
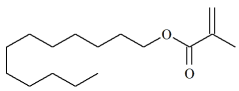
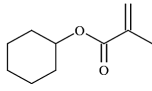


(※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。)

 <b>MLF Experimental Report</b>	提出日 Date of Report 8 August 2014
課題番号 Project No. 2013B0264 実験課題名 Title of experiment Structural Analysis of Polymer Brush Layer Sandwiched between Two Surfaces by Means of Neutron Reflectometry – Second Trial for Various Clearance 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.) BL16 SOFIA 実施日 Date of Experiment 28-31 March 2014

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.

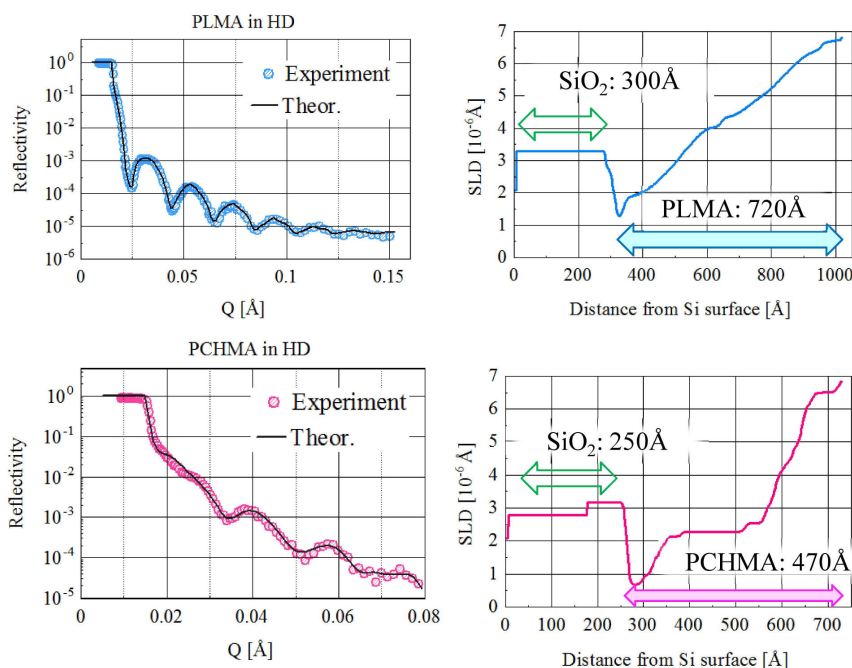
<p>1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.</p> <p>Concentrated polymer brush is one of promising materials grafted onto surfaces to reduce sliding friction drastically. Some reports say that the swollen brush in solvent supports load by osmotic pressure, but there are still rooms for discussion about the structure of polymer brushes between two surfaces.</p> <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 SiO<sub>2</sub> blocks was 10×10 mm, the dry thickness of the polymer brush layer was about 50-100 nm, and the target clearance between two surfaces was about 100-500 nm. The polymer brush on one side was grafted from the deuterated monomers for clear contrast for neutron.</p> <p>In this beamtime, we checked the structures of two kinds of polymer brushes, poly-lauryl methacrylate (PLMA) and poly-cyclohexyl methacrylate (PCHMA) shown in Fig. 1, in deuterated hexadecane as a model of base oil first. The obtained results were discussed with their friction coefficients previously measured in base oil.</p> <div style="text-align: right;">  <p>(a) LMA</p>  <p>(b) CHMA</p> </div> <p><b>Fig.1</b> Monomer structure</p>
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<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。)</p> <p>Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.</p> <p>First, we checked the thickness of the PLMA and PCHMA brushes grafted onto SiO<sub>2</sub> substrate in deuterated hexadecane as a model of base oil. The obtained reflectivity profiles and the SLD profiles analyzed through the fitting procedure were shown in Fig. 2. The PLMA brush thickness in hexadecane was about double of its dry thickness, while the PCHMA brush thickness in hexadecane was almost same as its dry thickness. It proved that hexadecane was a good solvent for PLMA, but it was a poor solvent for PCHMA. The swelling ratios, that is, the ratio of swollen thickness against full brush length were 56% and 41% for PLMA and PCHMA, respectively.</p>
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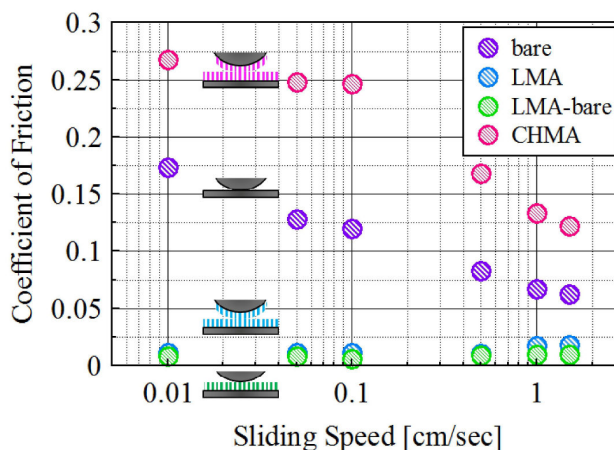
## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

We performed the friction tests for PLMA and PCHMA brushes in base oil by using a ball-on-disk nanotribometer (CSM Instruments). The friction coefficient of PLMA in base oil was always less than 0.01, while the coefficient of PCHMA was over 0.2 when the sliding speed was low, as shown in Fig. 3. This big difference is quite interesting – these experimental results proved that the brush must be swollen enough in solvent to exert their ultra-low friction property.

In addition, we performed pretest for the structural analysis of swollen polymer brushes sandwiched between two surfaces to check the operation, stability, and specifications of the special sample holder. We confirmed its stable operation, but we found that there were some points to be modified before the next beamtime.



**Fig.2** Reflectivity profiles (left) and SLD profiles analyzed through fitting operation (right); PLMA (top) and PCHMA (bottom)



**Fig.3** Friction coefficients of PLMA and PCHMA brushes