CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1. N-vinyl pyrrolidone (NVP) : Fluka
2. 2-hydroxypropyl methacrylate (HPMA) : Fluka
3. Ethyleneglycol dimethacrylate (EGDMA) : Aldrich
4. Azo-(bis)-isobutyronitrile (AIBN) : Aldrich
5. Titanium dioxide : Merck
6. Acrylic polymer UT-50 : Siam Chemical
7. Acrylic polymer T-45 : Siam Chemical
8. Ammonium hydroxide : J.T.Baker
9. Silicone oil 350s : Dow-corning
10. Hydroquinone : Aldrich
11. Poly(vinyl alcohol) : Nippon Synthetic
12. Xanthan gum : Rhone-Pouleng
13. PEG-150 : PIAS

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

3.2 Instruments and Apparatus

1.Mechanical stirrer

2.Scanning electron microscope : JSM

3.Universal testing machine : Instron Corporation

4.Brookfield viscometer : DV-I+

5.Thermogravimetric analyzer : Elmer Perkin TGA7

6.Differential Scanning Calorimeter : Perkin Elmer DSC7, STREC CU

7.PH meter

3.3 Procedures

3.3.1 Preparation of Compounding Ingredients

Temporary solder mask A

All of the ingredients were mixed by mechanical stirrer under room temperature. The rate of mechanical stirrer was 400 rpm. The formulations of compounding ingredients and the appropriate condition were investigated by varying the initiator concentration, the diluent reactive monomer, polymer, releasing agent, thickening agent, pigment and inhibitor as shown in table 3.1. The preparation procedure of composite product is presented in flow diagram 3.1. The apparatus set up is shown in figure 3.1.

Typically, reactive monomer I, reactive monomer II, and initiator were mixed in the reactor with stirring at 400 RPM, room temperature and 5-10 minutes. The second step, polymer, thickening agent, releasing agent and pigment are added in the

reactor with stirring at 400 RPM, room temperature and 15 minutes. Next, it is heat in Oven at 100 °C and 5 minutes. We will obtain the finish product.

