

USE OF MICROWAVE ENERGY FOR MATERIAL PROCESSING IN A SIMPLE LABORATORY

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Abstrak

Penggunaan microwave energy untuk pengolahan material telah dipelajari secara luas oleh para peneliti dan juga oleh penulis. Beberapa fenomena yang berbeda (*microwave effect*) berupa peningkatan kualitas hasil diperoleh seperti singkatnya waktu yang dibutuhkan untuk proses dan meningkatnya kualitas bahan setelah proses dibanding hasil pengolahan dengan cara konvensional. Beberapa bahan keramik yang telah diteliti secara mendalam menggunakan gelombang frekuensi tinggi ini seperti alumina, zirconia, barium titanate, mullite, dan silica. *Microwave effect* ini juga dilaporkan terjadi pada *reaction sintering* pada nitrides dan lead-barium titanate. Pengolahan keramik dengan microwave terkendala dengan peralatan yang ada karena umumnya butuh suhu tinggi sehingga butuh microwave dengan energi tinggi. Ini berbeda dengan aplikasi microwave pada reaksi kimia, polimer, dan material organik (*microwave chemistry*) yang tidak membutuhkan energi yang tinggi sehingga lebih mudah dilakukan. Sehingga di laboratorium yang sederhana pun kita bisa menggunakan microwave baik untuk percobaan mahasiswa maupun untuk penelitian. Hal ini telah dilakukan di laboratorium Fisika FMIPA UHO. Hasil eksperimen pendahuluan diuraikan secara singkat di tulisan ini.

Kata Kunci: Mikrowave, keramik, microwave effect, microwave chemistry.

I. INTRODUCTION

The possibility of processing ceramic materials with microwave energy was known by the 1950s and had been investigated by limited researchers until 1980s. Up to now microwave processing becomes more widely used in industry and investigated by researchers and professional societies worldwide [1]. For ceramic sintering in microwave furnace has been widely studied for alumina, zirconia, barium titanate, mullite, phosphates, and borides [2-5].

Enhancement of reaction sintering has also been reported for nitrides and in lead-barium titanate. Besides sintering, many other thermally activated processes have been studied in microwave furnaces and compared to conventional processing such as microwave-enhanced joining of ceramic parts, nucleation and crystallization, grain growth, annealing, combustion synthesis, calcinations, and other solid state reactions, surface penetration of ion diffusing in alumina, chemical diffusion in glass and ceramic, polymer and organic material processing [6-11].

Most used frequency for application until now is 2.45 GHz due to the fact that they were

designed to process foods and the water in the foods. Foods are a good absorber at this frequency even in room temperature. Due to wide availability of 2.45 GHz microwave ovens, this frequency is also used by many researchers for heating different materials. Unfortunately, for ceramics processing application, need high temperatures to absorb 2.45 GHz microwave energy appreciably. Their absorption may be increased by adding increasing temperature at first or changing the frequency of the microwaves. The latter often is not feasible for most laboratories due to the relative unavailability of equipment.

Recently, the use of microwaves for heating chemical reactions has increased dramatically [12-14]. The researchers focus on the use the energy to reduce the time necessary to achieve esterifications, hydrolyses, ether preparations, Diels-Alder reactions, oxidations, ene reactions, and halide exchange reactions [13]. Chemical reactions don't need a high temperature and also don't need high microwave energies. It opens possibility to apply microwaves even in a simple laboratory such as in Physic Laboratory FMIPA UHO.

II. MICROWAVE ABSORPTION THEORY

In conventional heating, the heating elements supply heat to the sample, the majority of heat is concentrated along the surface. Because energy absorbed only at surface and must be transferred into the bulk of part by conduction which taking a finite amount of time, surface must hotter than interior until the part achieves thermal equilibrium. In a microwave furnace, the material will absorb microwave energy and will convert it into heat. Heat is generated internally within the materials resulting volumetric heating. However, heat must lose from the surfaces by convection and radiation resulting in an interior temperature hotter than the surface. In general, the time-averaged power dissipated per unit volume in a material can be expressed as [1]

$$P_v = 1/2\omega\epsilon_0\epsilon''|E|^2 \dots\dots\dots(1)$$

Where ϵ'' is the imaginary part of the complex dielectric constant of the material, ω is the angular frequency of the electric field E , and ϵ_0 is the permittivity of free space. The complex and frequency-dependent dielectric constant is determined by the structural properties and thermodynamic state of the material.

