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Synthesis of silicon carbide using concentrated solar energy

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Abstract

Silicon carbide (SiC) has been prepared successfully using concentrated solar energy provided by the IER-UNAM solar furnace. This has led to the development of a low CO_2 emissions process for the production of this material via carbothermic reduction of a silica/carbon (SiO_2/C) nanocomposite, which has shown a more reactive carbon for formation of composite, being more thermally stable. Silica (obtained by a sol–gel process) and sucrose were used as precursors of silicon and carbon, respectively, at a temperature of 700 °C in controlled atmosphere (nitrogen) for the formation of the SiO_2/C composite. This composite was used in a second step to obtain SiC at a temperature of 1500 °C, in argon atmosphere. The experimental setup used a Pyrex® glass spherical vessel designed to work with concentrated solar power and controlled atmospheres. The structure and morphology of the solar obtained SiC were analyzed with FTIR, XRD, TGA/DSC, SEM and TEM techniques. Results show that it is feasible to use concentrated solar energy for the synthesis of SiC. The solar SiC obtained is nanostructured and is mainly β-SiC.

Keywords: Concentrated solar energy; Solar furnace; Silicon carbide; SiO_2/C composite; Solar SiC

1. Introduction

The use of concentrated solar energy provided by solar furnaces has many applications in the processing of materials, since with this type of systems it is possible to reach high temperatures (around 3000 °C) in a few seconds. Therefore, solar furnaces can be used for: thermal treatment of metals, producing ceramic materials, synthesis of nanomaterials, sintering and degradation of compounds, among other processes. A reported example of these applications has been the synthesis of fullerenes (Chibante et al., 1993; Fields et al., 1993; Laplaze et al., 1996a,b; Flamant et al., 2004). In addition, synthesis of some carbides have been achieved, such as tungsten carbide (Shohoji et al., 1999; Almeida Costa Oliveira et al., 2007a, 2007b), molybdenum carbide (Guerra Rosa et al., 1999), carbides of titanium and zirconia (Cruz Fernandes et al., 1999), calcium carbide (Paizullakhanov and Faiziev, 2006) as well as the vanadium-group metals carbides: V, Nb and Ta (Amaral et al., 2000), VIa-group metals carbides (Shohoji et al., 2000) and *d*-group transition metals carbides excluding hafnium (Rodríguez et al., 2001). In the case of silicon carbide, there are few experimental tests

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carried out in solar furnaces, some of them are: its synthesis through a mixture of powders of silicon and amorphous carbon (Cruz Fernandes et al., 1998; Cañadas et al., 2004) or a mixture of quartzite and coke (Gulamova et al., 2009), performing their analysis of the obtained SiC only through XRD. Also, the SiC synthesis from carbon nano-onions, graphite and multi-walled carbon nanotubes to form SiC nanowires (Lu et al., 2013).

SiC ceramics have been a focus of several research due to their high values of absorptance (Han et al., 2014), specific surface area, porosity, hardness, melting point (Krawiec and Kaskel, 2006), thermal conductivity, resistance to oxidation and mechanical strength, as well as excellent chemical stability at high temperatures (Ji et al., 2011; Najafi et al., 2011; Zhao et al., 2011; Guo et al., 2012, 2014; Saeedifar et al., 2013; Rajarao et al., 2014) and hostile atmospheres (Dey et al., 2011; Wu et al., 2013). SiC have several applications, such as catalytic supports, electronic and photoelectric devices, reinforcement in composites and materials for high temperature applications (Eom et al., 2013; Kong et al., 2013) for example, in volumetric solar receivers (Fend et al., 2004; Agrafiotis et al., 2007; Fend, 2010; Mey et al., 2014).

