

Electronic Supplementary Information (ESI) for

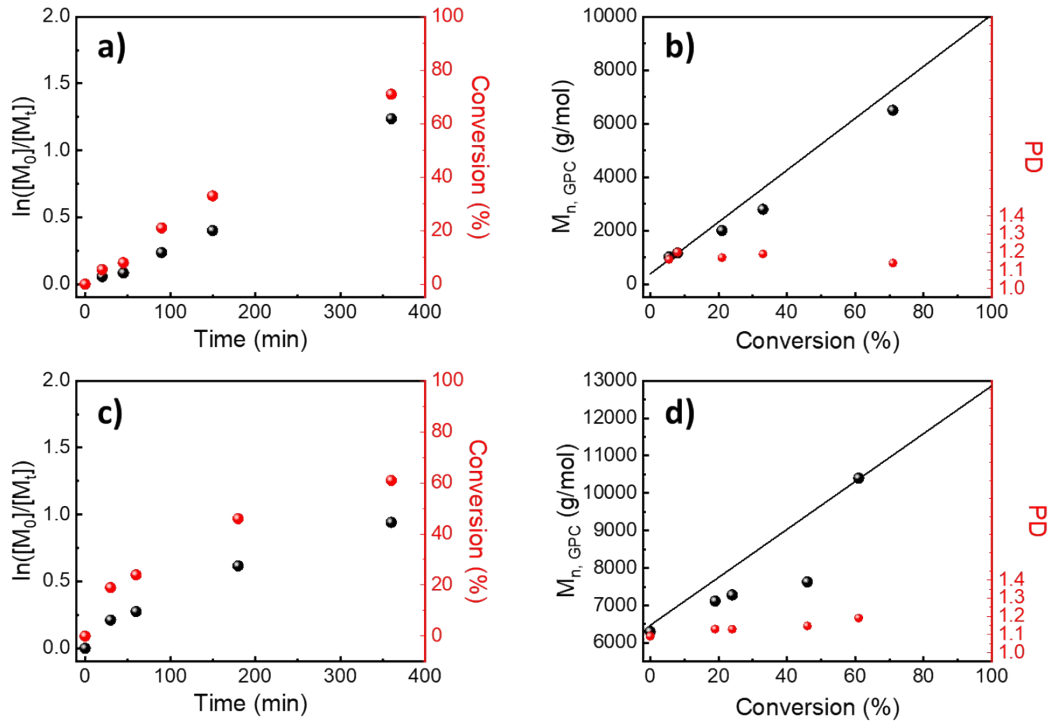
**Control over microphase separation and dielectric properties via para-fluoro thiol click reaction**

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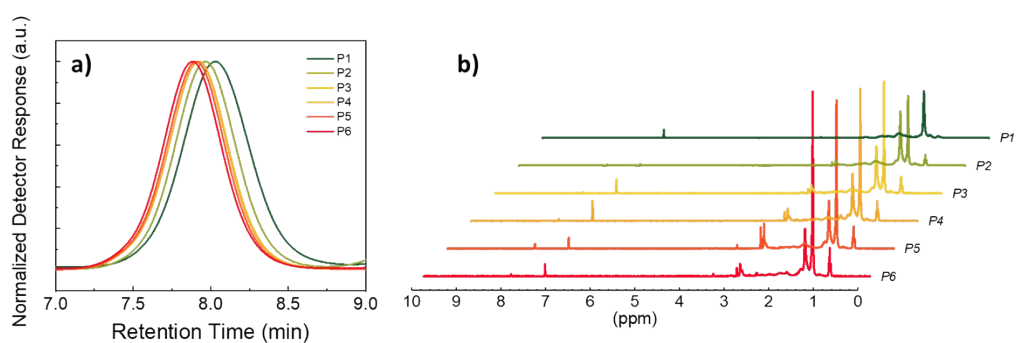
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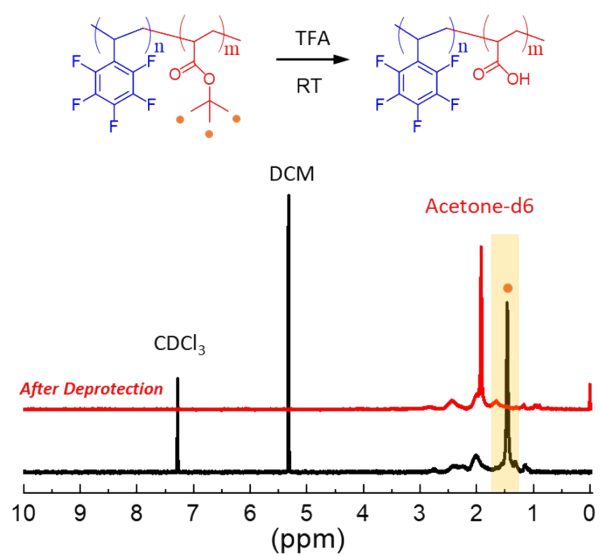
**Fig. S1.** (a, c) Semi-logarithmic kinetic plot and monomer conversion versus time data for polymerisation of 1<sup>st</sup> block (PFS) and 2<sup>nd</sup> block (tBA). (b, d)  $M_{n, GPC}$  and PD values versus monomer conversion for each block.

