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## Microwave Assisted Technology for Transparent Conducting Oxides Synthesis

Jayathilake DSY\* and Peiris TAN\*

Department of Chemistry, Loughborough University, Loughborough, UK

### Abstract

Transparent conducting oxides are essential component in large number of modern devices. Co-precipitation, sol-gel synthesis, hydrothermal and solvothermal methods are the common methods use to synthesize transparent conducting oxide (TCO) materials. The microwave assisted synthesis has already been used to synthesize inorganic materials with unique properties. Though, transparent conducting materials synthesis using microwave radiation is emerging as a novel and innovative technology that shows a great potential to be useful in a range of applications with many advantages over conventional thermal synthesis. Microwave synthesis provides new approaches for enhancing the physical, chemical and optical properties of TCO materials as well as economic advantages through energy savings. The fundamentals of microwave synthesis and a summary of recent developments in microwave synthesis of TCO materials are presented in this review. This review helps to guide researchers who are interested in using microwaves to synthesize low cost alternative TCO materials and improve their properties by making full use of the capabilities of microwave radiation.

**Keywords:** Transparent Conducting Oxides; Microwaves; Synthesis; Energy saving; Low-cost

### Introduction

In recent years, microwaves have been explored as an efficient alternative energy source to traditional forms of heating (i.e. isomantles, oil baths, hot plates etc.) in materials synthesis [1]. It has been shown that the use of microwave radiation for material synthesis has shown promising results over conventional heating methods in terms of the product yield and the reaction rate [2]. This is due to deference in the two heating mechanisms (i.e. microwave heating and conventional heating) [3,4]. In the microwave process, the heat is produced within the material instead of external heat source. Particularly, microwave heat generation is due to an energy conversion rather than a heat transfer process. As the material is being treated under microwaves, the absorbed radiation is transformed to heat energy and the material turn into its own heating source [5,6]. Hence, the thermal gradient in a microwave processed material is the reverse of that in a material processed by conventional heating [7,8]. The temperature profiles of microwave and conventional annealing for a reaction are shown in Figure 1.

### Microwave Absorbing Characteristics of Materials

The interaction of microwaves with matter is quantified by two complex physical quantities; dielectric permittivity,  $\epsilon$ , and permeability (magnetic susceptibility),  $\mu$  [9]. In reality, the permittivity and permeability are complex quantities which are defined as  $\epsilon^*$  and  $\mu^*$  respectively as below equations 1 and 2 [10],

$$\epsilon^* = \epsilon' - i\epsilon'' = \epsilon_o \left( \epsilon_r' - i\epsilon_{eff}'' \right) \quad (1)$$

$$\mu^* = \mu' - i\mu'' = \mu_o \left( \mu_r' - i\mu_{eff}'' \right) \quad (2)$$

where  $\epsilon'$  is the dielectric constant which represents the time-independent polarizability of a material in the presence of an external electric field, that is to say it measures the resistance encountered when forming an electric field in a medium.  $\epsilon''$  is the dielectric loss or loss factor of the material, and indicates the time-dependent component of the permittivity which quantifies the ability of the material to convert absorbed energy into heat.  $\epsilon_r'$  is the relative permittivity of the material which is the ratio of the permittivity of a substance to that of free space or vacuum.  $\epsilon_{eff}''$  is the effective relative loss factor.  $\mu'$  is the magnetic permeability,  $\mu''$  magnetic loss factor,  $\mu_o$  the permeability of free space ( $\mu_o = 4\pi \times 10^{-7} \text{ Hm}^{-1}$ ),  $\mu_r'$  is the relative permeability of the material and  $\mu_{eff}''$  is the effective relative

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#### \*Correspondence:

Jayathilake DSY, Department of Chemistry, Loughborough University, Loughborough, LE113TU, UK.

**E-mail:** dsyjayathilake@gmail.com

Peiris TAN, Department of Chemistry, Loughborough University, Loughborough, LE113TU, UK.

