Microwave Instruments Geared Towards Hotter Future

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Background

Many industrialists become aware of the advantages that microwave energy brings to their processes. Also, many scientists find new promising results when applying microwaves at their laboratories, even observing new reaction pathways not possible with conventional heating.

Exploiting the potential of microwave processing requires a deep understanding of the underlying chemo-physical processes at molecular level. A comprehensive understanding of these correlations requires new and complex microwave instruments and advanced measurement capabilities.

The Microwave Division (DiMaS)[1] of the TIC research institute ITACA undertakes scientific and applied research, technological development and technology transfer initiatives in the field of microwave engineering. DiMaS also offers consultancy services of advice, high frequency measurements, and expert feasibility studies in projects of technological development, applicable to the microwave sector.

The division is mainly composed by University staff with the assistance of contracted researchers, technicians and fellowship students. The team of researchers is now well established with close links to industry and has one of the best independent microwave design and test facilities (Fig. 1) in Europe.

Figure 1. One of the microwave laboratories at DiMaS facilities.

Knowing at first hand their needs, DiMaS researchers have been more than twenty years developing microwave technology within four main activity lines: Electromagnetic modeling and numerical techniques; design of microwave circuits; microwave metrology/sensing, and microwave heating processes.

These years gave us a holistic, integral vision of the microwave processes, including:

- Performing the characterization of materials and processes for the determination of the material interaction with the MW fields. As a result, a wide range of specific and accurate measurement devices can be found in our facilities for basic metrology. Measurement services are provided for power, noise, impedance, dielectric and magnetic material properties, and other basic quantities.
- Designing and manufacturing new bench-top laboratory applicators, including in-situ and real-time control of the process parameters, optimizing process efficiency and product quality.
- Providing complete engineering support for developing demo units; addressing industry heating processes, with more than 100 prototypes developed (Fig. 2), and more than 20 international patents covering the entire spectrum of heating applications.

Figure 2. A laboratory microwave system for materials sintering.

Recent results and future trends

Potential benefits of microwaves are not enough if they don’t occur in a repeatable, reliable and controllable process. In those cases, users which are commonly non-experts in microwaves do not trust the technology. Too many times, the underlying microwave-matter interaction and the resulting heating mechanisms are still unclear.

At the same time, the application of microwave energy is moving towards increasing temperatures. At present, microwave processes developed at DIMAS aim at temperatures well above 1,000ºC (Fig. 3). The requirements of the materials and structures involved in such processes arise as a complex challenge.

Figure 3. Examples of high temperature continuous microwave processes.

This situation impelled us to take a step forward in microwave metrology. In general, the methodologies employed for dielectric measurements at room temperature (Fig. 4) require some modifications to overcome the practical limitations at high temperatures. Thus, more sophisticated and advanced instruments were demanded to address these needs.

Figure 4. A portable system for the measurement of the dielectric properties of materials placed in standard vials.

As a result, a new system was developed at DiMaS consisting of a new microwave cavity and heating system for microwave processing and in-situ dynamic measurements of the complex permittivity of dielectric materials at high temperatures (~1,000ºC). The method is based on a dual-mode cylindrical cavity where heating and testing are performed by two independent microwave sources with a cross-coupling filter that avoids any interference between them (Fig. 5).

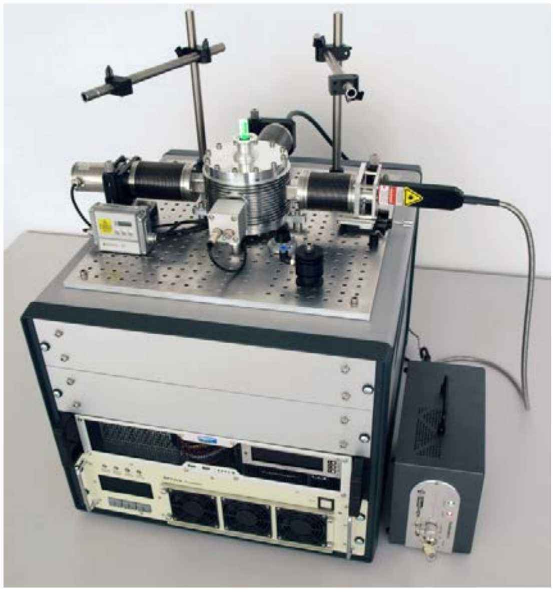

Figure 5. Dual-mode cylindrical cavity for microwave processing and in-situ dynamic measurements of the complex permittivity of dielectric materials at high temperatures.

This system provides the dielectric properties of materials as a function of temperature by an improved cavity perturbation method during heating, with an accuracy of the complex permittivity within 3% in the dielectric constant and around 10% in the loss factor within the range from 10^{-3} to 10^{-1}.

