

**Morphological Characterization and Analysis of Ion-  
Containing Polymers Using Small Angle X-ray Scattering**

Mingqiang Zhang

Dissertation submitted to the faculty of the Virginia Polytechnic Institute and State  
University in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

In

Chemistry

Robert B. Moore, Chair  
Timothy E. Long  
Hervé Marand  
Judy S. Riffle

October, 2014  
Blacksburg, Virginia

Keywords: perfluorosulfonic acid ionomer, Nafion<sup>®</sup>, ionomer, proton exchange  
membrane, block copolymer, morphology, small angle X-ray scattering, solution  
procedure

**Figure 1.3.** Yarusso-Copper's modified hard-sphere model (Adapted with permission from *Macromolecules* **1983**, *16*, 1871. Copyright, The American Chemistry Society, 1983)



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**Title:** Microstructure of ionomers:  
interpretation of small-angle x-  
ray scattering data

**Author:** David J. Yarusso and Stuart L.  
Cooper

**Publication:** Macromolecules

**Publisher:** American Chemical Society

**Date:** Dec 1, 1983

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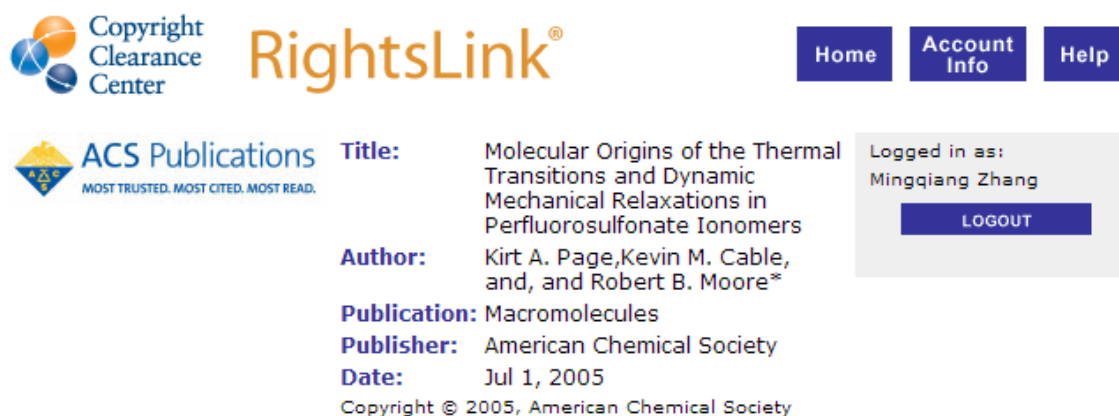
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**Figure 2.6.** Small-angle X-ray scattering (SAXS) profiles of TMA<sup>+</sup> (A) and TBA<sup>+</sup>-Nafion (B) subjected to thermal annealing at 100 and 200 °C for 10 min. Each plot contains two dimensional SAXS images before (left) and after (right) thermal annealing at 200 °C (Reprinted with permission from *Polymer* **2009**, 50, 5720. Copyright, Elsevier, 2009)



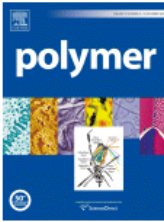
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**Author:** Jong Keun Park, Justin Spano, Robert B. Moore, Sungsool Wi

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**Publisher:** Elsevier

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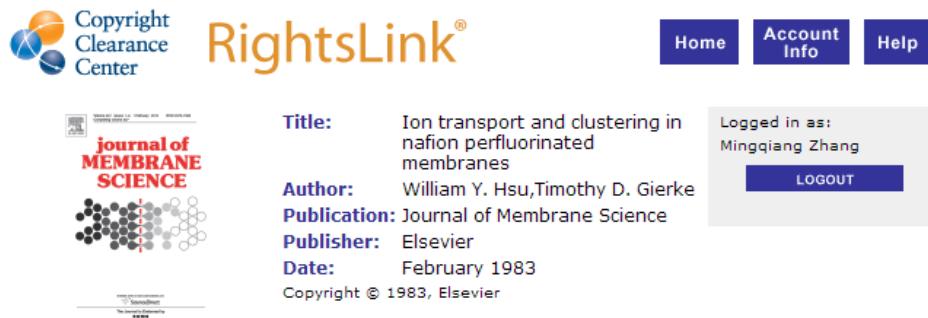
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**Figure 2.7.** Cluster-network model for Nafion membranes developed by Gierke and co-workers (Adapted with permission from the *Journal of Membrane Science* **1983**, 13, 307. Copyright, Elsevier, 1983)



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**Figure 2.8.** Core-shell Model developed by Fujimura (Adapted with permission from *Macromolecules* **1981**, *14*, 1309. Copyright, The American Chemical Society, 1981)



