# MICROWAVE SYNTHESIS AND SINTERING – THE GREEN PERSPECTIVE

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**Abstract:** An environmentally benign, highly efficient, novel, fast, and cheaper method with enhanced kinetics has been developed for the synthesis of stabilized zirconia ($ZrO_2$) using microwave energy and its superiority over conventional heating has been established. A kitchen multimode microwave oven was modified to work as microwave furnace. It has been observed for the first time that fully cubic calcium stabilized zirconia has been prepared of composition $Zr_{0.9}Ca_{0.1}O_{1.9}$ from precursor obtained by mixed oxide method at temperature as low as $1100^{o}C$ within 5 minutes of time-period by microwave processing. The preliminary speculation on the mechanism of microwave assisted solid state reactions is that the external electric field in the microwave method was the main cause for rapid synthesis of cubic calcium stabilized zirconia by comparing with the conventional synthesis which led to Microwave effect.

**1.1 Introduction:** Microwave synthesis and sintering of materials is a relatively new [1] and potentially important way of material processing and has opened a completely new area of research in the materials science and engineering. Microwave processing of materials is fundamentally different from conventional processing in its heating mechanism. The main advantages of microwave sintering technology over conventional methods have been said to be: 1) the material is heated internally by interaction with the microwaves resulting in uniform volumetric heating rather than heat conduction; 2) very high heating rate in short processing time are achieved which leads to low grain growth to densification ratio resulting in uniform microstructure, finer grain size and better mechanical properties; 3) heating efficiency in microwave is material dependent and thus is a function of dielectric loss of the material; 4) microwave processing, highly efficient and environmentally benign, results in saving of time and energy consumption; 5) due to fast heating rate and a novel method of heat generation, microwave processing may provide a new fast synthesis technique to synthesize better and less expensive materials. In addition, claims have been made in the literature of enhanced kinetics due to reduction in activation energy for sintering, although the micro mechanism responsible for this effect is yet to be investigated. During the processing of PZT materials the microwave has shown that the interaction is enhanced with vacancy in $TiO_2$ being used as one of the ingredient in the formation of PZT [2]. Microwave heating goes back to post-World War II era when old radar equipment was used for drying large white wares [3]. Early work on the use of microwave sintering was concentrated On dark ceramics, especially magnetic materials [1]. Extraordinarily rapid heating and high temperatures were reported by Haas [4] in urania gels. The first serious interest in microwave research formally appeared in the 1960's [5]. The International

Microwave Power Institute (IMPI) was the first society [6] (www.impi.org) developed for the purpose of further investigation of the various aspects of microwave science and technology, ranging from food processing and cooking to the processing of materials for industrial, scientific and communication applications [7]. As a result of the increased interest among scientists and engineers, many advances are being made in dielectric property measurements, modeling and processing. A better understanding of microwave- material interactions and the economics of microwave processing is evolving that will provide the basis for future industrial applications. In parallel, new equipment with variable frequencies, high-temperature capabilities, and better temperature monitoring and control systems are being designed and manufactured.

There is a few work reported in the area of microwave powder synthesis [8]. One of the necessities for successful microwave synthesis or processing is the ability of one or more of the phases to couple with the applied field, preferably at room temperature. This provides the basis for microwave material interaction that results in heat generation followed by material synthesis, even in the presence of other phases, which do not couple with the microwave effectively. If one phase couples with the microwave field, that phase will heat and react with other phases present in the system by thermal conduction and thus helps other phases to couple with the microwave field.

The most significant characteristic of microwaves is that they are not hazardous. From heat balance consideration of standard man in standard conditions, $100~Wm^{-2}$ ($10~mWcm^{-2}$) is considered to be the safe upper limit even during infinite exposure because thermoregulatory systems compensate for any power absorption. A power level of $10~Wm^{-2}$ is considered to give no heating effect even under adverse conditions of ambient temperature and humidity .

## 1.2 Experimental Procedure:

**1.2a: Fabrication Of Microwave Furnace:** A kitchen multimode microwave oven (LG MS-285SD 1200W, Frequency 2.45 GHz, 900W RF magnetron output power) with dimensions of the inner cavity as 34.4cm x 34.4cm x 22.5cm in volume was modified to work as microwave furnace as shown in Fig. I. To make this kitchen microwave oven suitable for high temperature sintering experiments, following modifications were made:

An insulating packet transparent to microwaves was made to fire the work piece to prevent the loss of radiations and to sustain the cavity temperature during firing.

