

# Nanoscale Flow Chip Platform for Laboratory Evaluation of Enhanced Oil Recovery Materials

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## ABSTRACT

We present a lab-on-chip platform for the experimental evaluation of Enhanced Oil Recovery (EOR) methods from the nanoscale to the scale of reservoir rock pore networks. We have employed semiconductor process technology to build lab-on-chip flow devices with features at the nanometer scale that allow us to perform controlled flow experiments for calibrating multi-scale flow models. The platform built on silicon semiconductor technology is highly customizable and allows for design adaptation of different physical model representations. The approach enables us to experimentally investigate and validate liquid flow in porous media below the micrometer scale and to deploy calibrated, multi-scale flow simulations in a digital representation of a given rock pore network. The chip implementations of the nanoscale, porous rock network enable systematic flow studies covering various parameters (e.g. effective porosity, viscosity, surface properties) under controlled conditions of physical parameters (e.g. temperature, pressure). High resolution optical microscopy measurement techniques enable us to track individual nanometer size fluorescent tags which allow us to directly determine fluid flow speeds even in sub-micrometer constrictions. We introduce the architecture of the flow chip, discuss how the flow experiments are performed and how the experimental results are used to calibrate the flow simulations. Ultimately, the calibrated flow simulations will be used for predicting the efficiency of a specific EOR agent for improving oil displacement in a pore scale network of reservoir rock.

**Keywords:** Nanofluidics, EOR, Digital Rock Physics

## 1 INTRODUCTION

Enhanced oil recovery (EOR) defines a set of exploration technologies and practices for tertiary oil production [1]. A fundamental question is which chemical and physical processes limit oil displacement at sub-micron scales. Flow simulations are needed that account for the complex interactions between reservoir constituents, such as rock and oil, and EOR materials, such as polymers and nanoparticles. Such multi-scale flow simulations with experimental calibrations, ideally at the

nanoscale, form the very core of next generation simulation technology that will provide recommendations for EOR methods to the reservoir engineer [2].

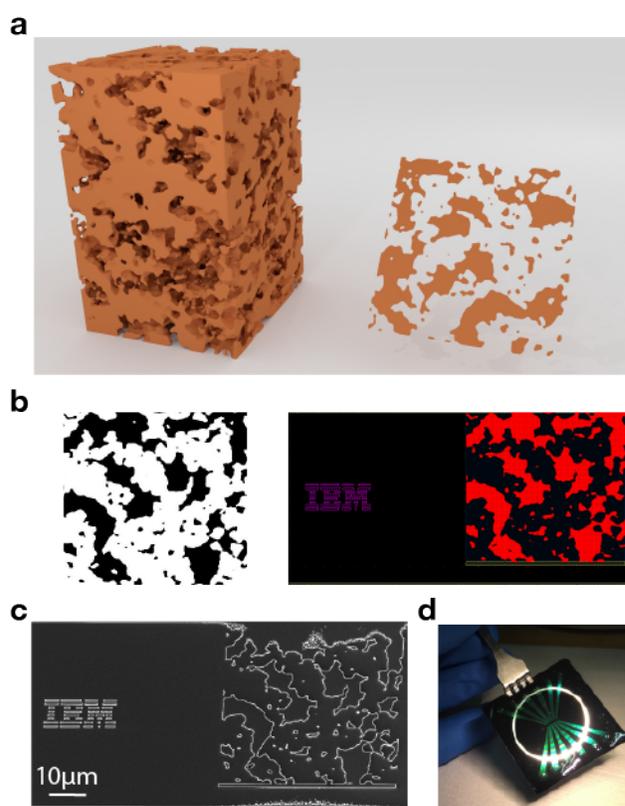


Figure 1: (a) 3D visualization of a porous medium obtained from  $\mu$ CT data together with a single slice extracted from the data cube. (b) Design template obtained by thresholding the slice in (a) (left) and the resulting CAD design used for nanoscale flow chip fabrication (right). (c) Scanning electron micrograph of an as-fabricated nanoscale flow chip. (d) Optical micrograph of a completed nanoscale flow chip sealed with a glass cover having inlet and outlet ports.

