産業技術国際交流会

マイクロ波効果・応用国際シンポジウム

International Symposium on Microwave Science and Its Application to Related Fields

～21世紀の革新的グリーンテクノロジー～

講演要旨集

2002年11月21日(木)～23日(土)
奈良県新公会堂

主　催　財団法人　日本産業技術振興協会
　　　　財団法人　産業創造研究所
共　催　マイクロ波応用技術研究会
　　　　日本電磁波応用研究会
　　　　奈良教育大学

EFFECT OF MICROWAVE ELECTRIC AND MAGNETIC FIELDS IN SINGLE MODE CAVITY ON MATERIAL PROCESSING

_D. Agrawal, J. Cheng, R. D. Peelamadu and R. Roy, Pennsylvania State University,
University Park, PA, 16802, USA

Thus far almost 100% work on microwave processing of materials has been confined to using a multi-mode cavity in which electric and magnetic fields cannot be separated. Using a 2.45 GHz, single mode TE103 cavity, it is possible to separate electric and magnetic components of microwave radiation and expose small size samples in pure E and H fields at microwave frequencies. Here we report the effect of electric and magnetic fields at 2.45 GHz frequency on the heating behavior of variety of materials. The survey of variety of samples of metals, ceramics, composites and magnetic materials showed remarkable differences in their heating behaviors and microstructural developments. In some cases when exposed to magnetic fields the material is found to have decrystalized in a matter of few seconds.

(dxa4@psu.edu)

Introduction: The vast majority of papers dealing with microwave heating of solids ascribe the heating to energy loss mechanisms of the electric vector. Very recently our experimental findings have demonstrated that magnetic losses play an important role in microwave sintering of bulk materials for a wide range of conductor and semiconductor materials. In 1994 Cherradi et al [7] published a paper in which they showed that the magnetic field must make substantial contributions to the heating of alumina (at high temperature) and semi-insulators, and metallic copper. In that work, their experimental design of using samples of 120mm length, was such that the sample was always heated in both H and E fields simultaneously, caused by a complicated interplay of the different absorption and conduction mechanisms. Our research in this study was based on our own unique success over the last several years with microwave sintering to full density in very short times of all major ceramics, including oxides, nitrides, and carbides [8 -9].

Experimental: A finely tuned 2.45 GHz cavity with a cross section dimension of 86 mm by 43 mm which works in TE_{103} single mode was used. The details of the system are reported elsewhere(??). The maximum electric field is in the center of the cross section, where the magnetic field is at a minimum. The maximum magnetic field is near the wall, where the electric field is minimum. A quartz tube was introduced in this location to hold the sample and also to enable us to control the atmosphere around the sample. A 2.45 GHz, 1.2 kW microwave generator (Toshiba, Japan) with power monitor was used as microwave source. Small cylindrical samples (5mm diameter and 5mm high) was placed inside the quartz tube at the two locations where maximum electric field and the maximum magnetic field are located, respectively. Sample temperatures were measured using an infrared. During the experiments, atmospheric pressure nitrogen gas was passed through the quartz tube to avoid oxidation of metal samples at high temperature.

Results and Discussion: Figure 1 shows the heating observed for a typical commercial powdered metal sample. As can be seen this sample is heated only in the magnetic field with a high heating rate. Other pure metal powder-compacts of Co, Fe and Cu were also heated in E and H fields. The Co and Fe samples exhibited the same behavior during the microwave heating. There were little heating in the electric field but high heating rates were observed in the magnetic field. The microwave heating of the Cu powder-compact sample was quite anomalous. The sample heated up very fast both in the electric and magnetic fields (Fig. 2). The sample's temperature rose to ~700°C in 1-2 minutes, then quickly dropped down to ~500°C and kept within that range during the continuous heating. Al_2O_3 is a proto-typical ceramic material with excellent dielectric properties. This material usually has very low dielectric loss, and it is not easy to heat up by microwave, especially at lower temperature. Since the dielectric loss of Al_2O_3 increases with temperature, microwave heating of Al_2O_3 becomes more efficient at higher temperature. The microwave heating of high purity Al_2O_3 samples in the electric field went slowly in the beginning, the heating rate speeded up after the sample reached a temperature of ~500°C. But in the magnetic field, Al_2O_3 could not be heated at all under the same microwave power for same exposing time (Figure 3). ZnO is another important dielectric material. It exhibited the same heating behavior as Al_2O_3 in E and H fields (Figure 4).