There are several conventional methods for the synthesis of SiC (Gerhardt, 2011). The traditional process for SiC manufacturing was invented by E.G. Acheson (Krstic, 1992) in 1892, in his method he used silica and coke at high temperature (2000 °C). Most widely used synthesis processes are carbothermic reduction methods, which involve lower temperatures than the traditional methods (1100–1800 °C) at different times of synthesis (Jin and Guo, 2003; Yao et al., 2007; Dhage et al., 2009; Moshtaghioun et al., 2011; Babić et al., 2012; Kong et al., 2013; Xingzhong et al., 2012; Guo et al., 2010), using different materials as precursors, for example: silicon powder, silica sol or tetraethylorthosilicate (TEOS) as a source of silicon; and graphite, phenolic resin, resorcinol–formaldehyde, bamboo or sucrose as precursor of carbon, among others.

In the present work, a solar furnace was used to provide the heat of the reaction. This appears as an alternative in the development of processes for the synthesis of materials that aims to promote unconventional energy sources with low CO_2 emissions. In this case, silicon carbide was synthesized by carbothermic reduction method using as precursors silica (obtained by the sol–gel method) and sucrose. The nanocomposite and SiC were synthesized using a Pyrex® glass spherical vessel (CIEViP by its acronym in Spanish) in a controlled atmosphere.

2. Experimental setup

2.1. Materials

In the silica synthesis (as silicon precursor) tetraethylorthosilicate (TEOS, 99.98%), absolute alcohol and HNO_3 5 wt% were used. For SiC synthesis, the following materials were used: sucrose (99.5%) as carbon precursor, sulfuric acid (H_2SO_4) 65 wt%, hydrofluoric acid (HF) 10% and distilled water.

2.2. Experimental procedure

The experimental procedure consisted of five stages as show in Fig. 1a, where the last two were carried out in the solar furnace. The first step was the synthesis of silica through the sol–gel process. This silica is the silicon precursor in the SiC synthesis. In this step TEOS–ethanol (1:3) and HNO_3, as a catalyst, were mixed and agitated. Subsequently the mixture was dried at room temperature, obtaining the SiO_2. The second stage consisted of mixing the synthetized silica with sucrose, H_2SO_4 and distilled water, in an alumina crucible forming a liquid mixture; in the third step this mixture was dried at 100 °C for six hours, raising the temperature to 160 °C for another 6 h; finally the fourth and fifth stage were the synthesis of SiO_2/C nanocomposite and SiC, respectively. In these last two steps, the heat for the reaction was provided by using concentrated solar energy from the IER-UNAM solar furnace (HoSIER by its acronym in Spanish), located in the city of Temixco, Morelos, Mexico. The HoSIER is a solar furnace of high radiative flux which is part of the "National Laboratory of Solar Concentrating Systems and Solar Chemistry" (LACyQS by its acronym in Spanish) located at the Institute of Renewable Energies (Instituto de Energias Renovables, in Spanish) of the National University of Mexico (Universidad Nacional Autónoma de México in Spanish). This solar furnace has a power of 25 kW with peak concentrations of 18,000 suns (1 sun = 1 kW/m^2) in the focal zone (3.68 m), producing a solar image of 8 cm-diameter (Riveros-Rosas et al., 2010). The main components of the HoSIER are three: a concentrator of 409 hexagonal mirrors of first surface (each mirror has a diameter of 40 cm); a heliostat of 81 m^2 surface area and a shutter of 42.2 m^2, which is located between the heliostat and the concentrator. This shutter regulates the amount of radiative energy reaching the focal zone, and thus can be used to control the temperature of the sample (Estrada et al., 2011; Pérez-Enciso et al., 2015).

The crucibles and sample holders (high purity alumina and graphite) used in the solar experimental tests were introduced in the CIEViP, which consists of a Pyrex® glass vessel of 25 l capacity, where experiments at high temperature in a controlled atmosphere can be conducted (Fig. 1b). Nitrogen and argon gases (99.95% purity) were introduced through the lower part of the CIEViP to provide an inert atmosphere. Nitrogen was used for the synthesis of SiO_2/C and argon for the SiC synthesis. A vacuum pump was connected at the CIEViP to ensure continuous gas flow and to control the pressure inside the chamber. The pressure was monitored with a MKS DualTrans vacuum transducer Mod. 910 (measuring range from 1×10^{-5} to 1500 Torr). For the low temperature synthesis (SiO_2/C synthesis), the temperature in the sample was measured with two type "K" thermocouples. For the case of the high