

Plasma etching technique for SEM observation of polymer blends

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Abstract

Plasma etching technique is utilized for preparing SEM specimen of polymers, and it is turned out that the plasma etching technique can be an alternative method replacing the conventional sample preparation methods. The conventional techniques including the staining and chemical etching methods, have been widely used for SEM observation, even though they have a couple of disadvantages such as the handling of the toxic chemicals and the time consuming procedures. For example, the staining method frequently needs to handle RuO₄ or OsO₄, which is very toxic, and it usually takes a couple of days due to the complex preparation steps. The chemical etching technique is also very difficult to choose a right solvent for selective etching specifically when the solubility parameters of each component are similar.

In this work, we developed a novel method to characterize the morphology of polymer and polymer blend without any chemical etching or staining process. This novel technique contains the plasma etching in the presence of carrier gas followed by the cryogenic microtoming process. Oxygen is chosen as the carrier gas in this study. After the plasma treatment, a clear contrast of the interface between polymer phases is observed because the plasma susceptibility of each polymer is different due to the different chemical structure. Morphologies of several ternary polymer blends after oxygen plasma etching are shown in Figure 1 as an example. An electrodeless plasma system (Plasma-Preen II, Plasmatic system Inc.) equipped with 2.45GHz microwave was used, and the etching conditions including etching time, oxygen concentration, and the intensity of plasma were properly controlled. This plasma etching technique was applied many other polymers and polymer blending systems, and it should be noted that we successfully obtained very clear images to investigate the morphologies, even for the cases that the chemical etching technique failed to give satisfactory contrast.

Therefore, it is believed that the plasma etching technique is a superior sample preparation technique over the chemical etching method or the staining method in the characterization of the polymer morphology.

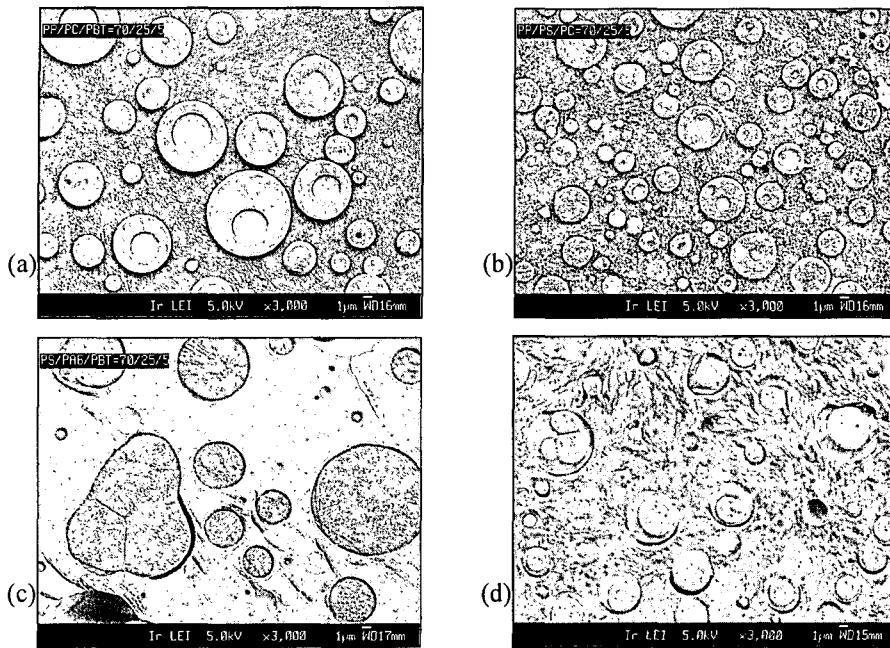


Figure 1. SEM images of various ternary blend (x3000);
(a) PP/PC/PBT (b) PP/PS/PC (c) PS/Nylon-6/PBT (d) PE/PMMA/P