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Development of microwave susceptors based on SiC composites and their application for a one-step synthesis of ZnO nanostructures

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ABSTRACT

We report on the synthesis of silicon carbide (SiC)-based composites containing different proportions of aluminum and/or vanadium III oxides. These composites have been successfully tested as susceptors into a commercial microwave oven operating at 2.45 GHz frequency. After 120 s only of microwave irradiation, the generated temperature has reached a plateau of 1750 °C, which was obtained for SiC composite containing 10 wt% of Al_2O_3 and/or V_2O_3. Furthermore, the structural properties of these composites were investigated by means of X-ray diffraction and scanning electron microscopy before and after exposure to microwaves irradiation. These SiC-based susceptors were then used as a source of heat to synthesize a nanostructured ZnO material through two different processes, namely the zinc metal evaporation/condensation occurring under air, and through a rapid thermal decomposition of zinc acetates and nitrates precursors. The structural analysis supported the possibility to grow nanostructures of controlled morphologies via the control of the microwave power and the type of precursor employed. We believe that this proposed one-step microwave assisted method provides a simple and efficient alternative to synthesize various oxide nanostructures in a very short reaction-time.

1. Introduction

Owing to numerous advantages over the conventional processing methods, the use of the microwave heating as a material synthesis and processing has attracted a growing attention and opened up a completely new area of fundamental and applied research [1–7]. Numbers of relevant reviews works have been already published on various microwave assisted processes for the synthesis of both organic and inorganic materials [8–10], where they offered the possibility to reduce the energy consumption and produce materials of enhanced physical properties [11]. Furthermore, it has been demonstrated that a large amount of oxide nanostructures of different morphologies can be successfully synthesized by thermal evaporation of metallic powders under air using SiC based susceptors as a microwave source of heat [12–15].

It's well established that the fundamentals of the absorption phenomenon occurring by solids through the microwave-matter interaction is essentially related to dielectric and/or magnetic losses [16–20]. In the ultra-high frequency domain (300 MHz to 3 GHz), the electromagnetic absorption mechanism is mainly related to the presence of $Si-C$ dipoles which follow the instantaneous change of the external alternative electric field but with a certain delay. Consequently, the resulting polarization phasor lags the applied electric field leading to a dissipation of the energy as a heat [13,14].

Recently, it was reported that SiC ceramic microwave absorption capability can be enhanced by doping the ceramic with materials of various nanostructures such as carbon nanotubes or nickel oxide nanorings [21]. It has been also demonstrated that enhanced microwave absorption capacity of SiC based composites(e.g. $SiC\text{-}Si_3N_4\text{-}SiO_2$ materials) is achieved by the modification of its effective dielectric property [22]. Furthermore, adding yttrium oxide (Y_2O_3) which does not absorb microwaves at temperature below 1000 °C to an absorbing phase of the iron oxide such as Fe_3O_4 has been reported to enhance considerably the heating rate of the composite. This enhancement effect has been assigned to the formation of a third phase which could absorb microwaves efficiently [23]. It was also suggested that "aniso-thermal" reactions could occur in the ceramics containing two or more phases that have different microwave absorbance behaviors [23,24]. In such cases, and as discussed by Peelamedu and Roy [23,24], the absorbing phase could act as a source of heat while the second phase acts as a heat sink. This dual behavior leads to a strong temperature gradient which induces in turns the matter transport and a fast reaction explaining there by the rapid sintering process observed under microwave exposure.

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Their experimental results suggest that it is possible to control the microwave absorption of ceramics through the engineering of their compositions.

We report in this work the development of microwave susceptors based on SiC composites and their application for a one-step synthesis of ZnO nanostructures. In the first part of this work, we analyzed the microwave absorption of a large series of SiC based composites containing various oxides, including Al_2O_3, and V_2O_3. We then tested the absorbing capacity of our samples to the microwave irradiation by recording their measured temperature during the exposure process. In the second section of this work, we discussed the synthesis of ZnO nanostructures using SiC based composites, where moreover, high purity ZnO nanostructures having different morphologies were found to be easily and efficiently synthesized through this one-step thermal decomposition of zinc rich precursors using our developed microwave assisted synthesis method.

2. Experimental

2.1. Preparation of SiC/oxide susceptors

Twenty (20) samples of SiC/oxides having different compositions were first prepared as displayed in Tables 1, 2. All the samples have approximately the same dimensions and the same weight (namely, 2.2 cm height, 2 cm diameter, and 13 g). Seventeen (17) samples were prepared by mixing manually inside a mortar a SiC powder (Aremco Products, INC.) of 10 µm grain-size with 10 wt% of a given oxide (Al_2O_3, V_2O_3, etc.) (see Table 1). A few samples were prepared by adding two different oxides (see Table 2). A sample made of 100% SiC was used as a reference. After mixing the powders, an appropriate amount of deionized water was added to the mixture to enhance the cohesion forces between the different grains prior to mechanically compact them into cylindrical shapes by a pressing machine. The obtained samples were then subjected to 900 °C treatment under air in a conventional furnace during one hour. The heating rate in the conventional furnace was 20 °C per minute.

A modified conventional microwave oven (General Electric, model NO. JES1142SF001, 2.45 GHz, 1.5 kW) was used for the synthesis experiments. As depicted in Fig. 1, a small hole was drilled to allow the temperature measurement by a pyrometer, and a thermal shield was also used. The temperature of the samples during their microwaves irradiation was measured with an optical pyrometer (Modline Plus™ infrared thermometer, Irconinc., USA). Three different materials of a known emissivity were used for temperature measurements: two single crystals of silicon carbide (emissivity = 0.80–0.85), nickel oxidized (0.80–0.95) and a platinum foil (emissivity =0.25–0.35). Subsequently, the temperature was also observed with a two-wavelength pyrometer (METI-MQ11) which enables the measurement of the temperature of the material independently of its emissivity. For the thermal insulation, a refractory shield made of fibrous alumina was used. Further details related to the experimental set up can be found elsewhere [13]. The thermal shield plays a critical role as it is transparent to the microwaves and ensures at the same time the heat confinement.

