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Breaking Barriers to the Commercialization of High Temperature Microwave Processing

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Abstract

The barriers to the practical use of microwaves are identified, microwave furnace companies are identified and compared, and the recent developments using microwaves for high temperature processes are discussed. The effectiveness of using microwaves to promote heating for drying, calcination, binder removal, glass melting, and sintering of ceramics and powdered metals has been demonstrated in laboratories throughout the world. Microwave drying of materials has become an industrial reality, however, high temperature processing (1000-2200 °C) is just beginning to find it's way into commercial products. The obstacles to the wide spread industrial up-take of this technology include, 1) the lack of commercial microwave furnaces, and 2) the lack of materials process know how. As practical recipes can be developed with less expense, and more furnace companies rise to the challenge, there will be a significant shift towards the production of advanced materials using high temperature microwave processes.

Introduction

New innovations require time to mature and to find their place in the manufacturing arena. Once a technology is proven in the laboratory, there are many challenges to commercialization. The utilization of microwaves for firing or sintering ceramics, metals, and glass can benefit many manufacturers with energy savings, time savings, and improved products. Some examples of the target industries for microwave firing technology (> 1200 °C) includes electroceramics (e.g. sensors, actuators, varistors, ultrasonics, magnets), advanced ceramic and carbide hardmetal wear parts, and bioceramics.

It is Ceralink's¹ assessment that manufacturers will incorporate microwave technology more readily when two major barriers are overcome. One of the barriers is the need for standardized commercially available microwave furnaces. Why do we need this when manufacturers of ceramic, glass, hardmetal, and sintered metal products will certainly have a microwave furnace system designed specifically for their product line at some stage of scale-up? The reason is simple. Material manufacturers must develop confidence and familiarity with microwave systems.

Manufacturers must make a difficult decision to invest in the design and building of a microwave furnace when they don't see microwave furnaces operating first hand. Collectively, we can make the decision easier by pointing to standard lab scale and small-scale microwave furnaces. Standard microwave furnaces should be available to universities and corporate research laboratories. Published results can be reproduced on standard equipment so that less time is wasted in rigging systems or repeating

experiments. Also, microwave furnace must be packaged to sell to companies familiar with conventional furnaces. Adequate operating instructions and possibly an "acceptance test" with a real material (e.g. zirconia) should be supplied.

Another barrier to the use of microwaves for high temperature materials processing is the need for a path to develop material process know-how. There should be a step by step method to provide data and develop a vision that will encourage the large investment needed in capital and technical expertise. The current sources for these investigations include university research, microwave manufacturers, and independent microwave testing facilities. Universities typically perform well in basic research, but are not focused on engineering or scale-up problems and generally have a slow response time by industry standards. Microwave furnace manufacturers have an obvious vested interest in the success of microwave feasibility and therefore can be innovative and helpful, however, customers may be skeptical of the results. By establishing an independent microwave testing facility, Ceralink tests feasibility, develops material processes, assists in product scale-up, and assists microwave manufacturers in growing the market for their furnaces.

Commercial Microwave Furnaces

There are many companies that will design and build microwave systems to their industrial clients specifications. The design process must be carried out jointly with the material manufacturer. Very few of these companies offer standard lab scale microwave furnaces. Table 1 gives a list of companies that offer standard systems with a range of prices and capabilities. Table 2 gives a summary of the different approaches to the use of microwaves for high temperature materials processing. Susceptors or receptors are often used with 2.45 GHz systems to start heating and prevent thermal runaway in the load. Susceptors are materials, usually semiconductor, such as silicon carbide that heat easily from room temperature in the field.

Table 1: Commercial Microwave Furnaces with Specifications

Company	Location	Frequency (GHz)
C-Tech Innovation (formally EA Tech.)	UK	2.45 + gas/ electric
Communications and Power Industries	USA	2.45, 18, 28
Dennis Tool Company	USA	2.45
Fuji Denpa	Japan	2.45
Gycom	Russia	30
Linn High Therm	Germany	2.45, 5.8
Microwave Materials Technologies	USA	2.45
Research Microwave Systems	USA	2.45

