Experimental evidence of redistribution of fields during processing in a high-power microwave cavity

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A sensing probe has been introduced to profile the magnetic fields in a 2.45 GHz, high-power single-mode microwave cavity. The treatment of a ferrite sample inside the cavity has established that, during the heating process, there is an unusual concentration of a magnetic field around the sample. During field heating, intense magnetic fields have been monitored, and are analogous to the redistribution of an electric field reported by Riedel and Svoboda [Proceedings of the Eighth Ampere Conference and Microwave Heating (2001)] in their simulation studies on microwave sintering. © 2004 American Institute of Physics. [DOI: 10.1063/1.1806542]

Synthesis and sintering of electronic materials using microwave energy is emerging as a promising technique with dramatic enhancements in reaction and sintering kinetics. The differences between microwave and conventional heating have been highlighted in many recent publications.\(^{1-4}\) Kriegsmann\(^{5,6}\) presented analytical models for heating of thin dielectric rods in a transverse electric (TE\(_{101}\)) cavity and showed that the field was perturbed and localized, which resulted in a stable hot spot. More recently, Riedel and Svoboda\(^{7}\) modeled microwave sintering by simultaneously solving Maxwell's equations along with heat conduction and sintering equations.

In this work, we report the experimental evidence of field redistribution in the cavity during microwave sintering. These studies show that the magnetic field in the cavity accumulates/concentrates in and around a ferrite sample during microwave heating treatment. This study also supports the idea of the localization of a field as proposed by Kriegsmann,\(^{7}\) and the results on simulation studies as reported by Riedel and Svoboda.\(^{7}\)

The imposition of boundary conditions on electromagnetic wave propagation in a confined space is largely responsible for typical field patterns and field distribution in waveguides and cavities. The field profiles for many TE and transverse magnetic modes have been analyzed and discussed in standard textbooks.\(^{8,9}\)

For all \(E\)- and \(H\)-field experiments, we use a TE\(_{103}\) rectangular single-mode cavity located at the Microwave Processing Center of the Pennsylvania State University. Cheng \textit{et al.}\(^{10}\) discussed the details about the design and functioning of this single-mode cavity.

During a microwave heating experiment, the location of two \(H\)\(_{\text{max}}\) regions along the side walls of the cavity made it convenient to measure and study changes in the magnetic field rather than the electric field. Accordingly, a magnetic field sensing probe was assembled using standard techniques.\(^{11}\)

Our probe consisted of a small loop antenna (diameter \(-4\) mm) at one end of a 50 \(\Omega\) coaxial copper (hard) cable, which was nearly 0.5 m long. At the other end of the cable, a HP detector mount with a rectifying diode (BAT 46—Schottky Barrier Diode from DIODES Inc. USA) was attached. The output of the detector was fed into a current meter. Figure 1(a) shows the schematic of the probe.

The present studies were carried out utilizing earlier studies which had shown that microwave energy coupled very well to a Magnetite sample when placed in the magnetic field of the cavity.\(^{12}\) In order to avoid cracking, the \(\text{Fe}_{3}\text{O}_{4}\) samples were presintered at 1000 °C for 3 min in air. The presintering treatment of \(\text{Fe}_{3}\text{O}_{4}\) however, left the material partially oxidized. Hence, the starting pellet predominantly consisted of \(\text{Fe}_{3}\text{O}_{4}\) with a considerable amount of \(\text{Fe}_{2}\text{O}_{3}\). Since all of the field treatment experiments were carried out in air (\(p\text{O}_2=0.21\) atm), this pellet continuously underwent oxidation. After the \(H\)-field treatment, the pellet recorded a weight gain of 0.25%–0.5%. Since we did not notice any corresponding changes in the measured microcurrents, we believed that these microcurrent changes reflected sintering rather than any phase variations.

