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REVIEW

Developments in Microwave Processing of Materials

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The principles of microwave heating are discussed with reference to dielectric properties and heating phenomena. Microwave processing has been emerging as a novel sintering method for many traditional ceramics, advanced ceramics and ceramic composites as well as polymer and polymer composites. Most recent potential applications involve steel making, used tires recycling and alternative sources for energy recovery (oil shale and capped oil wells). Many specialty ceramics, composites and metal powders have been successfully processed in microwave with improved properties. The aim of this paper is to provide an introduction to microwave heating, its mechanism and to illustrate its application in the field of synthesis.

Key Words: Microwave processing, Mechanism, Ceramics, Microwave effects.

INTRODUCTION

It has long been established that a dielectric materials, such as ceramics can be heated with energy in the form of microwave[1,2]. The use of microwave energy for processing ceramics and ceramic matrix composites has been the subject of a large amount of exploratory research. The potential advantages of microwave processes over conventional processes for ceramic processing include reduced processing time, improved product uniformity and yields, improved or unique microstructure and the ability to synthesize new materials. Excellent reviews have been provided on the general aspects of microwave processing by several experts[2-7]. Some of the key characteristic features and benefits of the use of the microwave for sintering and synthesis are summarized in Table-1. This paper summarises the mechanism, overview developments and discusses important applications of microwave processing.

Mechanism of microwave interaction with ceramics

Many different physical phenomena are involved in the microwave processing of ceramics. The interaction of microwave with matter takes place through the electric field vector E (V/m) and the magnetic field vector H (A/m) of the microwave. When a material is subjected to an electric field, it gets polarized creating an electric polarization. There are four polarization mechanisms in solids and three of them lead to the losses in the microwave region: (1) space charges arising from localized electrical conduction, (2) rotating electric dipoles, (3) ionic polarization associated with far-infrared vibrations and (4) electronic polarization[8].

The absorption of microwaves by a dielectric material depends on the material's complex permittivity, ε*(F/m), which is composed of a real part called relative permittivity (ε', dielectric constant). It is a measure of the ability of a material to store electromagnetic energy and an imaginary part (ε'', dielectric loss factor) is a measure of the ability of a material to convert electromagnetic energy to heat[9].

$$\varepsilon^* = \varepsilon' - \varepsilon'' = \varepsilon_0(\varepsilon'_r - j\varepsilon''_{eff})$$

where $j = (-1)^{1/2}$, ε_0 is the permittivity of free space ($\varepsilon_0 = 8.86 \times 10^{-12}$ F/m), ε'_r is the relative dielectric constant and ε''_{eff} is the effective relative dielectric loss factor.

When microwaves penetrate and propagate through a dielectric material, the internal electric fields generated within the affected volume of the dielectric, induce translational motions of electrons or ions thus producing electric dipoles which rotate. The resistance of these induced motions due to inertial, elastic and frictional forces, which are frequency dependent, cause losses and attenuate the electric field. Because of these losses, volumetric heating results[2]. The loss tangent (tan δ) is commonly used to describe these losses. Loss tangent (tan δ), a parameter used to describe how well a product absorbs microwave energy, is the ratio of relative dielectric loss factor ε_{eff}'' to the dielectric constant ε_r' and is given by eqn. 1:

TABLE-1
BENEFITS, CHALLENGES AND NEEDS OF MICROWAVE PROCESSING[3]