The correlation of the complex permittivity with the heating rate, temperature, absorbed power, and other processing parameters can help to better understand the interactions that take place during microwave heating of materials compared to conventional heating. Furthermore, the measurement of dielectric properties with the temperature permits to determine the energy of reactions and to obtain kinetic parameters such as the activation energy, defining the concept of *microwave calorimetry* in comparison with differential calorimetry by conventional heating.

For example, Fig. 6 shows the temperature dependence of the dielectric properties of a kaolin sample. During the heating process, the changes in the dielectric properties perfectly correlate with the known transformations in kaolin: below 200ºC the loss of water; at 500ºC the dehydration of kaolin; at 900ºC the formation of aluminium-silicon spinel; above 1100ºC crystallization of amorphous silica phase. Also, the video images reveal that an important expansion and shrinkage of the sample take place at certain temperatures. These changes in the sample volume are quantified as well and considered for an accurate determination of the kaolin dielectric properties.

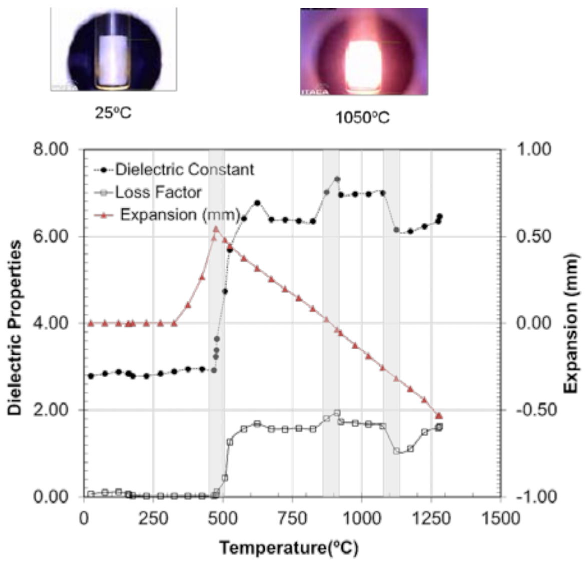

Figure 6. Dielectric properties of kaolin during microwave heating. The insets show the sample at 25 and 1,050ºC.

Modified versions of this system have resulted in a new generation of sophisticated microwave applicators and reactors. As an example, another version of this system allows performing chemical reactions in a continuous way, while maintaining all the control functionalities and the dielectric characterization of the reactants under perfectly controlled atmosphere and microwave conditions.

The functionality of the microwave dielectric measurement system has been demonstrated by heating and measuring a wide range of samples up to 1,200ºC. Some of these results have been already presented in Journals and conferences with the most updated state of the art in microwave science and technology[2-7].

Future plans

In view of the significant progress presented above, there is still a long way to go. Beyond the material dielectric properties, a comprehensive collection of data is obtained as a result of each measurement: absorbed power at each temperature, temperatures at which not forced acceleration/deceleration of the heating rate occurs, hysteresis between heating/cooling cycles, etc. Thus, a multidisciplinary collaboration is essential to understand the new insights that this system is providing about the microwave-matter interactions.

Aiming to give a deeper understanding of these interactions, the system combines different techniques to analyze the chemo-physical characteristics at molecular level. Recently, a Raman spectrometer was included to provide information about the materials' microstructure during microwave heating, simultaneously to the in-situ dielectric characterization[7].

The results given by all these developments allow predicting in a reliable way the behavior of materials under high frequency fields, which is essential to design and optimize new high temperature microwave processes. Our main goal is to enable getting the most out from the numerous benefits that microwave energy can give to applications in the entire range from fundamental science to technology.

For further reading

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About the Author

Jose M. Catalá-Civera was born in Valencia (Spain) in February 1969. He received the Dipl. Ing. and Ph.D. degrees from the Universidad Politécnica de Valencia, Spain, in 1993 and 2000, respectively. Since 1996, he has been with the Communications Dept., Universidad Politécnica de Valencia, where he received the Readership in 2000 becoming full professor in 2011. Currently he is head of the Microwave Applications Research Division of the Institute ITACA at the Universidad Politécnica de Valencia. His research interests encompass the design and application of microwave theory and applications, the use of microwaves for electromagnetic heating, microwave cavities and resonators, measurement of dielectric and magnetic properties of materials and development of microwave sensors for non-destructive testing. He has co-authored about 100 papers in referred journals and conference proceedings, more than 50 engineering reports for companies and he holds 13 patents. Dr. Catala-Civera is IEEE Member, IMPI Member, and he is reviewer of several international Journals. He is currently Board Member of the Association of Microwave Power in Europe for Research and Education (AMPERE), a European-based organization devoted to the promotion of RF and microwave energy.