Contents lists available at ScienceDirect

Chemical Engineering Research and Design

journal homepage: www.elsevier.com/locate/cherd

Prototyping a calorimeter mixing cell with direct metal laser sintering

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ARTICLE INFO

Article history:
Received 19 August 2015
Received in revised form 13 November 2015
Accepted 20 November 2015
Available online 30 November 2015

Keywords:
Additive manufacturing
Laser sintering
Flow calorimetry
Enthalpy of mixing

ABSTRACT

Herein we demonstrate the inherent strengths of an additive manufacturing method in the fabrication of a continuous-flow static mixing cell. The mixing cell was designed for the measurement of the enthalpy change related to mixing of fluids. The manufactured mixing cell consists of a single continuous structure and contains a highly tortuous internal channel, the construction of which would have been impossible using more conventional techniques. The design of the mixing cell is analyzed through both theoretical calculations as well as experimental testing. The manufactured calorimeter cells were tested with two well-known reference systems and were found to be fully functional and comparable to commercial ones. Inclusion of laminating mixer units inside the mixing cell was found to facilitate the mixing of the incoming streams.

1. Introduction

The heat of mixing (Vidal, 2003) is an important measurable thermodynamic property. Heat of mixing data can be used to validate the consistency of vapor–liquid–equilibrium (VLE) data (Olson, 1983), and together with the VLE-data to develop and validate thermodynamic equilibrium models. Flow calorimeters (Poledníček, 2000) are specifically designed for heat of mixing measurements. In a calorimetric mixing cell two streams are brought into contact and mixed thoroughly. The evolved heat is measured by monitoring the heat flow between the mixing cell and the surrounding calorimeter chamber. Typically, the mixing is assumed to be complete within the mixing cell. In reality the completeness of the mixing depends on the internal configuration of the mixing cell (Poledníček, 2000).

Additive manufacturing, colloquially known as 3D-printing, has developed rapidly and become more versatile in the past few years (Wohlers and Caffrey, 2013). One of the most scientifically interesting additive manufacturing methods is direct metal laser sintering (DMLS), which is able to process materials with good chemical and mechanical resistance, such as stainless steels. In DMLS process, a freeform metal object is fabricated by melting and fusing together fine metal powder under a focused laser beam in a layer-by-layer fashion (Gibson et al., 2009; Simchi et al., 2003).

Laser sintering is an automated fabrication process which is able to translate computer 3D-models (CAD-models) directly into corresponding physical objects. Likewise CAD-models have great compatibility with several computational and simulation methods. Many fluid dynamics simulation programs include the possibility of importing CAD-models as the geometry on which the simulations are based. The utilization of these connections allows very rapid prototyping and developing cycles covering design, test and redesign.

2. Methods and materials

CAD-models used in this work were done with AutoCAD–software (Autodesk Inc., version 18.0.0, 2013). The fluid dynamics simulations were done with computational software COMSOL Multiphysics (COMSOL Inc., version

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http://dx.doi.org/10.1016/j.cherd.2015.11.015

4.4.0.150, 2013). The simulation was based on the finite element method. The flow velocity field inside the mixing channel was calculated assuming a laminar flow, with uniform velocity profile (corresponding to total volumetric flow of 0.5 $cm^3\,min^{-1}$) at the inlets, and constant pressure at the outlet. For the flow computation the hydrodynamic properties of water at 25 °C and 1 atm were used. To visualize the flow propagation through the mixer, an imaginary tracer compound was introduced in one of the inlet streams with an initial concentration of 100. The presence of the tracer compound was not allowed to modify the stream properties. The diffusion coefficient for the tracer compound was set to 0, since the main interest was on the flow lamination behavior of the mixing units. Due to the nature of the finite element method, some numerical diffusion was present, which would have considerably reduced the accuracy of diffusion modeling. The computational mesh consisted of approximately of three million elements. The simulations were solved on a 330 GHz quad core processor with 16 GB of RAM. A model similar to the one used in this work is freely available at: http://www.comsol.com/model/micromixer-320.

The fabrication of the mixing cell prototypes was done on M 270 metal Direct Metal Laser Sintering workstation using Stainless Steel PH1 metal powder both supplied by EOS GmbH Electro Optical Systems (www.eos.info). After fabrication, the parts were annealed at 480 °C for 4 h to remove any residual thermal stresses. Parts were separated from the building platform by wire electrical discharge machining. The surface of all parts was finished with micro shot-peening.