The charges present in the material would respond to the electric field (that is associated with microwave) by the following two processes [2,3]: *Polarization*, short range formation and displacement of dipoles present in the material; quantified as (ϵ_d'') and *Conduction*, long-range displacement of charged particles; quantified as (ϵ_c''). The formation and displacement of dipoles is dominant at the microwave frequencies (2.45 GHz). Some of the other types of polarization observed in materials (at different frequencies) include electronic polarization (ϵ_e''), ionic polarization (ϵ_i''), and interfacial polarization (ϵ_s''). Due to the experimental limitation with differentiating each type of polarization, a termed referred to as *effective loss*, (ϵ_{eff}''), has been introduced [1]. The effective loss factor is,

$$\epsilon_{eff}'' = \epsilon_d'' + \epsilon_c'' + \epsilon_e'' + \epsilon_i'' + \epsilon_s'' \dots\dots\dots(1)$$

Microwave heating is mainly due to polarization and ionic conduction. It depend on microwave

frequency. Simplify can be described that the ionic conduction losses decrease with an increase in frequency. This behavior is due to the fact that as the frequency increases, a greater amount of time is available for the charge to transport in the direction of field. Another observation that is important to microwave processing is the shift in the losses due to dipolar rotation towards microwave frequencies with temperature. This behavior is due to the increase in kinetic energy of molecules with temperature. This phenomenon can lead to unstable accelerated heating often termed as thermal runaway. The absorption of microwave energy and conversion to heat is due to polarization and conduction would result in a rise in temperature, $\frac{\Delta T}{\Delta t}$, and this is given by the following equation:

$$\frac{\Delta T}{\Delta t} = \frac{2\pi f \epsilon_0 \epsilon_{eff}'' E_{rms}^2}{\rho c_p} \dots\dots\dots(2)$$

where, ϵ_0 is the permittivity of free space ($8.85 \times 10^{-12} \text{ V/m}^3$), ϵ_{eff}'' is the relative effective dielectric loss due to ionic conduction and dipolar reorientation, f is the frequency (Hz), E_{rms} is the root mean square of the electric field within the material (V/m), ρ is the bulk density of dielectric material (kg/m^3) and C_p is the heat capacity of the material at constant pressure($\text{J/kg}^\circ\text{C}$). The dissipation of microwave power is limited by the attenuation of electromagnetic waves within the material and this is quantified as depth of penetration, D_p .

III. EXPERIMENT RESULTS IN MICROWAVE HEATING

Experiments of material processing by using microwave energies were performed by using microwave devices with wide range frequencies up to 300 GHz. Some experiment results are presented in this section.

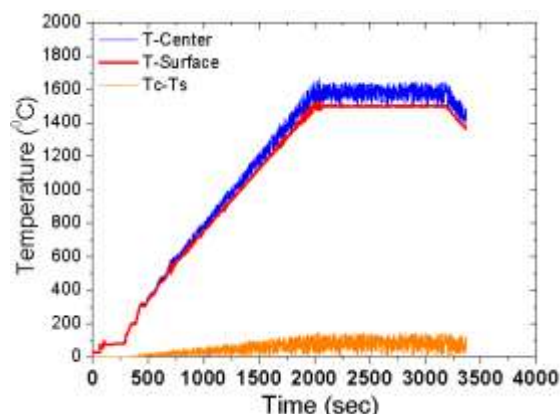


Fig. 1 Temperature profile of alumina heated by microwave

Figure 1 shows the temperature gradient between the center and the surface of alumina sample in microwave heating at very high temperature 1500 °C. The graph shows that temperature gradient exist between surface and bulk in the material being microwave heated. From experimental result exhibited that the center is hotter up to 100 °C than the surface. It much lower than gradient temperature during conventional heating at same temperature (1500 °C). It suggests that microwave processing can achieve a more homogenous heating compared than conventional one.

Another result of microwave processing is shown in Fig. 2.