Benefit
▪ Cost savings (time and energy, reduced floor space).
▪ Rapid heating of thermal insulators (most ceramics and polymers).
▪ Precise and controlled heating (instantaneous on/off heating).
▪ Selective heating.
▪ Volumetric and uniform heating (due to deep energy penetration).
▪ Short processing times.
▪ Improved quality and properties.
▪ Synthesis of new materials.
▪ Processing not possible with conventional means.
▪ Reduction of hazardous emissions.
▪ Increased product yields.
▪ Environmentally friendly (clean and quiet).
▪ Self-limiting heating in some materials.
▪ Power supply can be remote.
▪ Clean power and process conditions.
Challenges
• Heating low-loss poorly absorbing materials.
• Controlling accelerated heating (thermal runaway).
• Exploiting inverted temperature profiles.
• Eliminating arcing and controlling plasmas.
• Efficient transfer of microwave energy to work piece.
• Compatibility of the microwave process with the rest of the process line.
• Reluctance to abandon proven technologies.
• Timing.
• Economics.
Needs
✓ Availability of affordable equipment and supporting technologies.
✓ Kiln furniture, thermal insulation and other processing support hardware.
✓ Development of compositions and processes tailored specifically for microwave processing.
✓ Better fundamental understanding and modeling of microwave-material interactions.
✓ Better process controls, electronic tuning and automation (smart processing).
✓ Better communication among equipment manufacturers, technology developers, researchers and commercial users.
✓ More emphasis on microwave processing of magnetic materials.

$$\tan\delta = \frac{\varepsilon''_{eff}}{\varepsilon'_r} = \frac{\sigma}{2\pi f \varepsilon_0 \varepsilon'_r} \quad (1)$$

where σ is the total effective conductivity (Sm^{-1}) caused by conduction and displacement currents and f is the frequency (GHz).

The value of tan δ of an assembly of molecules depends on several factors *e.g.*, on the frequency of the electromagnetic waves, the temperature and the physical state and composition of the mixture. A product with a higher loss tangent is heated faster under the microwave field as compared to a product with a lower loss tangent[10]. Assuming no magnetic losses in the materials[9], the microwave power absorbed per unit volume (Q) in a material is given by the eqn. 2:

$$Q = 2\pi f \varepsilon_0 \varepsilon_{eff}'' E_{rms}^2 \quad (2)$$

where f is the frequency of the microwave in Hz, ε_0 is the permittivity of free space (8.86×10^{-12} F/m), ε_{eff}'' is the relative dielectric loss factor and E_{rms} is the root mean square value of the electric field.

Dielectric heating rates depend on the value of tan δ, but also on the size/quantity of the reaction mixture and (as for conventional heating) on the heat capacity of the medium. The consequence of an increasing volume of the reaction mixture is that incident microwaves suffer an absorbance loss factor and the depth of penetration of the incident radiation is related to tan δ and the penetration depth is given by eqn. 3:

$$D_p = \lambda_o \sqrt{\left(\frac{\varepsilon_r'}{\varepsilon_{eff}''}\right)} \quad (3)$$

where D is the penetration depth, λ_o is the wavelength of the microwave radiation.

The penetrating ability of microwaves in the material is also expressed by the skin depth, which is defined as the depth at which the electric field falls to 1/e or 37 % of its initial value[8] is shown by eqn. 4:

$$\text{Skin depth} = \frac{1}{(\pi f \mu \sigma)^{1/2}} \quad (4)$$

where f is the frequency, μ is magnetic permeability and σ is the conductivity. For copper, $\sigma = 5.8 \times 10^7\ \Omega^{-1}\ m^{-1}$, $\mu = \mu_0$, where μ_0 is the permeability of free space and the skin depth is less than 1 μm at microwave frequencies. Since the field penetration is proportional to $\sigma^{-1/2}$, the microwave energy heats semimetals and semiconductors better than any metal[11].

The power absorbed per unit volume P (Wm^{-3}) is expressed as:

$$P = \sigma|E|^2 = 2\pi f \varepsilon_0 \varepsilon'_r \tan\delta\, |E|^2 \quad (5)$$

where E (Vm^{-1}) is the magnitude of the internal electric field. This equation illustrates that the power absorbed varies linearly with the frequency, the relative dielectric constant, tan δ and the square of the electric field.

