Experimental report

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Proposal: CRG-2762 **Council:** 4/2020

Title: Investigation of Static SequentialInfiltration Synthesis into Polystyrene-block-Maltoheptaose by neutron reflectivity

Research area: Engineering

This proposal is a new proposal

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Samples: Maltoheptaose
Polysyrene
Polystyrene-block-Maltoheptaose

Instrument	Requested days	Allocated days	From	To
SUPERADAM	2	3	01/02/2021	04/02/2021

Abstract:

In block copolymer lithography, metal oxide is often infiltrated selectively into one block, to establish high etch selectivity between blocks. This work investigates two different infiltration methods, where Alumina is infiltrated into 12 nm pitch Polystyrene-block-Maltoheptaose. A previous data collection of multiple pulse infiltration was made at Super-ADAM, ILL in February 2020, where analysis is ongoing. The proposed data set focuses on static (single pulse) sequential infiltration synthesis, which is a less material consuming process, but an initial test still indicates infiltration of more material per cycle. The study is expected to be of use in nanofabrication of sub-10 nm features.

CRG-2762 Experimental Report

Investigation of Static Sequential Infiltration Synthesis into Polystyrene-block-Maltoheptaose by neutron reflectivity

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Background

The block copolymer (BCP) poly(styrene)-*block*-maltoheptaose (PS-*b*-MH) of molecular weight 4.5 kg/mol and 1.2 kg/mol, respectively, can be self-assembled into a 12 nm pitch pattern. The polymer can be infiltrated with trimethyl aluminium (TMA) and water in an atomic layer deposition (ALD) chamber, in a process known as sequential infiltration synthesis (SIS). Aiming for a quantitative characterization of the resulting alumina inclusion in respective block, thin film samples of homopolymer maltoheptaose (MH, 1.2 kg/mol), homopolymer hydroxyl-terminated poly(styrene) (PS-OH, 4.5 kg/mol), and self-assembled PS-*b*-MH were investigated, for varying number of infiltration cycles, in specular neutron reflectivity (NR) measurements in air at 5.12 Å wavelength, for incident angle 0-4°, at Super-ADAM neutron beamline, Institute Laue-Langevin (ILL), Grenoble, France, on February 1-4, 2021.[1] Also, experiments exploring any differences in alumina content in the polymer films after using two different SIS methods, dynamic and static infiltration, as well as influence of the process parameters temperature, exposure time, and pulse duration for static infiltration were performed. Some of the measurements are complementary to a previous experiment in 2020.[2]

Preliminary results

Table 1 and **Table 2** shows samples measured in specular NR, including their corresponding scan numbers.[1]

Table 1. Sample material measured in specular NR, and their corresponding scan numbers.

SIS method	Number of SIS cycles	Si substrate	MH	PS-OH	PS-*b*-MH (H)	PS-*b*-MH (V)	PS-*b*-MH (new)
N/A	0	120	78	82			112
Dynamic	1			130			
	2						119
	4			131			
	12		124	125	126	138	
Static	1		71	72	70	132	113
	2		83	89		136	114
	4		76	77		137	118

N/A Not applicable.

Table 2. Sample material, 2 cycles of static infiltration, measured in specular NR, and their corresponding scan numbers.

Parameter	Value	MH	PS-OH
Temperature / °C	64	84	90
	80	83	89
	100	88	94
Exposure time / s	20	95	100
	60	83	89
	100	96	101
TMA/H_2O pulse duration / ms	10/5	102	107
	25/15	83	89
	75/45	106	108

Analysis of PS-OH samples showed no indications of alumina inclusion, regardless of parameter change (see **Table 2**). The alumina content in MH was not significantly affected by temperature. A low exposure time increased the alumina content significantly, from 2 vol% at 100 s to 40 vol% at 20 s (see **Figure 1**). The TMA/H_2O pulse durations also had a significant effect on the amount of alumina inclusion; both the shorter and the longer pulses resulted in 40 vol%, compared to 8 vol% for the medium pulse duration (see **Table 2** and **Figure 2**). These results could potentially have an impact on possible aspect ratio in pattern transfer of the block copolymer pattern.[3]