Tungsten carbide (WC) belongs to a larger family of semiconductor materials. Pure WC powder compact samples were microwave heated in E and H fields respectively. The results exposed that the magnetic loss factor, not the dielectric loss, is the principle source leading to microwave absorption (Figure 5).

We also tried some composite samples in E and H fields: alumina-powdered metal, tungsten carbide-cobalt (WC-Co) and ZnO-Co. Depending on the field, we obtained quite different results. The WC-Co sample can only be efficiently heated in the magnetic field, as the pure WC and Co samples also did. The alumina-powdered metal and ZnO-Co composite samples can be heated up in both electric and magnetic fields, since these composites contain two components, one of which is more sensitive to the E field (Al_2O_3, ZnO), and the other more sensitive to the H field (powdered metal, Co). We assume that the temperature came from different contributions which depend on the sample located in different microwave fields. For example, when the ZnO-Co powder mixture sample was located in a "pure" H field, in the beginning only high magnetic loss Co powder absorbed microwave power and heated to high temperature. Meanwhile, there was no absorption occurring in the ZnO powder which remained at low temperature. The measured temperature resulted mainly from the Co absorption. As time passed, the ZnO powder got heated higher and higher due to conductive heat transfer. Conversely, when the sample was put in a pure E field, the temperature profile should be exactly reversed. Now the ZnO powder had higher temperatures and the heat transfer proceeded from ZnO to Co.

Many magnetic materials when exposed to magnetic field became decrystallized in a few minutes in the magnetic field, but nothing happened in E field. A typical example is shown of Fe3O4 in Figure 6. These and other latest results will be presented in this paper.

References:

1. A. Cherradi, G. Desgardin, J. Provost, and B. Raveau, *Elelctroceramics IV*, Vol.**II**, (eds. Wasner, R., Hoffmann, S., Bonnenberg, D., & Hoffmann, C.) RWTN, Aachen, 1219-1224 (1994)
2. J. Cheng, D. Agrawal, S. Komarneni, M. Mathis, and R. Roy, *Mater. Res. Innov.* **1**, 44-52 (1997)
3. R. Roy, D. Agrawal, J. Cheng, and M. Mathis, *Ceram. Trans.* **80**, 3-26 (1997)
4. J. Cheng, R. Roy and D. Agrawal, Mat. Res. Innovat., 5, 170-177 (2002).

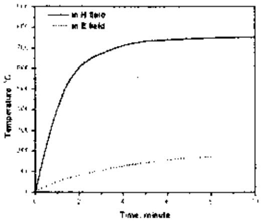


Figure 1

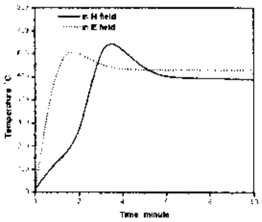


Figure 2

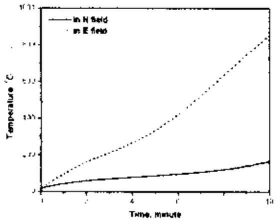


Figure 3

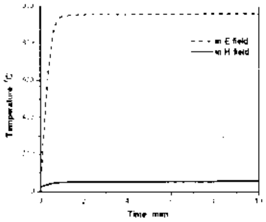


Figure 4

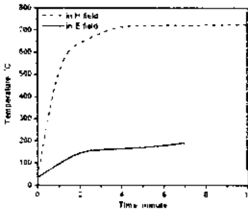
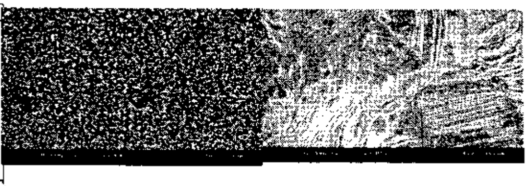


Figure 5



E-Field, 60 Sec. H-Field, 60 Sec.