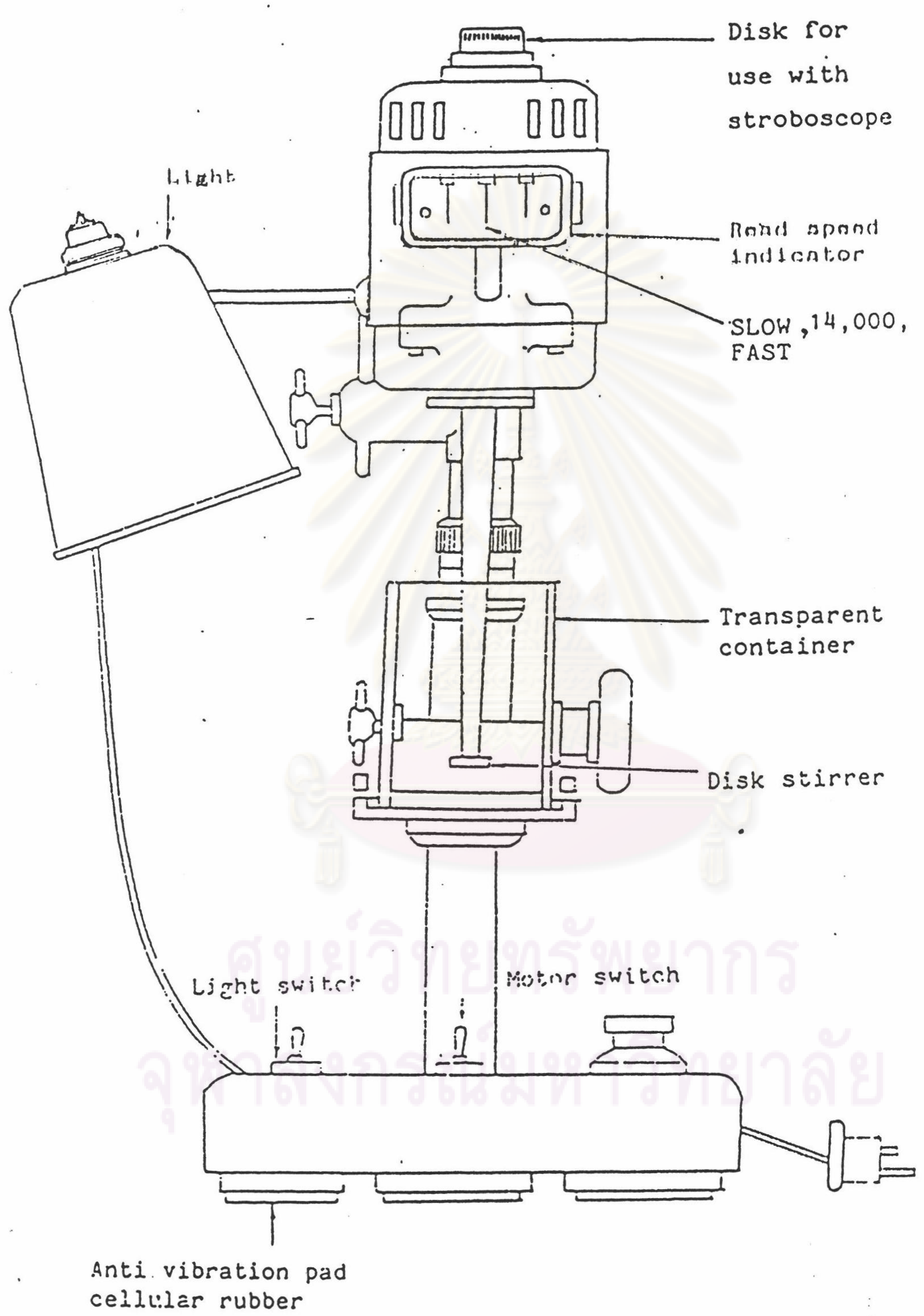

Figure 3.1 The apparatus set up

Table 3.1 The composition of solder mask A

Ingredient	Unit	Quantity
Reactive monomer I:Reactive monomer II	phr	80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 80:20
Polymer	phr.	80, 100, 120, 140
Thickening agent	phr.	2, 5, 10, 15
Releasing agent	phr.	2, 5, 10, 15
Pigment	phr.	0.1, 0.2, 0.3, 0.4
Ammonium hydroxide	%wt	1 %wt of thickening agent
Temperature	°C	100

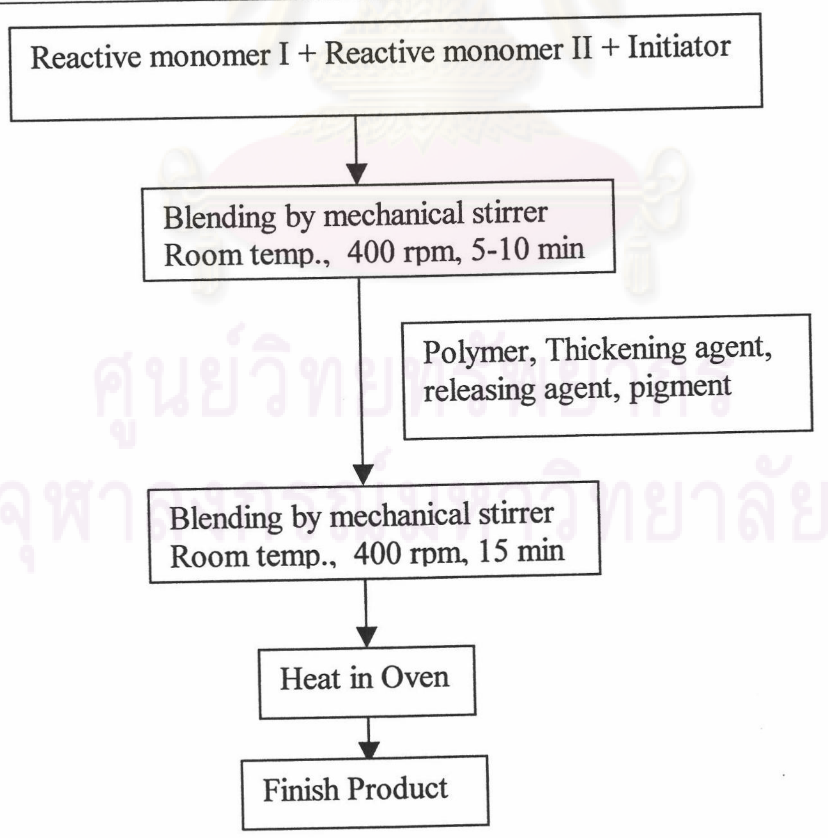

Diagram 3.1 The preparation procedure flow diagram of solder mask A

Temporary solder mask B

All of the ingredients were mixed by mechanical stirrer under room temperature. The rate of mechanical stirrer was 800 rpm. The formulations of compounding ingredients and the appropriate condition were investigated by varying the initiator concentration, the diluent reactive monomer, polymer, thickening agent, pigment and inhibitor as shown in Table 3.2. The preparation procedure summary of composite product is presented in Flow Diagram 3.2 The apparatus set up is shown in Figure 3.1.