**E-mail:** nirmalprs@gmail.com

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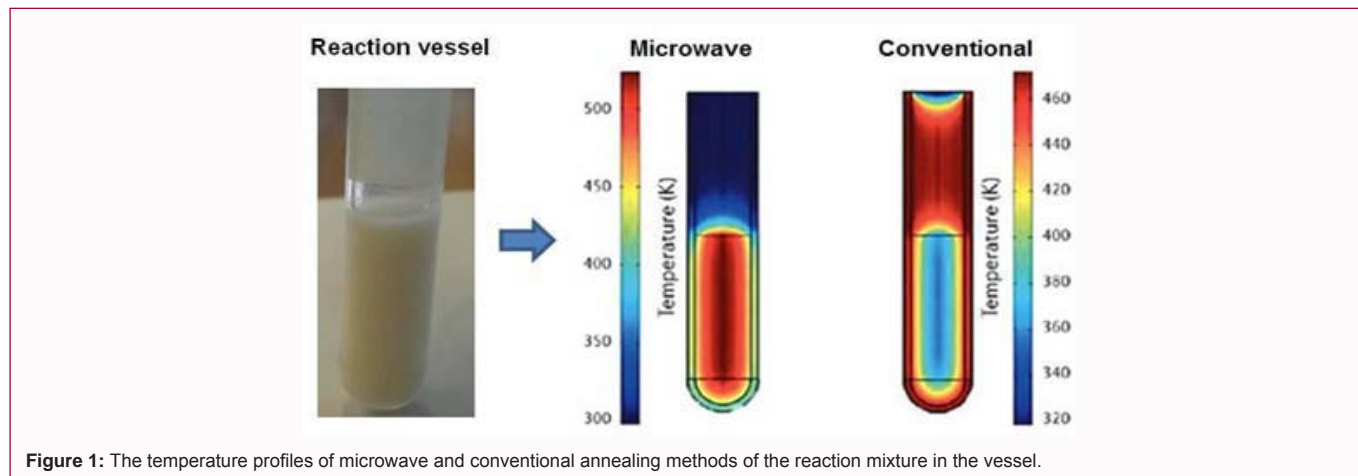


Figure 1: The temperature profiles of microwave and conventional annealing methods of the reaction mixture in the vessel.

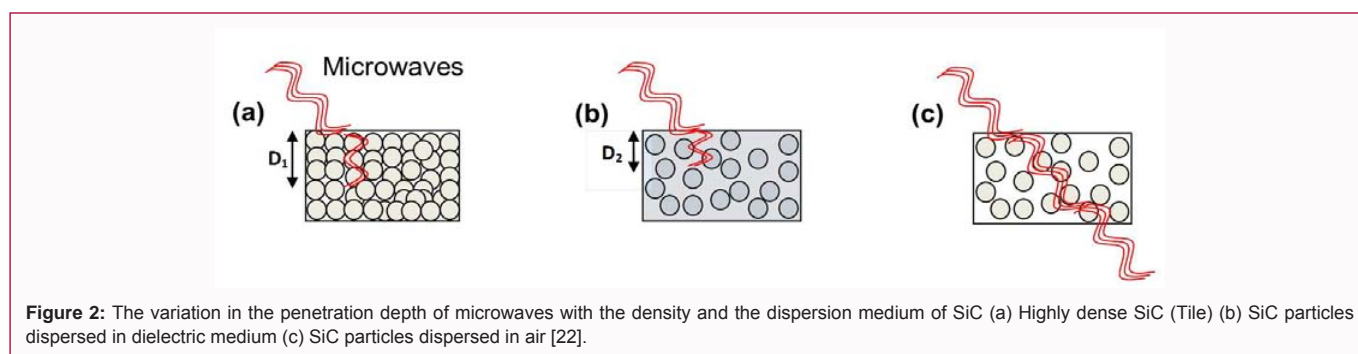


Figure 2: The variation in the penetration depth of microwaves with the density and the dispersion medium of SiC (a) Highly dense SiC (Tile) (b) SiC particles dispersed in dielectric medium (c) SiC particles dispersed in air [22].

magnetic loss factor.

The ratio of the dielectric loss to the dielectric constant is known as the loss tangent ( $\tan\delta$ ) or dissipation factor which is given in equation 3 [11],

$$\tan \delta = \frac{\epsilon''}{\epsilon'} \tag{3}$$

The angle  $\delta$  represents the phase lag between the polarization of the material and the applied electric field. The loss tangent is an indicator of the ability of the material to convert absorbed energy into heat. A good microwave absorber has  $\tan\delta \geq 0.1$  while those with  $\tan\delta \leq 0.01$  are transparent to microwaves. The heat generation by microwave energy can be quantified by equation 4 [12],

$$P = \sigma |E|^2 = 2\pi f \epsilon_o \epsilon'' |E|^2 = 2\pi f \epsilon_o \epsilon'' \tan \delta |E|^2 \tag{4}$$

where,  $P$  is power dissipation ( $W/cm^3$ ),  $\sigma$  is the total effective conductivity,  $E$  is the magnitude of the internal electric field, and  $f$  is the microwave frequency.

