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X-ray micro computed tomography characterization of cellular SiC foams for their applications in chemical engineering

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ABSTRACT

Open-cell SiC foams clearly are promising materials for continuous-flow chemical applications such as heterogeneous catalysis and distillation. X-ray micro computed tomography characterization of cellular β-SiC foams at a spatial voxel size of 13.6^3 μm^3 and the interpretation of morphological properties of SiC open-cell foams with implications to their transport properties are presented. In-situ draining experiments were carried out in order to understand the nature of the residual static liquid hold-up in SiC foams enabling a better modeling and design of structured reactors based on SiC foams in the future. In order to see more practical uses, μ-CT data of cellular foams must be exploited to optimize the design of the morphology of foams for a specific application.

1. Introduction

Cellular ceramic foams [1–5] have gained increasing popularity in chemical engineering, especially as catalyst supports, due to their irresistible combination of attractive features, such as, low pressure drop (in comparison with fixed beds with the same exchange area), and enhanced mixing (in comparison with monolithic reactors). Specifically, for continuous-flow catalytic processes with liquid-or multi-phases, in addition to the low pressure drop, enhanced transport phenomena such as mass and momentum transfer are also mandatory because the diffusivity of species in the liquid phase is generally five orders of magnitude lower than one in the gas phase. Therefore, cellular foams are attractive candidates for developing new multiphase catalytic processes.

Silicon carbide (SiC) is a popular substrate for the preparation of cellular foams due to its unique properties of lightweight, high mechanical strength (hardness = ca. 2800 kg/mm^2), high oxidation and chemical resistance and good thermal conductivity (360–490 W/m·K) [6,7]. Various hierarchical catalysts/SiC foams materials (e.g. zeolites on SiC foams [4,8,9], nanostructures on SiC foams [4,10] and titania on SiC foams [6,7,11]) have been developed and investigated for a variety of applications as summarized in a recent review [12] demonstrating their potential as promising alternatives to conventional catalytic reactors (e.g. packed beds and monolithic reactors). From the process design point of view, hydrodynamic and transport properties of reactor beds based on open-cell foams need to be understood or predicted from the geometric structural parameters of foams. Experimental investigations have been made to develop the knowledge on pressure drop of single-phase flow through foams [13,14] and hydrodynamics (such as residence time distribution and radial dispersion within SiC foams) [15–17]. Combined experimental and numerical studies were also performed for the understanding of multiphase (e.g. characteristics of gas phase distribution) [18] and coupled reaction-heat transfer phenomena [19]. However, lot of aspects that have not been fully understood yet, in particular how morphometric parameters of cellular foams affect their hydrodynamic and transport properties.

X-ray micro computed tomography (μ-CT) is a technique to perform the non-invasive and non-destructive investigation of the three dimensional (3D) interior structure of optically opaque materials at micron level spatial resolution [20–22]. μ-CT is a precise technique and it is acknowledged that the effect of the measurement resolution and segmentation parameters on morphometric parameters of materials is trivial when the structure size is much bigger than the measurement resolution [23]. μ-CT characterization of cellular foams was also carried out with the main focus on exploring morphometric properties and mechanical behaviors of materials [3,23–31] Transport and hydrodynamic properties of cellular foams are related closely to their morphological properties such as the cell size, porosity, strut thickness, etc. To date, only a few works endeavored to interpret μ-CT data of cellular foams for continuous-flow chemical applications [2,3,5,32,33].

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In this work, β-SiC open-cell foams were characterized by X-ray micro computed tomography (μ-CT) and comprehensive geometric structural parameters of SiC foams were extracted. Considering the potential application of SiC foams in the chemical engineering, the effect of morphometric parameters on the fluid flow and transport phenomena in SiC foams were discussed. Furthermore, in-situ experiments with the air-water system through SiC foams were also performed allowing the non-intrusive investigation of the static liquid hold-up in the SiC foam packing.

2. Experimental

2.1. Materials and X-Ray Micro Computed Tomography Characterization

SiC (β polymorph) cellular foams were provided by SICAT SARL (France). The materials (average pore sizes = 13 and 30 pores-per-inch, PPI; denoted as S13 and S30 respectively, bulk density = 360 g/cm^3; void fraction ≈ 80%) were received as pellets (as seen in Fig. 1) then cut into cubes with the size of 20 × 20 × 20 mm. Cubic foams were scanned with a Nikon Metris 225/320 kV CT walk-in bay system (Fig. S1) equipped with a Perkin Elmer 1621–16-bit amorphous silicon flat-panel detector (2 K × 2 K pixel). A tungsten X-ray target was used to generate the X-ray spectra at 55 kV and 300 μA. The sample was rotated through 360° using continuous mode during which 2001 projections were collected. The acquisition time for each projection was 1.5 s and the spatial voxel size is about 13.6^3 μm^3 allowing for fine differences in SiC foams to be distinguished.