Typically, PEG-150, xanthan gum, water were mixed in the reactor with stirring at 800 RPM and room temperature. Next, Polyvinyl alcohol is slowly added in this batch until good dispersion. Next, heat up temperature to 70-75 ^{o}C and stir until good solubility. And then cool down temperature to 35 ^{o}C. The last step, reactive monomer I, reactive monomer II and initiator are added in the reactor with stirring at 800 RPM, room temperature and 5 minutes. We will obtain the finish product.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

Table 3.2 The composition of solder mask B

Ingredients	Unit	Quantity
Water (deionized)	phr.	Balanced
Reactive monomer I:Reactive monomer II	phr.	1:1, 1:2, 1:3, 2:2, 2:1, 3:1
PVA (GH-17) : PVA (GL-05)	phr	5:5, 5:10, 5:15, 5:20 10:5, 15:5, 20:5
Thickening agent (Xantham gum)	phr	0.1, 0.2, 0.3, 0.4, 0.5
PEG-150	phr	0.1, 0.2, 0.3, 0.4, 0.5
Temperature	°C	100

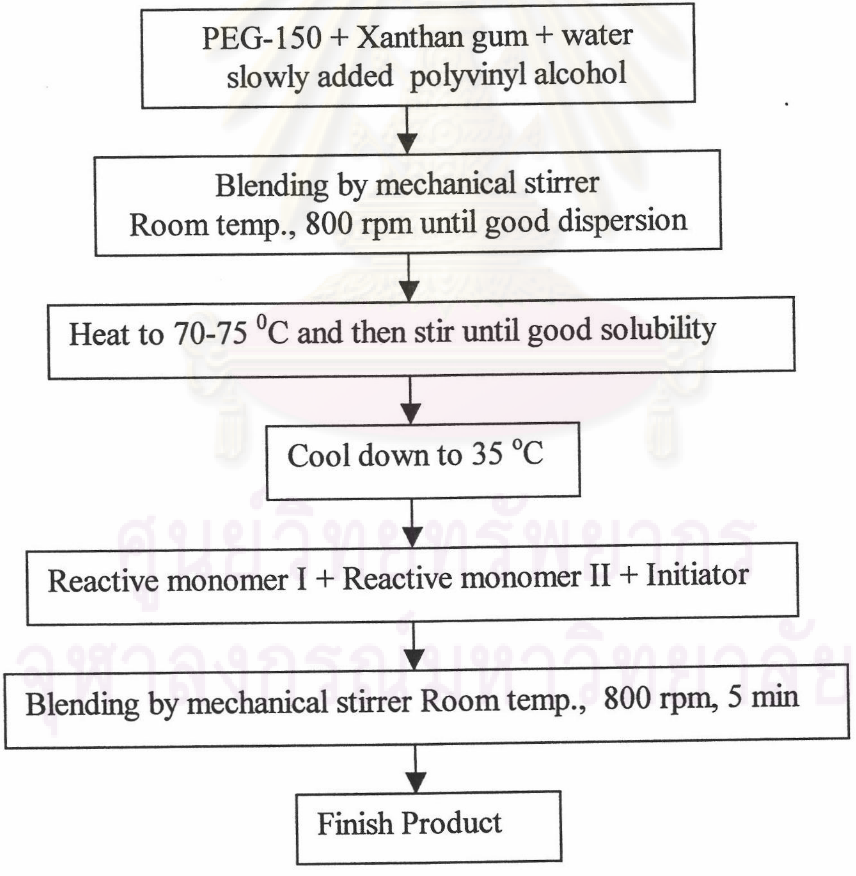

Diagram 3.2 The preparation procedure flow diagram of solder mask B

3.4 Characterization

3.4.1 Determination of the morphology of temporary solder mask

a) Scanning Electron Microscopy (SEM)

A JEM-35 CF scanning electron microscope was used for the examination of temporary solder mask which was prepared by coating with gold vapor deposition before viewing.