$$f_{PAA} = \frac{\frac{N_{PAA} \times m_{PAA}}{\rho_{AA}}}{\left(\frac{N_{PAA} \times m_{PAA}}{\rho_{AA}}\right) + \left(\frac{N_{PFS} \times (m_{PFS} + CR \times m_{thiol})}{[\rho_{PFS} \times (1 - CR)] + [\rho_{clicked} \times CR]}\right)} \quad (1)$$

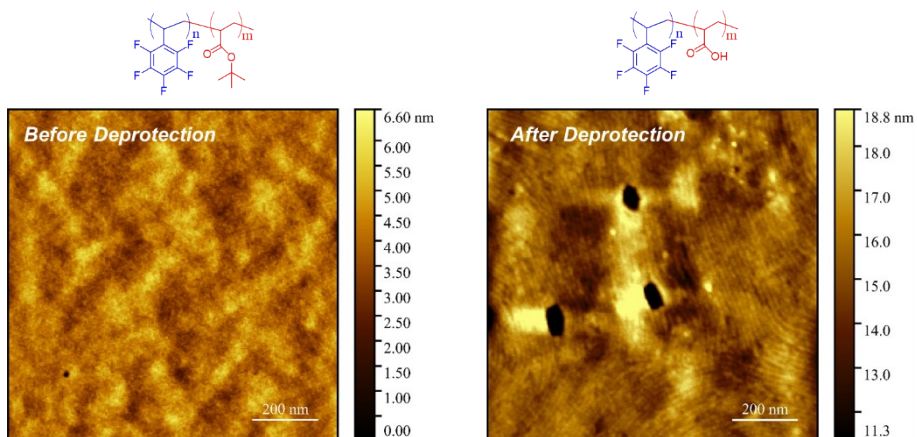
The volume fraction of blocks were calculated above formulation.  $N_{PAA}$  and  $N_{PFS}$  are the degrees of polymerisation of the AA and PFS blocks.  $m_{AA}$ ,  $m_{PFS}$ , and  $m_{thiol}$  are the molar masses of acrylic acid (72 g/mol), pentafluoro styrene (194 g/mol), and 1-dodecanethiol (202 g/mol).  $\rho_{PAA}$  and  $\rho_{PFS}$  are the densities of PAA and PFS blocks which are taken as 1.41 and 1.55 g/mL, respectively.  $\rho_{clicked}$  is the density of clicked part of PFS block and is assumed as 1.19 g/ml.  $CR$  is click ratio and varies between 0 and 0.86.