An aluminum plate was placed below the sintering packet to prevent the cavity from being overheated.

A voltage system consisting of two variacs was added to the power supply of the magnetron to control the heating rate and to achieve the required temperature for desired exposure time.

A hosepipe of copper was soldered onto the outside surface (under the outer shell) of the top of the cavity for continuous water circulation to circulation to efficiently cool the magnetron and to maintain the cavity at low temperature so as to avoid excess heat transferred from the cavity to the magnetron.

A small hole was drilled in the centre of the roof just above the axis of the fabricated furnace for insertion of the thermocouple.

A digital display (Pyrotron) was connected to the S type thermocouple to measure the cavity temperature with an accuracy of $\pm 1^{o}C$ during processing.
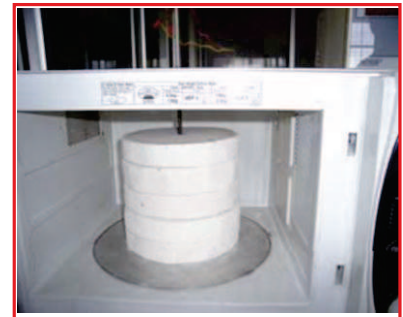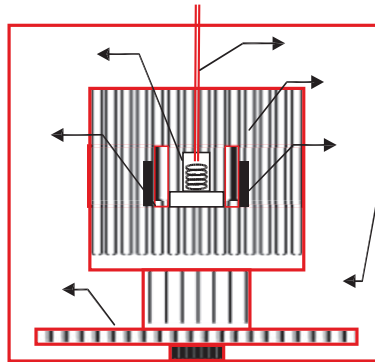
**Figure I : Microwave furnace; Figure II: Schematic display of the sintering packet; 1. Aluminium plate; 2. Zirconia cylinder; 3.Samples; Thermocouple; 5. Al-25/1700 insulator;7. SiC rods; 7. Microwave port; Figure III: Insulating packet in Microwave Furnace in microwave processing**

**1.2b: Insulating Packet:** The insulating packet was made of an alumina crucible, zirconia cylinder (Zircar Products, Inc. Florida, NY), silicon carbide (SiC) rods, and thermal insulating AL-25/1700 (Zircar Products, Inc. Florida, NY) as shown in Fig. II and III.

Zircar alumina insulation type AL-25/1700 is a combination of high purity alumina fibers, refractory fillers and inorganic ceramic binders. The typical chemical composition of the thermal insulating AL-25/1700 board is 80% $Al_2O_3$ and 20% $SiO_2$ with density 0.44 g/cc, modulus of rupture 1.38 MPa, compressive strength 0.24 MPa and thermal conductivity 0.27W/m$^{o}$K at 1650$^{o}$C. As claimed by the manufacturer, Al-25/1700 offers low thermal conductivity, high temperature stability, good hot strength, dimensional stability and excellent resistance to thermal shock and chemical attack, and is not affected by oil or water. It can withstand temperatures up to 1700$^{o}$C (3100$^{o}$F) for continuous use. AL-25/1700 is pre fired, contains no organic binders and produces no smoke or odor when heated. The zirconia cylinder vertically surrounding the samples functions both as a microwave susceptor and as an insulator. As a susceptor, it absorbs and converts microwaves to heat, thus preheats the sample if the sample does not absorb microwaves significantly at low temperatures. As an insulator, it prevents heat of the samples from dissipation to the surroundings. Also SiC rods were used as a susceptor which help in combination with the zirconia cylinder in preheating of the sample. Preheating up to 400$^{o}$C is necessary in case of zirconia sample because it shows lossy character only after this temperature.

