Structural study for Thermoplastic Elastomer Consist of Carboxyl-terminated Telechelic Poly(ethylene-butylene) and Stearylamine

Yoshiaki Takahashi and Akihiko Takada
Institute for Materials Chemistry & Engineering, Kyushu University

1. Introduction

Carboxyl-terminated telechelic poly(ethyrene-butylene) (CTPEB) and stearylamine (Octadecylamine; ODA) mixtures show a very interesting rheological properties which is quite similar to thermoplastic elastomers as mentioned below. To understand the origin of the rheological properties, it is essential to analyze the structure. Since the main repeating units of CTPEB and ODA are CH2, such study can be performed only by means of SANS measurements using deuterium-labeled samples. Unfortunately, we could not have beam time for this study but preliminary measurements are carried out during IRT work. Here we briefly report the results with rheological properties and other characteristics of the system.

2. Preparation, characterization and rheological properties of the sample

The number averaged molecular weight of CTPEB was 1400. CTPEB and ODA are dissolved in toluene and they are mixed at a condition the moles of carboxyl and amine residues become the same, then the solvent was removed in a vacuum oven. The sample thus prepared had a thermal transition peak in DSC thermogram at around 310 k. IR spectroscopy and WAXD analysis did not show any evidence of formation of large ionic complex but existence of small amount of crystalline of stearylamine at low temperature and its disappearance at elevated temperature.

Dynamic viscoelastic measurements were carried out in a temperature range of 283 ? 343 k. At low temperatures, elastomeric behavior was observed in full range of tested frequency (0.1 ? 100 rad/sec). That is, storage moduli G ' were always higher than loss moduli G "and G 'was almost constant at the order of 0.5 MPa. On the other hand, liquid like behavior similar to the behavior of pure telechelic polymer was observed at elevated temperature. Stress-strain curves obtained at 298 k with different shear rates showed that non-linear behavior starts at around 1 % strain and the maximum of stress is observed at around 100 % strain. Thus, dynamic viscoelastic properties of this system in linear region are similar to thermoplastic elastomers; that is, elastic at low temperature and liquid like at elevated temperature. However, it is a very fragile elastomer.

3. SANS measurements

Deutrated CTAB was prepared by adition reaction of deuterium gas to carboxyl-terminated polybutadiene. The SANS measurements are performed by SANS-U spectrometer at the Neutron Scattering Laboratory of the ISSP, The University of Tokyo, established at C1-2 beam line of JRR3M in JAERI (Tokai), Ibaraki, Japan. The wavelength and beam size of incident neutrons was 0.7 nm and 3 mm?, respectively. The temperature were 298 (elastic state) and to 338 k (liquid-like state).

The data at high q are almost the same irrespective of temperature. The data at low q become higher at the lower temperature, while it dropped to background level at the higher. At the lower temperature, there existed a small shoulder at around q = 1.2 (1/nm), which is close to the peak position for aluminum tri-stealyrate/decalin system studied previously. Note that aluminum tri-stealyrate/decalin system show gel-like behaviors and the peak position is very close to that of crystalline of stealyrate derivatives. Thus we speculate that stealyrate residues in CTPRB/ODA still have ordered structure inside the networked structure, though the regularity is much lower than that in the crystals. To

clarify the structure, further works are definitely needed.