ELSEVIER

Materials Science and Engineering B 137 (2007) 225–231

MATERIALS SCIENCE & ENGINEERING B

www.elsevier.com/locate/mseb

Preparation of a thermal insulating material using electrophoretic deposition of silica particles

R. Tomasi, D. Sireude, R. Marchand, Y. Scudeller*, P. Guillemet

Ecole Polytechnique de l'Université de Nantes, LGMPA, Rue C. Pauc, la Chantrerie, BP 50609, 44306 Nantes cedex 3, France

Received 23 January 2006; accepted 21 November 2006

Abstract

The electrophoretic deposition (EPD) of silica nanoparticles was performed to prepare consolidated thermal insulating material. Optimal conditions were found considering the loading contents, the suspension medium, and the electric field intensity. The density and thermal conductivity were changed by applying a mechanical pressure before drying of the gel. Density ranged from 120 to 500 kg m^{-3} with an average porosity comprised between 0.78 and 0.95. According to the porosity, the average pores size of compressed materials was between 7 and 20 nm. Such materials exhibit a thermal conductivity as low as 0.020 W m^{-1} K^{-1} at room temperature and 1 atm pressure. Thermal conductivity ranged from 0.020 to 0.045 W m^{-1} K^{-1}. Minimal conductivity exists for a certain distance between particles. Boundary resistance between adjacent particles was obtained as function of the porosity and its change interpreted as ones of conductive paths though air molecules and across direct solid–solid exchange between particles.

Keywords: Thermal properties; Insulating material; Electrophoretic deposition; Thermal conductivity; Solid–solid interfaces

1. Introduction

Nanoporous materials can provide higher thermal performance than many conventional insulating materials over a wide temperature range (from cryogenic to high temperature). Particles agglomerates are used in a large series of applications where suitable thermal performance is required. Efficient thermal insulation can be achieved by using agglomerates of ultra-fine particles, as homogeneous as possible, with small pores and suitable inter particle space size. The thermal conductivity can be reduced by decreasing inter particle space size and creating poor solid–solid contacts. Thermal conductivity of agglomerates strongly depends on their textures as well the morphology of the particles [10].

Among suitable materials, one can mention the organic and inorganic aerogels which form ultra-fine solid matrices with open cell morphology produced by sol–gel processes and dried by supercritical extraction. Monolithic and granular silica aerogels exhibit a thermal conductivity as low as 0.016 W m^{-1} K^{-1} at 300 K and 1 atm pressure (see for example [6,8,11]). The thermal conductivity of these materials is lower than ones of air at the same temperature and pressure.[1] Agglomerates of solid primary particles were also envisaged to form evacuated insulating panels with interesting thermal performance. Thermal conductivity of silica powder can reach 0.002 W m^{-1} K^{-1} under vacuum and 0.027 W m^{-1} K^{-1} at room temperature and 1 atm pressure [7].

The change in thermal conductivity versus the density, and porosity, was observed theoretically and experimentally for aerogels. One can mention the case of resorcinol-formaldehyde aerogels, as reported in Ref. [6], which offered a minimum in thermal conductivity when the density was changed from 50 and 300 kg m^{-3}. The minimum of conductivity was obtained with a density of 150 kg m^{-3}.

Electrophoretic deposition can offer an interesting route to agglomerate small size particles with desirable porosity. Electrophoretic deposition (EPD) is the process by which charged particles in a suspension medium move under an applied field to an oppositely charged electrode and coagulate to form a stable deposit [1]. EPD has been widely applied to fabricate matrices, fibres and coating [2,3,4].

* Corresponding author. Tel.: +33 2 40 68 31 21; fax: +33 2 40 68 31 99.
E-mail address: yves.scudeller@polytech.univ-nantes.fr (Y. Scudeller).

[1] Thermal conductivity of air in free space is 0.026 W m^{-1} K^{-1} at 300 K and 1 atm pressure.

0921-5107/$ – see front matter © 2006 Elsevier B.V. All rights reserved.
doi:10.1016/j.mseb.2006.11.027

This paper presents the use of electrophoretic deposition to fabricate different density fumed silica particle agglomerates with high thermal performance. Thermal conductivity was investigated after applying a mechanical pressure and drying the as prepared silica gel. This study aims to explain the effect of structural features on thermal properties of such materials.

2. Experimental procedure

2.1. Material

Ultra-fine fumed silica powder was used (AEROSIL 380). Particle size distribution was comprised between 5 and 20 nm. Average primary particle size was 7 nm in accordance with the BET specific surface area 380 $m^2\,g^{-1}$. Solid phase of the particle was amorphous. The tapped density of the powder was 50 $kg\,m^{-3}$.

2.2. Electrophoretic deposition

The suspension was prepared with a suitable weight of fused silica loading. The mixture was homogenised by magnetic agitation and ultrasound. Non aqueous suspension media were chosen to avoid any formation of macro-pores in deposits which usually occurs in electrophoretic deposition with aqueous suspensions [5]. In the present study, ethanol and acetone were used as suspension media.

