Using Microwave Radiation for Porcelain Tableware Sintering

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Abstract— This work presents an improved microwave multimode cavity for porcelain tableware sintering. It includes 6 magnetrons, each one with a nominal power of 1 kW at 2.45 GHz. The distribution of the electromagnetic field inside the cavity is calculated using the COMSOL Multiphysics software. The improved automated control system comprises the use of a pyrometer that allows the analysis of the thermal delay between the temperature given by a thermocouple and the real temperature in the porcelain material. Results exclude the existence of the so-called non-thermal microwave effect, recognized in some literature. Physical properties, such as density, rupture energy and impact resistance, are compared with those of tableware obtained by conventional methods. The improved microwave oven allows a better temperature measurement, with a faster sintering with the same quality standards than the conventional sintered porcelain.

1. INTRODUCTION

Microwave processing of materials has been increasingly used in different areas such as ceramics sintering [1–3], ceramics drying [4], metal powders heating [3], oil products [5], lime and concrete [6], wood [7], pyrolysis [8], chemistry [11], etc..

To avoid overheating, uniform electromagnetic field inside the cavities is required [3, 10]. However, the local electromagnetic field can be used to join materials with better heating control than with conventional convective heating methods [11]. The way how the radiation interacts with the material depends on a high number of parameters such as the radiation frequency, position of the microwave generators, geometric form of the load, dielectric properties, thermal conductivity, specific heat, density and electromagnetic and thermal behavior of the materials during the heating process [12]. To overcome the inhomogeneous heating some techniques like the use of turntables, mode stirrers, and others much more complex [1, 2] can be implemented. The absorbed power density of the microwave radiation can be expressed by

$$P = \frac{1}{2}\left[\left(\sigma_{dc} + \omega\varepsilon_r''\right)E^2 + \omega\mu_r'' H^2\right] \tag{1}$$

where σ_{dc} is the dc electrical conductivity, ε_r'' and μ_r'' are the imaginary parts of the permittivity and permeability, respectively and E and H are the electric and magnetic fields. The simplified unsteady heat transfer equation is given by

$$\rho\, C\frac{\partial T}{\partial t} = k\,\nabla^2 T + P \tag{2}$$

where ρ, C and k are the density, the specific heat and the thermal conductivity of the material, respectively.

Our study aims the understanding of the heating process of ceramic materials by microwave radiation in multimode ovens, its applications, limitations, as well as the optimization and opportunities of this heating method.

2. ELECTROMAGNETIC FIELD SIMULATIONS

Simulation studies are essential to understand the intrinsic heterogeneity of the electromagnetic field that leads to inhomogeneous heating of the material to be sintered. Specific technical solutions such as the orientation of the waveguides, and the locations where they are positioned, as well as the samples position inside the oven, were studied.

The analysis of the electromagnetic field at a macroscopic level implies the modeling of the Maxwell's equations, subject to the boundary conditions. The COMSOL Multiphysics software

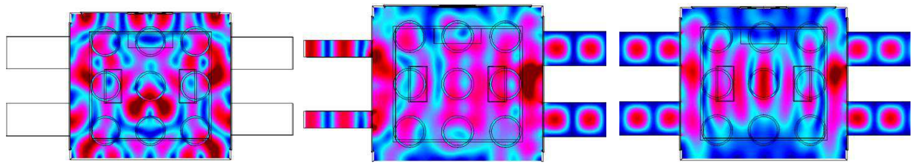

Figure 1: Simulated electric filed, for different magnetron configurations.

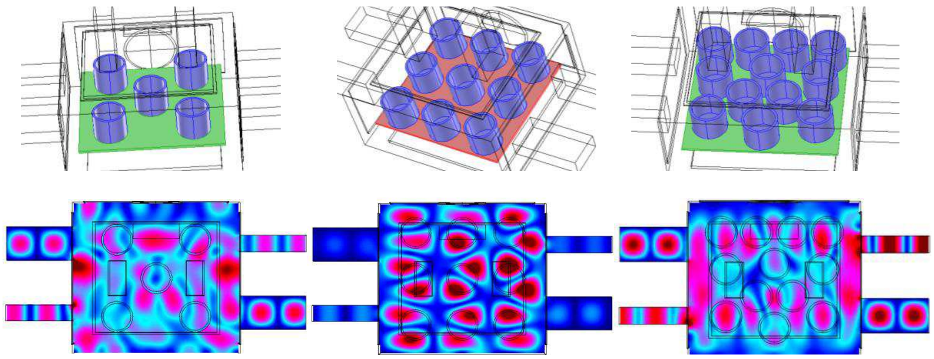

Figure 2: Load schedules and simulation results for different numbers and positions of the porcelain cups inside de cavity.

