Fabrication of quartz-etched micro-models for two-phase flow studies

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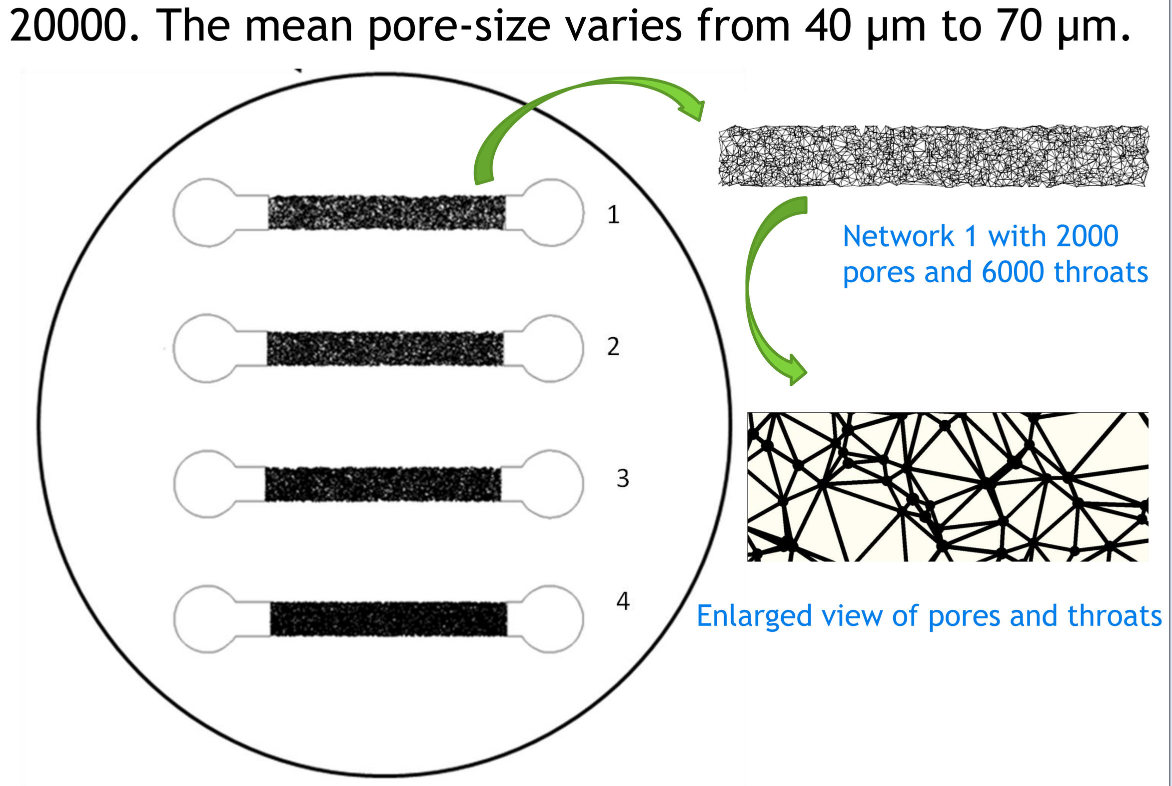
ABSTRACT

Micro-models representing porous media have been successfully fabricated for investigating two-phase fluid-flow through them. A combination of pore bodies and throats form the flow network in a micro-model. Different micro-models are prepared based on varying number of pores and throats, i.e., representing porous media of varying porosity and connectivity. These networks are etched on Quartz material along with reservoirs on each side of the network. A separate layer of Quartz with holes serving as inlets and outlets is connected to these reservoirs by molecular bonding. Quartz is chosen as the substrate because of its optical and wetting. Vertical walls in the channels are one of the primary fabrication goals, and it has been achieved by using Inductively Coupled Plasma Reactive Ion Etching (ICPRIE).

OBJECTIVE AND NETWORK DESIGN

Fabrication of micro-models for investigating fluid flow through a porous medium has been started as early as the '50s [1], and later the focus of such studies has been varied, from capillary fingering effects [2] to nanometer scale flow [3] to Invasion-Percolation Experiments [4]. In this study, the principal objective is fabrication of micro-model for two-phase fluid-flow experiments and subsequent visualization.