**1.2c:Temperature Measurement In Microwave Processing:** Temperature measurement in microwave sintering/heating is one area, which is very critical, and is a matter of concern for its accuracy and reliability. The microwave sintering/ heating in ceramics usually has been reported as improving ceramic properties at relatively lower processing temperature, or at the same temperature [9-12] . Hence the precise temperature measurement is essential for the comparison of results obtained by microwave and conventional processing

methods. In a microwave field, the interference of microwave with the electromotive force of the thermocouple makes the temperature readings random or erroneous [9,13,14]. To avoid the microwave interference with the thermocouple, the thermocouple was properly shielded and grounded to get reliable temperature readings. In this study, a platinum shielded S type Pt-10%Rh/Pt thermocouple was used to measure temperature. The thermocouple was fitted in the microwave so that the tip of the thermocouple was 1mm away from the work piece in the sintering packet. The temperature of the sample was measured with S type thermocouple with an accuracy of $\pm 1^{o}C$ during processing.

**1.2d: Conventional Furnace:** A programmable furnace (JEW, New Delhi) having silicon carbide rods as heating elements was used for the present study. The programmer has the facility of 99 segments in each program. The furnace was capable of operating at high temperature up to $1400^{o}C$ with high heating rate (upto $600^{o}C$ per hour) and low thermal inertia. The temperature in the conventional sintering was measured with S type Pt-10%Rh/Pt thermocouple. The samples were placed in the alumina crucible, which was in contact with the tip of the thermocouple. The heating and cooling rates were controlled by programs with desired soaking time.

**1.2e: Microwave Processing of Precursor of Partially Stabilized Zirconia:** Multi pellet runs are conducted in both microwave and conventional sintering experiments. In microwave sintering, the pellets, usually five to six (12.80 mm in diameter and 2.50 mm in thickness) were stacked one over the other in the alumina crucible of the sintering packet. To the stacked pellets, poly vinyl alcohol (6.6%) solution was added as a susceptor. Poly vinyl alcohol is a lossy having high tangent loss at room temperature. Hence, it interacts with microwave first and gets heated up to its decomposition temperature $(300^{o}C)$. In the course of decomposition, it heats the work piece to a temperature where it starts absorbing microwaves and thus the volumetric heating of work piece is initiated. In conventional sintering, the pellets, usually six to eight in number, were placed in the alumina crucible without any susceptor. Heating rate in microwave was kept $100^{o}C$ per

minute after $400^{o}C$ whereas in conventional method it was kept as $10^{o}C$ per minute. Since the sintering temperature in both the processing techniques was identical, the difference in the properties of the sintered samples can be attributed to the microwave effect.

**1.2f: conventional Processing of Precursor of Stabilized Zirconia:** The pellets of the samples prepared (composition x = 0.02, 0.04, 0.06, 0.08, 0.10) were fired conventionally at $1400^{o}C$ for 4 Hrs. The maximum temperature of $1400^{o}C$ was achieved in 3 hours in a programmable furnace and the temperature was measured with S type Pt-10%Rh/Pt thermocouple with an accuracy of $\pm 1^{o}C$ during processing.

**1.2g: Characterization Techniques:** The formation of different phases in each composition fired at different temperatures and for different soaking time periods were analyzed by X-ray diffractometer (PHILIPS, Model: PW1710). Densities of each pellet were measured by Archimedes Principle using distilled water.