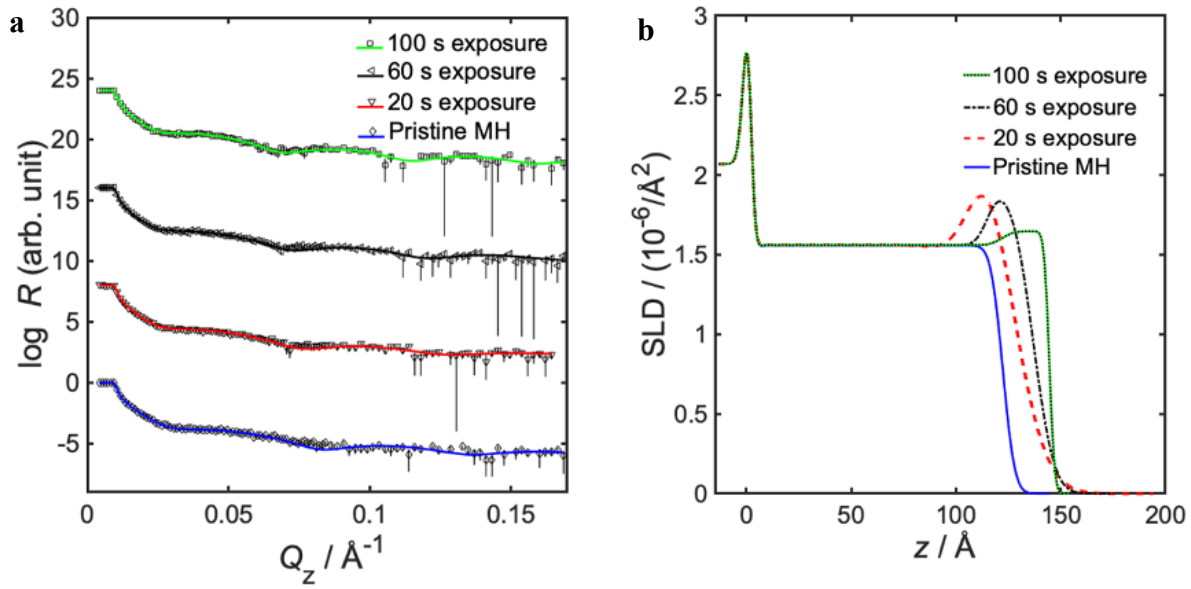

Figure 1. a) Measured MH specular neutron reflectivity R with error bars as a function of neutron momentum transfer perpendicular to substrate surface Q_z, and fitted models, and b) fitted scattering length density (SLD) as a function of perpendicular distance from substrate surface z. Figure reproduced with permission from the authors.[3]

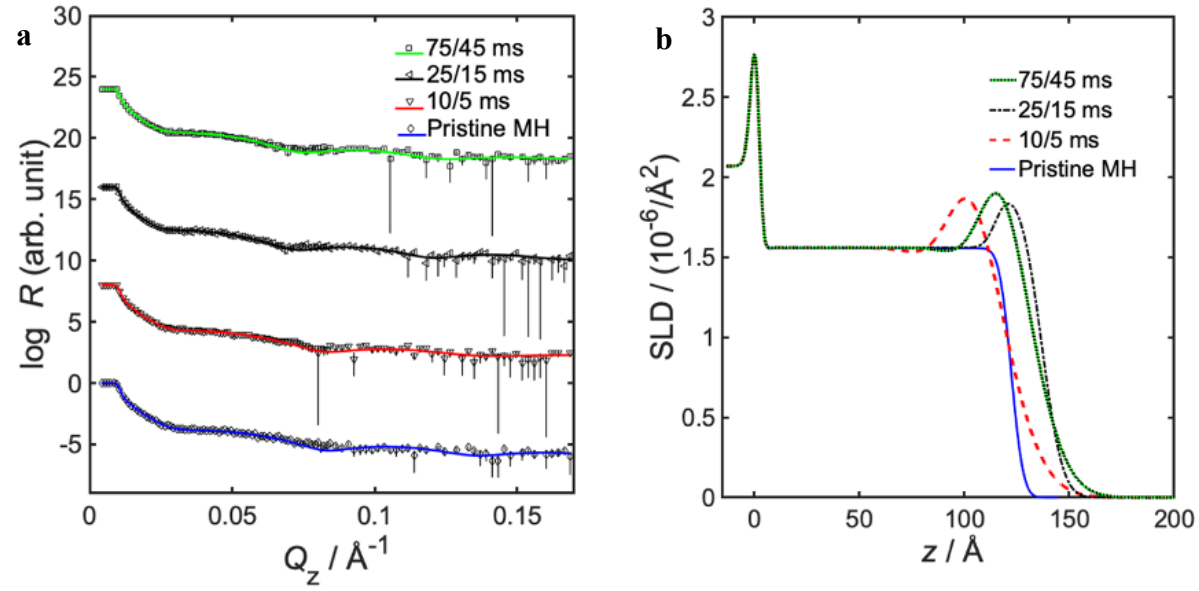

Figure 2. a) Measured MH specular neutron reflectivity R with error bars as a function of neutron momentum transfer perpendicular to substrate surface Q_z, and fitted models, and b) fitted scattering length density (SLD) as a function of perpendicular distance from substrate surface z. Figure reproduced with permission from the authors.[3]

Acknowledgements

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References

[1] Löfstrand, A., and Vorobiev, A., "Investigation of Static Sequential Infiltration Synthesis into Polystyrene-block-Maltoheptaose by neutron reflectivity", Institut Laue-Langevin (ILL): Grenoble, 2021, doi:10.5291/ILL-DATA.CRG-2762.

[2] Löfstrand, A., Mothander, K., and Vorobiev, A., "Neutron Scattering Study of Selective Infiltration Synthesis into Block Copolymer for Sub-10 nm Nanolithography Applications", Institut Laue-Langevin (ILL): Grenoble, 2020, doi:10.5291/ILL-DATA.CRG-2597.

[3] Löfstrand, A., Vorobiev, A., Mumtaz, M., Borsali, R., and Maximov, I., "Sequential Infiltration Synthesis into Maltpheptaose and Poly(styrene): Implications for sub-10 nm Pattern Transfer," 13 (2021), submitted.