incorporates the differential equations that the finite element method can handle. The microwave radiation is feed by rectangular ports, and its electric field vector is determined by,

$$\vec{\nabla} \times \left(\mu^{-1} \vec{\nabla} \times \vec{E}\right) - k_0^2 \left(\varepsilon' - \frac{i\sigma}{\omega\varepsilon_0}\right) \vec{E} = 0 \quad (3)$$

The losses in the conductive walls are sufficiently small to not significantly affect the distribution of the electromagnetic field, and so the boundary conditions of the cavity are approximated as perfect conductors, $\vec{n} \times \vec{E} = 0$. The electromagnetic field simulations of the cavity loaded with porcelain cups ($\varepsilon^* = 2.2 - i5.10^{-4}$) and a SiC plate ($\varepsilon* = 18.2 - 1.78i$), for a total power of 6 kW, are shown in Figures 1 and 2. The measurement of the complex permittivity, at 2.45 GHz, was made using the small perturbation theory [13, 14], with a cavity operating in the TE_{015} mode, using a HP 8753D network analyser.

The uniformity characteristics are higher when the number of pieces inside the oven cavity is increased; observed experimentally. Figure 2 shows three schemes for different porcelain positions and number of cups.

3. MULTIMODE CAVITY DESIGN

For the monitoring and the temperature control inside the 6 magnetrons (6M) oven, and consequently the energy applied to the sample, a LabView code was developed that uses a *power-time-indirect temperature control* that comprises a set of combinations of a number of magnetrons that are active (*power*) during a particular time interval (*time*) which will act in a range of a predetermined temperature (*temperature*). This combination ensures a higher uniformity of the electromagnetic field inside the cavity and so a quicker sintering of the porcelain, with lower power consumption. To avoid electromagnetic interference in the readings of the thermocouple, and even its damage, it is shielded with a platinum foil. Biscuit and glazed porcelain pieces were tested.

In the oven floor, a immediately above the thermal insulation, a silicon carbide (SiC) plate that works as a base for the porcelain and as microwave susceptor was placed immediately above the thermal insulation, absorbing part of the electromagnetic radiation and transferring heat to the surroundings materials.

4. PHYSICAL CHARACTERIZATION

We do not present a results' color analysis, but we can see by naked eye that the sintering of tableware in a microwave oven is incomplete not due to its physical characteristics but because it is not possible to obtain the white porcelain unless a reactive gas that bonds chemically to the iron oxides that exist in the porcelain raw material is introduced. We tested in a nitrogen atmosphere, although the porcelain obtained under those conditions was whiter, it does not meet the industrial standards.

Table 1 shows the physical characterization of samples MW1-7 (Biscuit) and MWG (Glazed). CVG and CVB are the glazed and biscuit conventional sintered cups. MWx are the cups sintered with microwave radiation. MW5, 6, 7 have a dwell time of 5, 10 and 20 min. 12 cups were sintered in each set.

From Table 1 we observe that the glazed porcelain sintered with microwave radiation (MWG) at 1358°C shows physical characteristics very close to those of the glazed porcelain conventionally

(a) (b) (c)

Figure 3: Photographs of (a) the 6M oven, (b) of the SiC plate and the relative porcelain positions, and (c) interior layout of the furnace.

Table 1: Physical properties of some sintered porcelains. T_P and T_T are the temperature measure by the pyrometer and by the thermocouple, respectively.

Sample	CVG	CVB	MW0	MW1	MW2	MW3
Impact resistance (J·cm^{-2})	0.89 ± 0.28	1.05 ± 0.21	0.90 ± 0.14	1.03 ± 0.25	1.00 ± 0.15	0.81 ± 0.15
Rupture energy (J)	0.18 ± 0.03	0.17 ± 0.05	0.18 ± 0.03	0.19 ± 0.03	0.19 ± 0.03	0.17 ± 0.03
Apparent density (g·cm^{-3})	2.408 ± 0.005	2.443 ± 0.002	2.452 ± 0.013	2.439 ± 0.027	2.439 ± 0.029	2.398 ± 0.083
Open porosity (%)	0.30 ± 0.15	0.51 ± 0.29	2.34 ± 2.02	0.43 ± 0.15	0.41 ± 0.17	1.75 ± 2.05
T_P (°C)	—		1315	1350	1365	1380
T_T (°C)	1380		1268	1313	1322	1349
Time (mim)	200		64	70	68	70

