently, new concepts towards multi-channel processing based on solid-state power supplies will have to be developed for facilitating high-power microwave applications with this technology.

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- "InnoREX" (Grant Agreement Number 309802) for the research into and the development of highly efficient microwave systems for heating processes during the production of extruded, biodegradable polymers, and
- "COALINE" (Grant Agreement Number 609149) for the research into and the development of microwave application in the drying and hardening of fiber-reinforced composites directly during the production of fiber-reinforced composite profiles in the process of pultrusion.

It is also worth noticing that a more comprehensive presentation about the emerging applications of microwave technology with illustrative information likewise this article's content is planned to be presented at 16th International Conference on Microwave and High Frequency Heating: AMPERE 2017.

About the Authors

Tunjar Asgarli joined Muegge GmbH in 2016. He is a plasma applications engineer, focusing on the development of microwave plasma sources.

Dr. Markus Reichmann joined Muegge GmbH in 2011. As a senior manager microwave technology, he focuses on the development of microwave applications.

Niko Voit joined Muegge GmbH in 2010. As a manager of research, development and engineering, he manages the R,D&E Department, and focuses on the development of power electronics.

About the Company:

Muegge GmbH is a solution provider in the field of industrial heating and plasma technology, offering solution packages and value added services with continuous improvement in quality and efficiency. The R,D&E Department of Muegge GmbH keeps close collaboration with customers to achieve product excellence, optimally matching the application needs. The department consists of 5 research teams, namely Power Electronics, Microwave Technology, Plasma Technology, Engineering and Funded Projects; working together to establish the basic understanding required to deliver a product according to the specified requirements and projects' scopes.

E-mail: info@muegge.de

Ricky's Afterthought:

Beamed Wireless Power-Transfer Using a Dynamic Metasurface Aperture

A. C. (Ricky) Metaxas

Life Fellow, St. John's College, Cambridge, UK
E-mail: acm33@cam.ac.uk

How often is one frustrated for having forgotten to charge a cell phone, an iPad or other similar devices? What a brilliant idea to be able to devise a system whereby any device within a room could be charged automatically without having to connect cables to the supply?

This is what researchers at Duke University, Intellectual Ventures' Invention Science Fund, and the University of Washington in the USA are working on. Two aspects of this work were discussed independently in our Afterthought articles, first in Issue 75 where I introduced WC Brown's concept of wireless power transmission highlighting the proposed Space Solar Power System, put forward by the Japanese Aerospace Exploration Agency. Then in Issue 82 I focused on a new type of artificial material, called a metamaterial. Well, a focused microwave beam and a metamaterial are at the heart of the scheme discussed here.

"Imagine an electromagnetic wave approaching the large number of tiny cells of the metamaterial, and say you can tune each cell to manipulate the wave in a specific way, you can then dictate exactly what the field looks like when it comes out on the other side.", stated Professor Smith who leads this research initiative at Duke University. According to this study, a flat metamaterial device no bigger than a typical flat-screen television could focus beams of microwave energy, envisaged in the X or K bands (8-12 or 18-26.5 GHz, respectively) down to a spot about the size of a cell phone within a distance of up to 10 m. Although such a scheme is capable of powering more than one device at the same time, one caveat is that each device to be processed must signal its presence in the room and communicate its location and orientation with respect to the transmit aperture. This is within the capability of present day technology.

The proposed Fresnel-zone (near-field) approach of this project takes advantage of widely used LCD technology to enable seamless wireless power delivery to all kinds of smart devices. One other important aspect of this wireless power-transfer scheme is the ability to safely direct focused beams of microwave energy to charge specific devices, while avoiding unwanted exposure to people, pets and other objects.

How near are the researchers in achieving such a goal? They claim that all the principles have been established and confirmed, and such a system can be made. It is simply a question of building such an array of different items and testing it. In their original paper, submitted in the arXiv archive of Cornel University Library in 2016, they state, "We find that approximate design formulas derived from the Gaussian optics approximation provide useful estimates of system performance, including transfer efficiency and coverage volume. The accuracy of these formulas is confirmed using numerical calculations".

For further reading:

D. R. Smith, V. R. Gowda, O. Yurduseven, S. Larouche, G. Lipworth, Y. Urzhumov, M. S. Reynolds, "An analysis of beamed wireless power transfer in the Fresnel zone using a dynamic, metasurface aperture", Submitted to arXiv pre-print repository, 2016, ID:1610.06799.

https://arxiv.org/ftp/arxiv/papers/1610/1610.06799.pdf

AMPERE 2017

16th International Conference on Microwave and High Frequency Heating

September 18-21, 2017, Delft University of Technology, Delft, The Netherlands

http://www.ampere2017.nl

The AMPERE-2017 conference is an occasion for researchers and engineers, from academia and industry, to exchange innovative research ideas and to promote the most recent advances in the applications of microwave and high frequency technologies, at both laboratory and industrial scales.

