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Polystyrene Freeze Dried from Dilute Solution:Ê Tg Depression and Residual Solvent[1] WEI ZHENG, SINDEE SIMON, Texas Tech University — The calorimetric glass transition temperature, Tg, was measured for both linear and cyclic polystyrenes freeze-dried from dilute solution in benzene for concentrations of 0.10%, 0.05% and 0.02% polymer by weight. Upon freeze-drying, Tg was found to be depressed by 4 to 15 K depending on the sample, solvent concentration, and freezing conditions. Annealing under vacuum (0.05 torr) at moderate temperatures, 40 to 140° C, resulted in the shifts of Tg back to its bulk value and was accompanied by a decrease in sample weight. The observed weight loss is presumably due to residual solvent. The amount of solvent present in the original freeze-dried samples was determined from the weight loss observed after annealing under vacuum (0.05 torr) for one hour at 100° C and one hour at 140° C. The calorimetric glass temperature was measured as a function of the fraction of the residual solvent. A linear correlation was found between the Tg depression and the residual solvent concentration, in agreement with data in the literature. In addition, the structure of the freeze-dried polystyrene was examined using Fourier transform infrared spectroscopy. Results show that the recovery of Tg is not due to the structure change during annealing. We conclude that the residual solvent has a significant effect on the Tg depression observed for polymers freeze-dried from dilute solution; no depression or even a slight increase in Tg was observed in the absence of residual solvent.

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