

CHAPTER III

EXPERIMENTAL

3.1 Raw Materials and Reagents

3.1.1 High Impact Polystyrene (HIPS)

Commercial grade of HIPS (grade : HI650) were used in this study. A thermal polymerized copolymer high impact polystyrene having rubber particle size between 3-4 microns and rubber content 7.0-8.0 %

It was supplied by Thai Petrochemical Industry (Public) Company Limited.

Typical data of high impact polystyrene grade HI650 are shown in Appendix I (Table 1).

3.1.2 Glass Fiber

“E” glass fiber used in this study were chopped strands and milled fibers. It were supplied by Nitto Boseki Co., Ltd. It is alumina-boron-silicate glass with alkali oxide content less than 0.8%. It is highly soluble in acids moderately soluble in alkalis, used in the electrical industry etc.

Visual Characteristics : In external appearance, the chopped strands shall be free from such defects as unevenness of binding agents, miscut strands, and dirties which are impedimental to service.

Product specification of glass fiber are shown in Appendix I (Table 2-3)

3.1.3 Coupling agent (Organofunctional silane)

gamma-Glycidoxypropyltrimethoxysilane (A-187) was supplied by O.S.I.Specialty Co., Ltd. This coupling agent is a clear liquid. The physical properties of silane A-187 are shown in Appendix I (Table 4)

3.2 Apparatus and Equipments

1. Thumber mixer
2. Extruder: Ishinaka Iron Works Model HS 50 mm. vent type
3. Injection machine: Sumitomo Model SH 80M
4. Impact tester & Computer system: Yasuda Model NO. 258-PC
5. Melt tester: Polymertechnik GmbH Model SWO
6. Vicat softening tester: Yasuda Model HD-PC
7. Material testing instruments: Instron Model 4465(5 kN),
2630-100 Series Extensometer
8. Gloss Measurement: GARDNER
9. Horizontal balancing machine model HBI-25

3.3 Experimental Procedures

3.3.1 Addition of Epoxysilane Coupling Agent to HIPS

The silane coupling agent was added to HIPS by initially in a dry mixing drum. This mixture was then extruded into a melt impregnation die by using a laboratory scale extruder, ISHINAKA IRON WORKS. The screw was a mixing-type, thus providing a homogeneous polymer blend. The

blended polymer was finally pulled through a shaping nozzle, cooled down and cut to the pellets. The following showed the composition of mixing.

Table 3.1 Formulation of HIPS compound.

| Compound | HIPS (phr) | Silane (phr) |
|----------|------------|--------------|
| A | 100.0 | 0.1 |
| B | 100.0 | 0.2 |
| C | 100.0 | 0.3 |
| D | 100.0 | 0.4 |

3.3.2 Dry Blending of HIPS with Glass Fiber

The glass fiber was introduced into the compound A-D prepared in section 3.3.1 and homogenized manually for approximately 10 minutes. It was then extruded through a single screw extruding and injected as the specimen according to ASTM. Table 3.2 shows the composition of each glass fiber reinforced HIPS or HIPS/GF. For each composition, it had been prepared three times. The physical properties of the specimens obtained from each of three times had been measured and then average as shown in Appendix V.

Table 3.2 Formulation of glass fiber reinforced HIPS.