Figure 6

1A−1

EFFECT OF MICROWAVE ELECTRIC AND MAGNETIC FIELDS IN SINGLE MODE CAVITY ON MATERIAL PROCESSING

_D. Agrawal, J. Cheng, R. D. Peelamadu and R. Roy, Pennsylvania State University,
University Park, PA, 16802, USA

Thus far almost 100% work on microwave processing of materials has been confined to using a multi-mode cavity in which electric and magnetic fields cannot be separated. Using a 2.45 GHz, single mode TE103 cavity, it is possible to separate electric and magnetic components of microwave radiation and expose small size samples in pure E and H fields at microwave frequencies. Here we report the effect of electric and magnetic fields at 2.45 GHz frequency on the heating behavior of variety of materials. The survey of variety of samples of metals, ceramics, composites and magnetic materials showed remarkable differences in their heating behaviors and microstructural developments. In some cases when exposed to magnetic fields the material is found to have decrystalized in a matter of few seconds.

(dxa4@psu.edu)

Introduction: The vast majority of papers dealing with microwave heating of solids ascribe the heating to energy loss mechanisms of the electric vector. Very recently our experimental findings have demonstrated that magnetic losses play an important role in microwave sintering of bulk materials for a wide range of conductor and semiconductor materials. In 1994 Cherradi et al [7] published a paper in which they showed that the magnetic field must make substantial contributions to the heating of alumina (at high temperature) and semi-insulators, and metallic copper. In that work, their experimental design of using samples of 120mm length, was such that the sample was always heated in both H and E fields simultaneously, caused by a complicated interplay of the different absorption and conduction mechanisms. Our research in this study was based on our own unique success over the last several years with microwave sintering to full density in very short times of all major ceramics, including oxides, nitrides, and carbides [8 -9].

Experimental: A finely tuned 2.45 GHz cavity with a cross section dimension of 86 mm by 43 mm which works in TE_{103} single mode was used. The details of the system are reported elsewhere(??). The maximum electric field is in the center of the cross section, where the magnetic field is at a minimum. The maximum magnetic field is near the wall, where the electric field is minimum. A quartz tube was introduced in this location to hold the sample and also to enable us to control the atmosphere around the sample. A 2.45 GHz, 1.2 kW microwave generator (Toshiba, Japan) with power monitor was used as microwave source. Small cylindrical samples (5mm diameter and 5mm high) was placed inside the quartz tube at the two locations where maximum electric field and the maximum magnetic field are located, respectively. Sample temperatures were measured using an infrared. During the experiments, atmospheric pressure nitrogen gas was passed through the quartz tube to avoid oxidation of metal samples at high temperature.

Results and Discussion: Figure 1 shows the heating observed for a typical commercial powdered metal sample. As can be seen this sample is heated only in the magnetic field with a high heating rate. Other pure metal powder-compacts of Co, Fe and Cu were also heated in E and H fields. The Co and Fe samples exhibited the same behavior during the microwave heating. There were little heating in the electric field but high heating rates were observed in the magnetic field. The microwave heating of the Cu powder-compact sample was quite anomalous. The sample heated up very fast both in the electric and magnetic fields (Fig. 2). The sample's temperature rose to ~700°C in 1-2 minutes, then quickly dropped down to ~500°C and kept within that range during the continuous heating. Al_2O_3 is a proto-typical ceramic material with excellent dielectric properties. This material usually has very low dielectric loss, and it is not easy to heat up by microwave, especially at lower temperature. Since the dielectric loss of Al_2O_3 increases with temperature, microwave heating of Al_2O_3 becomes more efficient at higher temperature. The microwave heating of high purity Al_2O_3 samples in the electric field went slowly in the beginning, the heating rate speeded up after the sample reached a temperature of ~500°C. But in the magnetic field, Al_2O_3 could not be heated at all under the same microwave power for same exposing time (Figure 3). ZnO is another important dielectric material. It exhibited the same heating behavior as Al_2O_3 in E and H fields (Figure 4).