PHYSICAL ELECTRONICS

MOLECULAR IMAGING
OF MICRON-SCALE FEATURES
With a C_{60}^+ Primary Ion Beam

OVERVIEW

Polymer laminates have been used for decades in many industries in order to blend the attributes of individual polymer layers into a useful product. Examples of material properties that are combined in a polymer laminate include gas (e.g. O_2, H_2O) impermeability, UV protection, flexibility, puncture resistance, corrosion resistance and optical transparency. Industries that rely heavily on polymer laminates to deliver products in the global marketplace include food packaging, medical devices and electronic components, to name just a few. In this Note, we use time-of-flight SIMS (TOF-SIMS) to chemically image the micron-scale layers in a Nylon-ethylene propylene copolymer laminate cross-section. To be specific, the sample was comprised of 1-4 µm thick layers of Nylon fixed between approximately 10-15 µm thick layers of ethylene propylene (EP) copolymer. Comparison is made between the information obtained using a 30

keV Au^+ liquid metal ion gun (LMIG) primary ion beam and a 20 keV C_{60}^+ primary ion beam. The experimental data demonstrates that micron-scale features may be imaged equally well with either primary ion beam. However, the high mass ion yield afforded by the C_{60}^+ ion source allows the micron-scale features to be chemically imaged using molecular ions.

EXPERIMENTAL

An unbunched 30 keV Au^+ primary ion beam, having a DC current of 1.5 nA, was used to image the laminate cross-section. In a similar manner, an unbunched 20 keV C_{60}^+ primary ion beam having a DC current of 30 pA was used to image the laminate cross-section. A raw data stream was collected in the negative polarity with each of the primary ion beams to allow post-acquisition evaluation (i.e. retrospective analysis) of the data. A 50 µm x 50 µm area of the laminate cross-section was imaged using Au^+ primary ions, and a 75 µm x 75 µm area of the laminate cross-
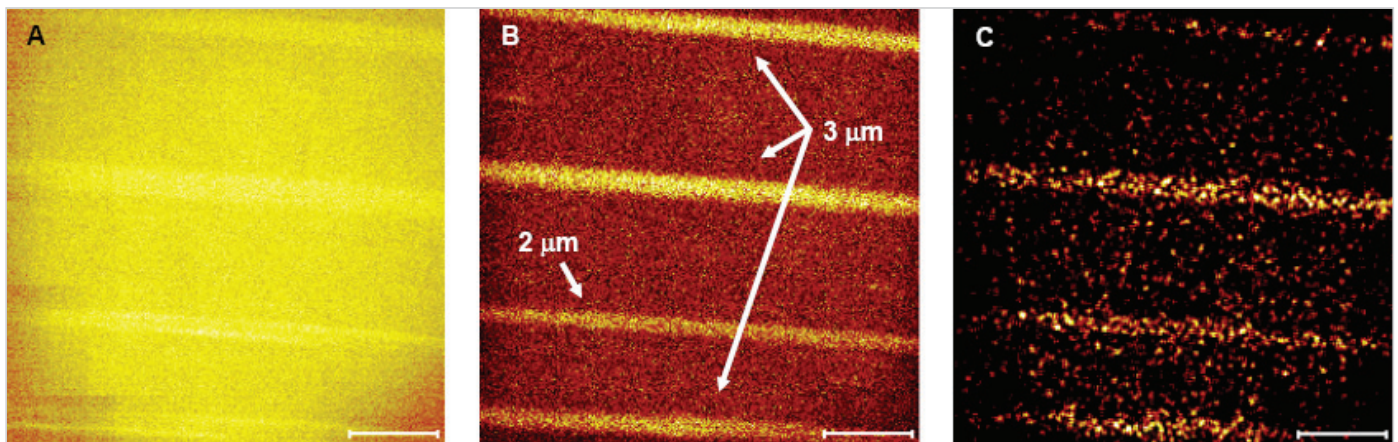

Figure 1: Negative polarity ion images of the Nylon-EP laminate cross-section acquired using an unbunched Au^+ primary ion beam. (A) Total ion. (B) O^- (16 m/z). (C) CN^- (26 m/z). The scale marker is 10 µm (50 µm field-of-view).

section was imaged using C_{60}^+ primary ions. Imaging was performed at different locations of the sample. Charge compensation was accomplished using 10 eV electrons.