## 2 FLOW CHIP PLATFORM

In Figure 1 we outline the design and implementation work flow of our nanoscale flow chip platform. We first visualize geometrical characterization data of porous media and then choose a particular data subset of interest (Fig. 1a). In the present case we source digital data of porous media from publicly available repositories [3]. After choosing the data set of interest we perform a segmentation step in order to obtain a template image that differentiates between solid material and open pore space. This image serves as a template CAD design input which is required for fabrication of the nanoscale flow chip (Fig. 1b). Based on the CAD design we then fabricate the nanoscale flow chip on an 8inch silicon wafer platform using semiconductor integration technology [4] with a series of process steps similar to those described in Wunsch et al. [5]. In brief, we first define larger flow channels for fluid input and output by a combination of optical lithography and reactive ion etching. Next, we pattern the porous media structure into the center of the flow channel by electron beam lithography and reactive ion etching. In Figure 1c, we show a scanning electron micrograph of an as-fabricated nanoscale flow chip close to the region where we transferred the porous structure onto the silicon wafer. The smallest constriction in the present case is approximately 200 nm. Next, we dice the silicon wafer into individual nanoscale flow chips. To enable flow measurements, we encapsulate the flow channels by anodically bonding [6] a glass cover slide having micro-machined ports for fluid injection and extraction. In Figure 1d, we show an optical micrograph of a finished nanoscale flow chip having a size of  $4 \times 4 \text{ cm}^2$ .

## 3 FLOW EXPERIMENTS

In Figure 2, we present the results of flow experiments conducted with our nanoscale flow chip platform. In order to avoid bubble formation during the measurements, we first perform a pre-wetting step by sequentially soaking the flow chip with Ethanol and de-ionized water until the flow channel is completely filled with liquid. Next, we insert the flow chip into a fixture that connects to an external feedback controlled pump system utilized to set the chip's pressure and establish a constant flow rate. As a representative test liquid for our experiments, we use de-ionized water driven at a pressure of 1 bar. We monitor the water flow by means of 17 nm fluorescent beads having an excitation and emission wavelength of 470 nm and 510 nm, respectively. For fluorescence imaging, we use a  $100\times$  oil immersion objective in combination with an EM-CCD camera (Andor iXon) operating at a frame rate of 18.1 ms.

In Figure 2a, we show an optical micrograph of the channel region inside the flow chip containing the ex-

tracted porous structure before admixture of the fluorescent beads. The acquired images have size of about  $(80 \mu\text{m})^2$  and consist of  $(512 \text{ px})^2$  which corresponds to an image pixel size of  $(160 \text{ nm})^2$ . The actual optical feature resolution achieved in the fluorescence images is about 250 nm. In Figure 2b,c we show fluorescence images at two representative times  $t$ . The fluorescent beads provide a high contrast which allows to track them even individually. By accumulating a series of image frames we are able to visualize the main flow patterns and constrictions on the chip (Fig. 2d). In the next section, we will quantitatively analyze the flow patterns and extract spatially resolved velocity distributions.

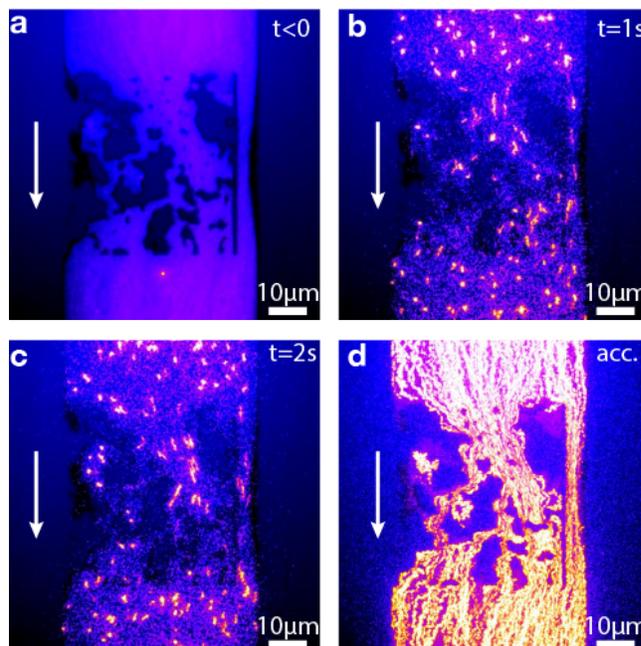


Figure 2: Nanoscale flow visualized by fluorescence microscopy. (a) Bright field optical micrograph of the flow chip, before insertion of fluorescence beads. (b)(c) Fluorescence micrographs overlaid with optical bright field micrographs at two selected times  $t$ . (d) Accumulated fluorescence signal integrated over all acquired frames. The arrows indicate the flow direction.

## 4 NANOSCALE PARTICLE IMAGE VELOCIMETRY

The quantitative flow analysis is based on the as-measured, high-resolution images of 17 nm fluorescent beads presented in the previous section. Note, that the experimental conditions allow us to discern the fluorescence of individual tracer beads from the much darker background. A total of 500 images were acquired per measurement at regular time intervals of 18.1 ms.