Table 2: Approaches to Microwave Processing

Method	Advantage	Disadvantage
2.45 GHz Mass produced magnetrons	<ul style="list-style-type: none">• Inexpensive	<ul style="list-style-type: none">• Often susceptors needed
2.45 GHz High power magnetrons	<ul style="list-style-type: none">• Good for large systems	<ul style="list-style-type: none">• Medium expense• Susceptors
5.8 GHz	<ul style="list-style-type: none">• Improved uniformity	<ul style="list-style-type: none">• Not tested
Hybrid 2.45 + 5.8 GHz	<ul style="list-style-type: none">• Improved uniformity	<ul style="list-style-type: none">• Not tested
Hybrid 2.45 + gas or electric	<ul style="list-style-type: none">• Most energy efficient• No susceptors• Retrofit capability	<ul style="list-style-type: none">• Availability
18, 24, 28, 30 GHz	<ul style="list-style-type: none">• Best field uniformity• No susceptors	<ul style="list-style-type: none">• Expensive

An inexpensive system that uses low power 2.45 GHz magnetrons is the Research Microwave Systems (RMS)² ThermWAVE shown in Figure 1. This modified kitchen microwave is a useful tool to explore processes.³ It is packaged as a system that includes a controller and the accessories, such as a casket and susceptors. It is essential that researchers with expertise in material science become comfortable with microwave systems. The ThermWAVE package opens the door for many material researchers who don't have time to develop their own system and can't justify investment in more expensive equipment prior to proof of concept.



Figure 1. Research Microwave Systems ThermWAVE 2.45 GHz 1300 Watts.

The Communications and Power Industries (CPI) Autowave⁴ is a system that uses high power magnetrons and can be fitted with a klystron (18 GHz) or gyrotron (28 GHz). The Autowave has two chamber sizes; the larger chamber is shown in Figure 2. CPI provides an Acceptance Package to demonstrate that the Autowave can be used to reproducibly sinter (fire) a ceramic.⁵ The system is versatile, can be used for research, scale-up, and/or production, but it is relatively expensive. Microwave

Materials Technology (MMT) offers a similar 2.45 GHz system to that shown in Figure 2. The chamber is not lined with refractory insulation. Microwave transparent refractory containers or caskets are used inside the chamber. This increases the versatility and cuts down on power requirements for small-scale tests.



Figure 2. The CPI Autowave station can be fitted with different frequency (2.45, 18, 28 GHz) and power levels.

An example of a commercial system that uses only 2.45 GHz magnetrons, shown schematically in Figure 3, is the Dennis Tool Co.⁶ system developed with Penn State University.⁷ This system is sold as a complete package including the materials process technology. The focus has been on carbide (hardmetal) manufacturing and the process is in the final stage of scale-up at a cutting tool company. There are several patents surrounding the technology including the "stoke type" configuration for feed through.^{8, 9} In scale up, it was found that smaller loads produced more uniform superior product, however the larger loads were still superior to conventionally fired tungsten carbides.

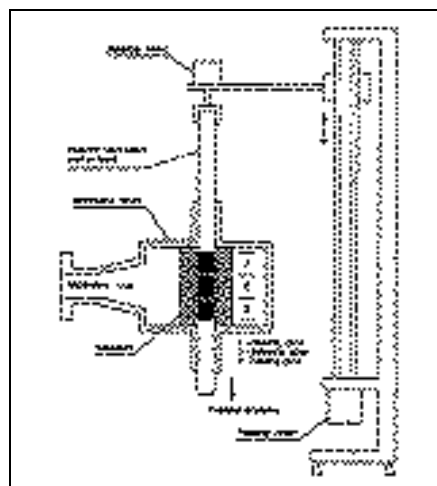


Figure 3. Apparatus for continuous microwave sintering of ceramics up to 1 meter long, and up to 10 cm in diameter. (from ref ⁷)

Linn High Therm¹⁰ has made a laboratory size 2.45 GHz microwave sintering furnace available for ~10 years. Linn's recent innovation is the development and inexpensive manufacture of 5.8 GHz magnetrons. This is an industrial frequency that has not been explored for high temperature processing of materials. There is potential for increased field uniformity, especially when combined with 2.45 GHz, and the possibility of heating more materials from room temperature without susceptors.¹¹

Linn High Therm offers two versions of a hybrid system using 2.45 GHz and 5.8 GHz magnetrons as shown in Figure 4.