RESULTS

The Nylon-EP laminate cross-section was imaged in the negative polarity using the Au^+ and the C_{60}^+ ion beams; the image data is disclosed in Figure 1 and Figure 2, respectively. Ion imaging with Au^+ primary ions allowed discrimination of the Nylon layers using the O^- and CN^- ion signals. The Nylon layers in the LMIG-acquired images are determined to be either 2 or 3 μm thick as indicated in Figure 1B. There is a significant O^- signal from within the EP matrix which may be observed in Figure 1B. The source of oxygen in this aliphatic hydrocarbon matrix may be due to the presence of an amide-based additive. This possibility is supported by the presence of CN^- signal in these same areas which may be observed
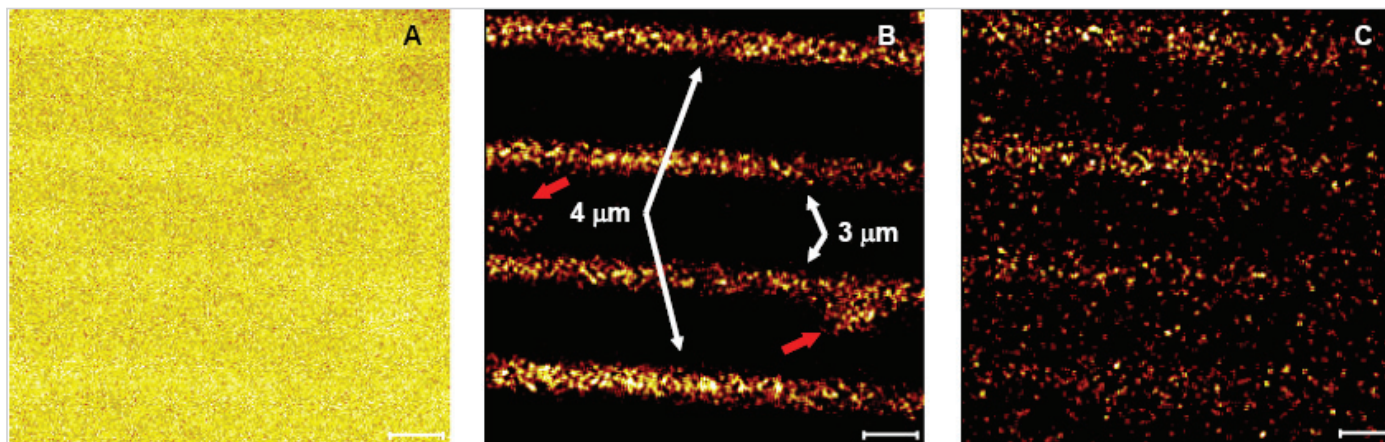

Figure 2: Negative polarity ion images of the Nylon-EP laminate cross-section acquired using an unbunched C_{60}^+ primary ion beam. (A) Total ion. (B) CN^- (26 m/z). (C) Additive molec. ion (474 m/z). The scale marker is 10 μm (75 μm field-of-view).

in Figure 1C. Ion imaging with C_{60}^+ primary ions also allowed differentiation of the Nylon layers from the EP matrix using the CN^- ion signal. As shown in Figure 2B, the Nylon layers in the C_{60}^+-acquired images are determined to be either 3 or 4 μm thick. In this ion image, the contrast was adjusted to highlight the defects that are indicated by red arrows; therefore, the CN^- signal originating from the EP matrix (i.e. between the Nylon layers) is not readily observed in this image even though it is present. A significant advantage of using the C_{60}^+ ion beam for chemical imaging is the terrific high mass and molecular ion signal enhancement. In the case of the Nylon-EP laminate cross-section, the molecular ion (474 m/z) of an amide-based additive is readily imaged with the C_{60}^+ ion beam whereas there was a negligible signal produced at 474 m/z using the Au^+ ion beam. As observed in Figure 2C, the molecular ion of the amide-based additive coincides with the Nylon layers but appears to have diffused into the surrounding EP matrix.

CONCLUSION

The use of a C_{60} cluster ion beam in TOF-SIMS analysis enables imaging of high mass and molecular ion species. Features on the micron-scale are easily imaged with a 20 keV C_{60}^+ primary ion beam. The capability to image small features, 2-4 μm thick in the case of the Nylon-EP laminate cross-section, using a high mass molecular ion was demonstrated in this Note. This capability can be a valuable asset for routine problem solving in an analytical laboratory.

ACKNOWLEGEMENTS

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