Network Design is performed in MATLAB, where Delaunay triangulation is used for closest resemblance to a porous medium. The pores and throats are designed as cylinders and rectangular sectioned, respectively. Four different micro-models were designed , with each network 35 mm long and 5 mm wide. The number of pores varies from 2000 to 6000 while the number of throats varies from 6000 to 20000. The mean pore-size varies from 40 μm to 70 μm.



Design of Four Networks (Network 1: 2000 pores, 6000 throats, mean pore-size 40 μm; Network 2: 3000 pores, 9000 throats, mean pore-size 40 μm; Network 3: 3000 pores, 9000 throats, mean pore-size 70 μm; Network 4: 6000 pores, 20000 throats, mean pore-size 50 μm

MATERIALS AND METHODS

Matlab (Math Works, Natick, MA, USA) is used in the network design for its Delaunay Triangulation mode, with FORTRAN used for assigning sizes to the pores and throats. The design is incorporated in AUTOCAD (Autodesk Inc., San Rafeal, CA, USA). L-edit has been used for final mask-design. Photomask Generator (Heidelberg DWL-200) is used for a negative mask fabrication, quartz substrates are used for the channel-fabrication (4" diameter circular, 0.5 mm thickness, Sensor Prep Services, Inc., Elburn, IL, USA). A UV Mask Aligner is used for the lithography purpose, Solitec Spinner used for spin-coating photo-resist on substrate before lithography as well as CEE Hotplate for post-baking of the exposed wafers. Finally, ICPRIE (Alcatel AMS110) is used for plasma-etching of the network.

FABRICATION

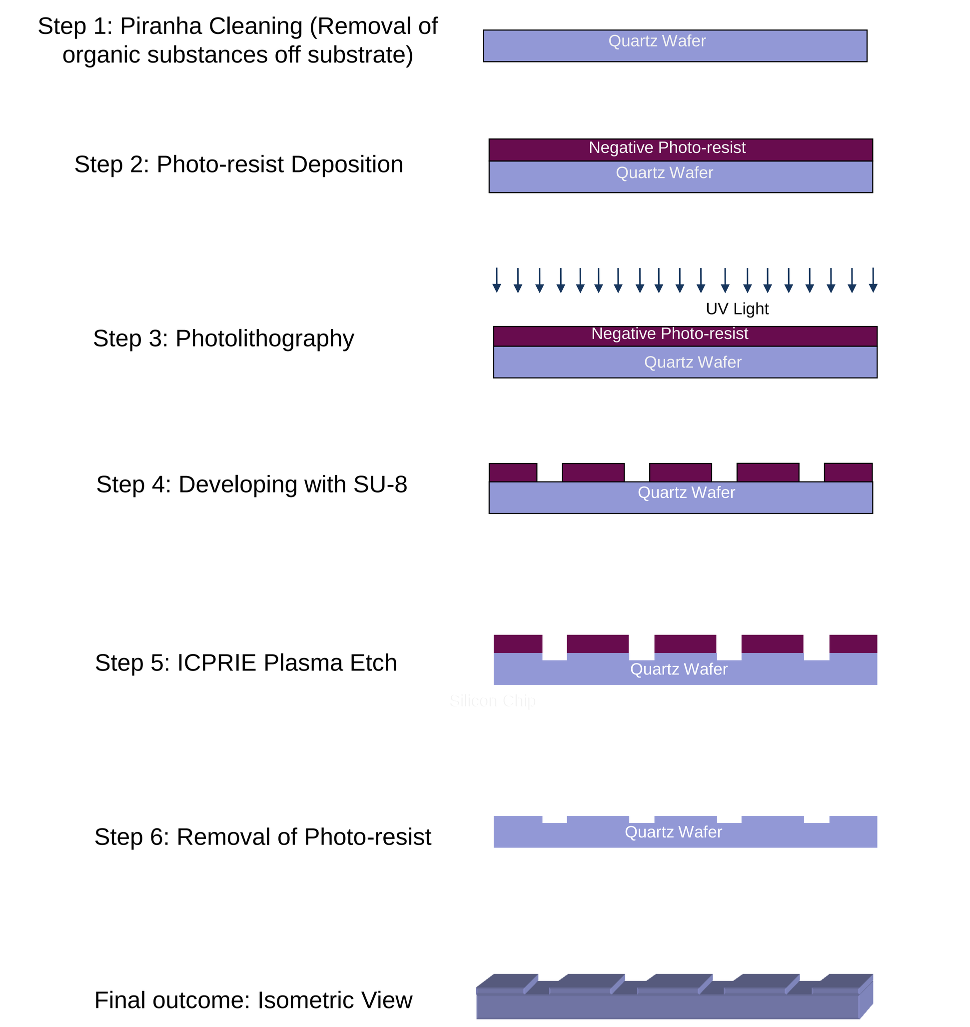
A negative photomask is prepared in the mask-generator (5" square, 2mm thick, resolution 2 μm, glass with chrome coating). Double-sided polished quartz substrate, 500 μm is chosen for etching the channels representing the networks in the design.

