CHAPTER III

EXPERIMENT

3.1 The apparatus

a) Mixing system

Agitator : ELECTRODYNE RLG 100

Power supply : 220 V, 50 Hz

Number of motor revolutions = 60 - 1250 rpm.

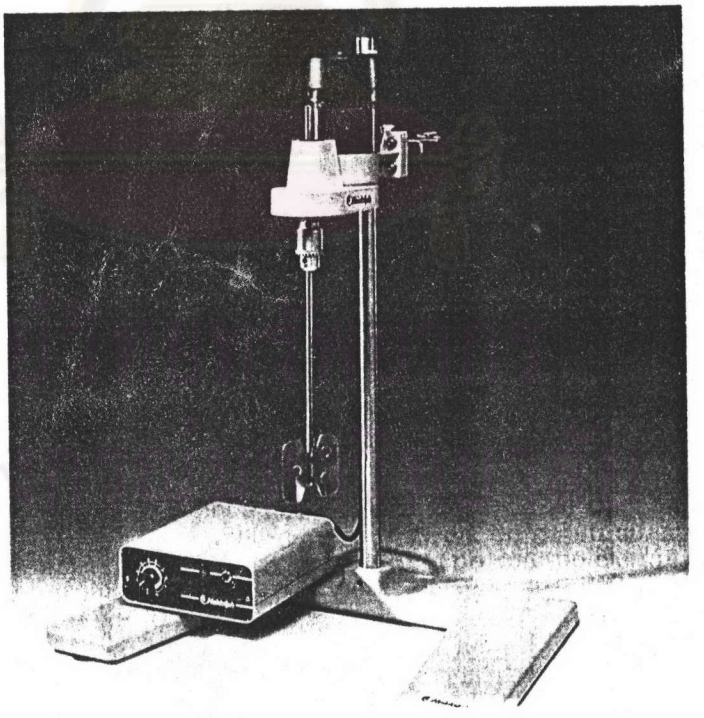


Figure 3.1 Mixing system

b) Adhesive curing system

Set temperature of convection oven at 150°C to cure epoxy adhesive composites 3 min. for epoxy adhesive A and 5 min. for epoxy adhesive B.

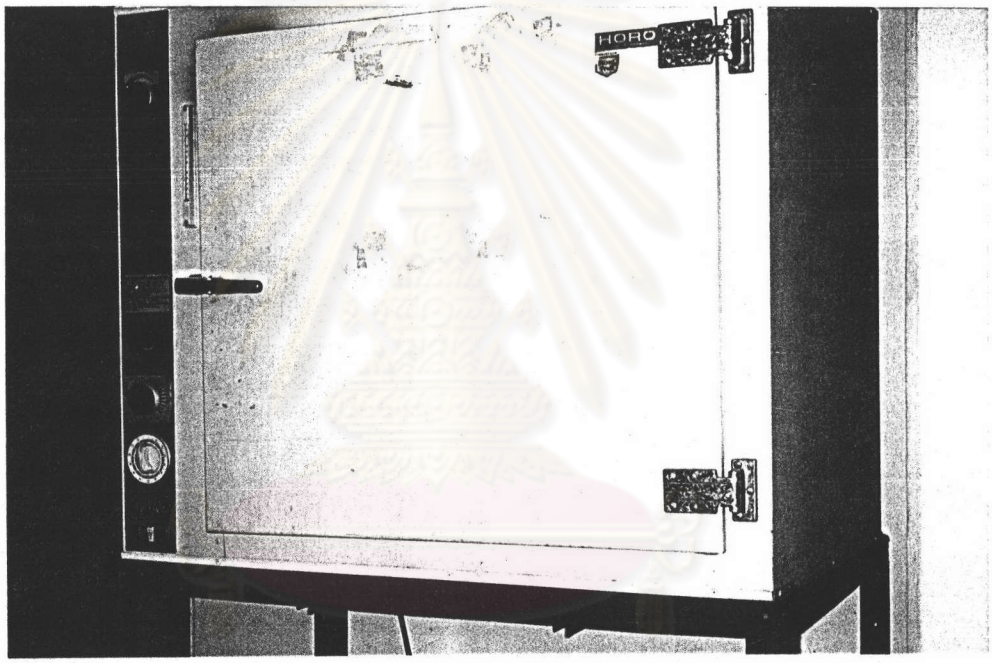


Figure 3.2 Convection oven

c) Thermal conductivity measurement system : -

The apparatus illustrated in Figure 3.3 is the standard thermal conductivity analyzer model 88 K-factor instrument of Anacon, Inc. , USA.