Program and special interest theme – Process Intensification

The program evolves around microwave and high frequency development and applications:

- Dielectric properties measurement
- Microwave and high frequency material interaction
- Industrial applications and scale-up
- Microwave and high frequency supply design
- Modeling of microwave and RF power applications
- Microwave and RF plasma applications

In addition, a special interest theme of the conference is chemicals and materials process engineering, including:

- Microwave assisted chemistry and processing
- Process intensification with electromagnetic energy
- Sustainable Chemistry& Biochemistry
- Biomass and waste processing
- Materials processing
- Medical and biological applications

Abstract submission deadline - April 30th 2017.

Plenary speakers (a partial list):

- José Manuel Catalá Civera (Universitat Politècnica de València)
  "Measurement technologies for emerging microwave heating processes"
- Carlo Groffils (Microwave Energy Application Consult) "Microwave applications in the Benelux"
- Richard van de Sanden (Dutch Institute for Fundamental Energy Research (DIFFER))
  "Nonequilibrium plasma chemistry to improve kinetics and selectivity of chemical transformations"
- Georgios Dimitrakis (University of Nottingham). "The use of dielectric spectroscopy for process monitoring and optimization"

Short course

Prior to the conference a short course will be organized on radio frequency and microwave heating, including:

- Basic Principles of RF and MW Heating (Prof. José Fayos-Fernández, Universidad Politécnica de Cartagena)
- Dielectric Property Measurements (Prof. José M. Catalá Civera, Universitat Politècnica de València)
- Microwave Applicator Design Criteria (Prof. Paolo Veronesi, University of Modena and Reggio Emilia)
- Modelling of RF and MW Heating Systems (Prof. George Dimitrakis, University of Nottingham)
- Review of Industrial Applications (Prof. Vaidhy Vaidhyanathan, Loughborough University)

Venue and social program:

- The conference venue is the Aula Congress Center of the TU Delft, located in the heart of the university campus.
- Gala Dinner at De Lindenhof, a 1880s cultural centre for the personnel of the Gist and Spirits Industry.
- Opening reception at Royal Delft, Royal Dutch Delftware Manufactory "De Koninklijke Porceleyne Fles".
- Boat tour and Reception in Town Hall Delft
- Royal Delft, tile workshop, during this workshop you create your own hand painted Delftware tile. You can choose for a classical design or make your own.
- Guided Tour Mauritshuis, The Mauritshuis in The Hague is home to the Best More than two hundred top works from Dutch and Flemish masters.
- September 19th 'Prinsjesdag' the annual address of the monarch to parliament, which is accompanied by celebration around the honorary procession of the monarch through The Hague.

Other Upcoming Events

IMPI's 51st Annual Microwave Power Symposium
June 20-22, 2017, Miami, FL, USA
http://impi.org/symposium-short-courses/

9th Int'l Conference on Advanced Materials (ROCAM 2017), and 2nd Int'l Symposium on Dielectric Materials & Applications (ISyDMA 2017)
July 11-14, 2017, Bucharest, Romania
http://rocam.unibuc.ro

Summer School in Ultrasound and Microwaves for Chemical Processing – Ultrasound & Microwave Technologies
Sept. 4-8, 2017, KU Leuven, Belgium

AMPERE-Newsletter Call for Papers

AMPERE Newsletter welcomes submissions of articles, briefs and news on topics of interest for the RF-and-microwave heating community worldwide, including:

- Research briefs and discovery reports.
- Review articles on R&D trends and thematic issues.
- Technology-transfer and commercialization.
- Safety, RFI, and regulatory aspects.
- Technological and market forecasts.
- Comments, views, and visions.
- Interviews with leading innovators and experts.
- New projects, openings and hiring opportunities.
- Tutorials and technical notes.
- Social, cultural and historical aspects.
- Economical and practical considerations.
- Upcoming events, new books and papers.

AMPERE Newsletter is an ISSN registered periodical publication hence its articles are citable as references. However, the Newsletter's publication criteria may differ from that of common scientific Journals by its acceptance (and even encouragement) of news in more premature stages of on-going efforts.

We believe that this seemingly less-rigorous editorial approach is essential in order to accelerate the circulation of ideas, discoveries, and contemporary studies among the AMPERE community worldwide. It may hopefully enrich our common knowledge and hence exciting new ideas, findings and developments.

Please send your submission (or any question, comment or suggestion in this regard) to the Editor in the e-mail address below.

AMPERE-Newsletter Editor

Eli Jerby, Faculty of Engineering, Tel Aviv University, Israel, E-mail: jerby@eng.tau.ac.il

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