The experimental apparatus, shown in Fig. 1, was composed of the electrophoretic deposition cell/1/placed under a magnetic stirrer/2/. Adjustable DC voltage ranging from 0 to 350 V was used as power source. A voltmeter and a current probe were incorporated in the EPD circuit. The EPD cell was formed of a beaker containing the suspension and two emerged titanium electrodes 2 cm apart from one another. This distance was keep constant during the deposition. The dimension electrode was of 60 mm × 60 mm and 1 mm in thickness. The electrode surfaces

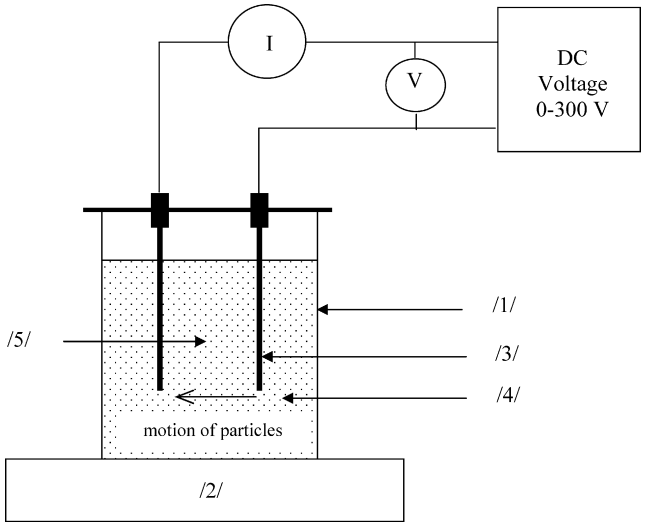

Fig. 1. Schematic diagram of the experimental apparatus using the electrophoretic deposition (EDP) cell: /1/ EDP cell, /2/ magnetic stirring, /3/ titanium electrodes, /4/ non aqueous suspension medium and /5/ charged silica particles.

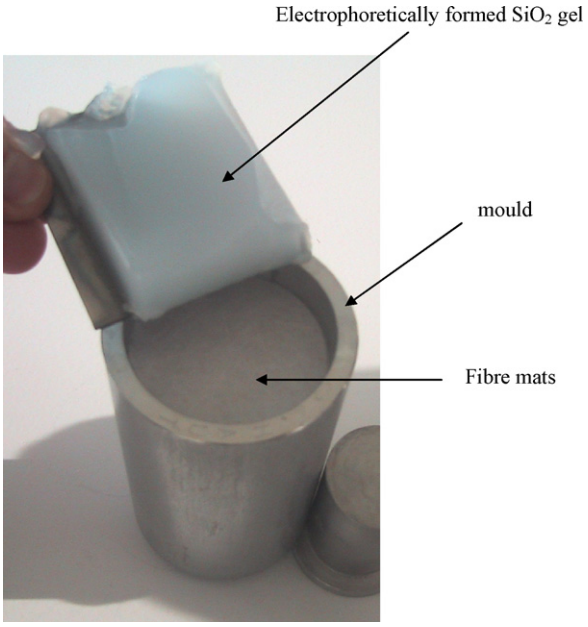

Fig. 2. Electrophoretically formed SiO_2 gel using a constant electric field $E = 150\,V\,cm^{-1}$ after a deposition time of 12 h.

were smooth to receive a silica deposit and remove it easily after the deposition. Various suitable working voltages were used. In practice, the electric field was comprised between 15 and 150 $V\,cm^{-1}$. Silica particles exhibit a negative charge with a pH higher than 2–3. Under suitable electric field, the motion of particles was made from the positive to the negative electrode.

The deposition time usually ranged from 12 to 24 h, depending on the silica loading and working electric field. After this time, one can mention that the suspension was clear. All the silica content of the suspension was recovered between the electrodes. After deposition, silica gel forming a green body was exposed to air at room temperature in order to extract the greater part of solvent (Fig. 2). The thickness of the gel body was defined by the distance of 20 mm between the electrodes. Samples of gel were taken and then placed into a cylindrical mold of 50 mm in diameter. Then, three layers of fibre mats, of 15 μm each, were incorporated at the bottom and top surfaces of the sample in order to consolidate the final material. After exposure to air for a couple of minutes, the sample was uniaxially compressed inside the mold to a given loading pressure with a suitable rate. Cold-pressing on wet surfaces was performed by using an automatic pressure system.

Under these conditions, the sample can be densified and consolidated at room temperature. Finally, after compression the sample was placed into a drying oven at 130 °C to eliminate the residual solvent. After these fabrication steps, the sample could be easily handled and cut up. The thickness of the sample was between 2 and 5 mm according to the loading pressure used and the amount of formed silica gel (see Fig. 3).

2.3. Structural characterization

Structure was investigated by using the following methods: