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A Programmable Gas Injection System for 3D Ice Lithography

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Introduction. Ice lithography (IL) is a novel nanofabrication method that utilizes electron beam (e-beam) to pattern ice thin-film that is condensed onto the cold sample surface [1] (Fig. 1A). The ice thin-film is formed by depositing vapor onto the sample that is cooled by a cryostage [1]. In 2017, Tiddi et al. reported resist patterns made of organic ice [2]. E-beam exposed organic ice is cross-linked and stable at room temperature. Similar to layer-by-layer 3D printing, proof-of-concept 3D structure were made by performing a repetition of “condense and expose” sequences. The final sublimation step removes the un-reacted ice, leaving a room temperature stable cross-linked 3D structure (Fig. 1B).

Today, we aim to expand the IL system into a full-fledged 3D ice lithography (3DIL) system for multi-material 3D printing of nanostructures. To obtain 3DIL process with high level of precision, the 3D process needs to be automated and the different modules need to be integrated. One of the critical modules is an advanced gas injection system (GIS) to handle several precursors for multi-material 3D printing.

Design. To achieve reliable and accurate gas injection with fast response time, we designed a GIS (Fig. 2A) that is based on atomic layer deposition (ALD) system . This GIS design utilizes continuous stream of carrier gas, which transports a controlled amount of precursor pulse into the SEM and over the cold sample. Each pulse is set to produce a consistent pressure drop in the precursor cell, which corresponds to a specific thickness of the deposited ice film, as described in equation (1).

$$\Delta h = \frac{V_{GIS} M}{\rho_i A R T} \Delta P_{GIS} \qquad (1)$$

Here, Δh is the ice thickness, V_{GIS} is the volume of the precursor cell, ΔP_{GIS} is the pressure drop in the precursor cell, M is the molar mass of the precursor, R is the gas constant, T is the precursor temperature, A is the deposition area, and ρ_i is the precursor ice density [3]. The precursor material is provided in a vial equipped with a temperature controlled heater. This ensures that the user can achieve enough vapor pressure by adjusting the precursor temperature. The precursor cell is the volume between the source (SRC) and the ALD valve. The injection (INJ) and vacuum (VAC) valves connect the GIS to SEM chamber and vacuum pump respectively. By opening or closing either INJ or VAC valves the user can direct the flow into the SEM chamber for precursor injection or to the vacuum pump for purging. The pressure gauge P1 to Pn monitor the precursor cell pressures, while Pv monitors the main vacuum line pressure. The carrier gas line and the mass flow controller (MFC) also mediate the purging of the main vacuum line between precursors switching.

Implementation. We assembled a single precursor GIS (Fig. 2B). The opening and closing of the valves, as well as the flow of the carrier gas can be integrated into a streamlined operation. A leak-valve is used as the SRC valve to carefully control the precursor cell pressure. We used a diapraghm valve and a motorised ball valve for INJ and VAC valve respectively. The ALD, INJ and VAC valves are pneumatically controlled by solenoid valves and relays. The SRC valve is directly controlled with analog voltage signal. The pressure values from P1 and Pv are read digitally. Labview and NI DAQ were used to automatically control all valves and the MFC (Fig. 2C). This way, the GIS can be programmed for consistent layer thickness during repeated injection for 3DIL.

Test and evaluation. We started with the pumping process to purge the system from residual air as well as to establish an optimal vapor pressure in precursor cell. The precursor pressure can be adjusted to condense a thin-film with desired thickness. While the ALD and solenoid valves are rated to 5 ms, we measured that 50 ms precursor pulse can be performed consistently, which results in an 80% pressure drop in precursor cell. The continuous N2 gas flow can be controlled down to 0.05 sccm, such that the SEM and Pv pressure are maintained at 10^{-2} Torr. Our preliminary test using iso-propyl alcohol precursor with a pressure drop of 4.29 Torr produced a film of 400±30 nm thickness.

[1] A. Han, et al., “Ice lithography for nanodevices,” Nano Letters. 10 (2010) 5056–5059.
[2] W. Tiddi, et al., “Organic Ice Resists,” Nano Letters. 17 (2017) 7886-7891.
[3] W. Tiddi, et al., “Organic ice resists for 3D electron-beam processing: Instrumentation and operation,” Microelectron. Eng. 192 (2018) 38-43.

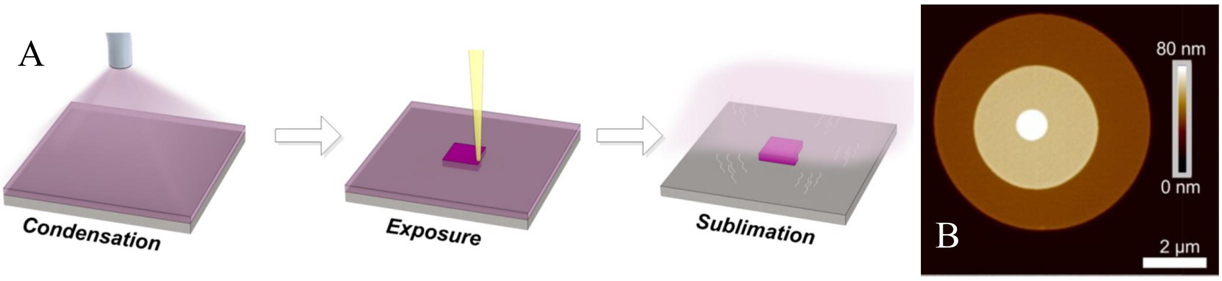
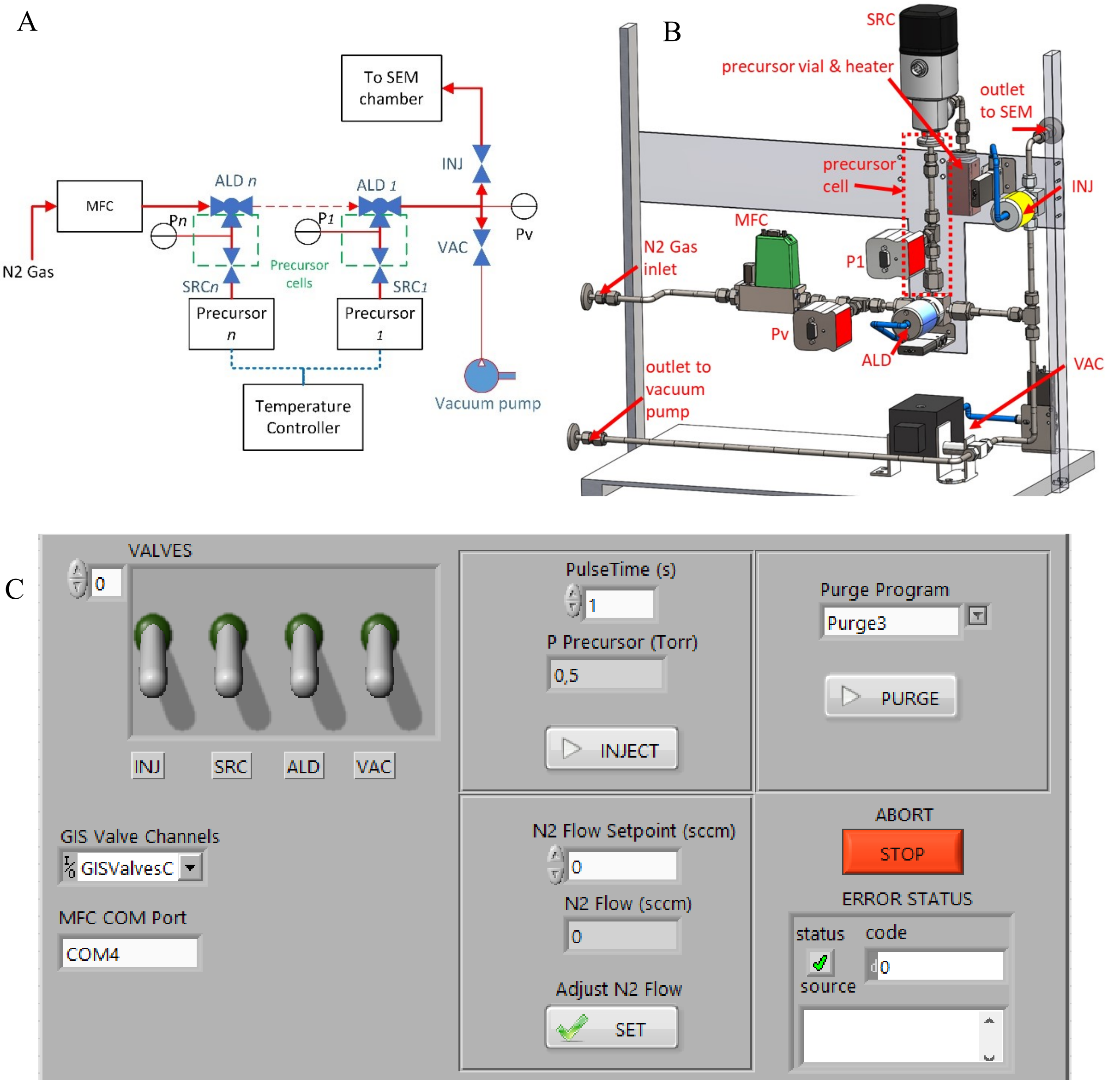


Figure 1. Ice lithography process (A) [2]. The 3D structure is made of organic resist (B).



VALVES: 0

PulseTime (s): 1

P Precursor (Torr): 0,5

Purge Program: Purge3

GIS Valve Channels: GISValvesC

MFC COM Port: COM4

N2 Flow Setpoint (sccm): 0

N2 Flow (sccm): 0

ERROR STATUS
status ✓ code 0
source

Figure 2. Schematic of the multi-material GIS module (A). Prototype of the assembled GIS for single material (B). Labview GIS user interface (C).