doi:10.1017/S1431927610060204
Microsc. Microanal. 16 (Suppl 2), 2010

The Sputtering Behavior of Polymeric Materials during Focused Ion Beam Nanomachining

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The ability to examine the topographical and morphological properties of polymeric material can provide significant insight into their functional properties. Focused ion beam (FIB) nanomachining and ion induced secondary electron imaging (ISE) is gaining acceptance as the method of choice to accomplish this [1]. The development of optimized nanomachining methodologies is essential to the continued expansion of FIB technology to the myriad of polymeric materials of technological importance. Recently, a FIB in situ technique was developed for revealing and imaging the cross sectional morphology of the polymeric components in a bicomponent polymeric fiber with the island-in-the-sea (I/S) structure [2]. This technique exploits the topographical contrast generated as a result of differential sputtering. When combined with the high surface specificity and high signal-to-noise ratio obtained using Ga^+ ISE imaging, the capability of FIB to provide a useful approach for efficient characterization of the cross-sectional morphology of bicomponent polymeric fibers without the necessity of staining or other sample preparation required for previously employed imaging techniques was demonstrated.

While the utility of FIB nanomachining and ISE imaging was demonstrated for two specific bicomponent polymer systems, harnessing the full potential of nanomachining and differential sputtering to reveal morphology hinges on the ability to optimize FIB sputtering conditions. Sputtering of polymers is likely a complex process dependent on the molecular structure, thermal sensitivity, and chemical reactivity of the polymers which differs significantly from sputtering of materials such as semiconductors, ceramics and metals [3]. In polymers, material removal rate will be strongly affected by the stability of the resulting radicals and ionic fragments produced as the Ga^+ looses energy in the polymer(s). The generation of radicals and other factors resulting in the destruction of the polymer can also be affected by sputtering conditions such as Ga^+ dose rate, beam overlap and other factors. To gain a better understanding of the process of the nanomachining of polymers, dose rate, beam overlap and other factors affecting the material removal rates of selected technologically important polymers were investigated.

Five technologically important thermoplastics were investigated: linear low density polyethylene (LLDPE), polypropylene (PP), polylactic acid (PLA), polyethylene terephthalate (PET), and nylon 6 (PA6). A FEI Quanta 200 3D DualBeam FIB system (FEI Company, USA) with a 30kV Ga^+ beam was used for all sputtering experiments. Polymers used for sputter rate determination were in the form of beads from the suppliers. A hand microtome was used to obtain flat areas on the polymer beads. To determine the sputter rate of each polymer, 5µm x 5µm craters were sputtered into the respective polymer beads (see example in Figure 1) under various experimental conditions. Craters were sputtered by scanning the Ga^+ beam in a serpentine pattern at normal incidence using a range of beam currents, specifically 0.05, 0.1, 0.5, 1 and 5 nA Ga^+ with sputter time varied to maintain a constant total dose. The depths of the craters were determined by measuring the crater side wall heights using a software available on the FEI Quanta 200 3D. Sputter rates were then calculated based on the observed crater volume divided by the Ga^+ dose required to remove that volume. The sputter rates and other nanomachining effects such as topography generation were studied under varying vacuum and chemical environmental conditions. Also, the influence of the FIB beam parameters such as beam overlap, dwell time per pixel, and the total sputtering time was investigated.

References
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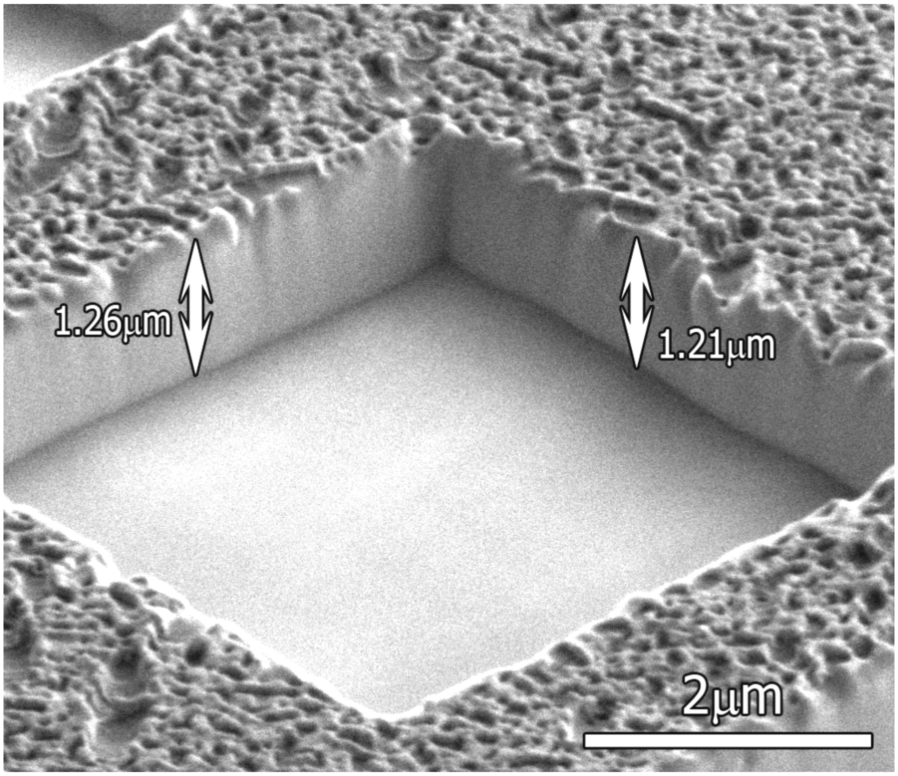

FIG. 1. An ISE micrograph of typical sputter crater (into LLDPE in this case) is shown. The crater shown was sputtered using a 30keV Ga^+ beam at normal incidence with 1nA beam current for 50 seconds. The crater depth measurements shown in the micrographs were adjusted for the 52^o ISE viewing angle.