| Sample No. | HIPS | Glass Fibers %by wt of HIPS | | Silane |
|------------|------|-----------------------------|---------------|--------|
| | | Chopped strands | Milled fibers | |
| 1 | 100 | 0 | 0 | 0 |
| 2 | 95 | 5 | 0 | 0 |
| 3 | 95 | 5 | 0 | 0.1 |
| 4 | 95 | 5 | 0 | 0.2 |
| 5 | 95 | 5 | 0 | 0.3 |
| 6 | 95 | 5 | 0 | 0.4 |
| 7 | 90 | 10 | 0 | 0 |
| 8 | 90 | 10 | 0 | 0.1 |
| 9 | 90 | 10 | 0 | 0.2 |
| 10 | 90 | 10 | 0 | 0.3 |
| 11 | 90 | 10 | 0 | 0.4 |
| 12 | 85 | 15 | 0 | 0 |
| 13 | 85 | 15 | 0 | 0.1 |
| 14 | 85 | 15 | 0 | 0.2 |
| 15 | 85 | 15 | 0 | 0.3 |
| 16 | 85 | 15 | 0 | 0.4 |
| 17 | 95 | 0 | 5 | 0 |
| 18 | 95 | 0 | 5 | 0.1 |
| 19 | 95 | 0 | 5 | 0.2 |
| 20 | 95 | 0 | 5 | 0.3 |
| 21 | 95 | 0 | 5 | 0.4 |
| 22 | 90 | 0 | 10 | 0 |
| 23 | 90 | 0 | 10 | 0.1 |
| 24 | 90 | 0 | 10 | 0.2 |
| 25 | 90 | 0 | 10 | 0.3 |
| 26 | 90 | 0 | 10 | 0.4 |
| 27 | 85 | 0 | 15 | 0 |
| 28 | 85 | 0 | 15 | 0.1 |
| 29 | 85 | 0 | 15 | 0.2 |
| 30 | 85 | 0 | 15 | 0.3 |
| 31 | 85 | 0 | 15 | 0.4 |

3.3.3 Molding

The manufactured HIPS/GF pellets were dried at temperature 80 °C for 2-3 hours. The pellets were then injection molded by using SUMITOMO injection machine. The testing specimens were prepared according to ASTM using a mold that is generally used for the commercial samples.

Different levels of fiber loading, from 5% to 15% (by weight of composite) and different levels of silane coupling agents from 0.1 to 0.4 phr were prepared. For each level of fiber-loading, six to eight specimens were tested in each case (see Appendix II).

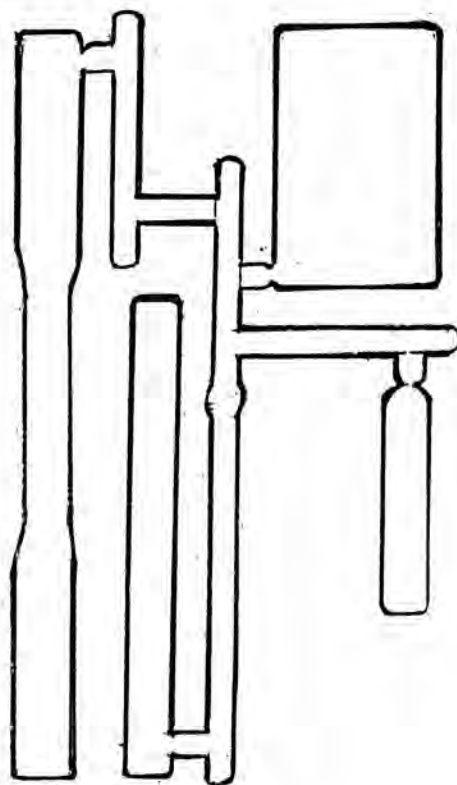


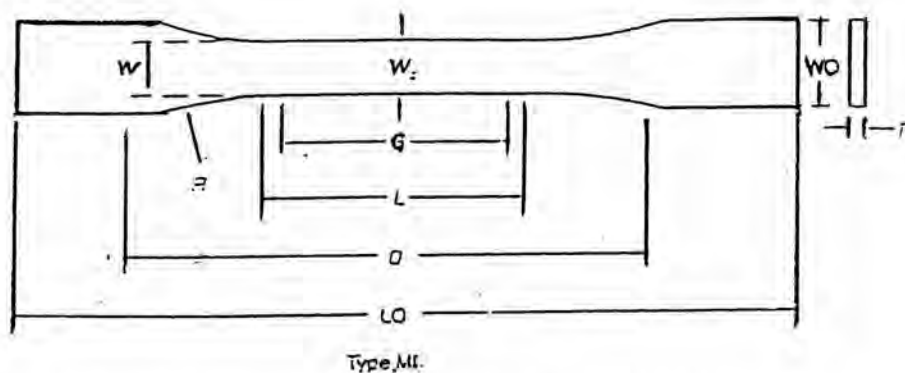
Figure 3.1 The standard specimens according to the ASTM test method.