Table 2
Samples made of three components.

Sample no.	Component of sample	Ceramic A (SiC)	Ceramic B	Ceramic C
20	84%SiC + 8% ZrO_2 + 8%MnO_2	(84%) 10.92 g	(8%)1.04 g ZrO_2	(8%)1.04 g MnO_2
21	85%SiC + 8% ZrO_2 + 7%SiO_2	(85%) 11.059	(8%)1.04 g ZrO_2	(7%)0.91 g SiO_2

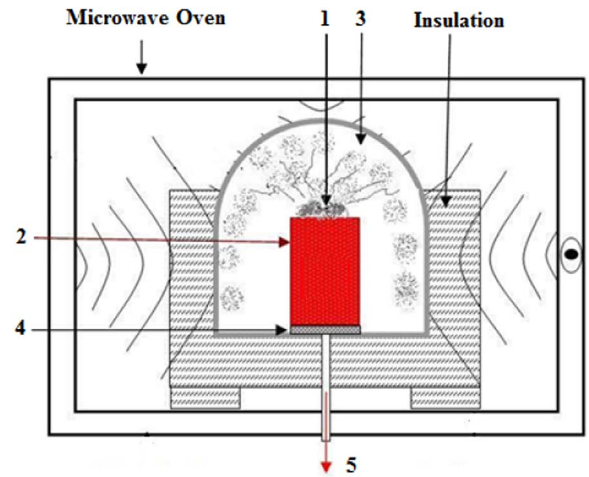

Fig. 1. A schematic diagram of the set-up used for the synthesis of ZnO nanostructures: 1. Flakes of metallic zinc. 2. A cylindrical microwave susceptor. 3. An alumina crucible. 4. A thin disc of SiC used as a source of radiation to measure the temperature by the pyrometer. 5. A small hole to allow the temperature measurement by the pyrometer.

Table 1
Samples made of two components.

Sample no.	Component of sample	Ceramic A (SiC) (AremcoProducts,INC)	Ceramic B
1	90% SiC + 10% ZrO_2	(90%)11.7 g	(10%)1.3 g ZrO_2(Zirconium oxide 99.9975%, JMC.Ltd)
2	90% SiC + 10% SiO_2	(90%)11.7 g	(10%)1.3 g SiO_2 (Silicon Dioxide 99,9%, Unaxis Materials Aktiengesells chaft Schlosswegll)
3	90% SiC + 10% Y_2O_3	(90%)11.7 g	(10%)1.3 g Y_2O_3 (Yttrium oxide, 99.99% red)
4	90% SiC + 10% Al_2O_3	(90%)11.7 g	(10%) 1.3 g Al_2O_3 (Aluminum Oxide powder 99%, Aremc Products, INC.)
5	90% SiC + 10% clay	(90%)11.7 g	(10%)1.3 g clay (thermal clay, Qaisumah, KSA)
6	90% SiC + 10% Ni_2O_3	(90%)11.7 g	(10%)1.3 g Ni_2O_3(Schwarz, Fluka-GARANTIE, Switzerland)
7	90% SiC + 10% Fe_2O_3	(90%)11.7 g	(10%)1.3 g Fe_2O_3 (Johnson Matthey, U.K)
8	90% SiC + 10% NiO	(90%)11.7 g	(10%)1.3 g NiO (Black, Fluka-GARANTIE, Switzerland)
9	90% SiC + 10% V_2O_3	(90%)11.7 g	(10%)1.3 g V_2O_3 (Vanadium *III* Oxide,99%, Aldrich Chemical Company, Inc. USA)
10	90% SiC + 10% MgO	(90%)11.7 g	(10%)1.3 g MgO (Powder, Mallinckrodt Chemical Works, U.S.A)
11	90% SiC + 10% TeO_2	(90%)11.7 g	(10%) 1.3 g TeO_2 (Tellurium dioxide, 99.995%, Addrich Chem. Co. Canada)
12	90% SiC + 10% PbO	(90%)11.7 g	(10%)1.3 g PbO (mono-yellow, Fisher Seientific Company, U.S.A)
13	90% SiC + 10% CuO	(90%)11.7 g	(10%)1.3 g CuO (Copper(II) oxide, ultrapure, Alfa Products, Morton Thiokol, Inc)
14	90% SiC + 10% TiO_2	(90%)11.7 g	(10%)1.3 g TiO_2 (Titanium dioxide, BDH Chemicals Ltd Poole, England)
15	90% SiC + 10% ZnO	(90%)11.7 g	(10%)1.3 g ZnO (Zinc oxide, dry process, Fisher Scientific Company, USA)
16	93% SiC + 7% MnO_2	(93%)12.09 g	(7%) 0.91 g MnO_2 (Manganese (IV) oxide, 99.99%, Aldrich Chem. Co.)
17	88% SiC + 12% MnO_2	(88%)11.44 g	(12%)1.56 g MnO_2 Manganese (IV) oxide, 99.99%, Aldrich Chem. Co.
18	90% SiC + 10% CaO	(90%)11.7 g	(10%)1.3 g CaO (Calcium oxide,Johnson Matthey, U.K)
19	90% SiC + 10% Co_3O_4	(90%)11.7 g	(10%)1.3 g Co_3O_4 Cobalt(II,III), Packed in Switzerland, Fluka-Garantie