The values of ε'_r and tan δ increase with the increase in the temperature. The increase in ε'_r with the temperature is due to an increase in the polarizability caused by the volumetric expansion[12]. For example, Al_2O_3 has a significantly greater coefficient of thermal expansion[2,13], which leads to a greater increase in its ε'_r. In addition, the composition and the density have a major influence on the value of ε'_r. Higher density tends to give a greater value of ε'_r. Fig. 1a. and 1b shows the influence of the temperature on the dielectric constant and tangent loss of the material, respectively[2,14].

Review of microwave processing of materials

Research on the microwave processing of ceramic materials has been carried out since Von Hippel[15] began the examination of the interaction between microwaves and the oxide materials and the variation of the loss characteristics of the materials as a function of microwave frequency. Soon after, Feiker[16] reported that the dielectric materials could be heated rapidly by the microwave at 915 MHz, Ford[17] conducted the high-temperature chemical processing using the microwave irradiation. Among the pioneers, Tinga *et al.*[18,19] studied the interaction of the microwave and the materials along with the thermal processing of the oxides and composite materials using microwaves of 915 MHz and 2.45 GHz. They also carried out the dielectric property measurements in the microwave field and developed the reaction cavities for a uniform microwave heating.

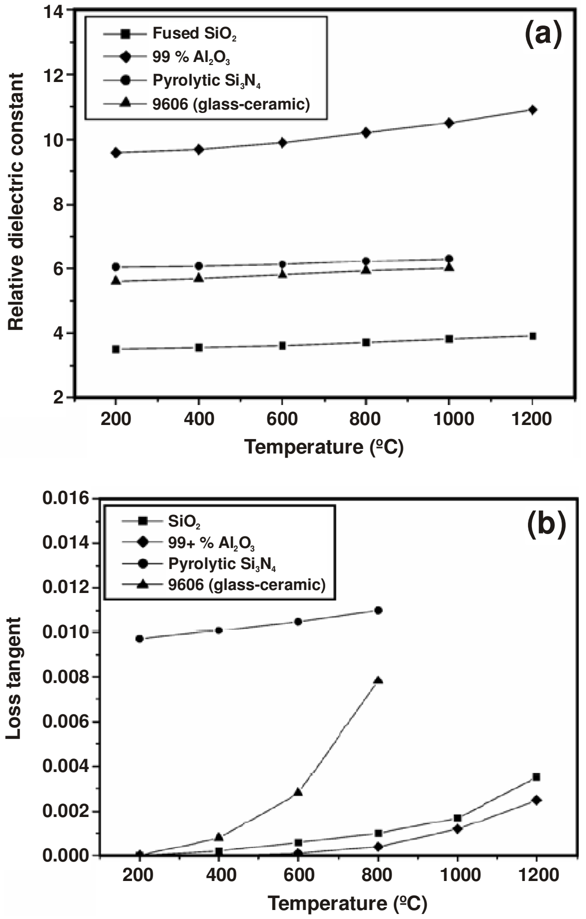

Fig. 1. (a) Relative dielectric constant *vs.* temperature and (b) loss tangent *vs.* temperature[2]

Alumina is the most common ceramic and has been widely used in microwave-sintering research by many scientists. Because of its highly refractory nature, it is difficult to sinter to full densification unless suitable sintering aids or some special processing techniques are adopted. Almost full sintering of alumina has been achieved much faster and at Bulk alumina, zinc oxide, silicon carbide, zirconia-toughened alumina, $MgAl_2O_4$,Y-PSZ, hydroxyapatite, Y-TZP 20 wt % Al_2O_3 composites and glass matrix composites etc have been prepared by this technique[20-25]. In addition, sintered 9Y-PSZ with very high relative density (*ca.* 97 %) and equiaxed grains (*ca.* 15 μm) was also achieved[26] by microwave sintering for only 35 min at 1625 °C.

Zirconia being a refractory oxide ceramic often requires high sintering temperatures and soaking time to obtain high degree of densification. In microwave fine grained zirconia ceramics were sintered at 1360 °C/2 min in a multimode, 2.45 GHz system[27]. In another study, single phase fully cubic calcium stabilized zirconia has been prepared from the precursor obtained by the mixed oxide method at a temperature as lower as 1100 ºC within a time period of 5 min[28]. Binner *et al.*[29] have reported the fabrication of the transparent zirconia ceramics from the nanopowder by the microwave hybrid heating at 1600 °C.

