



# Effects of reducing the reactor diameter on the fluidization of a very dense powder

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## ABSTRACT

The impact of decreasing the column diameter from 5 to 2 cm has been studied on the fluidization of a very dense powder both at ambient and high temperature. The final objective was to coat very dense nuclear fuel particles with silicon using a Fluidized Bed Chemical Vapor Deposition (FB-CVD) process with bed weights as low as possible. A surrogate tungsten powder, 19300 kg/m<sup>3</sup> in density, was used. Wall effects only appeared in the column with a 2 cm diameter. This was evidenced by an increase in the hysteretic behavior of the pressure drop curves and an increase of the minimum fluidization velocity, as well as by a decrease in the bed voidage. This critical diameter is higher than those found elsewhere for Geldart's group A and B powders, which is probably due to the very high density of the tungsten powder that increases the friction forces in the bed. Satisfactory thermal conditions were reached at 650 °C in a reactor with a 3 cm diameter using only 740 g of powder, thereby opening the way for future silicon coating experiments.

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## 1. Introduction

New nuclear fuels with limited enrichment in <sup>235</sup>U are under development for research reactors. Pulverulent U(Mo) metallic fuels dispersed in an aluminum-based matrix are among the most promising materials today. The coating of U(Mo) powder by a barrier material is also being considered to limit interfacial interactions between the fuel and its matrix under irradiation [1]. Silicon coatings formed by Fluidized Bed Chemical Vapor Deposition (FB-CVD) from silane (SiH<sub>4</sub>) is being investigated for the barrier [2].

For economic reasons, the FB-CVD process must be developed to treat fuel powder weights that are as low as possible. The fuel powder in question has a very high density (about 17500 kg/m<sup>3</sup>) which is close to that of tungsten (19300 kg/m<sup>3</sup>) [3]. This explains why a tungsten powder with mean diameter similar to that of the fuel powder has been used as a surrogate powder in previous studies [4]. Some preliminary experiments have demonstrated that the FB-CVD technology can uniformly coat 1500 g of tungsten powder with silicon in a reactor with a diameter of 3.8 cm.

The aim of this study is therefore to greatly decrease this weight while maintaining convenient fluidization and thermal conditions to obtain uniform coating characteristics in terms of thickness, chemical composition and morphology. Preliminary experiments have shown that a simple decrease in the bed weight is ineffective because the target

deposition temperature cannot be reached due to a too low heat exchange between the reactor walls and the particles. This means that a reduction in the reactor diameter must be considered.

In the literature, the impact of reducing the reactor diameter on gas-solid fluidized bed hydrodynamics has only been studied for powders belonging to Geldart's groups A and B [5], which are easily fluidizable [6–13]. To the best of our knowledge, all studies have been performed at ambient temperature. The early experimental study by Werther [13] has shown that the bed diameter influences bubble development and the distribution of the fluidizing gas between the dense phase and the bubble phase when the bed diameter is smaller than 500 mm. More recently, fluidized beds with inner diameters ranging between 0.7 and 32 mm – called micro-fluidized beds – have been tested for fixed bed heights with reactor diameter ratios ( $H_0/D$ ) between 0.6 and 7 [6–12]. Below a critical column diameter, there is evidence of changes in the fluidized bed hydrodynamics, known as wall effects. The most significant wall effects are an increase in the minimum fluidization velocity  $U_{mf}$ , an increase in the minimum bubbling velocity  $U_{mb}$ , and a modification in the bed voidage. Some articles also report a deviation in the experimental pressure drops from the theoretical ones calculated by the Ergun equation, the presence of hysteresis between the ascending and descending pressure drop curves, and overpressure near the minimum fluidization zone [8,12]. The authors found that wall effects were significant for critical column diameters between 5 and 12 mm depending on the conditions tested. Wang et al. [11] have also shown by CFD simulations that the onset of turbulent fluidization is advanced significantly in micro fluidized beds. Wang and Fan [12] have established

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a flow regime map from experimental results obtained in column diameters starting from 0.7 mm, thus confirming this trend.

In a fluidized bed, electrostatic charges can be generated through frictional charging when particles come into contact with other particles or with the column wall. Some significant consequences of charge generation are particle agglomeration and particle build-up on the reactor's inner wall [14]. The existence of electrostatic charges between the reactor walls and the particles (depending on the nature of the wall and of particles) can increase wall effects, especially in micro 2D columns [7]. For Liu et al. [9], the  $H_0/D$  ratio has no influence, whereas this ratio increases wall effects for other authors [8,10]. Delebarre [15] and Roche et al. [16] showed that the interaction forces between particles and walls increase with the particle diameter or density. The non-sphericity of particles is also known to increase solid frictions [17]. Liu et al. [18] showed that non-spherical particles can intensify the hysteresis phenomenon between pressure drop curves at ascending and descending gas velocities.

This study analyzes the impact of reducing the column diameter on the fluidization of a very dense tungsten powder under pure argon, first at ambient temperature and then at 650 °C. The aim is to reduce the weight of the surrogate tungsten powder to below 1500 g before reducing the weight of U(Mo) treated by FB-CVD, while maintaining convenient hydrodynamic and thermal conditions in order to produce uniform silicon coatings.

## 2. Experimental

### 2.1. Experimental setup and materials

In our experiment, we used glass and steel columns that were 1 m high but with different inner diameters: 5, 3.2 and 2 cm for the glass columns and 5, 3 and 2 cm for the steel columns. A photograph of the 2 cm glass column and a schematic diagram of the steel fluidized bed system are given in Fig. 1a and b. All were equipped with a similar Inox™ porous plate distributor. A differential fast response pressure sensor (GE Druck LPX) with taps under the distributor and on top of the column was used to measure the total pressure drop across the bed. Its accuracy was  $\pm 0.5\%$  full scale.