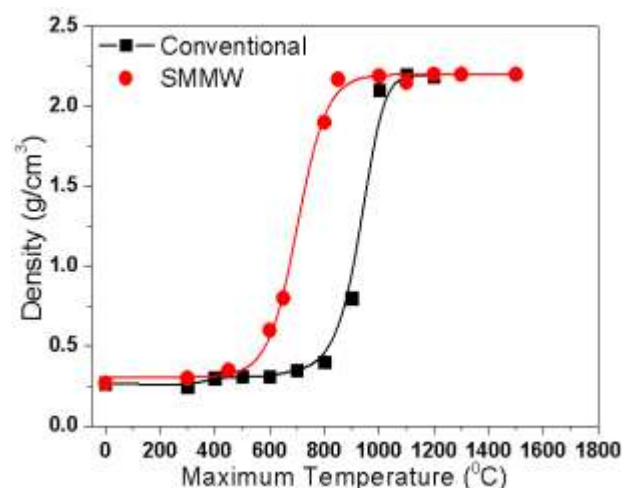


Fig. 2 Densification of microwave and conventionally sintered silica xerogel [15].

The graph shows the evolution of open and closed pores with increasing temperature of silica xerogel. For the temperature range from 450°C to 850°C, the increase in bulk density is proportional to the decrease in the percentage of the open pores. This behavior suggests that the

condensation reactions occurs on the surface of the silanols groups (Si-O-H) left in the porosity of the silica xerogel that are responsible for decreasing the open porosity which is one of main factors that influence the increase in bulk density. In these reactions, the silica xerogel structure tends to shrink due to the loss of the hydroxide (OH) groups and eventually forms Si-O-Si bridges to rearrange the Si and O atoms. That evident have been confirmed by FTIR characterization of sintered silica xerogel samples.

The two previous described previously in Fig. 1 and 2 were found from experiments by using a high frequency and high power microwave processing systems in Japan. Unfortunately, such devices aren't available in laboratories of Universitas Halu Oleo (UHO). However, experiments by using microwave energies still can be performed by using domestic microwaves (microwave oven 2.45 GHz). Though materials which can be processed and temperatures are limited. Moreover, need more careful and patient in preparing the experiment.

Figure 3 shows the domestic microwave oven which can be used for experiment even in simple laboratories.



Fig. 3 Microwave oven 2.45 GHz

Figure 4 shows the experiment results of drying characteristics of cocoa bean by using this microwave oven. The drying system was a domestic microwave oven which operated at three power levels. Conventional drying by using electric oven was also performed as a comparison. From the graph we can see that the faster drying was shown in microwave drying.

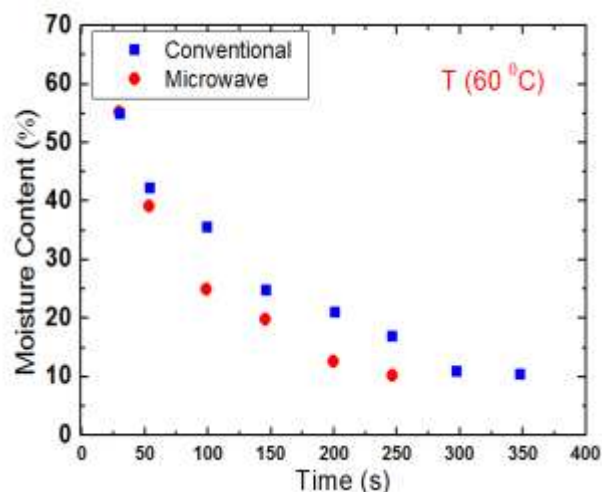


Fig. 4 Fast drying of cocoa bean by using microwave

Next experiment will be prepared for other materials as well as for chemical reactions by using this microwave oven in the Physic Laboratory FMIPA UHO. It is feasible because chemical reactions need not so high temperatures and microwave energies which can be provided by the oven.

IV. CONCLUSION

The experiments by using microwave energies have been performed for ceramics and organic materials. Some enhancements were found in ceramic processing. However, some experiments need high temperatures and need high microwave energies which can't be performed in a simple laboratory such as Physic Laboratory FMIPA UHO. Then we create a new experiment that does not require a high temperature and energy. The experiments were performed by using a domestic microwave oven. Preliminary results were shown in this paper. More experiments and further attempt to apply microwave energy in experiment and research are needed.