Single-phase PZT[30] has been obtained using a microwave-assisted process at 600 °C with non-stoichiometric titanium oxide (TiO_{2-x}). The use of TiO_{2-x} enhances the microwave absorption and increases the reaction kinetics substantially. The use of the non-stoichiometric precursors also leads to a different reaction pathways for the formation of PZT. In another study, PZT samples have been microwave sintered at a temperature 150 °C lower than the conventional process[30], which has resulted in finer grain size and the minimal PbO loss.

Vaidhyanathan *et al.*[31] synthesized single phase materials BMT in the microwave using reduced oxide precursors and sintered. The use of Ta_2O_{5-x} remarkably enhanced the reaction kinetics and produced single phase material at 1300 °C/20 min with higher densification than normally obtained by conventional processes.

Agrawal *et al.*[32] sintered various types of zinc oxide varistor under different processing conditions using microwave heating.

The hydroxyapatite (HAp) ceramics were sintered in microwave with significant time and energy savings[33,34]. Rodriguez-Lorenzo *et al.*[35] fabricate hydroxyapatite ceramics with tailored mechanical properties using microwave energy. Fang *et al.*[36] sintered hydroxyapatite fully in to a transparent ceramic at 1100 °C in 10 min by microwave processing. Fang *et al.*[37] fabricated transparent ceramics of spinel and alumina. Cheng *et al.*[38] made fully transparent AlON ceramics using multimode microwave system at 1800 °C.

Cheng *et al.*[39] achieved full sintered composites of WC-Co green bodies in only 2 h. The sintered products exhibit better mechanical properties than conventional parts. Multilayer ceramic capacitors (MLCCs) are used in almost all electronic devices. Microwave sintering of nickel-electrode MLCCs has been conducted in an intermediate reducing atmosphere. At a temperature of *ca.* 1250 °C, these chips are well sintered, which results in dense and uniform parts without delaminations or cracks[40].

Microwave energy has been used to melt and resolidify oxide and non-oxide refractory ceramic eutectic compositions in yttrium aluminum garnet (Al_2O_3-$Y_3Al_5O_{12}$; YAG) (T_m = 1827 °C) and boron carbide-titanium boride (B_4C-TiB_2) (T_m = 2310 °C) systems in a multimode 2.45 GHz microwave furnace[41].

Batches of Si_3N_4 cutting tools, with 90 parts per batch, were sintered using a cylindrical multimode cavity[11]. Energy consumption was estimated to be 80 % less than experienced with conventional heating.

An examination of the microstructures and the porosity distributions of the conventional and the microwave-sintered samples reveals that the microwave processed samples have more uniform microstructures than the conventional samples in which the core has more pores than the surface. An important distinction in the microstructures of the conventional and the microwave-sintered samples is that the pores in the microwave-sintered samples have more rounded edges than those of the conventional samples. It is commonly known that the sintered products exhibit an increasing ductility when the pore shape is more spherical. This has been proved by conducting a standard test for measuring the ductility and the toughness of the hollow cylindrical samples of FC208. It has been found that the conventional part failed at a load of 320 lbs whereas the microwave processed part at 430 lbs, indicating an increase of *ca.* 30 % in the strength[6].

Roy *et al.*[42] reported the first attempt at microwave sintering of powder metals and since then many other researchers have reported successful sintering of many metallic materials[43,44]. The application of microwaves to metallic materials has been extended from sintering to melting, brazing and joining of bulk metals[45].

Agrawal *et al.*[6] have shown that the powdered metals are in fact efficient microwave absorbers and as a result can be rapidly heated to their sintering temperatures. Many commercial powder-metal components of various alloy compositions, including Fe and steel, Cu, Al, Ni, Mo, Co, W, WC and Sn have also been sintered successfully with the microwaves producing essentially fully dense bodies.

