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Solar preparation of SiO_x ($x \approx 1$) nanopowders from silicon vaporisation on a ZrO_2 pellet. XPS and photoluminescence characterisation

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Abstract

SiO_x nanoparticles were prepared by vaporisation and condensation of melted silicon droplets put on zirconia pellets in a solar reactor at the focus of a 2 kW solar furnace. The size of the grains were nanometric, generally included in the range 20–40 nm, and the O/Si atomic ratio values were close to stoichiometry (O/Si $\approx 1 \pm 0.2$). XPS, DRX and TEM analyses show that these nanoparticles are amorphous with various silicon chemical environments which can be described as constituted with polysubstituted Si-($O_{4-n}Si_n$) tetrahedral configurations. The estimated oxygen atomic concentrations for these nanoparticles was in good agreement with thermodynamic equilibrium calculations for the system ZrO_2–Si at high temperature. The predominant gaseous species is the SiO molecule. The SiO_x nanoparticles present photoluminescence property similar to those currently reported for electrolytic porous silicon.
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Keywords: Silicon; Nanomaterials; Solar process; XPS

1. Introduction

The photoluminescence of porous silicon when activated by external ultraviolet sources has been observed over the entire visible spectrum from red to blue (Tamir and Berger, 1996; Ma et al., 1997; Koch and Petrova-Koch, 1996). It is currently attributed to the quantum confinement effect in the presence of an enlarged gap. This property is observed for bulk silicon too, but with considerably lower conversion efficiency. Porous silicon is considered as a promising material for photonic and electro-optical applications (Parkhutik, 1999; Bisi et al., 2000). Now, intensive research efforts have been invested for this type of materials including the ones for biological applications (Parkhutik, 1999; Desai et al., 2000). Various fabrication processes of silicon based photoluminescent nanoparticles have been proposed (Zhang et al., 1998; Kim et al., 1997; Terekhov et al., 2001; Guyot et al., 1997; Rinnert, 1999; Knápek et al., 1998). The present paper deals with a new way to create silicon based nanoparticles by using the evaporation and

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Nomenclature

T_m	temperature of the liquid (K)	E_B	binding energy (eV)
λ	thermal conductivity ($W m^{-1} K^{-1}$)	P_X	partial pressure of the species X (atm)
E_K	kinetic energy (eV)		

condensation of melted silicon pieces at the focus of a 2 kW solar furnace. Some benefits were expected from the use of such a solar preparation method against conventional techniques. First, the implementation of the solar route is simple and probably faster than those methods using electrochemical etching of silicon wafers or CVD, PECVD and ICVD systems. Secondly, the vaporisation–condensation method avoids to use toxic or dangerous chemical products, and the substitution of concentrated solar energy to other energy sources for high temperature preparation of nanoparticles seems to be a clean alternative proposal. The aim of this work was not to prepare pure silicon nanoparticles, but to obtain condensed SiO_x nanopowders and to characterise them with respect to the structure, the chemical composition and possible photoluminescence properties. XRD, electron diffraction, TEM, XPS and photoluminescence measurement were performed on the condensed nanopowders. Preliminary experiments have shown that the rate of vaporisation was very low when the silicon pieces were set down on a water-cooled metallic plate. Therefore a zirconia plate was inserted between the evaporated silicon piece and the cooled metallic plate in order to reduce the heat transfer from the silicon sample to the metallic plate. By this way, the temperature of the surface of the silicon sample could be increased, leading to better efficiency for the vaporisation process. Thermodynamic calculations were performed in order to estimate the consequences of possible interactions between silicon and the ZrO_2 insulator at high temperature. The equilibrium partial pressures of the gaseous species as a function of temperature were calculated taking into account of the presence of the ZrO_2 plate.

2. Experimental procedure

2.1. SiO_x nanoparticles preparation: the solar reactor

SiO_x nanopowders were prepared using a solar reactor depicted in Fig. 1. It is made of a Pyrex balloon ② which can be put at the focus of a 2 kW solar furnace previously described (Coutures et al., 1973). A little silicon powder mass ⑥, between 0.5 and 1 g, is placed either on a water cooled metallic plate ①, or on an insulating material ⑦ which is inserted between the silicon powder sample and the water-cooled plate. In fact, the conductive heat transfer from the silicon target to the refrigerated metallic plate resulted in too low temperature of the heated silicon piece for the vaporisation efficiency.

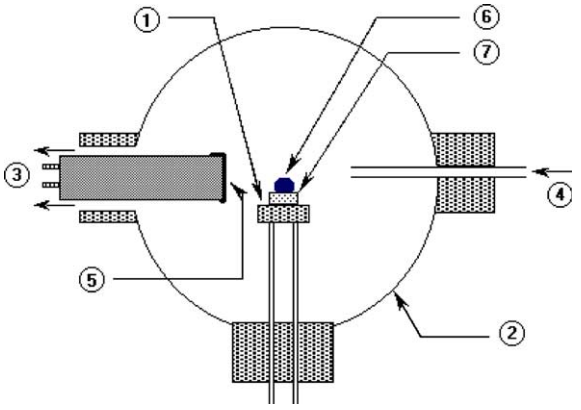

Fig. 1. Schematic diagram of the reactor for the production of SiO_x nanopowders.

The presence of the insulating material is essential to reach enough vaporisation flux of gaseous silicon species resulting in the formation of condensed SiO_x powders. Preliminary experiments made by focusing the solar radiation on the silicon powder just placed on the metallic plate resulted in producing very low amounts of condensed powders, even with maximum solar energy.

Moreover, the SiO_x condensed powders, obtained when the silicon target was just placed on the refrigerated plate, were highly oxidised in spite of the very low oxygen partial pressure P_{O_2} into the Pyrex balloon ($P_{O_2} \approx 10^{-11}$ atm, not tacking into account of possible microleaks). This high oxidation state of silicon, with an average O/Si atomic ratio = 1.7 was presumably due to the very low vaporisation flux of gaseous silicon species which was favourable to their oxidation.

Condensed SiO_x powders resulting from the vaporisation of silicon powders samples and condensation of the gaseous silicon species are collected on a cool copper foil ⑤ held on a water-cooled copper tube located at the beginning of a gas outlet ③ including a submicronic powder filter and a primary vacuum pump. Evaporation–condensation experiments were performed under an hydrogen pressure fixed in the range: 7–1013 mbar in the reactor with an H_2 flow rate in the range 1–15 $l min^{-1}$. H_2 was injected into the balloon through a gas inlet ④ including a gas mass flow meter. A Pirani gauge was used to measure the pressure into the glass balloon. The pres-

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