3.4 Mechanical Testing

Mechanical properties of the composites were measured by the following ASTM test methods

3.4.1. Tensile properties

Tensile properties were measured using an Instron universal testing machine model 4465. Specimens having size of $60 \times 150 \times 115 \text{ mm}^3$ were used for testing. Tensile strength of the sample was determined as per ASTM D638M-93.



W : 10 mm. W0 : 20 mm. G : 50 mm. R : 60 mm.

L : 60 mm. L0 : 150 mm. D : 115 mm.

Figure 3.2 Schematic dimension of tensile test specimen (type M).

3.4.2. Flexural Properties

Flexural properties were measured using an Instron universal testing machine model 4465. Specimens having 12.6 mm in width and 3.16 mm in depth were used for testing flexural strength of the sample as per ASTM D790M-93.

For a 3 point bending flexural testing, the length of the support span depends upon the thickness. In these cases, 50.8 mm span length was used in order to have support span to thickness ratio of 16:1. A test specimens were loaded via a diameter of crosshead loading nose by using the rate of crosshead motion at 1.3 mm/min.

3.4.3 Heat Distortion Temperature

Heat distortion temperature of plastics was measured using a heat distortion tester of Yasuda seiki model 148 HD-PC. The sample was determined as per ASTM D648-88.

The specimens having 127 mm. in length, 13 mm. in width and 3 mm. in thickness were cut by the circular saw cutting machine . At least two test specimens having smooth flat surface free from saw cuts, excessive sink marks, or flash were used.

The condition in testing were shown as follows:

| | | |
|---------------------|-------------|--------------------------------|
| Temperature : | 23 ± 2 | $^{\circ}\text{C}$ |
| Relative humidity : | 50 ± 5 | % |
| Rate B : | 120 ± 5 | $^{\circ}\text{C} / \text{hr}$ |
| Loading : | 5 | kg. |

Place the specimen under the needle , and gently lower the red needle (without the additional weight) so that the needle rests on the surface of the specimen and held it in position. The bulb of the thermometer or the thermocouple should be as close as practical to the specimen.

The load was applied, then set the penetration indicator at zero, and increase the temperature. Rate of temperature increasing shall be 120°C/hr uniformly throughout the test.

3.4.4 Flow Rates

Flow rates were measured using a melt flow indexer model Meltflicker HT. Melt flow index of sample was determined as per ASTM D1238-90b.

The temperature of the cylinder with piston and die in place shall have been at the test temperature (200°C) for at least 15 min before a test is begun.

Flow Rates testing conditions were as follows:

| | |
|-------------|-----------------------|
| Temperature | 200°C |
| The weight | 5 kgs. |

3.5 Manufacturing the Finished Products

The composite pellets were dried at temperature of 80°C for 2-3 hours before injection molding. DONG SHIN injection machine was used with following condition : cylinder temperatures 200°C , 210°C , 220°C , nozzle temperature 240°C , mold temperature 60°C , injection speed 30 mm/s, injection pressure 120 bar, holding pressure 60 bar, holding pressure time 12 s, back pressure 2 bar and screw speed 60 r/min.

The same mold usually used to produce a blower of an air-conditioner was utilized.

The injection molding of HIPS/GF sample no.8 was performed. A blower from HIPS/GF no.8 was then subjected to balancing measurement

using horizontal balancing machine model HBI-25 with the standard condition for testing a commercial blower. Its measurement has been done once in every month for a consecutive 3 months.