**1.3. Results and Disussion:** It has been observed for the first time that fully cubic calcium stabilized zirconia has been prepared of composition $Zr_{0.9}Ca_{0.1}O_{1.9}$ from precursor obtained by mixed oxide method at temperature as low as $1100^{o}C$ within 5 minutes of time-period by microwave processing. However fully cubic yttrium and magnesium stabilized zirconia of the same composition could not be synthesized from precursors obtained by mixed oxide method at temperature $1100^{o}C$ within 5 minutes of time-period by microwave processing [Fig. IV (a-c)]. In the YSZ system, single-phase cubic stabilized zirconia has formed by microwave processing at a temperature $1400^{o}C$ within 5 minutes of soaking time-period for the composition $Zr_{0.9}Y_{0.1}O_{1.95}$ [Fig. V(a-c)]. Fully cubic magnesium stabilized zirconia could not be prepared by microwave processing at a temperature $1400^{o}C$ even in 20 minutes of soaking time period for composition $Zr_{0.9}Mg_{0.1}O_{1.9}$ prepared by mixed oxide method [Fig. VI (a-c)] .
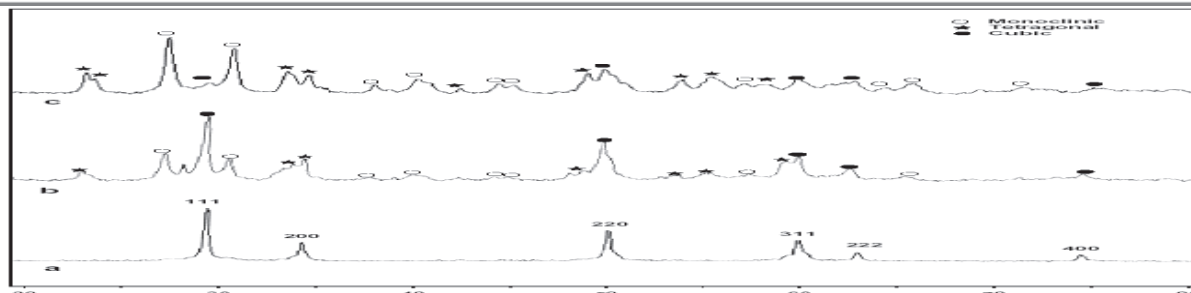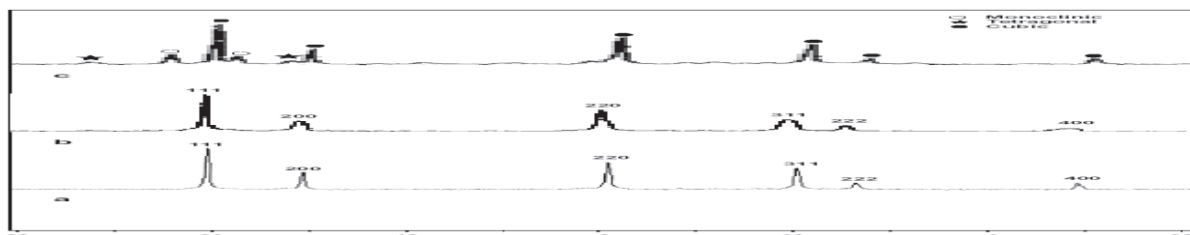
**Figure IV: XRD pattern of; a. Microwave Fired $Zr_{0.9}Ca_{0.1}O_{1.9}$ at $1100^oC$ for 5 min; b. Microwave Fired**



$Zr_{0.9}Y_{0.1}O_{1.95}$ at $1100^oC$ for 5 min; c. Microwave Fired $Zr_{0.9}Mg_{0.1}O_{1.9}$ at $1100^oC$ for 5 min

**Figure V: XRD pattern of Microwave fired; a. Microwave Fired $Zr_{0.9}Ca_{0.1}O_{1.9}$ at $1400^oC$ for 5 min; b. Microwave Fired $Zr_{0.9}Y_{0.1}O_{1.95}$ at $1400^oC$ for 5 min;c. Microwave Fired $Zr_{0.9}Mg_{0.1}O_{1.9}$at $1400^oC$ for 5 min**



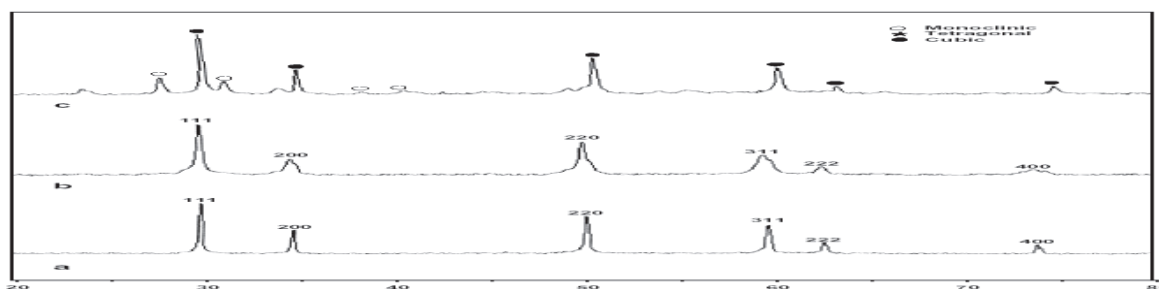**Figure VI: XRD pattern of a. Microwave Fired $Zr_{0.9}Ca_{0.1}O_{1.9}$ at $1400^oC$ for 20 min; b. Microwave Fired $Zr_{0.9}Y_{0.1}O_{1.95}$ at $1400^oC$ for 20 min; c. Microwave Fired $Zr_{0.9}Mg_{0.1}O_{1.9}$ at $1400^oC$ for 20 min:**

