CERAMIC PROCESSING VIA MICROWAVE IRRADIATION

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ABSTRACT. Nowadays, several thermal treatment procedures and technologies are being used for in ceramic processing. The application of microwave irradiation in ceramic manufacturing, for the consolidation of the final ceramic structure, has many energy and economical avails, while at the same time it is environmentally benign. In this work, the microwave irradiation potential usage in energy-efficient ceramic manufacturing is explored via a comprehensive literature review. Specifically, the advantages/disadvantages of the microwave irradiation compared to conventional heating technologies is discussed. Three different classes of ceramic materials, with industrial significance, are considered: a) bulk ceramics, b) electroceramics and c) bioceramics. Furthermore, future applications which may attract commercial concernment are also pointed out.

KEY WORDS: ceramics, processing, microwave irradiation, sintering.

1 INTRODUCTION

Ceramic materials have many technical advantages and can be successfully used in diverse technological applications and environments. Sintering is the foremost process, not only in ceramic industry but also in powder metallurgy, which has been significantly evolved during the past 70 years. During the sintering process, compressed particles are heated to sufficiently high temperatures. In this way chemical bonds between the particles are formed due to enhanced atomic mobility. The sintering process's driving force is the particle's surface tension, which leads to the minimization of surface area. Many factors may affect the sintering process: the type of material being used, its melting temperature, the particles' geometrical characteristics (surface, size, volume,) and other processing aspects. The basic mechanisms of the sintering procedure are: the grain boundary diffusion, the viscous flow, the evaporation and the recondensation. The conditions during sintering designate the microstructure evolution in the ceramic material. The attained microstructure significantly affects several physiochemical properties of the final ceramic material. Clayey raw materials solid-state sintering, via conventional thermal treatment, has been regarded as the most common manufacturing procedure for the fabrication of industrial ceramic materials (Kingery et al., 1976; Gotoh et al., 1997; Olevsky, 1998; Olevsky et al., 2006; Ch'ng & Pan, 2007; German 2010; Kirchhof et al., 2012; Matrenin et al., 2015).

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On the other hand, microwave irradiation is an innovative, emerging and environmentally benign technology. It can be used in several technological fields. Most common of them include biomass processing, green chemistry, biosciences, sewage sludge, waste and wastewater treatment (specifically in the oxidation/degradation of pollutants which have poor biodegradability). Moreover, microwave irradiation can be used in the nanoparticles' synthesis, the processing (consolidation) of nanocomposites, the production of nanoporous materials, and in metallurgical processing routes which include the agglomerates' drying and reduction of ore's concentrates. The main benefits of using microwave irradiation in industrial processes are the substantial decrement in processing time and energy and equipment's dimensions. Moreover, one other benefit is the increment in selectivity, product yield and purity when microwave irradiation is applied in purification reactions or chemical synthesis (Appleton et al., 2005; Tompsett, Conner et al., 2006; Viswanathan et al., 2006; Bai et al., 2007; Das et al., 2008; Yuen & Hameed, 2009; Budarin et al., 2010; Leonelli & Mason, 2010; Remya & Lin, 2011; Bassyouni et al., 2012; Motuzas et al., 2014). In particular, it has been reported that microwave-assisted sintering can be successfully employed for the processing of a broad range of oxide and non-oxide containing ceramic materials (Riedel & Svoboda, 2006; Mascarenhas et al., 2008; Oghbaei & Mirzaee, 2010; Grundas, 2011).

In this work, the microwave irradiation potential usage in energy-efficient ceramic manufacturing is explored via a comprehensive literature review. Specifically, the advantages/disadvantages of the microwave irradiation compared to conventional heating technologies are discussed. From material's perspective the significance of microwave sintering is underlined in this work; in order to evade unwanted growth of the material's grains, which leads to the deterioration of the mechanical performance, fast heating is highly desirable for the advanced ceramics' sintering. In that sense, three different classes of ceramic materials with industrial significance is considered: a) bulk ceramics, b) electroceramics and c) bioceramics. Furthermore, the impact of the processing parameters, during the microwave irradiation procedure, on the formation of the final ceramic microstructure is also described. Finally, future applications which may attract commercial concernment are also pointed out.

2 MICROWAVE IRRADIATION

Microwave irradiation has the ability to provide significant amounts of energy directly to the material. Microwave irradiation causes the internal heating of the material. Microwave irradiation is considered as an energy efficient heating method due to the fact that the provided energy causes internal heating of the material via molecules' interactions with the electromagnetic field. The microwave irradiation interplay with a dielectric material causes dipoles' rotation and translational motions of bound or free charges, due to the molecular-level energy transfer. In that sense, heat loss occurs due to the material's impedance to the indicated motions. Therefore, heat is produced in the total volume of the material. In this way, the processing time and cost for the thermal treatment of the material is significantly reduced.

