Depth Profiling of $^{13}$C Labeled Polymers using Secondary Ion Mass Spectrometry

SHANE HARTON, FRED STEVIE, HARALD ADE, NC State University — Deuterium labeling is known to change the thermodynamic properties of polymers and polymer blends (e.g. surface tension and bulk phase behavior). Anomalous segregation of dPS to an hPS:dPS/hPMMA interface has been recently observed, where the hPS:dPS blend is well within the single phase region of the phase diagram. Therefore, to probe various physical phenomena at polymer interfaces, such as chain mobility or reactive chain coupling, alternative labels must be established in order to provide a true tracer. It has been found that $^{13}$C labeling provides a true tracer for depth profiling of $^{13}$C-PS in $^{13}$C-PS:hPS and $^{13}$C-PS:hPS/hPMMA blends, with no observable segregation of $^{13}$C-PS to the $^{13}$C-PS:hPS surface or $^{13}$C-PS:hPS/hPMMA heterogeneous interface (Harton et al. Patent Pending). $^{13}$C-PS was synthesized with $\alpha,\beta-^{13}$C substituted styrene (33% v/v with 77% unlabeled styrene) using atom transfer radical polymerization with a low polydispersity ($M_w/M_n = 1.2$). A magnetic sector instrument was used for this analysis to separate $^{13}$C from $^{12}$C$^1$H. This requires a mass resolution ($m/\Delta m$) of $\sim 3000$, which cannot be achieved with a quadrupole (typical $m/\Delta m \sim 300$) thereby eliminating its use for depth profiling of $^{13}$C labeled polymers. Mass spectra of labeled and unlabeled PS were also analyzed using time-of-flight (ToF) SIMS to demonstrate the possibility for detection of high molecular weight fragments as a function of depth.

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Date submitted: 30 Nov 2005

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