Recently, Hwang *et al.*[46] have succeeded in combining microwaves with the electric arc furnace to develop a new clean and green steel making technology in which the CO_2 reduction has been substantially reduced and energy consumption has been cut down by 25 % over the conventional basic oxygen furnace technology. The unique aspect of this technology utilizes the advantages of rapid volumetric heating, high energy efficiency and enhanced reaction kinetics microwaves offer.

Several researchers[47] have demonstrated that a range of particulate metals and alloys can be heated using microwaves and can result an overall 60 to 90 % reduction in the processing time. Besides cost advantage, a reduced processing time, microwave sintered compacts have more homogenous and refined microstructure which leads to an improved mechanical properties.

Recently, model experiments on initial stage of microwave sintering of binderless tungsten carbide spheres have been carried out. Pure microwave heating was used during sintering experiments in 2.45 GHz multimode applicator. An anomalous neck growth in the initial period during microwave sintering was also revealed, which was then followed by neck growth obeying mechanisms of volume and surface diffusion. The value of activation energy of neck growth process has been significantly lower then any data on activation energy of diffusion processes in W-C system and may be explained by overheating in the neck zone, or even formation of liquid phase in the neck area[48].

Mondal *et al.*[49] were successfully sintered 90W-7Ni-3Fe alloys both through solid-state as well as liquid phase sintering in microwave furnace. As compared to conventional sintering, microwave sintering resulted in about 80% reduction in the overall processing time.

Mondal *et al.*[50] successfully sintered compacts of W-18Cu alloys in a microwave furnace for temperatures corresponding to solid and liquid phase sintering in relatively much shorter time than in the conventional heating method. In spite of higher heating rate in microwave sintering, no micro- or macrocracks in the sintered samples were observed which essentially supports the volumetric heating nature of microwave sintering. Microwave sintering lowers the sintering temperature to achieve the optimum densification as well as optimum mechanical properties.

Recent attempt by Glenn Research Center using a variable frequency microwave recovery process seems to have potential of economical, efficient and effective method for recycling used tires. Almost 100 % content of the tire is shown to have converted into useful products such as steel, oil, solid carbon and heating gas[27].

When oil shale (sedimentary rock) is heated in a special process (retorting) the petroleum products are released. Global resource Corp. in a US patent application has shown that variable microwave frequency based technology can be applied to heat oil shale to produce gases which can be condensed into petroleum products with high calorific value[27].

Microwave effects

The effect of microwave irradiation on a chemical reaction is very complex in nature and involves thermal and non-thermal effects.

Thermal effects arise from the different characteristics of microwave dielectric heating. The thermal effects observed under microwave irradiation conditions are a consequence of the inverted heat transfer, the inhomogeneities of the microwave field within the sample and the selective absorption of the radiation by polar compounds.

Overheating of polar liquids is an effect that can be exploited practically. Baghurst and Mingos[51] detected this effect in polar liquids on using microwaves, where overheating in the range 13-26 °C above the normal boiling point may occur. This effect can be explained by the "inverted heat transfer" effect since boiling nuclei are formed at the surface of the liquid. This effect could explain the enhancement in reaction rates observed in organic and organometallic chemistry. This thermal effect, which is not easily reproduced by conventional heating, can be used to improve the yields and the efficiency of certain processes.

The overheating effect has been demonstrated by Mingos[52] in the decomposition of H_2S over γ-Al_2O_3 and MoS_2-γ-Al_2O_3.

Zhang *et al.*[53] found that the higher conversion under microwave irradiation was attributed to the presence of hot spots. The authors estimated the temperature in the hot spots to be *ca.* 100-200 °C higher than the bulk temperature.

Zhang[54] found that the hot spots may be created by the difference in dielectric properties of materials, by the uneven distribution of electromagnetic field strength or by volumetric dielectric heating under microwave conditions. Selective heating has been exploited in solvents, catalysts and reagents.

