Assessing the local nanomechanical properties of self-assembled block co-polymers thin films by Peak Force tapping

Matteo Lorenzoni[1], Laura Evangelio[1], Célia Nicolet[2], Christophe Navarro[2], Francesc Pérez-Murano[1]

[1]Instituto de Microelectrónica de Barcelona (IMB-CNM, CSIC) Campus de la UAB. 08193 - Bellaterra. Barcelona. Spain.

[2]Arkema France, R.N. 117, BP34- 64170 Lacq, France.

Atomic force microscopy (AFM) is widely used to image surface topography at the nanometric scale or to map the qualitative differences of local surface properties such as friction, adhesion or elastic modulus. Using PeakForce^{TM} quantitative nanomechanical mapping (QNM)[1] is possible to reliably measure Young's modulus of materials with high spatial resolution and surface sensitivity. Exploiting such technique we could map and identify the different phases of PS and block co-polymers (BCP) randomly oriented.

Thin films of block copolymers can self-assemble into ordered periodic structures at the molecular scale (5 to 50 nm) with a rich variety of nanophase-separated structures (lamellar, pillars, cylinders etc.)[2]. The AFM nano-indentation brings important information regarding the changes in mechanical properties of the different oriented domains, being able to overcome typical limitations of surface analysis (as in the case of friction maps) by probing few nm underneath the surface. In order to get reliable quantitative information the optimization of the indentation conditions become crucial.

In terms of lateral resolution, probing nanometric length structures like the ones created by the self-assembly of BCP, brings the technique to its spatial limits.

In the work presented[3,4], we define what are the optimal conditions that allow BCP ultra-thin film measurements in PeakForceTM and present the data collected on poly(styrene-n-methilmethacrylate) (PS-b-PMMA) BCPs with various pitch and orientations.

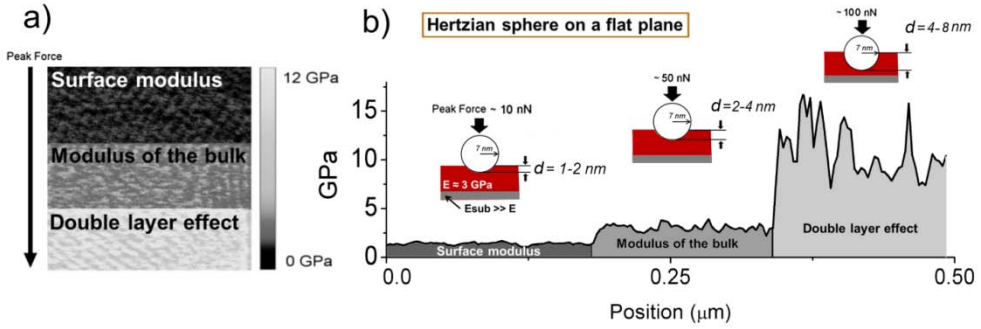

Figure 1. Reduced modulus map (a) of PS-b-PMMA BCP thin film 40 nm thick film. Three force set point (10, 50, 100 nN) are applied during the same scan in order to identify the optimal indentation conditions. Average modulus values of the whole image along the slow scan direction are plotted in (b). E related to the bulk is measured for deformation *d* between 2 and 4 nm (center part of panel b). In this case as *d* exceeds 10% of film thickness the proximity of the stiff substrate begins to influence the measure and the so called "double layer effect" becomes visible.

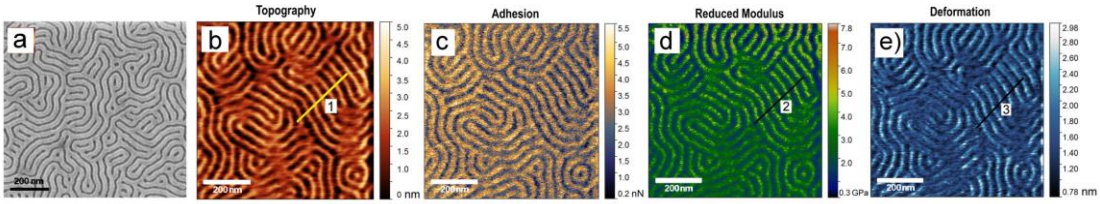

Figure 2 (a) SEM image of L37 PS-b-PMMA BCP thin film after removing PMMA by oxygen plasma. Height (b), adhesion (c), modulus (d) and deformation map of L37 BCP in the so called "fingerprint" configuration (random assembly). BCP characteristic length is 37 nm.

1. Young, T. *et al.* The use of the PeakForceTM quantitative nanomechanical mapping AFM-based method for high-resolution Young's modulus measurement of polymers. *Meas. Sci. Technol.* **125703,** (2011).

2. Abetz, V. & Simon, P. F. W. Phase behaviour and morphologies of block copolymers. *Adv. Polym. Sci.* **189,** 125–212 (2005).

3. Lorenzoni, M. *et al.* Assessing the local nanomechanical properties of self-assembled block co-polymers thin films by peak force tapping. *Langmuir* 151007141801001 (2015). doi:10.1021/acs.langmuir.5b02595

4. Lorenzoni, M. *et al.* Nanomechanical properties of solvent cast polystyrene and poly(methyl methacrylate) polymer blends and self-assembled block copolymers. *J. Micro/Nanolithography, MEMS, MOEMS* **14,** 033509 (2015).