It was found that microwave processed pellets of CSZ, YSZ and MSZ of composition x = 0.02, 0.04, 0.06, 0.08, 0.10 fired at $1400^oC$ for 20 minutes have higher densities as compared to conventionally sintered pellets and density increases with concentration of dopant ion and was maximum corresponding to composition x = 0.10 (relative density in CSZ = 98.97% [Table I], YSZ = 99.16% [Table II], MSZ= 98.81%)[Table III]. It was also found that the density of the pellets of composition x = 0.10 increased with increase in soaking time and were maximum for the soaking time of 20 minutes in microwave firing at $1400^oC$ [Table IV, V and VI]. The density further decreased for soaking times 25 and 30 minutes in CSZ and MSZ whereas it remained constant in YSZ [Table IV, V and VI]. The density of microwave fired x = 0.10 composition of CSZ, YSZ and MSZ at different sintering temperatures indicated the density increase with sintering temperature corresponding to maximum density at $1400^oC$ for 20 minutes soaking time period [Table VII, VIII and IX]. It appears that apart from thermal diffusion which occurs in the conventional processing some non-thermal phenomenon is also occurring responsible for single phase solid solution formation and densification.

**Table I : Variation in relative density of calcium stabilized**

| Composition (x) of Ca in $Zr_{1-x}Ca_xO_{2-x}$ | Relative Density of Conventional Sintered pellets at $1400^0C$ for 4Hrs | Relative Density of Microwave Sintered pellets at $1400^0C$ for 20 min |
|---|---|---|
| 0.02 | 87.18 | 90.60 |
| 0.04 | 88.89 | 94.87 |
| 0.06 | 87.69 | 95.73 |
| 0.08 | 82.39 | 97.78 |
| 0.10 | 82.05 | 98.97 |

**Table II: Variation in relative density of yttrium stabilized zirconia with different compositions (x) $Zr_{1-x}Ca_xO_{2-x}$ with different compositions (x) zirconia $Zr_{1-x}Y_xO_{2-x/2}$**

| Composition (x) of Y in $Y_xZr_{1-x}O_{2-x/2}$ | Relative Density of Conventional Sintered samples at $1400^0C$ for 4Hrs. | Relative Density of Microwave Fired samples at $1400^0C$ for 20 Min. |
|---|---|---|
| 0.02 | 83.67 | 93.67 |
| 0.04 | 88.17 | 96.17 |
| 0.06 | 88.50 | 96.67 |
| 0.08 | 94.50 | 98.17 |
| 0.10 | 95.33 | 99.16 |

**Table III: Variation in relative density of magnesium stabilized**

| Composition (x) of Mg in $Zr_{1-x}Mg_xO_{2-x}$ | Relative Density of Conventional Sintered pellets at $1400^0C$ for 4Hrs | Relative Density of Microwave Fired pellets at $1400^0C$ for 20 min |
|---|---|---|
| 0.02 | 83.45 | 89.86 |
| 0.04 | 88.51 | 93.75 |
| 0.06 | 94.26 | 95.78 |
| 0.08 | 95.61 | 96.62 |
| 0.10 | 92.23 | 98.82 |

**Table IV:  Variation in relative density of cubic calcium zirconia $Zr_{1-x}Mg_xO_{2-x}$ with different compositions (x) stabilized zirconia $Zr_{0.9}Ca_{0.1}O_{1.9}$ Microwave fired at $1400^0C$**

| Soaking time period in min | Relative Density |
|---|---|
| 5 | 89.74 |
| 10 | 91.45 |
| 15 | 89.23 |
| 20 | 98.97 |
| 25 | 90.43 |
| 30 | 89.74 |