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**Title:** Small-angle x-ray scattering study of perfluorinated ionomer membranes. 1. Origin of two scattering maxima

**Author:** Mineo Fujimura, Takeji Hashimoto, and Hiromichi Kawai

**Publication:** Macromolecules

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**Date:** Sep 1, 1981

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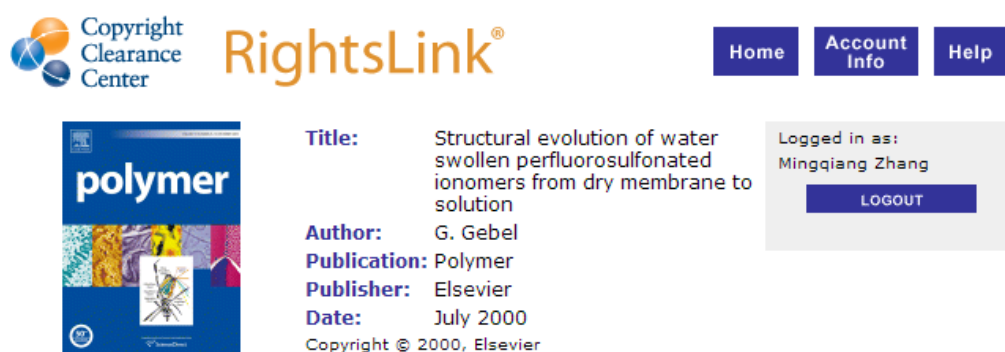
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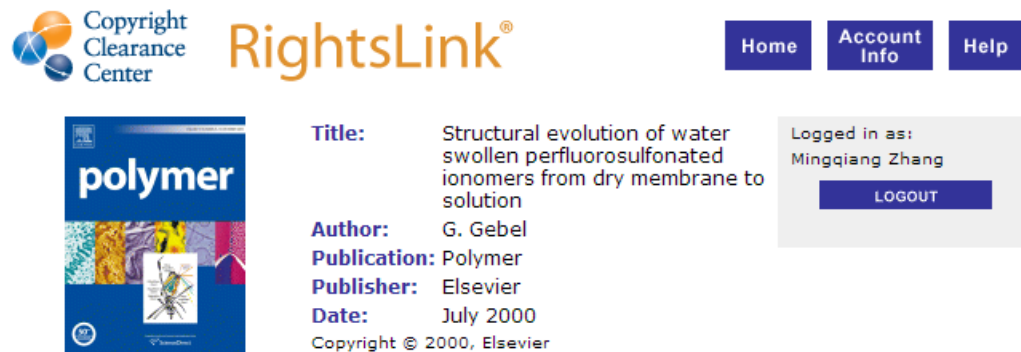
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- Publication:** Macromolecules
- Publisher:** American Chemical Society
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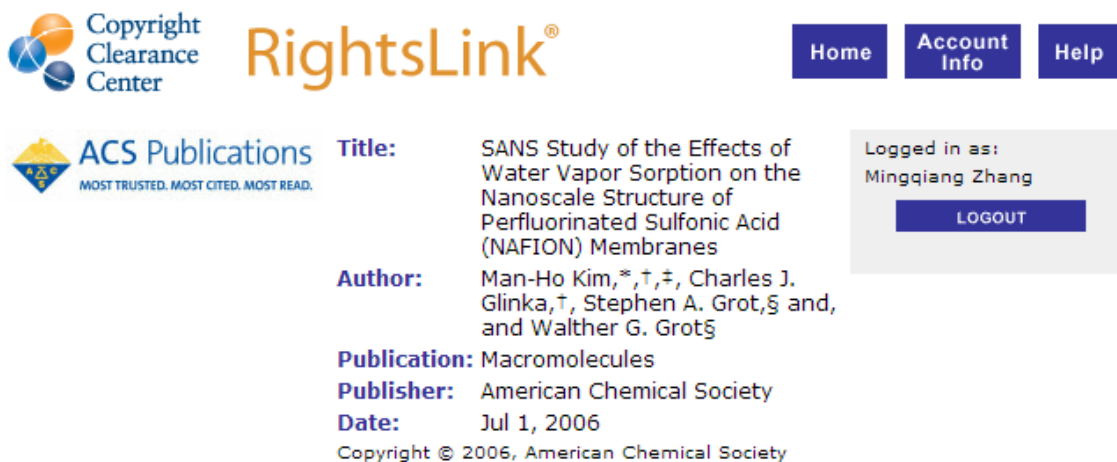
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**Author:** Man-Ho Kim,<sup>\*</sup>,<sup>†</sup>,<sup>‡</sup>, Charles J. Glinka,<sup>†</sup>, Stephen A. Grot,<sup>§</sup> and, and Walther G. Grot<sup>§</sup>