Sample	MW4	MW5	MW6	MW7	MWG
Impact resistance (J·cm^{-2})	0.67 ± 0.15	1.05 ± 0.27	0.78 ± 0.13	0.89 ± 0.14	0.86 ± 0.26
Rupture energy (J)	0.15 ± 0.02	0.20 ± 0.05	0.14 ± 0.03	0.16 ± 0.03	0.17 ± 0.05
Apparent density (g·cm^{-3})	2.331 ± 0.047	2.438 ± 0.029	2.436 ± 0.047	2.428 ± 0.047	2.410 ± 0.027
Open porosity (%)	3.70 ± 5.65	0.56 ± 0.027	0.36 ± 0.15	0.29 ± 0.07	0.31 ± 0.18
T_P (°C)	1400	~ 1365			1358
T_T (°C)	1372	~ 1325			1315
Time (mim)	70	$70 + 5$	$70 + 10$	$70 + 20$	66

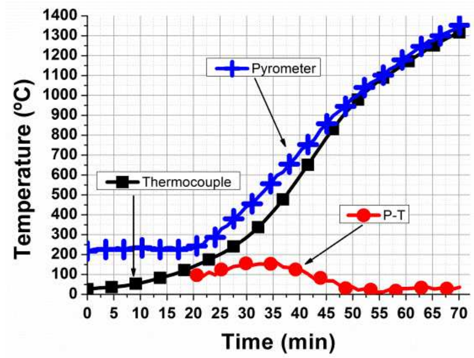

Figure 4: Sintering cycle for the MW1 sample.

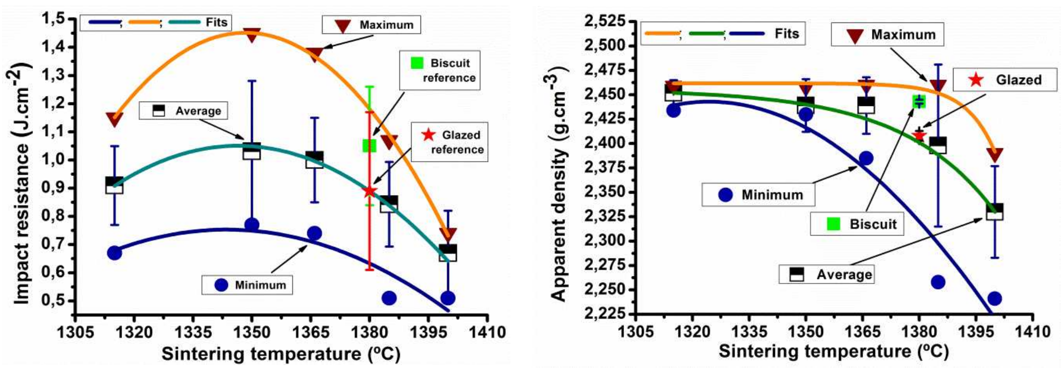

Figure 5: Impact resistance and apparent density as function of the sintering temperature. (read with the pyrometer).

sintered (CVG — 1380°C). Figure 4 presents the sintering cycle for the samples MW1 and Figure 5 the average, minimum and maximum impact resistance and apparent density for some samples as function of sintering temperature. References (conventionally sintered) of biscuit and glazed porcelain cups are also presented.

From Figure 5 we conclude that the best sintering temperature for the biscuit samples is around 1350–1365°C. Although in industrial conventional heating the maximum sintering temperature is close to 1380°C, this temperature is measured with thermocouples whose tips are measuring the air temperature. Industrial practice gives that the effective porcelain temperature is lower, normally between 1350°C and 1360°C. Comparing with the temperature (measured with the pyrometer) of the samples sintered with microwave radiation, these temperatures are in accordance with the temperatures of our best samples, MW1&2, for the biscuit and MWG for the Glazed sample.

The thermal delay between the temperature given by the thermocouple and the real temperature in the porcelain material at the minute 35 it is about 160°C. This difference decreases for values close to 35°C for higher temperatures. This difference is not very high, but it can be significant enough to produce the bloating phenomenon (bubbles formation) [15, 16]. Nevertheless, it must be taken into account that the measurements reported from compared measuring instruments were obtained from different spots.

5. CONCLUSION

The number of samples and their positions are very important in the heat homogenization.

The relationship between the porcelain density and the sintering dwell time duration is not conclusive, but results indicate that the dwell time can be eliminated. The tip of the thermocouple lies fairly close to the material sample, approximately 1 cm from its surface, and for the maximum temperature its reading is nearly 35°C lower than the temperature measured by the pyrometer. This difference is not very high, but it can be significant enough to produce the bloating phenomenon. The best results are for the samples MW1 & MW2, with sintering temperatures close to 1355°C. With the adequate control of a few microwave generators, it is possible to create a uniform electromagnetic field and therefore a more uniform heating, even at high heating rates. It is shown the viability of microwave radiation sintering of utilitarian porcelain. This technology is a good alternative to the conventional heating, allowing shorter sintering times. It is also shown the relevance of accurate temperature measurements. As consequence, we exclude the existence of a non-thermal microwave effect in the microwave sintering of porcelain tableware.

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