**Table V: Variation in relative density of cubic yttrium stabilized**

| Soaking time period inmin | Relative Density |
|---|---|
| 5 | 95.00 |
| 10 | 96.50 |
| 15 | 97.50 |
| 20 | 99.16 |
| 25 | 99.14 |
| 30 | 99.15 |

**Table VI: Variation in relative density of cubic $Zr_{0.9}Y_{0.1}O_{1.95}$**

**Microwave fired at $1400^O$C with soaking time periods magnesium zirconia stabilized zirconia $Zr_{0.9}Mg_{0.1}O_{1.9}$. Microwave fired at $1400^O$C with soaking time period**

| Soaking time period in minutes | Relative Density |
|---|---|
| 5 | 91.89 |
| 10 | 92.06 |
| 15 | 95.44 |
| 20 | 98.82 |
| 25 | 94.60 |
| 30 | 94.00 |

The microwave-material interaction depends strongly on the dielectric and magnetic properties of the material. The interactions between microwaves and matter are of many types. Among them, four important mechanisms are in solids and three of them lead to losses in microwave region: space charges arising from localized electrical conduction, rotating electric dipoles, and ionic polarization associated with far-infrared vibrations [15].The mechanism of dielectric heating in zirconia and other ionic solids is understood as a result of impurity-vacancy dipolar relaxation in the microwave frequency range. Doped zirconia contains a large concentration of lattice defects species. The defect reaction in YSZ system is expressed as Kroger-Vink notation as:$Y_2O_3 = 2Y'_{Zr} + VO^{¨} +3O^{.}$ Results obtained by different authors, for the dielectric properties of zirconia, diverge considerably. Data given by Batt et al [16] were considered here. ~ 0.04). The loss factor increases markedly to $\varepsilon"$~100 around

$1000^oC$ ($\varepsilon" Al_2O_3$ ~0.3 at the same temperature) to a level similar to that of conductive ceramics such as SiC. At room temperature the loss is entirely due to polarization. The high value of the dielectric constant $\varepsilon"$ 35±40, suggests a significant contribution from to the diffusion of the cations. Here the ionic radius of dopant cations varies as $Mg^{2+}$ (0.0189 nm) $<Y^{3+}$ (0.102 nm) $<Ca^{2+}$ (0.112 nm) [17]. From the results it is evident that higher the ionic radius of the dopant higher is the diffusion during the stabilization of zirconia. Further the polarisibility as per Fajan rules is highest of $Ca^{2+}$ among dopant cations being discussed, therefore microwave interaction is maximum in dopant $Ca^{2+}$ and its diffusion is highest in the stabilization of zirconia to form single phase cubic solid solutions. Booske etal [18] suggested the excitation of non-thermal phonons to be responsible for the mobility of crystal lattice ions. According to their model, microwave