A specimen pellet of \(\text{Fe}_{3}\text{O}_{4}\) (6 mm diameter \(-6\) mm thick) was placed inside a quartz tube, which was introduced into the cavity through a hole located at the center of the narrow sidewalls of the cavity. The quartz tube was open on both ends that permitted the insertion of a probe (loop) into the cavity from one end and facilitated the temperature measurement on the other end. Figure 1(b) illustrates the schematic of the cavity with the field sensing loop. Other details of the complete experimental setup are available elsewhere.\(^{13}\)

For the measurement of a ferrite specimen, there were two convenient positions of the maximum magnetic field in the cavity, [A and B in Fig. 1(b)]. The temperature of the specimen was measured using a digital infrared pyrometer (model MA2SC, Raytek Co., Santa Cruz, CA). The microwave power in the cavity was controlled by the input power (measured using a Digital power meter) applied to the magnetron. In order to check the field-detection characteristics of the assembled probe, the detector output current, \(I_o\), was measured as a function of the microwave power, \(P_{\text{in}}\), in the cavity.

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Figure 1. (a) Schematic of magnetic field probe. (b) Processor cavity with magnetic field probe positioned in the $H_{\text{max}}$ (region A). Another location for $H_{\text{max}}$ is around region B. "$a$" is the broad dimension of the cavity.

Figure 2 shows the results obtained from such a measurement without any specimen in the cavity. During these and later measurements, the field sensing loop was positioned at the maximum magnetic field, region A only [Fig. 1(b)]. The detector diode characteristics as well as the relationship between $P_{\text{det}}$ and the microwave power $P_{\text{mic}}$ had a nonlinear relation, which are reflected in Fig. 2.

In the next experiment, the Fe$_2$O$_3$ pellet was placed closer to the probe loop [position A in Fig. 1(b)] in the maximum magnetic field in the cavity. The sample was placed in the $H_{\text{max}}$ position right next to the loop as shown in Fig. 1(b).

The $c$ axis of the cylindrical Fe$_2$O$_3$ pellet was placed parallel with the $z$ axis of the quartz tube. In other words, the pellet face was placed perpendicular to the axis of the quartz tube and the heating was independent of specimen orientation. A variation of $I_{\text{sh}}$ (representing microwave power near the sample) and sample temperature ($T$) with microwave exposure time was recorded. These results are illustrated in Figs. 3(a) and 3(b). In Fig. 3, it can be seen that during the initial 300 s of the experiment, the current rose fast in parallel with temperature. When the sample started sintering, the current stayed high (>2 mA) and temperature also recorded a value somewhere between 850–900 °C. After field treatment of 900 s, the Archimedes's density obtained on the Fe$_2$O$_3$ sample was 93% of theoretical density while the starting material only had a density of 75%. At this point, the microwave absorption dropped rapidly. This indicated that the field concentration did not exist any more. In spite of that the
specimen temperature continuously stayed at a maximum temperature $900^\circ$ C. The variation of applied microwave power with time during measurement is shown in Fig. 3(c).

The experiment was repeated by placing the specimen pellet in another $H_{\text{max}}$ region on the opposite side of the cavity (position B in Fig. 1(b)). The detector loop remained at the same position (position A). The output current, specimen temperature, and applied microwave power were recorded as a function of time that are shown in Fig. 4. Here, the temperature profile of the specimen was similar to that shown in Fig. 3, but the current profile was inverted. As sintering of the specimen began, due to accumulation of field near the specimen (region B), it should fall at the position of the loop lying on other side in the cavity (region A).

The results on the microwave sintering of a $Fe_2O_3$ sample in the magnetic field, as shown in Figs. 3 and 4, clearly indicate that an intense concentration of a magnetic field exists around the specimen and confirm the redistribution of field in the cavity during microwave sintering. In the present work, we have experimentally recorded the magnetic field concentration around a ferrite sample in the cavity, as simulated previously.\(^\text{7}\)

Direct profiling of an electric field in the cavity requires the placement of a field sensing probe or loop in the $E$ field. Unfortunately, these field sensing metallic objects quickly burn/melt in high microwave power in the cavity due to spark and plasma formation as mentioned in one of our earlier publications.\(^\text{13}\)

The reason for field accumulation of high magnitude in a microwave cavity is still not clear. This is perhaps yet an

other effect, like others,\(^\text{14}\) that needs a satisfactory explanation and reflects our limited understanding on microwave-material interactions.

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