The Quartz substrates are Piranha -treated (H_2SO_4 : H_2O_2 in 3:1 volumetric ratio for 30 min) in order to clean them based on micro-fabrication requirements. KMPR-1025 negative photoresist is used in the process, and a 15 μm thick resist is spin-coated on the wafers and dried (200^0 C for 1 hour). Then the wafer is exposed to the UV light under the negative photomask. During the exposure, exposure time is decided taking into consideration that double sided polished substrate would require less time under UV than a single-sided substrate. After exposure, the substrate is developed with SU-8 developer for 90-100 seconds (Microchem Corp., Newton, MA, USA).

In order to ensure the correctness of exposure time, the developed substrates are studied under micro-scope. A post-exposure-bake is performed on quartz (100^0 C for 3 hour) and then Alcatel plasma etch is used for the final etch up to a depth of 40 μm. The etch-rate selected in AMS 110 (Alcatel, France) is 4 μm each pass.

The micro-models have been etched approximately at a depth of 40 μm keeping in mind that the mean pore size of the network is close to this, treating it as an equivalent of 3D porous media.

Finally, the substrate is cold-piranha treated for a day and O_2 plasma treated for complete removal of resist.



Schematic of Fabrication along with diagrams along the process-flow

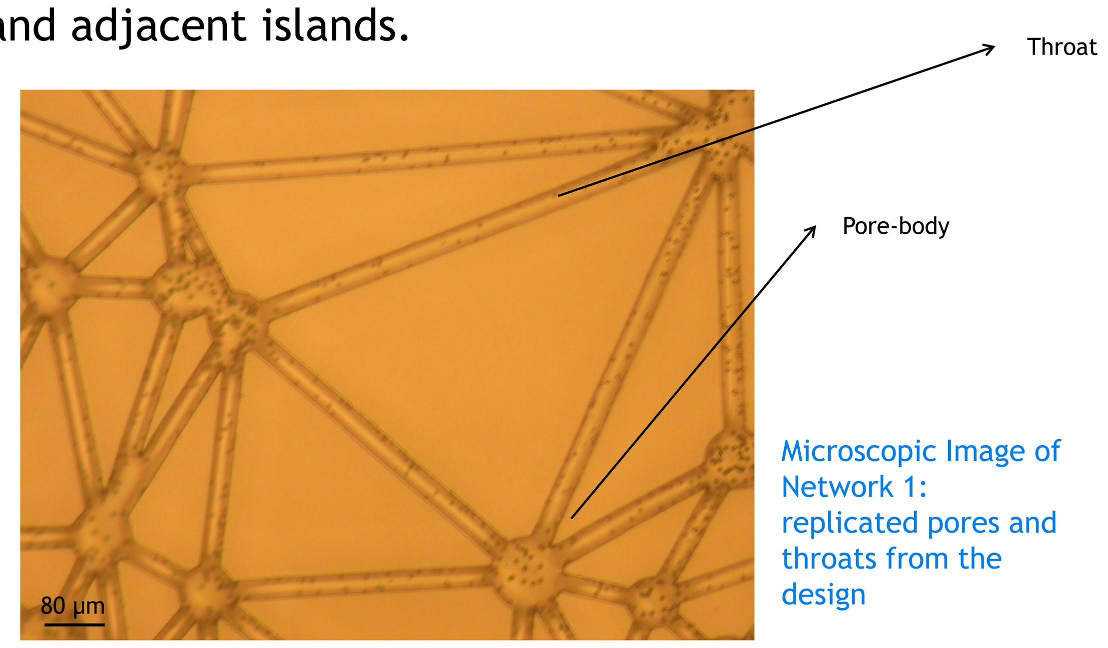
RESULTS

Fabrication of the mask at 2 μm reproduces the design with accuracy, as certain degree of smoothness is desired in the final micro-model in order to conduct experiments.

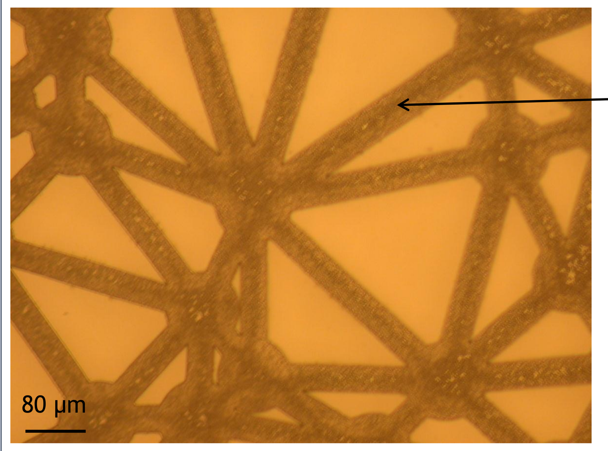
Final Quartz substrates, with the micro-models etched in them, are examined under microscope . The designed features have been replicated in the micro-model with fair degree of accuracy. The edges of the network are sharp, and hence the exposure-time and post-baking parameters have been reasonable.