We use an image-processing technique referred to as Particle Image Velocimetry (PIV) [7] for extracting flow velocities from each image stack. This technique divides each image into interrogation windows and calculates the cross-correlation between pairs of subsequent images, so that a local displacement vector can be obtained. Averages over 250 pairs of snapshots are taken in order to improve the statistics and signal-to-noise ratio of the flow field determination. We use  $(16 \text{ px})^2$  interrogation windows with 50% overlap (Nyquist sampling criterion) so that the resulting flow field has a velocity vector associated with each  $(1.28 \mu\text{m})^2$  domain.

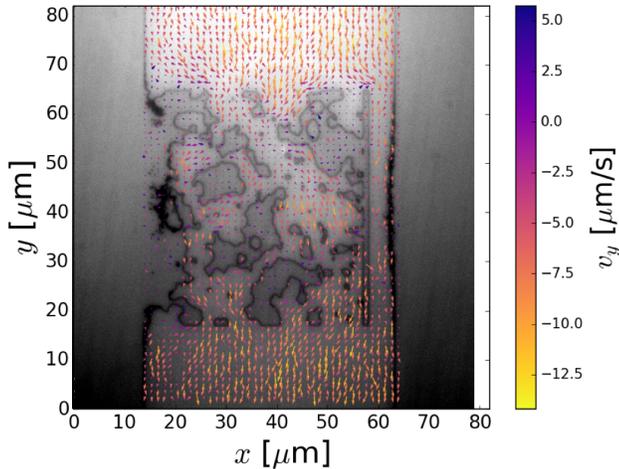


Figure 3: Flow velocity field obtained through PIV analysis. Each arrow corresponds to a local velocity vector representing an interrogation window having a size of  $(1.28 \mu\text{m})^2$ . The color code corresponds to the vertical (y) velocity component.

As a result, the 2 dimensional velocity distribution captures the local variation of flow speeds and directions, quantifying the flow properties in the porous structure. The extracted experimental flow distribution can now be compared to the results of a computational flow simulation based on the same porous geometry.

## 5 FLOW SIMULATIONS

An important application of the experimental flow analysis is the calibration of computational flow simulations in porous media. We have simulated liquid flow through the same porous structure that was used to create the flow devices by applying the Lattice Boltzmann Method (LBM) [8]. LBM simulations approximate solutions to the flow equations using an approach based on lattice gas automata, and are particularly well suited for simulation in complex geometry. Once calibrated, the LBM models can be applied directly to simulate flow through porous media at larger scales. Figure 4 shows

the magnitude of the velocity field simulated by LBM for the porous structure shown in Fig. 2 which is also used to extract the experimental velocity field pictured in Fig. 3. Note, that the flow velocities in the LBM simulation were calibrated from the measured results in Fig. 3.

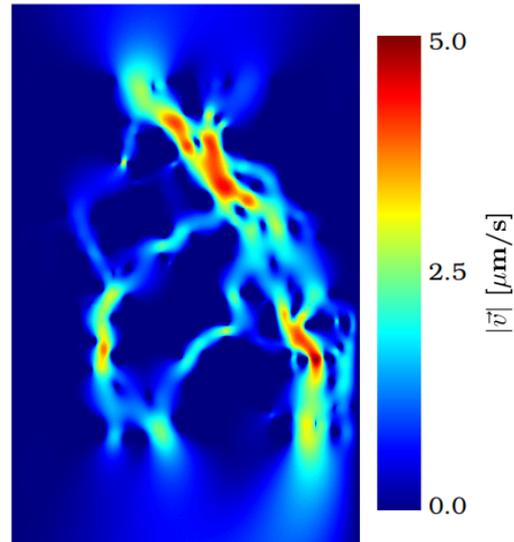


Figure 4: Simulated velocity field for flow through the porous media structure pictured in Fig. 2, from which was produced the experimental velocity profile of Fig. 3. The simulation was performed using the Lattice Boltzmann Method (LBM), and the flow velocities were calibrated from the experimental results.

## 6 CONCLUSION

In summary, we presented a method including (1) a digital porous medium, (2) a flow chip with a representation of the porous medium, (3) a nanoscale flow experiment, and (4) a corresponding flow simulation with a calibration provided by the experimental results. The method and the flow chip are designed to enable laboratory testing of the influence on flow of materials that are considered for application in Enhanced Oil Recovery.

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