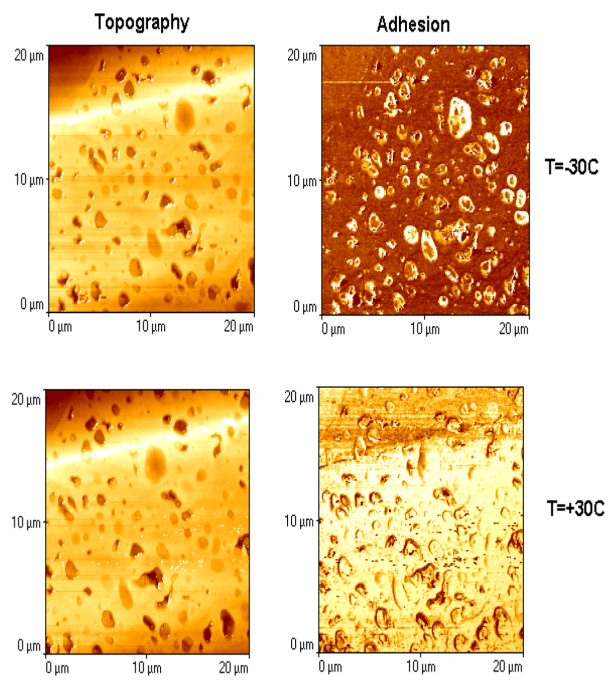
THERMAL SOLUTIONS

Two Phase Polymer System Studied by Temperature Dependent Pulsed Force Microscopy and Force versus Distance Curves

Fig.1



This note describes the study of a two-phase polymer system at different temperatures using pulsed force microscopy (PFM) and local adhesion measurements. The sample is held at various temperatures by using the temperature stage accessory of the 2990 Micro-Thermal Analyzer. In PFM a standard high resolution Si probe tip is mounted in the 2990 and used to measure stiffness and adhesion variations over surface. This is accomplished by measuring a force versus distance curve, at each point in the scan. In a force versus distance curve the probe tip is brought into contact with, indented into, and pulled-off of the surface. Relative measurements of stiffness and adhesion can be made during the indention and pull-off processes respectively. Therefore, in addition to topographic images, surface maps are collected where contrast is based upon the variation of these two physical properties.

Figure 1 shows topography and adhesion maps of a two-phase polymer system collected at two different bulk sample temperatures. The upper set was collected at a stage temperature of -30°C. Contrast between a continuous and a dispersed phase exists in both images. Interpretation of the adhesion image contrast leads to the conclusion that the continuous phase is less adhesive than the dispersed phase (darker colors correspond to less adhesion, and brighter colors to more). This is opposed to the adhesion image collected at a stage temperature of +30°C. This image displays almost no contrast between the continuous and dispersed phase. The image contrast indicates that the continuous phase has become more adhesive at this temperature. This information could not be derived from the topography images, which, except for some small drift, are identical.

Fig.2

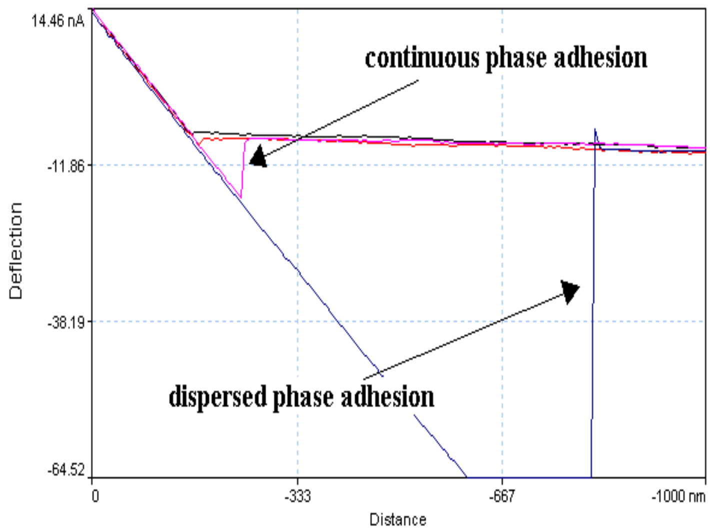
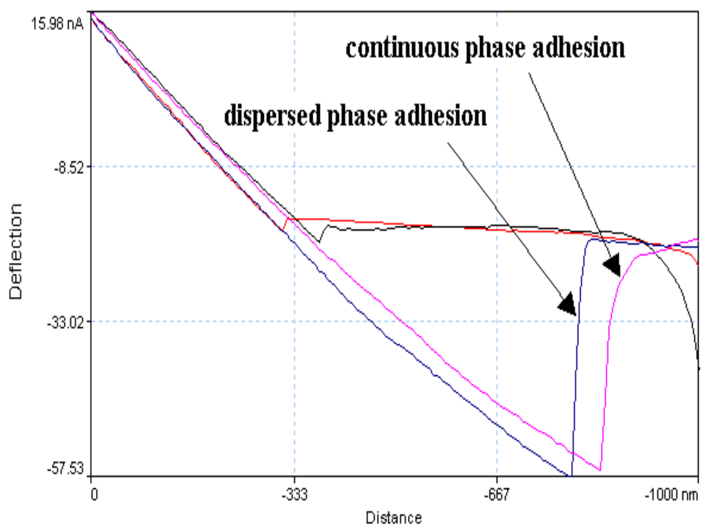


Fig.3



That the continuous phase has gone though a transition between –30 and +30°C is supported by the data shown in figures 2 and 3. These show individual force versus distance curves collected with the probe positioned at single points within the continuous and dispersed phases. Again the temperature stage is used to hold the sample at the two temperatures. These curves represent the force variations acting on the probe as it is brought into contact and pulled away from the sample surface. The arrows indicate the parts of the curves used to measure adhesion between the probe and the surface. The setup of the temperature stage allows for measurements to be made in a dry nitrogen atmosphere when there is LN_2 in the stage dewar. Thus, water vapor on the sample surface, which can induce large adhesive forces, should be minimal in these experiments. At -30°C (fig. 2) the adhesion of the dispersed phase is much greater than the continuous phase, in agreement with the images in figure 1. The spring constant of the cantilever is between 0.1 and 0.3 N/m. Taking the average value of 0.2 N/m, the adhesion force of the continuous phase at -30°C is estimated to be ~17 nN, while that of the dispersed phase is ~130 nN. At +30°C (fig. 3) there is little adhesion difference, also in agreement with the adhesion image in figure 1. Again assuming a spring constant of 0.2 N/m, the adhesion forces for both the continuous and dispersed phases are ~100 nN. This confirms that the continuous phase has increase in adhesion, while little or nothing has happened to the dispersed phase.