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 MLF Experimental Report	提出日 Date of Report 24 July 2013
課題番号 Project No. 2012B0021 実験課題名 Title of experiment Structural Analysis of Polymer Blush Layer Sandwiched between Two Surfaces by Means of Neutron Reflectometry 実験責任者名 Name of principal investigator Tomoko Hirayama 所属 Affiliation Doshisha University	装置責任者 Name of responsible person Dr. Norifumi Yamada 装置名 Name of Instrument/(BL No.) SOFIA (BL No.16) 実施日 Date of Experiment 23-26 March 2013

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
 Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.
<p>We prepared a special sample holder with an ultraprecise tilting stage supported by three piezoelectric elements for parallel adjustment of two surfaces. The face size of the SiO₂ blocks was 10×10 mm, the dry thickness of the poly methyl methacrylate (PMMA) polymer blush layer was about 50-100 nm, and the target clearance between two surfaces was about 100-500 nm. The polymer blush on one side was grafted from the deuterated monomers for clear contrast for neutron. The analyses by ellipsometry for dry thickness of the polymer blush layer and FT-IR for chemical bonds of the layer were checked previously for obtaining complementary data.</p>

2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。)
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.
<p>Tribology is one of academic disciplines covering friction, wear and lubrication in engineering for a lot of industrial applications such as bearings, gears, engine pistons in car and so on. Adsorption of polymer blushes on metal substrates has attracted attention as an effective way to reduce friction by means of assisted lubrication as found in natural lubrication systems. There are several studies investigating the relationship between the brush structures and their tribological properties, and some of the architectures of polymer brushes have been found to be highly effective in achieving super-low friction under lubrication. However, it is still unclear why such polymer brushes could contribute to attain super-low friction sliding. Some of previous studies expected that osmotic pressure occurred between the polymer blushes adsorbed onto top and bottom surfaces could support a part of load, resulting that the friction coefficient became super-low, less than 0.001. However, nobody knows about the probability of interpenetration of the polymer blushes adsorbed onto both sides in the narrow clearance sandwiched between the surfaces.</p>

2. 実験方法及び結果(つづき) Experimental method and results (continued)

First, we checked the thickness of the poly methyl methacrylate (PMMA) polymer blush grafted onto SiO₂ substrate in deuterated water and toluene. Water is poor solvent and toluene is good solvent for PMMA blush. The obtained reflectivity profiles in d-water and d-toluene were shown in Fig. 1. The PMMA blush thickness in water was almost same as its dry thickness, while the blush thickness in toluene was about three times larger than its dry thickness.

As a next step, we tried to measure the polymer blush thicknesses sandwiched by two SiO₂ blocks. The construction of the SiO₂ blocks without polymer blush is shown in the left illustration in Fig. 2. The procedure we conducted is as follows:

- (1) The Cr and SiO₂ layers on SiO₂ blocks were checked first. Then, since the SLD of Cr and SiO₂ are close, we used one layer model of Cr for the fitting.
- (2) The reflectivity profile of the sample with PMMA blushes sandwiched by the blocks was obtained.

The fitting operation in (1) showed that the total thicknesses of Cr+SiO₂ layer on the top and bottom blocks were 29 and 41 nm, respectively, as illustrated in the right in Fig. 2. The reflectivity profile in (2) is shown in Fig. 3. From the fitting operation, total polymer blush layers sandwiched by the blocks were 51nm; the upper polymer blush thickness was about 10nm, while the lower blush thickness was about 41 nm. We succeeded to obtain the clear reflectivity profile from the polymer blushes sandwiched by SiO₂ blocks. We wish to retry to do the same experiment again with changes in the clearance in the next beam time.

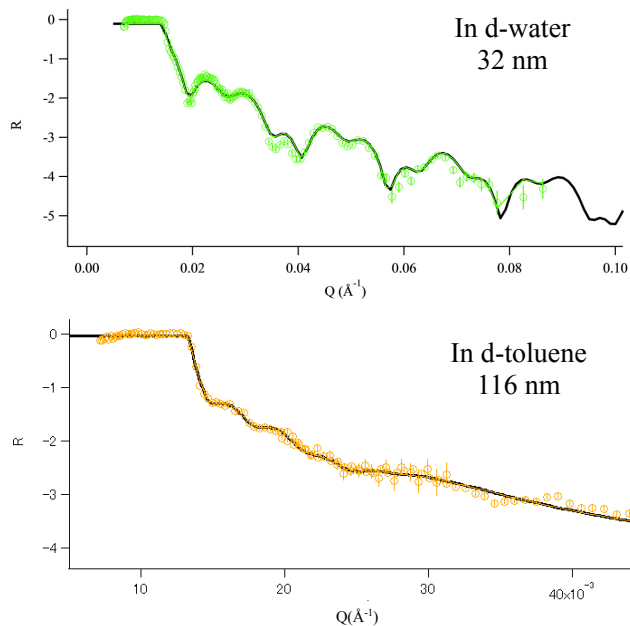


Fig. 1 Reflectivity profiles of PMMA blush in solvent

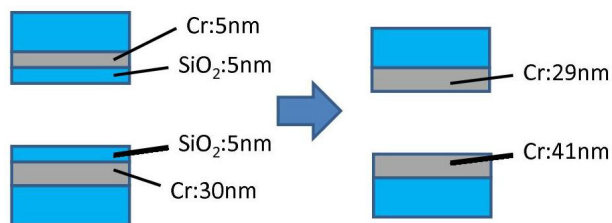


Fig. 2 Construction of SiO₂ blocks

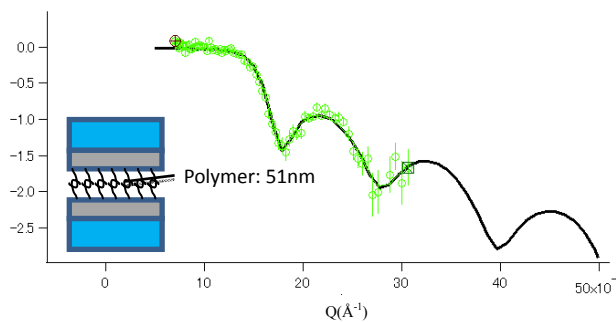


Fig. 3 Reflectivity profile from the polymer blushes sandwiched by SiO₂ blocks