2.2. In-situ X-ray Micro Computed Tomography Characterization of Static Liquid Hold-up

In order to perform the draining experiment to determine the residual liquid hold-up in SiC foams, a sample holder was designed (Fig. 2), in which a u-shape silicone tube was connected to the bottom of a foam (via a 1/8 in SS tube) to allow the fluid to drain from the foam. SiC foams were sit onto the sample holder and stabilized by gluing the lateral boundaries of the foam and sample holder (Fig. 2a). For the draining experiment, the perimeter (or lateral boundary) of the foam sample was also sealed by silicon (as illustrated in Fig. 2b) to prevent the water leak. The draining experiment was performed by varying the differential pressure head of a water-saturated foam (i.e. the pressure difference between the static water head in the foam and of the end of the silicon tube), which was achieved by moving the end of the silicon tube vertically with measurements to record the static differential pressure. As shown in Fig. 1b, by moving the end of the silicon tube vertically downward, the water level in the foam needs to change accordingly to maintain an equivalent static pressure letting the air penetrate into the open-cell structure from the top of the foam and hence draining the water out from the foam through the tube. Prior to the in-situ X-ray scanning experiments, water retention properties and critical pressure heads were determined in the laboratory under hydrostatic conditions following the procedure described by reference [34]. Saturated SiC foams (with water) were drained and scanned by μ-CT in-situ at these pressure heads. At each pressure head, sufficient time (>2 h) was allowed to achieve the equilibrium of three phases (i.e. air, water and solid).

2.3. Image Processing and Segmentation

Raw μ-CT data were firstly reconstructed by Nikon Metrolasis CT-Pro software (Metris XT 1.6), and then converted to a stack of tiff images using an in-house code (a Matlab based GUI) [35]. Resulting raw image data was loaded directly by the standard Avizo software (FEI, version 8) for subsequent image processing and segmentation based on the principles of morphological image analysis [36,37]. Batch processing of filter applications to raw image data was carried out using in-house developed scripts to save time. Detailed steps of image processing and segmentation are provided in the Supplementary Material. The results

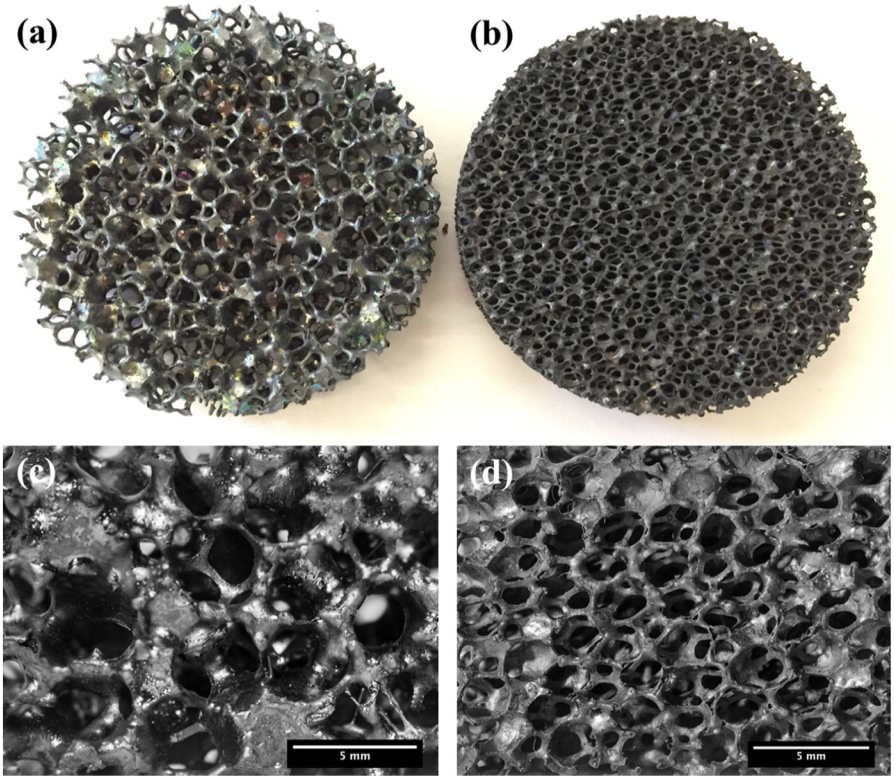

Fig. 1. SiC foam pellets as received: (a) 13 PPI (S13); (b) 30 PPI (S30); and (c) and (d) according microscopic images of foams.