The intensity of heat evolution in a sample depends on the materials properties, the frequency and intensity of the applied field, penetration depth of microwaves into the substance and geometric size of the sample [13]. The distribution of energy within a material can be determined by the attenuation factor,  $\alpha'$  expressed as equation 5,

$$\alpha' = \frac{2\pi}{\lambda} \left[ \frac{\epsilon'}{2} \left\{ \sqrt{(1 + \tan^2 \delta)} - 1 \right\} \right]^{\frac{1}{2}} \tag{5}$$

The attenuation factor can be used to calculate the penetration depth ( $D$ ) as suggested by von Hippel [14]. It is inversely proportional to the penetration depth of the material as shown in the below equation (6),

$$D = \frac{1}{\alpha'} \tag{6}$$

Penetration depth is an important parameter which determines the heat generation of materials by microwaves. When microwaves are incident perpendicularly to the surface of the materials, its intensity decreases progressively due to dissipation inside the volume of the materials. The penetration depth is defined as the distance from the surface of the material at which the power drops to  $1/e$  from its value at the surface. This can be also expressed as equation 7 [15],

$$D = \frac{3\lambda_o}{8.686\pi \tan \delta \sqrt{\epsilon'}} \tag{7}$$

At a frequency of 2.45 GHz, the free space wavelength  $\lambda_o$  is 12.24 cm. Penetration depth ranges between several micro metres to several tens of meters depending on the dielectric nature of the material.

In metals, microwave propagation is usually described in terms of skin depth ( $\delta$ ) which is defined as the depth at which the magnitude of electric field drops to  $1/e$  of the value at the surface and it is expressed as,

$$\delta = \frac{1}{\sqrt{f \pi \mu_{o\sigma}}} \tag{8}$$

In general, if the penetration depth exceeds the sample thickness, the material behaves as if it is transparent to microwaves. Volumetric heating only results when the penetration depth is comparable to the sample thickness. If the sample is too thick, absorption is limited to the surface [16].

Microwave interaction with materials can be predicted if dielectric properties such as dielectric constant and dielectric loss are known. The loss tangent is an indicator of the ability of the material to convert absorbed energy into heat and the penetration depth determines the penetration ability of microwaves into the material.

**Table 1:** Transparent conducting metal oxides synthesized using microwave radiation are listed by the dopant, synthesis/thin film fabrication method, reported conductivity/resistivity with the references.