The heating efficiency of the microwave irradiation may be expressed by the material's absorbed power. The expression of the material's absorbed power per unit volume, P (W·m-3) is given below (Das, Mukhopadhyay et al. 2008).

$$P = \sigma \, |E|^2 = 2\,\pi\, f\, \varepsilon_o\, \varepsilon'_r \tan\delta\, |E|^2 \qquad (1)$$

Material's dielectric parameters (i.e. tanδ and ε′r) determine the microwave energy field distribution inside the material. Particularly, ε′r (i.e. the dielectric constant of the material) indicates the microwave energy storage ability of the material. On the other hand, tanδ (i.e. the loss tangent) designates the electric field's penetration efficiency in the material and the material's dissipation ability to convert some part of the supplied energy into heat (Al-Harahsheh & Kingman, 2004).

3 MICROWAVE SINTERING OF CERAMIC MATERIALS

Microwave sintering procedures can significantly improve the densification efficiency of ceramic materials. Moreover, these procedures can significantly reduce the time needed for a thermal treatment cycle due to the uniform and rapid heating of the ceramic material. This processing time reduction significantly reduces the energy consumption and the cost for the ceramic materials' processing.

Due to the aforementioned benefits of using microwave irradiation to ceramic processing, its industrial usage should be promoted. Since the processing conditions have a significant impact on the microstructure and physiochemical properties of the produced ceramic materials, it would be interesting to comparatively investigate microwave sintering versus the various conventional sintering methods.

Notable progress has been made already regarding the microwave irradiation usage for the ceramic materials' sintering. In particular, the advanced ceramic materials fabrication demands ultra-fast sintering processes to avert unwanted growth of the material's grains. This unwanted growth leads to the deterioration of mechanical performance, as aforementioned. The densification (consolidation) of ceramic materials can be completed in very short times via microwave heating. The very fast and the superior densification quality, which is achieved by using microwave irradiation during the production of ceramic materials, may be explained by considering the microwave irradiation as wave. The heating of a ceramic material via microwave irradiation may be ascribed to non-thermal phenomena. Specifically, it can be explained by the accumulation of electrical energy amounts inside sealed pores of the non-sintered ceramic material (Agrawal, 1998). Nevertheless, another study related to the study of microwave-irradiation-assisted sintering of compressed ceramic powders, in the microscopic level, has shown that the electric field is not evenly spread throughout the material's grains. Specifically, the electric field strength was found to be higher in areas which are closer to grain boundaries and rough surfaces. These microscopic areas appear to promote the intense concentration of electric field which cause a non-uniform overall energy distribution inside the ceramic material (Birnboim et al., 1999). Consequently, the optimal parameters of the microwave irradiation procedure should be carefully determined in order to obtain the desired microstructure. In that sense, various process monitoring and quality control techniques have been developed, in order to understand, in a more complete manner, a sintering process which involves the use of microwave irradiation. For instance, the real time (in-situ) shrinkage measurement and control, during microwave sintering, in a specially designed microwave cavity, was proposed (Marinel & Savary, 2009).

On the other hand, there are several types of ceramic materials (e.g. red ceramics, glass-ceramics, nano-ceramics etc.) with different characteristics and industrial applications. Nevertheless, advanced ceramics have attracted much attention lately due to their broad field of application. Three different classes of ceramic materials with industrial significance are considered here: a) bulk ceramics, b) electroceramics and c) bioceramics. Our most significant literature findings, related to the aforementioned classes of ceramic materials are summarized in Table 1 (in the end of section 4).

3.1 Bulk ceramics and glass-ceramics

There are several literature findings which underline the significance of the microwave sintering for the production of bulk ceramics and glass ceramics. Croquesel J. et al. have successfully employed microwave irradiation in the sintering process of fine-grained pure α-Al_2O_3 powder (Croquesel et al., 2015). Specifically, they designed an appropriate cavity and suitable cells, in order to perform not only direct microwave sintering but also hybrid sintering. Finite Element Modelling (FEM) was used so as to be able to perform reliable comparative studies regarding the efficiency of microwave sintering versus conventional sintering, during the same thermal treatment cycle. Moreover, they found out that the degree of densification of α-Al2O3 upon microwave sintering was much higher than the one recorded upon conventional heating, during the first two stages (initial and intermediate) of the thermal treatment cycle. It is worth noting that no doping element was used for the heating initiation (Croquesel et al., 2015). On the other hand, Chatterjee A. et al. studied the microwave sintering procedure of Al2O3 during conventional and microwave sintering (Chatterjee et al., 1998). They found no significant differences in grain growth and densification between the two heating procedures, during the last stages of the sintering procedure. They concluded to this result by analyzing the heating and the microwave power absorption profiles (Chatterjee et al., 1998).