Microwave irradiation is a selective mode of heating. Characteristically, microwaves generate rapid intense heating of polar substances while apolar substances do not absorb the radiation and are not heated[52].

Non-thermal effects were claimed initially by Gedye *et al.*[55] when they observed significant rate enhancements for hydrolysis and esterification reactions. Jacob *et al.*[56] published an excellent review on synthetic results to which the microwave effect has been attributed.

Binner[57] concluded that molecular mobility can increase in the presence of a microwave field and that in this case it is the Arrhenius pre-exponential factor A that changes and not the energy of activation. An increase by a factor of 3.3 in the Arrhenius pre exponential factor could explain the acceleration in reaction rate obtained with microwaves. The Arrhenius pre-exponential factor depends on the frequency of vibration of the atoms at the reaction interface and it has therefore been proposed that this factor can be affected by a microwave field.

The use of microwaves leads to a temperature reduction of 80-100 °C in the sintering temperature of partially stabilized zirconia[57], an effect that is non-thermal in nature. Wroe *et al.*[58] showed that a microwave field improves either the volume or grain-boundary mechanism rather than improving diffusion at the surface that is dominant at low temperatures.

Another interesting study was reported by Zhang *et al.*[59] on the synthesis of aromatic esters by esterification of benzoic acids in refluxing alcohols. The authors used microwave radiation at a frequency of 1.0 GHz, where there is no microwave heating action but only an athermal microwave effect. Interestingly, under these conditions a reduction in reaction time was still observed.

Other reports include non-thermal effects in solid phase separation processes[60] partitioning of *p*-nitroaniline between pseudo-phases[61] structural transformations in amphiphilic bilayers[62] and protein-catalyzed esterifications and trans-esterifications[63].

'Anisothermal' process occurs only in microwave heating, where the components of the system heat at different rates to different temperatures. A large temperature difference leads to a reaction path and diffusion mechanisms different from those of the conventional isothermal heating but there is hardly any information is available in the literature on anisothermal reactions and associated diffusion mechanisms. Microwave enhancement effects have not been observed universally. In the synthesis of PZT and BT the anisothermal approach enhanced reactivity between the starting phases and produced the desired phase in a few minutes. The reaction of the mixture of these two phases in a microwave field occurs radically different from the conventional isothermal heating situation. In the microwave case, at 250 °C, no soak time hexagonal BT appears and at 900 °C in 5 min nearly phase pure tetragonal BT is formed. On the other hand the conventional process even at 1300 °C for 1 h soaking time does not produce any X-ray diffraction detectable tet-BT phase[64].

The sintering of ZrO_2-toughened Al_2O_3, ZrO_2 8 % Y_2O_3 and zirconia/12 % CeO_2 at 2.45 GHz provides an example of how a hybrid heating process can improve unstable heating[65]. SiC rods were inserted into the insulation that surrounded the specimens in what was referred to as the "picket fence" arrangement. The microwave energy initially heated the SiC rods, which, in turn, transferred heat to the insulation and eventually to the specimens. As the specimen temperature increased, it more effectively coupled with the microwave energy and began to heat directly.

Another hybrid microwave-heating scheme involved applying a thin layer of SiC powder to the interior of the thermal insulation chamber that is placed within the microwave oven[66].

In our laboratory, we have successfully exploited microwave energy to reduce the temperature required for sintering of a of doped and codoped ceria electrolytes which are potential electrolytes for IT-SOFC so we have obtained electrolytes for SOFC applications with improved mechanical and electrical properties.

In conclusion, microwave processing is emerging as a novel and innovative technology with many advantages over conventional processing. There is, therefore, increasing industrial application of microwave energy for heating, drying, curing and sintering of materials. Until 2000, microwave processing of materials mostly was confined to ceramics, semimetals, inorganic and polymeric. The most recent significant development in the microwave processing has been the sintering, brazing, joining and melting of metals also.

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