3.4.2 Determination of thermal properties of temporary solder mask

a) Thermogravimetric Analysis (TGA)

The initial degradation temperature was measured using the thermogravimetric analyzer (TGA) using a Perkin Elmer, model TGA 7 which was used to follow the weight loss of a 10 mg. sample between 50 and 800 ^{O}C. Data were recorded at scanning rate of 20 ^{O}C/min against a baseline scan.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

b) Differential Scanning Calorimetry (DSC)

A 10-20 mg. of a dried sample was placed into aluminum pan and measured between 50 and 150 ^{O}C in a Perkin-Elmer DSC 7 thermal analyzer. The heating and cooling rate was 10 ^{O}C/min.

The ASTM and ISO test methods was used for investigating the mechanical properties of composite product as follows:

a) Tensile Properties

(ASTM D638-99 : standard test method for tensile properties of plastics)

The tensile strength and elongation were determined. The form of standard dumbbell-shaped test specimens is shown in Figure 3.2. The test length shall be 25 ± 0.5 mm. for type 1.

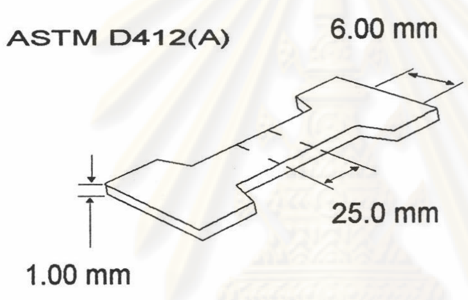

Figure 3.2 The dimensions of dies for dumb-bell test pieces

Dimension	Type 1 (mm.)
A Overall length (minimum)	115
B Width of ends	25.0 ± 1.0
C Length of narrow portion	33.0± 2.0
D Width of narrow portion	6.0+0.4
E Transition radius outside	14.0 ± 1.0
F Transition radius inside	25.0 ± 2.0

ศูนย์วิทยทรัพยากร จุฬาลงกรณ์มหาวิทยาลัย

The tensile testing condition

Temperature	25 °C
Humidity	50 %
Crosshead speed	500.0 mm./min.
Full scale load range	5.0 kN

3.6 Physical Testing

Physical properties of the temporary solder masks was measured by following the ASTM and the ISO test method as follows:

Adhesives of coating films

a) ASTM D 816-82 : Standard Test methods for rubber cements

These test methods measure the properties of adhesives, commonly called Rubber cements, that may be applied in plastic or fluid form.

Test method B – Adhesion strength in shear

The two strips of specimens were 25 mm (1 in.) in width and 125 mm (5 in.) in length were bonded over an area 625 mm^2 (1 $in.^2$).

The machine parameters and testing condition of the adhesion strength in shear test are listed below:

Temperature :	23 ± 2 °C
Relative humidity:	50 ± 5 %
Test speed:	48.0 mm/min

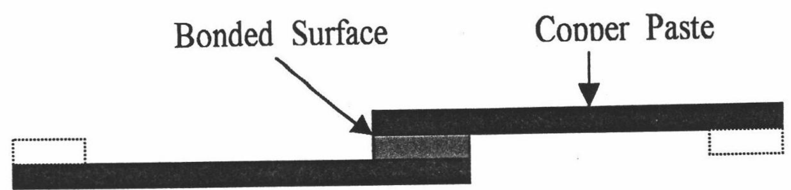

Figure 3.3 Standard Test methods for rubber cements

b) ASTM D3359-97: standard test methods for measuring adhesion by tape test

The dry films have thickness between 2.0 mils (50µm) and 5 mils (125µm). The cuts are 2 mm. apart and make six cuts. Then, this dry films are closed with tap that is 25 mm. width and 75 mm. length. After that this tap is removed from the dry film.

Classification	Percent area removed	Surface of cross-cut area from which flaking has occurred for six parallel cuts and adhesion range by percent
5B	0% none	
4B	Less than 5%	
3B	5-15%	

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

To.

Classification	Percent area removed	Surface of cross-cut area from which flaking has occurred for six parallel cuts and adhesion range by percent
2B	15-35%	
1B	35-65%	
0B	Greater than 65 %	

Figure 3.4 Classifications of adhesion test results

Viscosity

(ISO 1652-1985 (E): Rubber latex-Determination of viscosity)

The viscosity of solder masks (samples) were determined by Brookfield viscometer models DV-I+ small sample by pouring the samples into the container that was maintained at 25 ± 2 °C. A spindle no.7 was carefully inserted into the samples, in such a way as to avoid air being trapped. The spindle was placed vertically in the

samples and in the center of the container. The rotational frequency of spindle was slowly adjusted to appropriate the pointer to be indicated in the range of 5-100. Viscosity of solder masks was expressed in millipascal seconds (centipoises), using the appropriate factor obtained from Appendix A.