

Nanoanalysis of Gas Assisted Ion Etching of High Density Polyethylene using FIB/SEM Platforms for the Development of Functional Surfaces

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ABSTRACT

Although soft matter, such as polymers, are very much prone to react on electron/ion beam irradiation, it is well possible to turn degradation dependent physical/chemical changes from negative to positive when materials are exposed to beams in a controlled manner. This sort of processes allow tuning of surface properties in intended directions and thus provides the use of the ultimate materials and their systems towards desired and pre-defined targets out at the micro/nano-scale for creating functional surfaces. The recent work mainly focuses on using gas injections systems (GIS) in the FIB-SEM instrument to combine the gallium beam irradiation effects with GAE to optimize, design and develop new polymer surface properties and to create functional polymer surfaces, such that those materials can serve as novel materials for many different fields of science and technology. For GAE experiments in this work, XeF₂ and I₂ gases was used in combination to gallium ion etching of High Density Polyethylene (HDPE) surfaces with different beam currents and accordingly different gas exposure times resulting at the same ion dose. The alterations in the surface morphologies due to gas assisted etching based nanostructuring with various processing parameters were tracked using HR-SEM.

Keywords: Focused Ion Beams, Beam Induced Deposition, Gas Assisted Etching, Surface Modification, Nanotechnology, Polymer Surfaces, Functionalized Surfaces

1 INTRODUCTION

This paper mainly focuses on gas assisted etching of high density polyethylene (HDPE) surfaces to obtain different surface morphologies on the defined regions. For GAE experiments in this work, XeF₂ and I₂ gases was used in combination to gallium ion etching of bulk polymeric material surfaces using different ion beam currents and related processing times. The alterations in the surface morphologies due to gas assisted etching based nanostructuring were tracked using Atomic Force Microscopy (AFM) investigations.

Functional organic materials such as polymers or small molecules are preferred for a broad range of applications owing to rapid development towards their optimized structural properties. Besides that, the shrinkage in the dimensions of the novel materials, structures and systems down to atomic scale as an output of nanotechnology [1,2] has assigned Electron Microscopy as the inseparable part of this field [3-5]. However, it should be noted that irradiation damage, caused by the use of beams in the electron microscopes leads to undesired physical/chemical material property changes or uncontrollable modification of structures that are being processed. Nevertheless, it is well possible to turn degradation dependent physical/chemical changes from negative to positive use when materials are intentionally exposed to beams. Especially, controllable surface modification allows tuning of surface properties in intended directions and thus provides the use of the ultimate materials and their systems towards desired and pre-defined concepts. Most importantly, all those mentioned tasks can be carried out at the nanoscale when electron microscopy is in charge for surface modification tasks for creating functional surfaces.

2 EXPERIMENTAL

Surface modification processes of bulk High Density Polyethylene (HDPE) samples were carried out through ion etching and gas-assisted etching (GAE) in the FIB-SEM dual-beam platforms to develop functional polymeric surfaces. The etching gases used in this study were selected as I₂ and XeF₂ which assisted gallium etching processes. The preliminary tests for the observation of micro/nano sized morphological alterations were performed using HR-SEM and Atomic Force Microscopy (AFM) techniques.

The surface of bulk HDPE materials were exposed to ion beams in square patterns via, I₂ assisted gallium etching and XeF₂ assisted gallium etching respectively. The ion energy used for gallium etching was 30 keV. For these experiments, FEI NanoLab 600 Dual-Beam platform and JEOL 4601F MultiBeam System were used. By means of complimentary analysis techniques, the effect of using different assisting gas species and

gas exposure durations for ion Also detailed AFM investigations were performed on the irradiated surfaces revealing the nano sized alterations such as topographical changes and roughness values of the treated surfaces.

3 RESULTS AND DISCUSSION

Fig. 1, shows the Secondary Electron (SE) and related AFM images, taken inside the regions on HDPE where different conditions of I₂ assisted gallium etching was applied. Here, 3 nA, 5 nA and 7 nA ion beam currents used for the GAE processes for 50x50 μm areas of irradiation, while a similar ion dose (6.292 x 10¹⁷ ions/cm²) was selected for each square with decreasing processing times of 840 s, 504 s and 360 s, respectively.

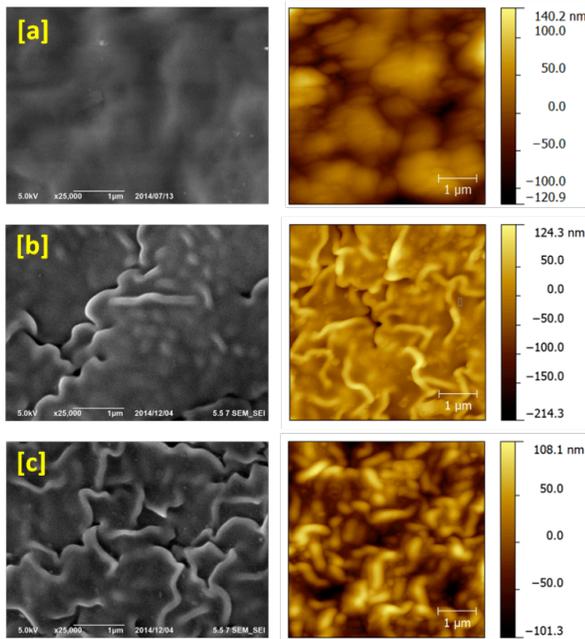


Figure 1: Iodine assisted gallium beam etched regions on bulk HDPE. The figure shows the HR-SEM and AFM images for: (a) 3 nA, (b) 5 nA and (c) 7 nA ion beam current for the irradiation process. The ion dose for all regions was set to 6.292 x 10¹⁷ ions/cm², changing the duration of the irradiation process for each region; which is the longest for 3nA and the shortest for 7 nA. Accordingly, the duration for I₂ exposure also varies.

