Appendix C

Volume Determination

The effects of temperature on polymer density have been determined using a technique based upon optical microscopy. A small polymer sample is placed into an Olympus BH-2 microscope with a Linkam THM 600 hot stage. Samples are ramped and held isothermally at each various temperatures for an equilibrium time of approximately 10 minutes. Olympus video calipers were used to measure the changes in length of the x and y-axes. A heating cycle and cooling cycle were both done to check for consistency. A description of the data and data analysis is given below (Table III-1,2).

Table III-1

Heating cycle	Temperature (°C)	Y-axis (microns)	X-axis (microns)
	23	453.9	655.1
	45	455.6	659.1
	55	456.3	661.6
	65	457.0	664.0
	85	458.9	665.0
	106	460.5	666.0
	126	460.5	667.0
	150	465.5	671.9
	170	470.4	673.9

Table III-2

Cooling Cycle	Temperature	Y-axis (microns)	X-axis (microns)
	170	470.4	673.9
	150	465.5	671.9
	126	460.5	667.0
	106	460.5	666.0
	85	458.9	665.0
	65	457.0	664.0
	55	456.3	661.6
	45	455.6	659.1
	23	453.9	655.1

Ratios of the change in length in going from a reference temperature to a new temperature to the initial length at the reference temperature ($\Delta l/l$) are calculated for each test temperature. An example calculation and Table III-3 of results is given below.

For 150°C:

$\Delta l/l = \Delta y/y = (465.5 - 453.9) / 453.9 = 0.025556$

$\Delta l/l = \Delta x/x = (671.9 - 655.1) / 655.1 = 0.025645$

average $\Delta l/l = 0.0256005$ (+/- 0.0000445)

Table III-3

Test temperature	Δy/y	Δy/y	Average Δl/l
25°C (reference)	0	0	0
45°C	3.7453 e-3	6.1059 e-3	4.3926 e-3
55°C	5.2875 e-3	9.8458 e-3	7.5667 e-3
65°C	6.8297 e-3	1.3586 e-2	1.0208 e-2
85°C	1.1016 e-2	1.5112 e-2	1.3064 e-2
150°C	2.5556 e-2	2.5645 e-2	2.5601 e-2

Average values for Δl/l are used to determine density of the polymeric adhesive as a function of temperature. This is done by converting a linear expansion ratio to a volumetric expansion ratio by assuming an isotropic state. The relationship for doing so is given as equation III-1.

$$\Delta V / V = \left(1 + \Delta l / l\right)^3 - 1$$ **Equation III-1**

For 150° C

$\Delta V/V = (1 + \Delta l/l)^{3} - 1$

$\Delta V/V = (1 + 2.5601\text{ e-2})^{3} - 1$

$\Delta V/V = 7.8783\text{ e-2}$

Thus, for a ΔV/V ratio of 7.8783 e-2, a unit volume of 1 cm^3 will have a final volume of 1.078783 cm^3. Hence, for a polymer of density(23°C) = 1.1206 g/cm^3, at 150°C, the density will decrease to 1.0388 g/cm^3. Densities of the polymer for each test temperature are tabulated below (Table III-4).

Table III-4

Test temperature	Average ΔV/V	Density (g/cm^3)
25°C (reference)	0	1.1206
45°C	1.4850 e-2	1.1042
55°C	2.2287 e-2	1.0955
65°C	3.0937 e-2	1.0870
85°C	3.9707 e-2	1.0778
150°C	7.8783 e-2	1.0388