CHAPTER II

EXPERIMENTAL DETAILS

2.1 Apparatus

1. Differential Scanning Calorimetry (DSC) was carried out on a Netzsch instrument, DSC 200 cell with a sealed aluminum pan, and the N_2 flow rate was 40 ml/min.

2. Dynamic mechanical analysis (DMA) was carried out on a DMA 7e, Perkin Elmer with a TAC 7/DX thermal analysis controller. An extension probe technique was used for cast films in creep scan mode using Helium purge with pressure head of 30 psi.

2.2 Materials

1. Poly(methyl methacrylate) (PMMA) : PMMA was from Shinkolite VP from Thai Petroleum Industry Co.,Ltd., its molecular weight of 75,000 was determined by a Water -150C GPC.

2. Poly(styrene-co-acrylonitrile) (SAN) : SAN was SAN25 from Polyscience. Inc., with acrylonitrile content of 25 wt %, its molecular weight of 79,000.

There is a strong exothermic interaction between SAN25 and PMMA (Akiyama and Jamieson, 1994).

2.3 Sample Preparation

Binary blends contained SAN copolymer and PMMA. Blends of composition ranging from 0 to 100 % wt % SAN were prepared by making

stock solution containing 15 % by weight of total polymer in methyl ethyl ketone (MEK). The solution were stirred at a very slow rate at room temperature. The solution was cast on the petridish glasses (diameter =10 cm). The solution was slowly allowed to evaporate at room temperature for 1 day. Final trace of the solvent were removed by annealing at 70°C for 3 days at the atmosphere pressure and then for 1 days under vacuum.

2.4   Experimental Procedure

T_g determination : Differential Scanning Calorimetry (DSC) was used to determine the glass transition temperature of each blend which is shown in Fig 3.1 The sample film was cut into fine grains. A small weighed sample of polymer (approximately 10 mg) was put in a sealed aluminum pan. For DSC measurement, the N_2 flow rate was 40 ml/min with a heating rate of 10 °C/min.

Tensile creep measurement: the dimension of the samples were rectangular: with a length of 8 mm (no more than 10 mm., DMA will detect the length automatically), a width of 3 mm, and a thickness ≈ 0.3 mm. Dynamic Mechanical Analysis (DMA 7 e) was used in the test, using the extension probe and the creep scan mode. We annealed the specimen to 30 °C above T_g for 4 minutes. After that the sample was quenched to the testing temperature which was 45 °C below T_g . The stress-strain curve was obtained at different aging time on the creep mode by setting creep ramp "on". The force rate was 500 mN/min and the final static force was 200 mN. After that the specimen was cooled to the room temperature for a period of time. The tensile creep was measured on the same specimen by annealing the specimen to 30 °C above T_g The sample was quenched to the testing temperature which was 45 °C below T_g. All creep tests were carried out with the force of 300 mN. The same procedure was applied to the five different ratios of blend (100 %

SAN : PMMA (30 : 70, 50 : 50, 70 : 30) and 100 % PMMA). The heating rate was 40 °C/min and cooling rate was 10 °C/min. At the ratio 50 : 50 the cooling rate was varied to 5 °C/min and 20 °C/min to study the effect of the cooling rate.

As pointed out above, creep measurements were carried out after the sample from a temperature above T_g to the test temperature, T_a, and then the sample was stored at this temperature for a certain time (aging time), t_a. By heating the sample above T_g all previous thermal history is erased. In this way, we can do several tests with one sample, and the possible different samples has been circumvented. The duration of creep experiment was limited to less than 10 % of the aging time.

It is very time consuming to have to heat and store the sample for very test. Struik[1978] suggests that a more convenient procedure: Once the sample is quenched and inserted in the thermostat chamber, it is subjected to a series of creep experiment. The experiment with the shortest aging time is carried out first, and subsequent experiments are performed after increasing the aging time by a factor of 2. The aging time is the total time elapsed since the quenching to the test temperature.(Struik, 1978)

We examine the applicability of the empirical equation Eq.(1.6). To determine β, all curves were fitted to Eq.(1.1) individually. For each curve, the parameter β and ε (0) were calculated. The values for β for all curve were compared, and an average value was used for furthering fitting. All curves are superimposed, and then the generated master curve is compared.

Aging shift factor thus can be obtained in two ways: We can use the t_0 values for creep curves of different t_a generated by fits to the master curve and compute the shift factor, a , as in Eq.(1.5). We can also determine a by using the superimposed curves of $\Delta\varepsilon$ (t) against log time (log t_a).