heating involves a time harmonic perturbation of the lattice through ionic polarization. If the microwave frequency satisfies resonance with one or more normal mode oscillations, the considerable amount of coherent, oscillatory motion will be pumped into those resonant modes (the net energy distribution resulting from microwave heating will not be pure Maxwell Boltzman distribution). Therefore one of the important factors for resonance with phonon is the creation of localized excess charges in the lattice. Since vacancies can create localized excess charges in the lattice, this localized charge concentration can lead to direct microwave frequency resonance with phonons. One can make only preliminary speculations on the mechanism of microwave assisted solid state reactions. But comparing with the conventional synthesis, the external electric field in the microwave method would be the main cause for rapid synthesis of cubic calcium stabilized zirconia [19]. This can be considered as microwave effect, which is maximum in case of CSZ [Fig. VII] as compared to YSZ [Fig. VIII] and MSZ [Fig. IX] systems corresponding to same dopant concentration 10 mol % and similar processing conditions at $1400^{\circ}$C soaked for 20 minutes.
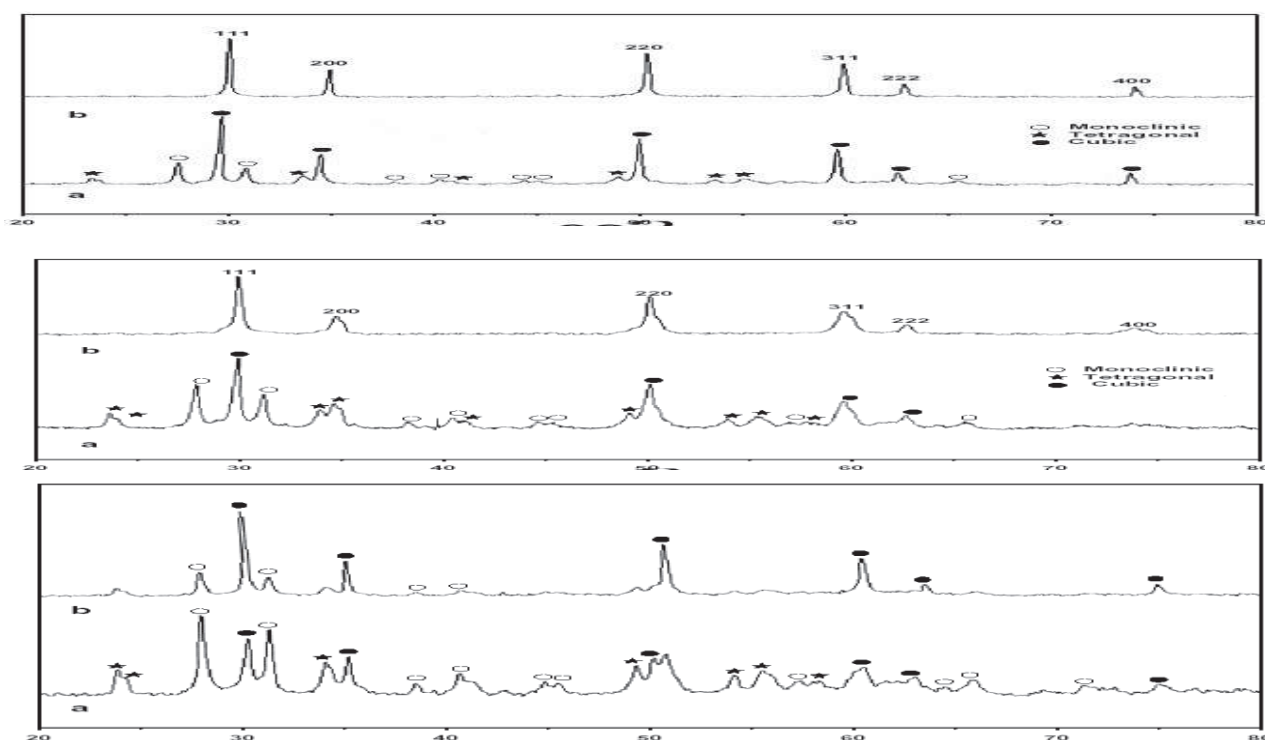


**Figure VII: XRD pattern of**     a. Conventional Fired $Zr_{0.9}Ca_{0.1}O_{1.9}$ at $1400^{\circ}$C for 4 hrs
b. Microwave Fired $Zr_{0.9}Ca_{0.1}O_{1.9}$ at $1400^{\circ}$C for 20 min
**Figure VIII: XRD pattern of**     a. Conventional Fired $Zr_{0.9}Y_{0.1}O_{1.95}$ at $1400^{\circ}$C for 4 Hrs
b. microwave Fired $Zr_{0.9}Y_{0.1}O_{1.95}$ at $1400^{\circ}$C for 20 min
**Figure IX: XRD pattern of**     a. Conventional Fired $Zr_{0.9}Mg_{0.1}O_{1.9}$ at $1400^{\circ}$C for 4 hrs
b. Microwave Fired $Zr_{0.9}Mg_{0.1}O_{1.9}$ at $1400^{\circ}$C for 20 min

**1.4: Conclusions:** A Kitchen microwave oven was modified to work as microwave furnace. Kinetic study of phase transformation in microwave processing was done and superiority of microwave processing over conventional processing has been established. Fully cubic calcium-stabilized $ZrO_2$ with composition $Ca_{0.1}Zr_{0.9}O_{1.9}$ was prepared by microwave processing at a temperature as low as $1100^{\circ}$C within 5 min. The present work has recognized the fact that microwave processing is environmentally caring, highly proficient, original, hasty, and economic method for the synthesis of stabilized zirconia ($ZrO_2$) and is superior over conventional heating.

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