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	承認日 Date of Approval 2013/12/7 承認者 Approver Jun-ichi SUZUKI 提出日 Date of Report 2013/12/04
課題番号 Project No. 2013A0005 実験課題名 Title of experiment Observation of amorphous hydrophobic crosslinked polyacrylonitrile gelation process 実験責任者名 Name of principal investigator Go Matsuba 所属 Affiliation Yamagata University	装置責任者 Name of responsible person Jun-ichi SUZUKI 装置名 Name of Instrument/(BL No.) BL15 実施日 Date of Experiment 4/29-5/1

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. Sample: Polyacrylonitrile (PAN) (synthetic, organic polymer resin) with the linear formula (C <sub>3</sub> H <sub>3</sub> N) <sub>n</sub> . Solvent: Deuterated dimethyl sulfoxide (d-DMSO)/Deuterated Water (D <sub>2</sub> O).
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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. We carried out time-resolved small-angle and wide-angle neutron scattering measurements to clarify structural formation process in nm-scale of PAN gel with hydrophobic cross-links in the atactic-PAN(a-PAN)/deuterated DMSO (dDMSO)/deuterated water (D <sub>2</sub> O) system. The neutron beam could transmit even dDMSO solvents, therefore we could observe nm-scale structure, especially atactic PAN network with small angle neutron scattering (SANS) measurements. Figure 1 shows that the SANS curves in various solvent conditions. The scattering intensity in low Q-region increases with the concentration of D <sub>2</sub> O. Below 6wt% (D <sub>2</sub> O/DMSO = 6/94 by weight), the scattering curve looks constant, then the gelation processes were prevented. However, after gelation, the scattering curve strongly decreases with scattering vector Q.
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## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

Figure 2 shows that the time dependence of SANS curves in the D2O 9wt% condition. The scattering intensity in low Q region increases with time. The density fluctuations have a size of 10 ~ 80 nm. These density fluctuations grow with time. For more detailed analysis, we evaluated the size of density fluctuations with Ornstein-Zernike Plot. Figure 3 shows that time evolution of correlation length of PAN gel. Before gelation time (36 min), the correlation length increases with time. Around the gelation time, the correlation length became significantly large. After gelation, it became the constant value (= 20nm). This correlation length, about 20 nm, is similar as the PAN gel in DMF/H2O system. From these results, the correlation length depends on the inter-cross linking point length.

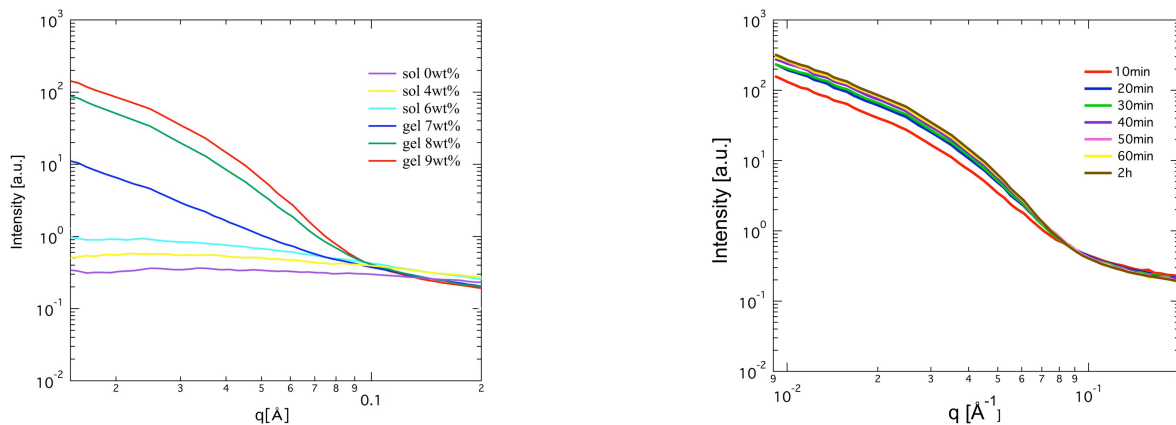


Figure 1 SANS intensity in various D2O conditions.

Figure 2 Time evolution of SANS profile in D2O 9wt %.

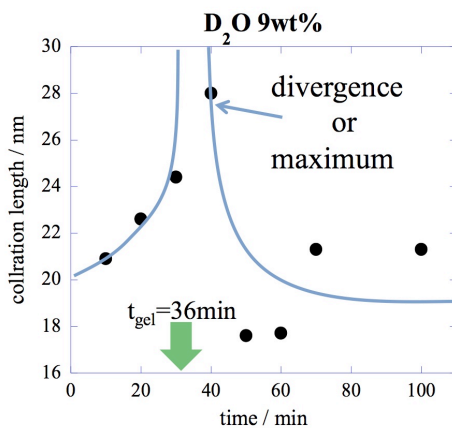


Figure 3 Time evolution of correlation length form SANS profile in D2O 9wt % (Figure 2).