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	承認日 Date of Approval 2015/9/15 承認者 Approver J. Suzuki 提出日 Date of Report 2015/6/24
課題番号 Project No. 2014B0081 実験課題名 Title of experiment Structure Analysis of Amphiphilic Phase-separated Gels of Poly(dimethyl siloxane) Based Copolymer in Water and Methanol Mixture 実験責任者名 Name of principal investigator Katsuhiko Yamamoto 所属 Affiliation Nagoya Institute of Technology	装置責任者 Name of Instrument scientist Jun-ichi Suzuki 装置名 Name of Instrument/(BL No.) TAIKAN (BL15) 実施日 Date of Experiment 2015/4/28-2015/4/30

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. Poly(dimethyl siloxane)-co-poly(<i>N,N</i> -dimethyl acrylamide) swollen with D ₂ O and/or methanol-d ₄ -(SiO(CH ₃) ₂)-co-(C ₅ H ₉ NO)-

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. Polymer gel samples were synthesized by radical polymerization of poly(dimethyl siloxane)- α,ω -diacrylate with <i>N,N</i> -dimethyl acrylamide. The obtained materials have a bicontinuous phase separated structure composed of hydrophobic PDMS and hydrophilic poly(<i>N,N</i> -dimethyl acrylamide) PDMAA domains. The samples were immersed in deuterated solvents (water, methanol, and their mixture). Small angle neutron scattering (SANS) of the swollen (equilibrium) samples were conducted at BL15 (TAIKAN). Also small angle X-ray scattering of the sample were measured at SPring-8 and Photon Factory. Figure 1 (left) shows SAXS profiles of the swollen samples. The profiles are dramatically varied with an increase in methanol contents in solvents. The swelling ratio of the samples depends on the mixture ratio of water/methanol. Additionally, it was confirmed that polar solvents never swells hydrophobic PDMS domain but hydrophilic PDMAA domain. The electron density of PDMS, PDMAA, water and methanol was 0.518, 0.600, 0.555 and 0.445, respectively. When solvents are incorporated in hydrophilic domain, the electron density of the hydrophilic domain decreases. Therefore, the electron density profile of the samples changed with an increase in
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

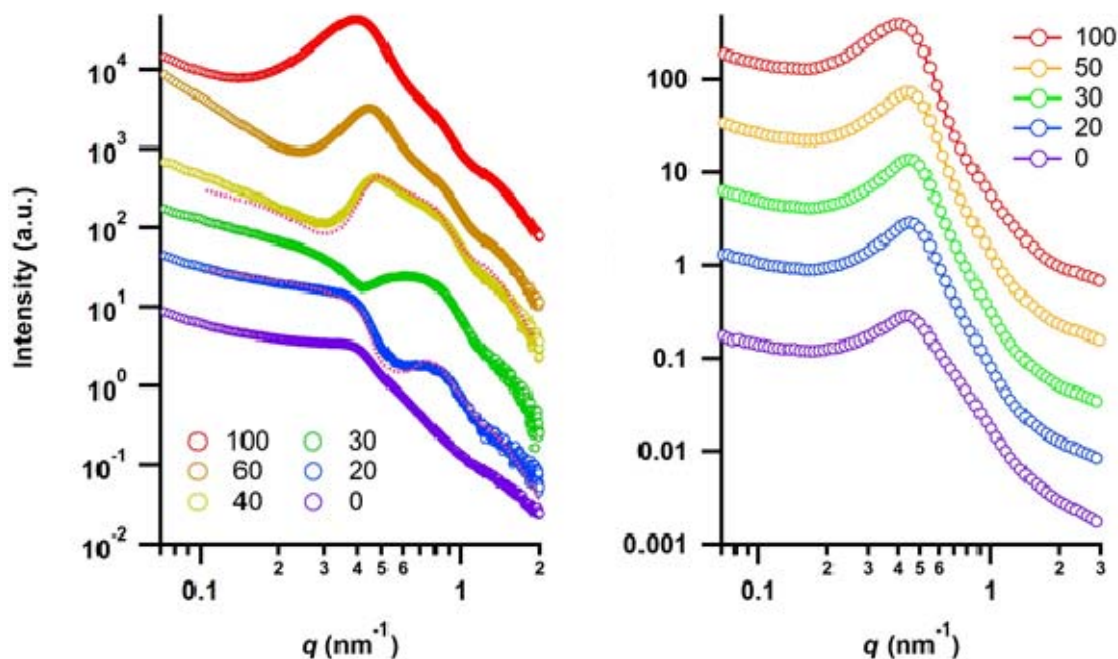


Figure 1. SAXS (left) and SANS (right) profiles of the swollen samples. Numerical numbers indicate the water weight fraction in mixture solvent. Dotted lines were obtained by calculation.

methanol. Moreover, the change in the profile depends on not only electron density but structure variation due to swelling. Here, in comparison SAXS with SANS with the same swelling ratio. There is nothing difference in structure between them but in contrast, *i.e.*, the electron density for SAXS and scattering length density for SANS. Figure 1 (right) shows the SANS profile of the swollen samples. In contrast to SAXS, the SANS profiles almost the same even if water fraction in solvent changed.

The SAXS profiles can be explained assuming that the hydrophobic domain formed cylindrical domain wrapped with a skin layer (pure hydrophilic PDMAA) cylindrical domains and hydrophilic region composed of PDMAA and solvents (core-shell cylindrical model) as shown partially with dotted lines in Figure 1. The hydrophobic PDMS domain was surrounded by a solvent-poor layer composed of PDMAA in polar solvents. Using the same structural parameter, we can fit the both SAXS and SANS profiles of the sample swelled with water as shown in Figure 2. Other SANS profiles can be fitted by the same procedure. We will validate the phase separated structure in the gel samples.

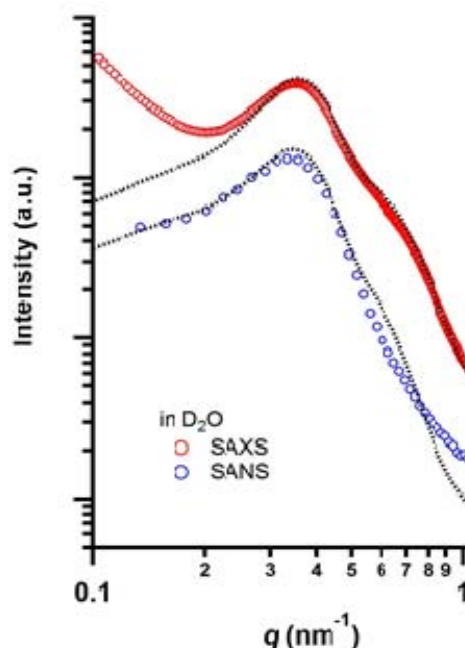


Figure 2. SAXS and SANS profiles of the sample swelled with deuterated water. Dotted lines are obtained by calculation.