**Publication:** Macromolecules

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**Date:** Jul 1, 2006

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**Title:** Parallel cylindrical water nanochannels in Nafion fuel-cell membranes

**Author:** Klaus Schmidt-Rohr and Qiang Chen

**Publication:** Nature Materials

**Publisher:** Nature Publishing Group

**Date:** Jan 1, 2008

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**Publication:** Soft Matter

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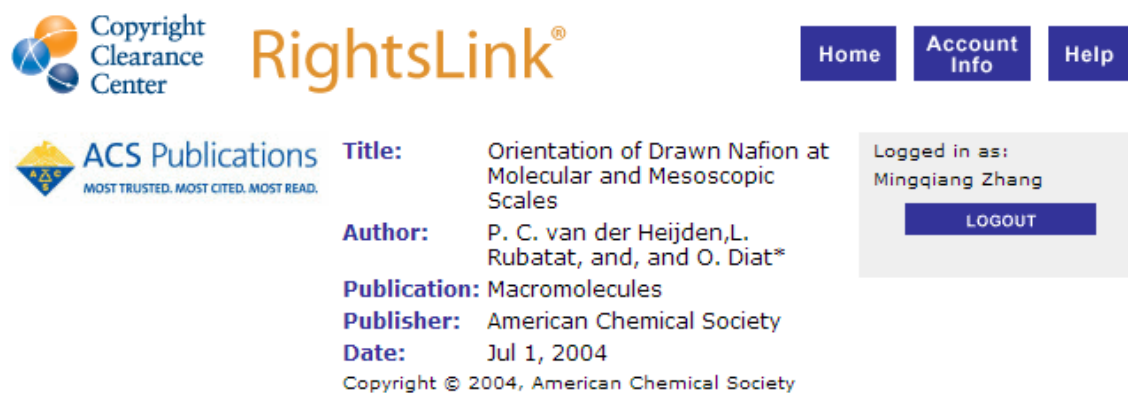
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**Figure 2.17.** Sketch of fibrillar structure of Nafion proposed by Heijden and co-workers (Reprinted with permission from *Macromolecules* **2004**, *37*, 5327. Copyright, The American Chemistry Society, 2004)



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**Publication:** *Macromolecules*

**Publisher:** American Chemical Society

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**Publication:** Macromolecules

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**Figure 8.3.** Common lattice structure. (a) Lamellae; (b) hexagonal packed cylinders; (c) BCC; (d) FCC; (e) hexagonal close-packed spheres; (f) primitive cubic; (g) double gyroid; (h) double diamond (DD); (i) Pm3n; and (j) Fddd (Reprinted with permission from Burger, C.; Hsiao, B. S.; Chu, B. In *Polymer Science: A Comprehensive Reference*; Matyjaszewski, K., Mödler, M., Eds.; Elsevier: Amsterdam, 2012, p 363. Copyright, Elsevier, 2012)

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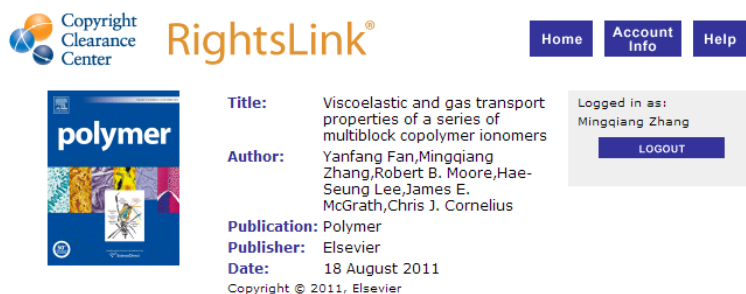
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**Figure 9.3.** SAXS patterns as a function of the applied magnetic field strength. The magnetic field direction is horizontal with respect to the orientation of the X-ray detector. The curves show the peak intensities and peak shapes of the microdomain scattering at  $q=0.07 \text{ \AA}^{-1}$  (triangles) and scattering at  $q=0.18 \text{ \AA}^{-1}$  (circles), respectively as a function of field strength (Reprinted with permission from *Journal of the American Chemical Society* 2010, 132, 17516. Copyright, The American Chemistry Society, 2010)

**Figure 9.4.** The average conductivity of 120:1 EO:Li<sup>+</sup> sample aligned in 5 T magnetic field in two directions under room temperature, with conductivity of nonaligned sample shown for comparison (Reprinted with permission from *Journal of the American Chemical Society* 2010, 132, 17516. Copyright, The American Chemistry Society, 2010)

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ACS Publications Title: Anisotropic Ionic Conductivity in Block Copolymer Membranes by Magnetic Field Alignment  
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