Manufactured mixing cells were tested in a SETARAM–C80 calorimeter with well-known reference mixing pairs: Cyclohexane + *n*-hexane and ethanol + water. Cyclohexane and n-hexane were obtained from Sigma-Aldrich, ethanol from Altia and pure water was obtained from in-lab Millipore Elix 20 water purification system.

3. Results

The designed mixing cell consists of a mixing section where the incoming streams are laminated in four consecutive split-and-recombine–type mixing sections. The stream lamination increases the diffusion interface area and reduces the diffusion distance between the mixing compounds (Branebjerg et al., 1996). The combined stream is conducted through a helical mixing channel (square cross section, side length 1.77 mm, channel length 0.58 m) that circulates the cell near its outer surface. In this channel, the final mixing and the evolution of the mixing enthalpy occur through diffusion. The external and internal structures of the mixing cell are shown in Fig. 1. Liquids are introduced into the mixing cell in a concentric inlet tube, schematically presented in Fig. 1(c). The liquids entering the cell are initially at the same temperature with each other as well as with the measurement chamber of the calorimeter. In the mixing channel the enthalpy of mixing is released (or consumed) and the stream temperature is no longer in equilibrium with the surroundings. The heat exchange between the mixing cell and the calorimeter chamber is detected by the thermopile sensors of the calorimeter. The thermopile signal is related to the heat of mixing of the liquids with a calorimeter specific calibration factor (Poledníček, 2000).

The accuracy of the mixing enthalpy measurement is heavily dependent on the completeness of the liquid mixing in the mixing channel. The split-and-recombine mixing sections were designed to facilitate the mixing by reducing the required maximum diffusion length. The repetitive unit of the split-and-recombine mixer is shown in Fig. 2. During the design process a liquid flow and diffusion simulation in COMSOL Multiphysics software was consulted. The simulation results for the mixing section are presented on the right side in Fig. 2. The color scale gives the concentration of one of the mixing compounds.

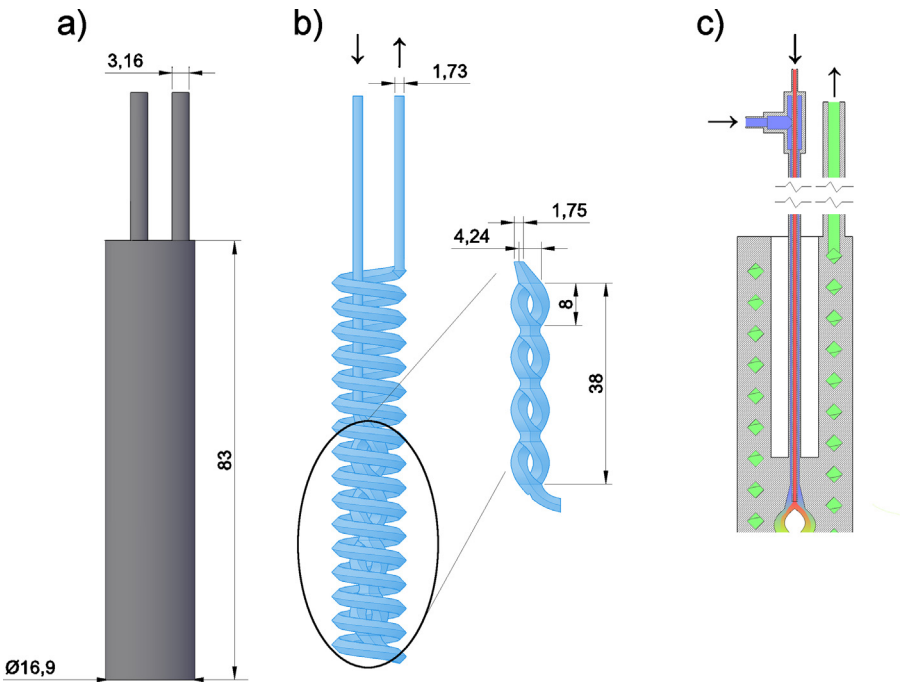

Fig. 1 – Structure of the mixing cell: (a) outer dimensions, (b) whole internal channel structure (represents "empty" space inside the cylinder), laminating mixer part shown also separately. (c) Inlet structure: Concentric tube structure used before the mixing cell to feed the two flows (marked as red and blue, green is the mixed out-coming flow). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)