Another series of ion beam irradiation using the same instrumental parameters such as, ion beam currents and fixed dose of ion exposure was repeated this time for XeF₂ assisted ion etching on HDPE. The correlated SEM and AFM images taken from the regions of interest are given in Fig. 2.

The roughness values measured by AFM are given in Table 1, revealing the Ra (arithmetic average of absolute values) and Rq (root mean squared-RMS) for each area of irradiation by gas assisted ion etching processes. The results

clearly demonstrate the alterations in the surface morphology of HDPE after focused ion beam etching with the use of different ion beam current and the exposure XeF₂ and I₂ gases simultaneously.

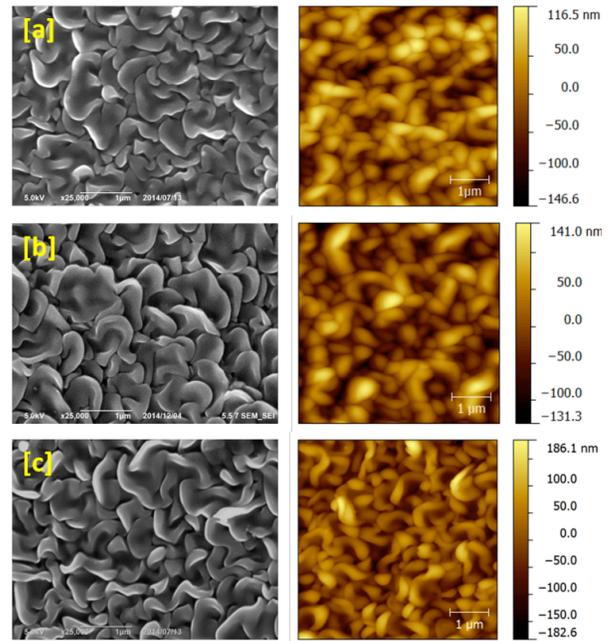


Figure 2: Xenon difluoride assisted gallium beam etched regions on bulk HDPE. The figure shows the HR-SEM and AFM images for: (a) 3 nA, (b) 5 nA and (c) 7 nA ion beam current for the irradiation process. The ion dose for all regions was set to 6.292 x 10¹⁷ ions/cm², changing the duration of the irradiation process for each region; which is the longest for 3nA and the shortest for 7 nA. Accordingly, the duration for XeF₂ exposure also varies.

Ion beam current (nA)	I ₂		XeF ₂	
	R _a (nm)	R _q (nm)	R _a (nm)	R _q (nm)
3.0	26.1	32.9	34.5	42.5
5.0	26.4	33.4	35.2	43.8
7.0	27.8	34.7	39.9	50.1

Table 1: Roughness values of the HDPE surface after gas assisted ion etching.

The overall images reveal the fundamental physical differences on the HDPE's treated surfaces, such as alterations in the texture, morphology and roughness, when the applications of different etching gasses are used during focused gallium ion-beams bombardment. This reveals the behavior of the polymer against different conditions of FIB processing to form targeted surface modification as far as developing functional surfaces is concerned. The SEM images indicate that HDPE surface form different types of texture when

exposure of different gas species combined with different conditions of ion etching are applied.

4 CONCLUSION

In this study, gas injections systems (GIS) in the FIB-SEM instrument are used to perform gas assisted etching (GAE) processing in order to optimize, design and develop new polymer surface properties and to create functional polymer surfaces with different textures. For GAE experiments in this work, XeF₂ and I₂ gases was used in combination to gallium ion etching of High Density Polyethylene (HDPE) surfaces with different beam currents and accordingly different gas exposure times resulting at the same ion dose. The alterations in the surface morphologies due to gas assisted etching based nanostructuring with various processing parameters were tracked using HR-SEM imaging and Atomic Force Microscopy (AFM) investigations.

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The images in Fig. 1 show the formation of "flake-like" surface texture due to I₂ assisted ion etching process, having more curves, with increasing ion currents. Related AFM images confirm the development of flake-like surface texture with the increase in the ion current used for the etching process. On the other hand, it is demonstrated in Fig.2 that, the use of XeF₂ for the enhancement of ion etching process caused "worm-like" surface texture on bulk HPDE, not very much varying in shape due to increased ion currents. AFM images confirm the surface morphology of XeF₂ assisted ion etched HDPE, in a good correlation with SEM images. The differences in the surface texture due to the exposure of different gas species during ion etching is presumably due to different chemical and physical interactions of the gas with the target polymeric material. When roughness values extracted from the AFM measurements are compared, it can be observed that there has been an increase in the surface roughness with increasing ion currents for all conditions of FIB etching. However, it has been detected that the average roughness values for XeF₂ assisted etched regions are higher than the ion etched regions with the exposure of I₂.

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