The steel columns were also equipped with a two-zone electrical furnace to simulate FB-CVD conditions with a target bed temperature of 650 °C on average. Five thermocouples were placed into a vertical 6 mm-diameter stainless steel tube inside the reactor to measure the bed temperature within the fluidized bed with an uncertainty estimated at  $\pm 2.5$  °C. They were respectively located at 1, 2.5, 5, 7 and 12 cm above the distributor.

All measurements were registered online with a Dasy Lab® acquisition system using an acquisition frequency of 0.1 Hz and a data resolution of  $10^{-6}$ . The fluidization gas was argon (Alpha 1, Air Liquide). Its flow rate was controlled by a mass flow controller (Aera FC-7710 CO, 0–20 slm). All experiments were conducted at atmospheric pressure.

We used a tungsten powder (CERAC, Inc. T-1220) supplied by Neyco. Its grain density was equal to 19300 kg/m<sup>3</sup>. Laser grain-size analyses were performed with a Malvern Master Sizer Sirocco 2000 in dry mode. The particle size distribution of the tungsten powder is given in Fig. 2a. The Sauter diameter ( $D_{3,2}$ ) was 70  $\mu\text{m}$  and the  $D_{10}/D_{90}$  diameters were equal to 50/105  $\mu\text{m}$  respectively. Fig. 2b shows a SEM photograph of the particles (Philips XL30 FEG) which are clearly non-spherical and faceted.

### 2.2. Experimental procedure

The fluidization hydrodynamics was first studied by plotting the bed pressure drop versus increasing and decreasing superficial gas velocity in the glass and steel columns at room temperature. Experiments were repeated three times. The distributor pressure drop was first measured within the appropriate range of superficial gas velocities in

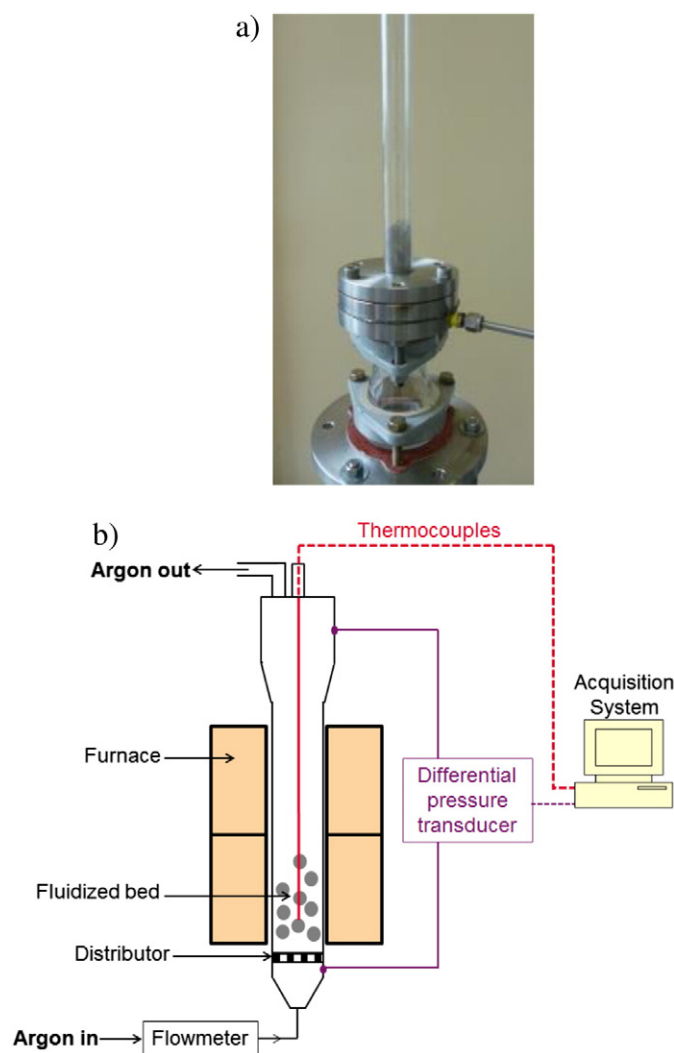


Fig. 1. (a) Photograph of the 2 cm glass column – (b) schematic diagram of the steel fluidized bed system.

each empty column and then removed from the experimental pressure drop. Bed pressure drops were averaged over 10 s periods when significant fluctuations occurred. The average expanded bed height was measured visually in the glass columns at decreasing gas velocity. A dimensionless bed expansion  $H^*$  was calculated by dividing the experimental expanded bed height by the fixed bed height. A minimum bubbling velocity,  $U_{mb}$ , was estimated by visually catching the first bubbles that appeared on the surface of the bed [9]. For each superficial gas velocity, the experiments were run 60 s to measure stationary fluidization data. The minimum fluidization velocity  $U_{mf}$  at room temperature was then deduced either from the pressure drop curves using the Davidson and Harrison method [19], or from the bed expansion results [8].

Experiments were performed using two  $H_0/D$  ratios (1.6 and 3) for each column diameter and column wall type (glass or steel). The corresponding powder weights used in each case are detailed in Table 1. A  $H_0/D$  ratio close to 3 had already been used for silicon deposition experiments on tungsten particles by FB-CVD using a reactor diameter of 3.8 cm, corresponding to a powder weight of 1500 g.

The second step involved studying the impact of reducing the reactor diameter on the fluidized bed thermal profiles. The aim was to obtain an average bed temperature of 650 °C with a minimal bed thermal gradient in steady-state conditions. The  $H_0/D$  ratios and weights of powder tested in the steel reactors are detailed in Table 2. The superficial velocity of argon was fixed around 4  $U_{mf}$ .

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