Further, the depth of the micro-models are measured in a surface profiler (Alpha Step) and it is found that, for four different micro models they vary approximately from 40.5 μm to 43 μm, which is reasonably close to the desired depth of 40 μm.

Certain features of the models prepared are observed in the microscopic images as well. There is considerable 'pitting' effect on the models, which is quite common for quartz substrate. Also, for Network 3 and Network 4, the surfaces are more rough than the first two, as observed from the contrast in the channels and adjacent islands.

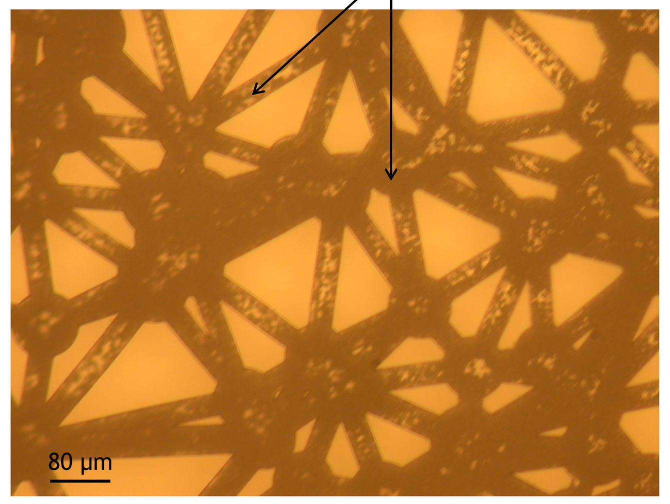


Microscopic Image of Network 1: replicated pores and throats from the design



Fabricated network in model 3, with the enhanced contrast in channels pointing out the roughness introduced in the channel during fabrication

Pronounced pitting Effect in Network 4



Sample images displaying the micro-models etched on Quartz substrate. Here, representative sections in Networks 3 and Network 4 are shown

CONCLUSION

Fabrication of four different pore-throat networks is carried out, where the micro-model represents a porous media. Novel fabrication recipe has been applied for obtaining the micro-model in quartz substrate, which is chosen for proper visualization of flow-experiment in the channels. For vertical walls in the channels, state-of-the-art plasma etch process has been adopted.

The experiment and analysis of two-phase fluid flow would be facilitated in such a micro-model and back-illumination in quartz would enhance the visualization.

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