

Abstract Submitted  
for the MAR07 Meeting of  
The American Physical Society

**Self-Organization and Chain-Folding in Hybrid Coil-Coil-Cube Triblock Oligomers of Polyethylene-*b*-Poly(ethylene oxide)-*b*-Polyhedral Oligomeric Silsesquioxane (POSS)<sup>1</sup>** JIANJUN MIAO, LI CUI, LEI ZHU, Institute of Material Science and Department of Chemical, Materials and Biomolecular Engineering, University of Connecticut, Storrs, CT 06269-3136 — In this work, the crystallization and self-assembly behaviors of well-defined triblock oligomers polyethylene-*b*-poly(ethylene oxide)-*b*-polyhedral oligomeric silsesquioxane (POSS) (PE-*b*-PEO-*b*-POSS) were studied. The samples were characterized by differential scanning calorimetry, synchrotron small angle X-ray scattering (SAXS), and wide angle X-ray diffraction (WAXD). The orientations of PE and POSS crystals in a shear-oriented sample were determined by 2D WAXD and SAXS. The results also suggest that POSS molecules form an ABCA-stacked four-layer lamellar (trigonal) crystal sandwiched by two PE-PEO layers. The solution-cast sample shows a long period of 13.37 nm, corresponding to an extended chain conformation in the PE crystals. When grown from the melt, the *d*-spacing decreases to 10.13 nm, indicative of once-folded chain conformation for the PE block.

<sup>1</sup>This work was supported by NSF CAREER award DMR-0348724, DuPont Young Professor Grant, and 3M Nontenured Faculty Award.

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Date submitted: 02 Dec 2006

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