In another study regarding Al_2O_3 ceramics, Bykov Y. V. et al. studied the microwave processing impact on the mass transport phenomena and phase transformations during the microwave-assisted annealing of nanoporous Al_2O_3 membranes (Bykov et al., 2010). Moreover, their study revealed that faster mass transport, which primarily depends from the intensity of the microwave energy field, is recorded during the microwave sintering process of Al_2O_3. Furthermore, the preferred orientation of pores inside the nanoporous Al_2O_3 membranes may be predicted and contingent phase transformations may be quantitatively characterized (Bykov et al., 2010).

Microwave irradiation has also been used for the production of magnesium-based composites. Specifically Wong, W. et al. underlined the importance of applying microwave irradiation for the production of light weight (and energy saving magnesium based materials (Wong & Gupta, 2015). The microwave sintering usage resulted in a significant reduction (around 80%) in the processing times and energy requirement comparing to conventional sintering, while the properties of the produced composites was not significantly affected (Wong & Gupta, 2015).

D’Arrigo M. C. et al. studied the microwave sintering process applied to the system CaO-ZrO_2-SiO_2, for the production of glass-ceramics (D'Arrigo et al., 2002). They found that the fabrication procedure, when microwave irradiation applied, appeared to be accelerated compared to the conventional sintering procedure. Nevertheless, the microstructural evolution and the densification, which were studied during the crystallization of glass-ceramics, was not affected by the different sintering procedures (microwave vs conventional) applied (D'Arrigo et al., 2002).

There are also some studies which indicated the applicability of microwave sintering to ceramic materials composed of raw materials deriving from industrial byproducts. Specifically, Karayannis, V. G. et al. reported the microwave sintering of ceramic compacts consisting of different types of fly ash and bottom ash. Dense ceramic microstructures, composed of highly calcareous lignite fly ash and bottom ash, were produced after applying a microwave sintering procedure for 30 min (at 1000°C) (Karayannis et al. 2013). Moreover, the ceramic samples composed of siliceous fly ash exhibited higher densification degree values, upon microwave sintering, than the conventionally-sintered samples, by applying the same thermal treatment procedure (800-1000°C) (Fang et al., 1996).

Some other studies underline the importance of using microwave irradiation during the sintering procedure applied to ceramic materials containing various types of waste. Chou S.-Y. et al. applied microwave irradiation to enhance the sintering process aiming at the transformation and the stabilization of calcium carbonate containing washed fly ash (produced by municipal solid waste incineration) into block ceramic materials (Chou et al., 2009). Moreover, they found out that the microwave sintering procedure was much more efficient comparing to the other conventional sintering procedures, which use electric furnaces operated in the temperature range 800-1100°C (Chou et al., 2009). Makul N. et al. reported the rapid sintering, in the temperature range 800-1200°C, of rice husk ash (Makul & Agrawal, 2010). Rice husk ash is a SiO_2-rich material, having α-SiO_2 and SiO_2-cristolbalite as the main mineralogical phases. After applying the microwave sintering procedure crystalline percentage of the silica phase was increased. This led to the significant improvement of the mechanical properties of the final ceramic materials (Makul & Agrawal, 2010).

3.2 Electroceramics

Electroceramics, is a category of advanced ceramics which exhibit interesting electrical properties. The electroceramics category includes ferroelectric materials, ferrites, solid electrolytes and piezoelectric materials, which can be used in many industrial applications.

Various electroceramics were tested during microwave sintering. Specifically, Angalakurthi R. et al studied the microwave sintering procedure of strontium bismuth titanate (SBTi) (Angalakurthi & Raju, 2011). The resulting SBTi electroceramics exhibited much higher densification degree, finer microstructure, better ferroelectric and mechanical properties than the samples processed by conventional sintering procedures. Furthermore, these enhanced properties were achieved in shorter sintering times than the times required for conventional sintering (Angalakurthi & Raju, 2011). Sonia Patel R. K. et al. employed microwave sintering (at 1100^{o}C for 1h) to produce Calcium-doped barium titanate ($Ba_{(1-x)}Ca_xTiO_3$) consolidated electroceramics (Sonia et al., 2011). The microstructural and compositional analysis of the produced electroceramics revealed a dense but homogeneous arrangement of the sub-micrometer sized grains and the formation of a single perovskite phase. Moreover, Yan Y. et al. fabricated SiO_2 doped $BaTiO_3$ electroceramics at relatively low temperatures by using microwave sintering (Yan et al., 2016). With the addition of 0.5wt% SiO_2 the produced electroceramics revealed homogeneous microstructures with relatively small grains, while the addition of larger amounts of SiO_2 (1 and 2 wt%) in the electroceramic led to densification degree enhancement and the formation of a secondary phase ($Ba_2TiSi_2O_8$) with columnar grains. These microstructural and mineralogical phase changes in the electroceramics resulted to their electrical break-down strength improvement (Yan et al., 2016).