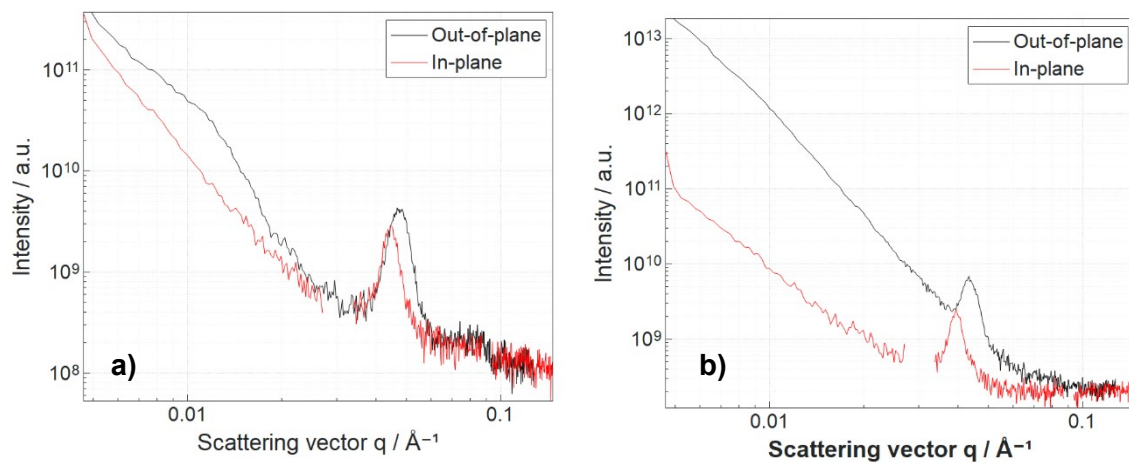
**Fig. S2.** (a) GPC traces and <sup>1</sup>H NMR spectra of P(PFS-b-tBA) for different click ratio



**Fig. S3.** <sup>1</sup>H NMR spectra of polymers before and after deprotection.



**Fig. S4.** AFM images of thin films of polymers before and after deprotection. The deprotection induces the phase separation and fingerprint-like lamellar structures appear after deprotection.



**Fig. S5.** The measured SAXS signal in the in-plane and out-of-plane directions for (a) without PFTR (P1) and (b) with PFTR (P2) samples.

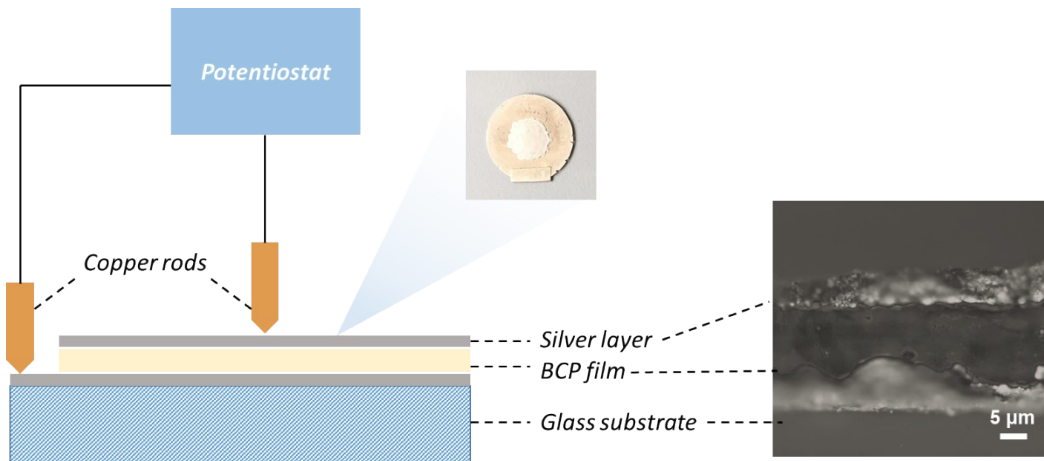
$$x = R_g^2 q^2 \quad (2)$$

$$g(f,x) = 2[f x + \exp(-f x) - 1]/x^2 \quad (3)$$

$$F(f,x) = \frac{g(1,x)}{g(f,x) g(1-f,x) - \frac{1}{4}[g(1,x) - g(f,x) - g(1-f,x)]^2} \quad (4)$$

$$S(q)^{-1} = \frac{F(f,x)}{N} - 2\chi \quad (5)$$

According to the Leibler theory, Equations 2 to 5 were used to calculate the  $\chi$  parameter of P(PFS-*b*-AA).  $R_g$  is the radius of gyration of an ideal (Gaussian) chain and  $q$  is the wave vector that is measured by SAXS.  $g(f,x)$  is the Debye function, where  $f$  is the volume fraction of any block. The dimensionless structure factor,  $S$ , is a function of  $q$  and obtained by SAXS, and  $N$  is the total number of segments.  $R_g^2$  is found to be  $64.76 \text{ nm}^2$  and  $q$  is  $0.46 \text{ nm}^{-1}$ .



**Fig. S6.** Schematic presentation connections for dielectric measurement. Cross sectional microscope image and photo of EIS samples.