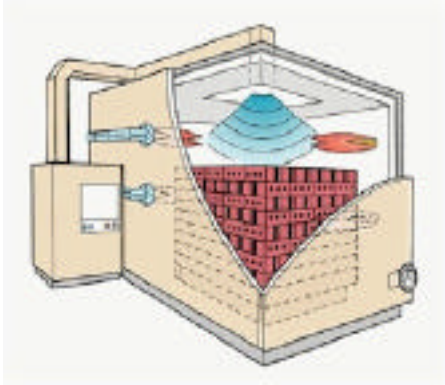
a) 800 W 2.45 GHz + 500 W 5.8 GHz

b) 1600 W 2.45 GHz + 1000 W 5.8 GHz

Figure 4. Linn High Therm laboratory scale systems with hybrid combination of 2.45 GHz and 5.8 GHz magnetrons.

Linn High Therm has also developed an efficient modular continual microwave drying system with a circular distribution of low power magnetrons. It would be of interest to see this system modified for high temperature processing.

Another hybrid system was developed at EA Technologies and now owned by C-Tech Innovations.¹²⁻¹⁴ This method uses a combination of microwaves and gas or electric heating, represented in Figure 5. There are several advantages to this system including even heating, energy efficiency, and the capability to retrofit magnetrons to existing furnaces. The method has been tested and initial scale up performed for many ceramic materials. The technology has recently been licensed to a Japanese kiln manufacturer. The availability in Europe and the US is an issue for the utilization of this technology.



a) Period kiln

b) Tunnel kiln

Figure 5. C-Tech Innovations (formally EA technologies) Gas Assisted Microwave Firing systems.

Microwave Testing and Feasibility

Companies are slowly, and some quietly, developing microwave processes and working with microwave system manufacturers. An important stimulus in this direction is the visibility and availability of proof of concept testing. Based on this need, Ceralink has developed a Microwave Testing Center in Alfred, New York. This Center offers a central location where companies have access to the various types of microwave systems. Companies can become informed and run feasibility studies prior to capital investment. In addition, companies can outsource engineering skills from Ceralink, with combined microwave and materials knowledge, to work with the microwave manufacturers and smooth the way for scale-up.

There are over 200 ceramic and glass manufacturing companies in New York State alone. Many companies have thought about microwave firing, but have not taken action. Alfred University's New York State College of Ceramic Engineering is world renowned, with graduates in the ceramic and glass industry throughout the US. Since the establishment of a Microwave Testing Center in Alfred, NY, many companies have showed an active interest. These companies will become customers for microwave systems.

Conclusions

Two major barriers to the widespread up-take of microwaves in high temperature (1000-2200 °C) processing of materials have been identified. One is the need for standardized microwave furnaces or kilns. This is important to increase the confidence level of the material manufacturers and give them the opportunity to understand the systems prior to purchase. Another important ingredient is the easy access to feasibility testing prior to purchase, which is solved with a Microwave Testing Center.

References

1. Ceralink Inc. Website: www.ceralink.com
2. Research Microwave Systems Website: www.thermwave.com
3. Walker, W.J.W., Shulman, H.S. Fall, M.F., Wolfe, L., *Sintering Wear Parts with Microwave Heating. 104th Meeting of The American Ceramic Society.* 2002. St. Louis.
4. Communications and Power Industries Web Site: www.autowave.tv.
5. Shulman, H.S., Fall, M., Walker, W., Treado, T. Sintering Uniformity and Reproducibility with 2.45 GHz Microwaves in an Industrial Sized Chamber. *104th Meeting of The American Ceramic Society.* 2002. St. Louis.
6. Dennis Tool Company Website: www.dennistool.com
7. Roy, R., et al., eds. *Microwaves: theory and application in materials processing IV.* Ceramic Transactions, ed. D. Clark, W. Sutton, and D. Lewis. Vol. 80. 1997. 3-26.
8. Dennis, M., et al., *Process and apparatus for the preparation of particulate or solid parts, United States Patent 6,126,895.* 2000.
9. Dennis, M., et al., *Method and apparatus for transporting green work pieces through a microwave sintering system, in United States Patent 6,066,290.* 2000.
10. Linn High Therm Website: www.linn.de
11. Moeller, M., H.S. Shulman, and H. Giesche. A Novel Approach to Understanding Microwave Heating of Zirconia, *104th Meeting of The American Ceramic Society,* 2002. St. Louis
12. Bond, S.M. *Microwave-Assisted Processing-from laboratory to production.* in *Microwaves: Theory and Application in Materials Processing V.* 2000. Orlando, FL, USA: The American Ceramic Society.
13. Wroe, F.C.R., *Method of processing ceramic materials and a microwave furnace therefore, in Unites States Patent & Trademark Office.* 2001, EA Technology Limited: United States. p. 1-18.
14. C-Tech Innovations Website: www.capenhurst.com.