| Metal Oxide                      | Dopant | Synthesis method and additives           | Precursors  | Specialty and reaction conditions                  | Reference |
|----------------------------------|--------|--|---|--|-----------|
| ZnO                              | Al     | Glycolysis, H <sub>2</sub> O             | Zn(OAc) <sub>2</sub>  | Spheres ca. 12 nm MW - 30 min                      | [26]      |
|                                  |        | DEG                                      | AlCl <sub>3</sub>   | 200°C  |           |
| ZnO                              | In     | Glycolysis, H <sub>2</sub> O             | Zn(OAc) <sub>2</sub>  | Spheres 15 nm, 30 min                              | [26]      |
|                                  |        | DEG                                      | InCl <sub>3</sub>   | 200°C  |           |
| In <sub>2</sub> O <sub>3</sub>   | Sn     | Alkaline hydrolysis, DMF/EtOH            | InCl <sub>3</sub>   | Irregular polyhedrons 25 nm, 3–15 s                | [27]      |
|                                  |        | N(CH <sub>3</sub> ) <sub>4</sub> OH      | SnCl <sub>4</sub>   | 300°C  |           |
| In <sub>2</sub> O <sub>3</sub>   | Sn     | Glycolysis                               | InCl <sub>3</sub>   | Spheres 15–19 nm, 120 min                          | [28]      |
|                                  |        | DEG N(CH <sub>3</sub> ) <sub>4</sub> OH  | SnCl <sub>4</sub>   | 200°C, 1 atm                                       |           |
| ZnGa <sub>2</sub> O <sub>4</sub> | Cu     | Alkaline Hydrolysis, H <sub>2</sub> O    | Ga <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> , ZnSO <sub>4</sub> | Aggregates of spherical 10–20 nm Particles, 30 min | [29]      |
|                                  |        | NH <sub>2</sub> OH                       | CuCl <sub>2</sub>   | 150°C  |           |
| ZnO                              | Au     | Alkaline hydrolysis, EtOH                | Zn(NO <sub>3</sub> ) <sub>2</sub>                                   | Nanorods 10–15 nm x 100–600 nm 10 min              | [30]      |
|                                  |        | NaOH, PEG                                | H[AuCl <sub>4</sub> ]   | MW cycles: 10 s on/20 s off                        |           |
| TiO <sub>2</sub>                 | La, Zr | Acidic hydrolysis, iPrOH                 | Ti(OiPr) <sub>4</sub> , ZrCl <sub>4</sub>                           | Aggregates of 15–25 nm particles                   | [31]      |
|                                  |        | AcOH, citric acid, PEG, H <sub>2</sub> O | La(NO <sub>3</sub> ) <sub>3</sub>                                   | 4 min  |           |
| ZnO                              | Al, Ga | Glycolysis, H <sub>2</sub> O             | Zn(OAc) <sub>2</sub> , AlCl <sub>3</sub>                            | Smallest spherical particle size 80 nm, 30 min     | [24]      |
|                                  |        | DEG                                      | Ga(NO <sub>3</sub> ) <sub>3</sub>                                   | 200°C  |           |

The power required to raise the temperature of material from initial ( $T$ ) to final temperature ( $T_o$ ) is given by equation 9,

$$P' = mC_p(T - T_o) / t \quad (9)$$

where,  $P'$  is the required power to raise the temperature in  $\text{Js}^{-1}$ ,  $m$  is the mass,  $C_p$  the specific heat capacity and  $t$  is the time taken. If the microwave frequency is constant and the variations of materials density ( $\rho$ ) and specific heat capacity are relatively small, the temperature increase by microwave irradiation will be a function of  $\epsilon''$  as shown by the below derived equation from equations 4 and 9,

$$\frac{2\pi f \epsilon_o |E|^2 \epsilon''}{\rho C_p} = \frac{(T - T_o)}{t} \quad (10)$$

This confirms that materials' dielectric loss plays an important role in elevating the temperature of the material by absorbing microwave energy. Microwave penetration is determined by the material's dielectric constant. The optimum combination of these two properties (expressed by  $\tan\delta$ ) along with the matching of the penetration depth and the sample thickness enables efficient heat generation throughout the material.

### Microwave Assisted Synthesis of TCOs

Over the past few decades, most of the microwave heating in chemistry has been limited to organic synthesis. More recently the use of microwave radiation for heating reactions in the laboratory has expanded to inorganic and materials chemistry. Reviews by Bilecka [17] and Rao [18] give an overview of microwave-assisted liquid phase routes to synthesize inorganic nanomaterials. A review by Kitchen et al [19], analyses the significant advances in the area of solid-state microwave synthesis. Hoz et al. provides an overall picture of non-thermal microwave effects on organic synthesis [20] whilst Lidström [21] delivers a critical review on microwave assisted organic synthesis and its advantages over conventional synthesis methods.

In organic and inorganic material synthesis, some or all the reactants involved in the reaction should have a high coupling

efficiency with microwaves. This provides adequate energy to overcome the activation energy barrier, allowing the reaction to proceed. The microwave absorbing properties of the reactants facilitate the rapid heating and lead to the successful synthesis of the target material within a very short period. In microwave-assisted synthesis, one often selects alternative reactants with favorable dielectric properties to achieve the maximum product yield. If there are no alternative microwave absorbing reactants available for material synthesis, it is usually possible to find a secondary material that can act as a heating source; this is commonly referred to as a microwave susceptor. Microwave susceptors are substances that have the ability to absorb microwave energy and convert it to heat. In other words, a susceptor is a material that has a high dielectric loss tangent. Graphitic or amorphous carbon, silicon carbide (SiC) and copper (II) oxide are some examples of commonly used susceptors [22,23]. These susceptor materials can be kept either in direct contact with the sample (by mixing with the other reagents) or placed outside (surrounding the sample). In some cases, adding a susceptor into a reaction mixture can cause problems with the final product purity, yield and reaction rate. Microwave susceptors can also be used in material post heat treatments to ramp up the temperature of poorly microwave absorbing materials [24]. In such cases, the penetration depth plays a vital role in the heat generation. If the penetration depth exceeds susceptor thickness, the susceptor behaves as if it is transparent to microwaves. Volumetric heating only occurs when the penetration depth is comparable to the sample thickness. If the sample is too thick, absorption is limited to the surface. The physical nature of materials, such as particle size, density and the medium the material is present in, also determines the microwave penetration depth [25].

