Internal Morphology of Diblock Copolymer Brushes Determined by Neutron Reflectivity. BULENT AKGUN, WILLIAM J. BRITTAİN, MARK D. FOSTER, Maurice Morton Institute of Polymer Science, The University of Akron, Akron, OH 44325, CHARLES F. MAJKRZAK, National Institute of Standards and Technology, Gaithersburg, Maryland 20899 — Although diblock copolymer brushes (BCB) have been extensively studied in recent years, their internal structure is still unknown. To elucidate the interface width and internal structure of BCBs, neutron reflectivity (NR) has been used. BCBs of deuterated polystyrene (dPS) and poly(methyl acrylate) (PMA) with dPS adjacent to the surface (d-PS-b-PMA) or with PMA adjacent to the surface (PMA-b-dPS) and having different thicknesses were synthesized using atom transfer radical polymerization. In ultra-thin BCBs a gradient in composition perpendicular to the surface extends essentially through the entire thickness of the brush. The interface width, defined as the full-width at half-maximum of a Gaussian function needed to represent the broadening of the step interface profile, is found to be smaller for PMA-b-dPS than for dPS-b-PMA brushes. The interface width for a film spun from untethered chains of dPS-b-PMA has been measured and the results compared with those for the BCBs. BCBs have been measured in both good solvent and poor solvent vapors with NR and the concentration profiles and extent of swelling determined.