Electroceramic materials that contain lead zirconate titanate (PZT) perovskite species exhibit enhanced piezoelectric effect and are being used in several industrial applications. Kumar A. et al. produced novel electroceramics from lanthanum-doped PZT (PLZT) fine powder (Kumar et al., 2016). The production route they followed involved high energy mechanical ball milling and subsequently microwave sintering at 1150 ^{o}C. The processing times were much shorter in the microwave sintering procedure than the ones for the conventional heating procedure. However, the samples processed with both processes exhibited similar dielectric and piezoelectric properties (Kumar et al., 2016).

Lanthanum gallate electrolytes consist an interesting class of electrocermaics which exhibit enhanced ionic conductivity. Lanthanum gallate electrolytes can be thermally treated (sintered) in lower temperatures than Yttria-stabilized zirconia (YSZ), specifically in the case of the lanthanum by alkaline rare earth elements (i.e. strontium and magnesium) towards the formation of lanthanum gallate doped with alkaline rare earths (LSGM) electrolytes. Kesapragada S. V. et al noted that, by using microwave sintering, it is possible to avert the secondary phases formation and grain coarsening, which are observed in elevated temperatures, during conventional sintering procedures. Therefore, stable and dense electrolyte layers can be produced due to the fact that the in situ heat generation leads to much faster sintering kinetics, by applying microwave irradiation. The fabricated LSGM materials may be used solid oxide fuel cells operated in intermediate temperatures (Kesapragada et al., 2003).

On the other hand, microwave sintering can be applied in the thermal treatment procedures for the production of ferrites. For example, microwave sintered MnZn-ferrites, which are destined to be used in high magnetic permeability applications, presented much coarser microstructures and higher densification degree, compared to the conventionally sintered samples. Therefore, the produced MnZn-ferrites exhibited much higher magnetic permeability values, compared to the conventionally sintered samples (Tsay et al., 2001; Tsakaloudi et al., 2004; Thota et al.; 2014).

3.3 Bioceramics

Bioceramics is also an important industrial category of advanced ceramic materials, in which ceramic processing via microwave irradiation increasingly applied. Mirhadi B. et al. reported the production of dense β-tricalcium phosphate (β-TCP) ceramic materials, via microwave sintering, starting from sub-micrometer β-TCP powder (synthesized by in-situ wet chemical precipitation) (Mirhadi, 2014). The microwave-sintered (at 1100°C for 15 min) samples have shown higher degree of densification and hardness than conventionally-sintered (at 1100°C for 2h) samples. Consequently, the microwave sintered β-TCP ceramics exhibited enhanced mechanical properties, even though they have been processed for significantly lower times (Mirhadi 2014). Veljovic D. et al. produced microwave-sintered bioceramics composed of agglomerated hydroxyapatite (HAP) and tricalcium phosphate (TCP) powder. The resulting porous microwave-sintered bioceramics exhibited interesting characteristics: shapeless inter-agglomerate pores and spherical intra-agglomerate pores. The bioceramics density achieved during microwave sintering appeared to be slightly higher than the one achieved for the concentionally-sintered sampled, while the fracture toughness and hardness was also increased in the microwave-sintered ceramics, compared to the conventionally-sintered ones (Veljović et al., 2010).

Another study related to bioceramics, from Upadhyaya D. et al., indicated the applicability of microwave sintering for the production of stabilized zirconia (3Y-TZP) bioceramics (Upadhyaya et al., 2001). 3Y-TZP is considered as an excellent microwave irradiation absorber due to the large number of point defects which are present in the structure of the bioceramic. This led to the production of highly consolidated (high density) bioceramic materials with rather uniform grain size distribution and, consequently, improved fracture toughness and hardness. In order to achieve similar properties' values for this type of bioceramics, via conventional sintering, much longer processing times were needed (Upadhyaya et al., 2001).