The variation of the penetration depth of SiC with the above physical properties is shown in figure. As indicated in the Figure 2, the penetration depth of microwaves in to SiC varies with the density and the nature of the particle dispersion in the medium. This affects the microwave coupling and heat generation. Generally, SiC

is an abrasive material with a high density. The microwaves couple with SiC as a whole and penetrate well into the material to generate heat effectively because of its high density (Figure 2a). When the SiC particles are dispersed in a dielectric medium, (such as water, ethanol etc.) microwaves interact with SiC particles as well as the dielectric medium to generate heat. Since both media couple well with microwaves, the penetration depth of such a system (which is determined by the sum of the contributions from each material's dielectric properties) are reasonably high, and this leads to heat generation (Figure 2b). In the case of SiC powders the voids between the particles are filled with air, which is transparent to microwaves. Thus, heat generation is only influenced by the particle size of SiC (figure 2c). To generate volumetric heating in SiC powders the penetration depth of microwaves needs to be comparable with the particle size of the SiC (diameter of the particles). Therefore, it is not possible to heat SiC nanoparticles (<100nm) by microwaves unless they are mixed with another dielectric matrix.

Over the past decade many colloidal inorganic metal nanocrystals, metal oxides and chalcogenides have been synthesized using microwave radiation. Table 1 gives an idea about the transparent conducting metal oxides synthesized using microwave radiation.

In most of the literature it is claimed that use of microwaves in microwave-assisted synthesis shortens the reaction times and reaction temperatures significantly compared to conventional synthetic techniques [32]. According to the reports, microwave synthesized TCO materials possess significantly improved conducting properties by enhancing crystallinity and modifying the intrinsic defects. For instance, Feldmann et al. stated ITO (Sn doped  $\text{In}_2\text{O}_3$ ) and AZO (Al doped ZnO) pelleted powders, microwave synthesized in polyol media with resistivity values of  $1.1 \times 10^{-2} \Omega\cdot\text{cm}$  and  $5.1 \times 10^{-1} \Omega\cdot\text{cm}$ , respectively [26,28]. Niederberger et al. synthesized AZO by a microwave-assisted non-aqueous sol-gel route in benzyl alcohol and then processed it into a TCO thin film. They stated a minimum resistivity value of  $2.35 \times 10^{-2} \Omega\cdot\text{cm}$  for their AZO thin film after post-synthesis heat treatment [33]. Furthermore, a recent study reported a lower resistivity of  $5.6 \times 10^{-4} \Omega\cdot\text{cm}$  for the microwave synthesized AGZO (Al and Ga co-doped ZnO) powders signifying the microwave assisted technology could be a promising way forward on producing low-cost electrical conducting materials for TCO applications [24].

## Conclusions

Over the past few years there has been a significant increase in research into the synthesis and thin film fabrication of doped metal oxides for transparent conducting applications. The demand for low-cost conducting thin films has pushed research towards new TCO materials. Similarly, efforts to reduce the costs associated with TCO fabrication have driven research towards low-cost energy efficient synthesis technologies. Hence, recent research has directed towards microwave synthesis of TCO materials. Since microwave synthesis is a relatively new technology for transparent conducting materials, more research is required to understand the nature of interaction and heat generation with different TCO materials. It is hoped that significant advancements will take place in microwave assisted synthesis of TCOs because of the unique advantages which cannot be achieved by the conventional radiant heating techniques. This review demonstrates that microwave assisted synthesis is a promising alternative method to conventional synthesis as it opens up a new research strategy for the engineering of TCO materials properties for use in advanced electronic and optoelectronic devices.

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