REFERENCES

- [1] W. H. Sutton, *Microwave processing of Ceramic Materials*, Microwave Solutions for Ceramic Engineers, Am. Cer. Soc., Ed. by D.E. Clark, D.C. Folz, C.E. Folgar, M.M. Mahmoud, pp.35-65 (2005).
- [2] I. N. Sudiana, I.N., R. Ito, S. Inagaki, K. Kuwayama, K. Sako, S. Mitsudo, *Densification of Alumina Ceramics Sintered by Using Sub-millimeter Wave Gyrotron*, Int. J. of Infrared, Millimeter, and Terahertz Waves. 34 (2013) 627-638.
- [3] R. R. Thridandapani, C. E. Folgar, A. Kulp, D. C. Folz, D. E. Clark, *Effect of Direct Microwave Sintering on Structure and Properties of 8 Mol% Y_2O_3 - ZrO_2* , J. Applied Ceramic Technology, 8[5] (2011), 1229-1236.
- [4] S. Mitsudo, K. Sako, S. Tani, I.N. Sudiana, *High Power Pulsed Submillimeter Wave Sintering of Zirconia Ceramics*, The 36th Int. Conf. on Infrared, Millimeter and THz Waves (IRMMW-THz 2011), Hyatt Regency Houston, Houston, Texas, USA, October 2-7, 2011.
- [5] Adam, D., *Microwave chemistry – out of the kitchen*, Nature, 421 (2003), pp. 571-572.
- [6] Y. Pang, D. K. Agrawal, D.M. Roy, R. Roy, *"Microwave Sintering of Calcium Stronium Zirconium Phosphate Ceramics,"* Microwave: Theory and Application in Material Processing II, Cer. Trans. 36, Am. Cer. Soc., Ed. by D.E Clark, W. R. Tinga, and J. R. Laia, (1993), p. 109.
- [7] C. E. Holcombe and N. L. Dykes, *"Microwave Sintering of Titanium Diboride,"* J. Material Science, 26[14] (1991), p.3730.
- [8] T. N. Tiegs, J. O. Kiggans, H. T. Lin, and C. A. Willkens, *"Comparison of Properties of Sintered Reaction-Bonded Silicon Nitride Fabricated by Microwave and Conventional Heating,"* Microwave Processing of Materials IV, Matls. Res. Soc. Proc., 347, Ed. by M.F. Iskander, R.J. Lauf, and W. H. Sutton, (1994), p. 501
- [9] Y. C. Kim, S. C. Koh, D. K. Kim, and C. H. Kim, *"Effect of Microwave Heating on Sintering of Silicon Nitride-Doped with Al_2O_3 and Y_2O_3 ,"* Microwave: Theory and Application in Material Processing III, Cer. Trans. 36, Am. Cer. Soc., Ed. by D.E Clark, W. R. Tinga, and J. R. Laia, (1993), pp. 109
- [10] J. Zhang, L. Lao, and H. Xia, *"Microwave Sintering of Si_3N_4 Ceramics,"* Microwave Processing of Materials III, Matls. Res. Soc. Proc., 269, Ed. by R.L. Beatty, W. H. Sutton, and M. F. Iskander, (1992), p. 329.

- [11] P. Boch, N. Lequeux, and P. Piluso, “*Reaction Rate of Ceramic Material by Microwave Sintering*,” Microwave Processing of Materials III, Matls. Res. Soc. Proc., 269, Ed. by R.L. Beatty, W. H. Sutton, and M. F. Iskander, (1992), p. 211
- [12] Kingston, H.M. and S. J. Haswell, 1997. *Microwave Enhanced Chemistry*. American Chemical Society, Washington DC, USA.
- [13] Hakim, L., P Lidström, J Tierney, B Wathey and J Westman, 2003. *Microwave assisted organic synthesis – a review*, Tetrahedron, 57, pp. 9,225–9,283.
- [14] L., P Lidström, J Tierney, B Wathey and J Westman, 2003. *Microwave assisted organic synthesis – a review*, Tetrahedron, 57, pp. 9,225–9,283
- [15] Aripin, I. N Sudiana, B. Sunendar, “*Preliminary study on silica xerogel extracted from sago waste ash*,” Indonesian J. of Mater. Sci., 6, pp.24- 30 (2010).