4 CONCLUDING REMARKS

The ceramic materials sintering based on microwave irradiation arises emerges as a novel and powerful ceramic processing technique for the production of advanced ceramics in relatively short times. Considerable progress has been made towards the commercialization of microwave sintering technology and the development of novel applications, especially in the area of advanced ceramics.

The comparison of microwave sintering method with conventional sintering procedures revealed the several advantages of microwave sintering, regarding the physiochemical properties and the microstructure of the final ceramic material:

• Through rapid heating during the microwave sintering the fast densification (consolidation of the ceramic material can be achieved. Moreover, rapid heating hinders excessive coarsening of the grains. Therefore, uniform and fine-grained ceramic microstructures are received which leads to improved mechanical properties.

• Considerable temperature gradients, between the internal volume of the ceramic material and its surface, occur during conventional sintering procedures. However, during microwave sintering these temperature gradients are eliminated due to high heating rates. This has to be considered in the fabrication of ceramic products which have large volume, since it may significantly affect the quality of the ceramic product.

• The microwave sintering procedure needs to be optimized for each specific class of ceramic materials, due to the fact that microwave irradiation may change various characteristics of the ceramic material. Specifically, the heat transfer mechanisms, the distribution of the electromagnetic field, the material's mineralogical phase transformations and the interaction between the material and the microwave irradiation should be considered during process optimization.

Table 1. Microwave technology in ceramic manufacturing

Ceramic materials	Applications / processing	Mesures	Factors	Main Results	References
Bulk ceramics & glass-ceramics	Microwave sintering via polymer impregnation and pyrolysis (PIP) process	Mechanical properties of SiC/SiC composites	The flexural properties, the interfacial debonding strength between SiC fiber and matrix of the fabricated SiC/SiC composites	Decreased the residual strength of SiC fibers as reinforcement and increased the interfacial debonding strength	(Yang et al., 2015)
	Hybrid and direct microwave sintering for ceramic powders		Wide range of heating rates	Significant enhancement of sintering	(Croquesel et al., 2015)
	Microwave sintering for synthesized magnesium composites	Energy efficient environment friendly microwave	Various types of metallic and ceramic reinforcements	Significant reduction of 80% in both processing time and energy consumption	(Wong & Gupta 2015)
	Microwave sintering to produce a glass-ceramic material	Densification and microstructural evolution of a CaO-ZrO_2-SiO_2 glass powder	Soaking times and temperatures	Speed up the sintering process	(D'Arrigo et al., 2002)
	Microwave sintering for powdery materials	Ceramic micro-structures	Ash particle size and shape, green density of the compressed powders, and sintering time	Rapid sintering	(Karayannis et al., 2013)
Electro-ceramics	Microwave sintering of Strontium Bismuth Titanate (SBTi)	Micro-structure, and mechanical, ferroelectric properties	Conventional and microwave furnaces	higher densification,fine microstructure, good mechanical and ferroelectric properties, shorter time	(Angalakurthi & Raju, 2011)
	Microwave sintering of ceramic pellets from powders of lanthanum	Sintering behaviour, microstructure, dielectric, piezoelectric properties	Sintering time and temperatures	Highest relative density	(Kumar et al., 2016)
	Activated microwave sintering of LSGM powders	Microstructure and phase formation	Sintering power,time and temperatures	Dense stable electrolyte layer	(Kesapragada et al., 2003)
	Microwave firing for MnZn-ferrites	Microstructures	Sintering power, time and temperatures	Materials to be used in high magnetic permeability appl..	(Tsakaloudi et al., 2004)
Bio-ceramics	Microwave sintering of ground RHA (Grha)	Microstructures	Sintering temperatures	Crystalline silica phase has been increased	(Makul & Agrawal, 2010)
	Microwave sintering of nano sized beta tricalcium phosphate (β-TCP) powder	Dense β-TCP ceramics	Phase composition and mechanical properties of materials	MW sintered β-TCP samples have superior mechanical properties	(Mirhadi, 2014)
	Microwave sintering of 3Y-TZP ceramics	Microstructu-ral and mechanical property	Conventional and microwave methods	Benefits in terms of microstructural design and mechanical properties	(Upadhyaya et al., 2001)

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6 NOTATION

The following symbols are used in this paper:
E = the magnitude of the internal field (V m-1),
σ = the total effective conductivity (S m-1),
f = the frequency (Hz). It is usually around 2.45 GHz, which is considered sufficient medical, scientific and industrial purposes.
εo = free space’s permittivity which is constant (εo = 8.86×10–12 F m-1).
ε′r = the relative dielectric constant and
tanδ = the loss tangent..