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Alumina coatings on silica powders by Fluidized Bed Chemical Vapor Deposition from aluminium acetylacetonate

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HIGHLIGHTS

▶ The Fluidized Bed Chemical Vapor Deposition process has been studied to coat SiO_2 powder by alumina.
▶ A metal organic precursor, aluminium acetylacetonate $Al(acac)_3$ has been used as single source.
▶ Between 400 and 500 °C, the deposits are formed of Al_2O_3, non-decomposed $Al(acac)_3$ and impurities.
▶ At 600 and 620 °C, the deposits are mainly formed of Al_2O_3 and acetylacetone $C_5H_8O_2$.

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ABSTRACT

A SiO_2 powder has been coated by alumina using the Fluidized Bed Chemical Vapor Deposition process and a metal organic precursor, aluminium acetylacetonate ($Al(acac)_3$ or $C_{15}H_{21}AlO_6$) as single source. A range of low temperatures, i.e. 400–620 °C has been explored at atmospheric pressure. Systematic characterizations were performed by Field Emission Gun Scanning Electron Microscopy (FEG-SEM) coupled with Energy Dispersive X-ray Spectroscopy (EDS), Fourier Transform Infra-Red (FT-IR) spectroscopy and Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). The process involves a first step of gas phase reactions producing reactive intermediates, themselves leading to Al_2O_3 and carbon containing deposits. Between 400 and 500 °C, the deposits are lamellar and constituted of mixtures of Al_2O_3, non-decomposed $Al(acac)_3$ and impurities, leading to a C/Al molar ratio close to 2. For this range of temperature, the precursor is not totally decomposed and the limiting parameter of the process is the deposition temperature. For 600 and 620 °C, the deposits are nodular and mainly formed of Al_2O_3 and acetylacetone $C_5H_8O_2$, one of the main intermediate species formed in the gas phase. The $Al(acac)_3$ decomposition seems to be complete, but a deposition temperature of 620 °C is not high enough to allow a complete decomposition of carbon ligands of the chemisorbing intermediate species. For these conditions, the C/Al molar ratio increases with the deposition temperature, to reach values between 4 and 6, in agreement with the observed darker colors of the deposits.

1. Introduction

The control of surface properties of powders by an appropriate coating is of main concern to enhance their performances for many applications [1]. One of the most efficient technologies to coat the outer surface of powders is the Fluidized Bed Chemical Vapor Deposition (FBCVD) process. CVD offers several advantages compared to wet preparation routes, in particular the absence of solvent and of additional steps of calcination or separation [2]. The Fluidized Bed (FB) is well suited for powders coating due to intense solid mixing, excellent heat and mass transfers and uniform temperatures [3–5]. To illustrate its large range of applications, the FBCVD process has been used to prepare supported catalysts [2], to produce photovoltaic materials [4], to improve surface properties of powders towards wear, oxidation and temperature resistance [5], to coat nuclear fuel particles and to produce multi- or single-walled carbon nanotubes [6].

Alumina coatings have attracted much interest this last decade, due to their interesting physical and chemical properties [7]. Alumina is a relatively hard material, chemically inert and stable at high temperature, which makes it interesting as diffusion barriers [8], or hard coatings [9]. CVD is still today the economically most favorable technique for producing high-quality alumina coatings [10]. Various precursors have been tested including halides or metal organic chemicals like trimethyl aluminium, aluminium

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