Supplementary material

Sub-Nanometer Expansions of Redox Responsive Polymer Films Monitored by Imaging Ellipsometry

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Surface Characterization

Traces of Fe is not seen from the XPS analysis of the characterization sample of MCU. The characterization sample is prepared by first immersing the gold substrate into the MCU solution overnight to form the thiol layer on the whole area of the gold surface. This sample is then washed with the same amount of ES-PFS solution and THF/ethanol as in the micro-patterned ES-PFS/MCU sample preparation. This means that by washing the sample with large amounts of THF removes the physisorbed ES-PFS from the MCU layer.
**Fig. S1.** XPS element spectra scan of Fe in characterization sample of MCU. See the text for the details.

The surface coverage is calculated using the cyclic voltammogram of a full ES-PFS layer shown in Fig. S2. The sample is prepared on a gold substrate by the same procedure of the ES-PFS filling of the micro-patterned ES-PFS/MCU sample. This full layer of ES-PFS is used to determine the surface area of the ES-PFS more accurately.

![Cyclic voltammogram of ES-PFS layer on gold captured at 50 mV s⁻¹ scan rate. The reference electrode is Ag/AgCl, the counter electrode is Pt and 0.1 M NaClO₄ is used as an electrolyte.](image)

**Fig. S2.** Cyclic voltammogram of ES-PFS layer on gold captured at 50 mV s⁻¹ scan rate. The reference electrode is Ag/AgCl, the counter electrode is Pt and 0.1 M NaClO₄ is used as an electrolyte.

The real time ellipsometric contrast imaging of the ES-PFS/MCU sample in situ under electrochemical contrast is also provided with a movie. The movie shows the intensity variation of the ES-PFS layer against the stationary MCU layer.