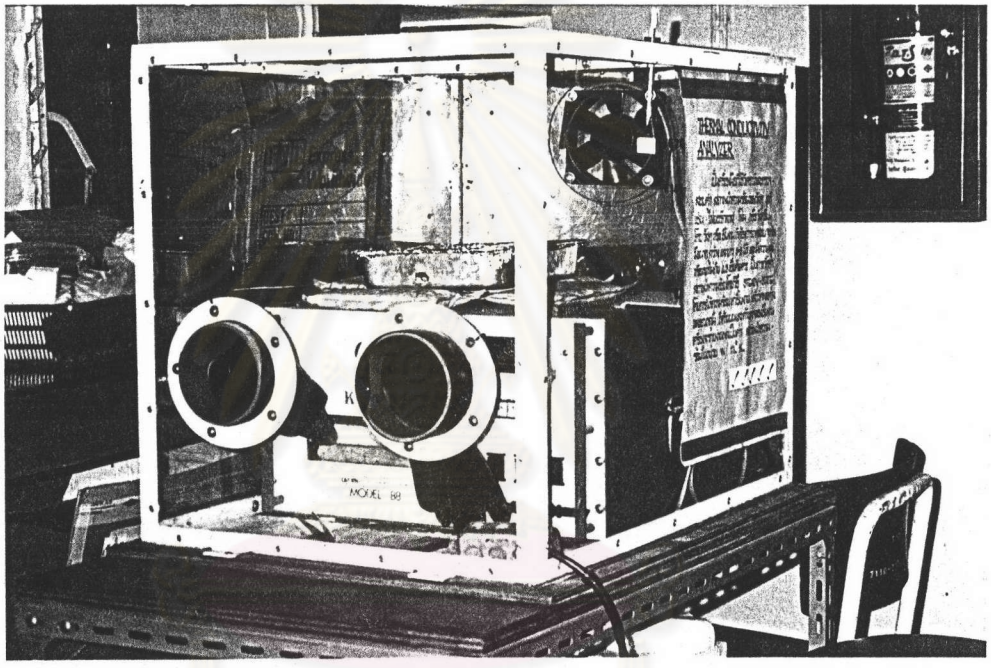
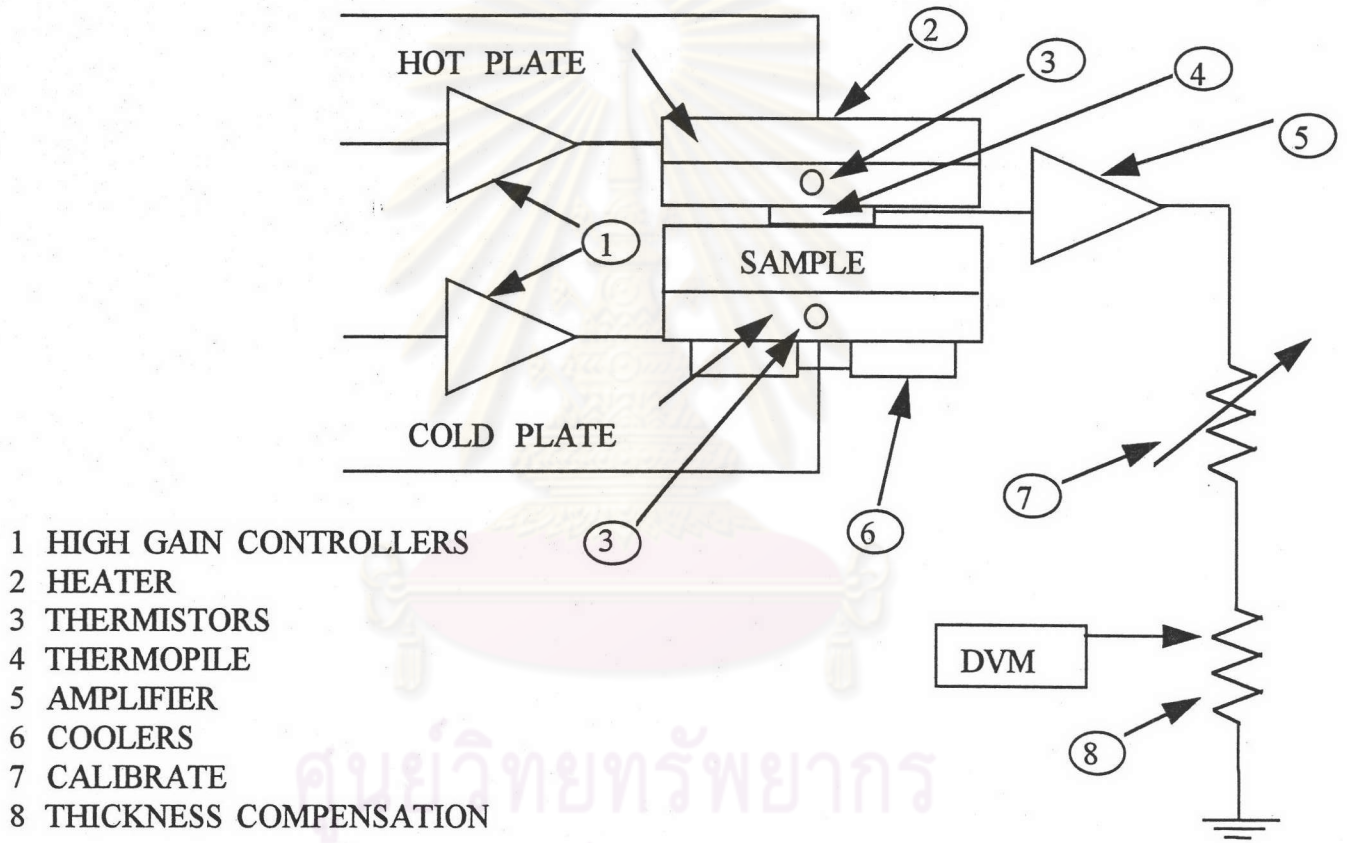


Figure 3.3 Thermal conductivity analyzer

The thermal conductivity measurement system is contained in an enclosure, dimensions 19 3/4" wide , 11 1/4" high and 19" deep. A 2" x 8" opening in the front panel (left side of instrument) is provided for purposes of inserting the sample into the controlled environment. The opening is sized to accommodate a (1/4"-2") thick x 6-8" x 6-8" square test sample. The power requirement is 115 V / 220 V AC, 50 / 60 Hz.

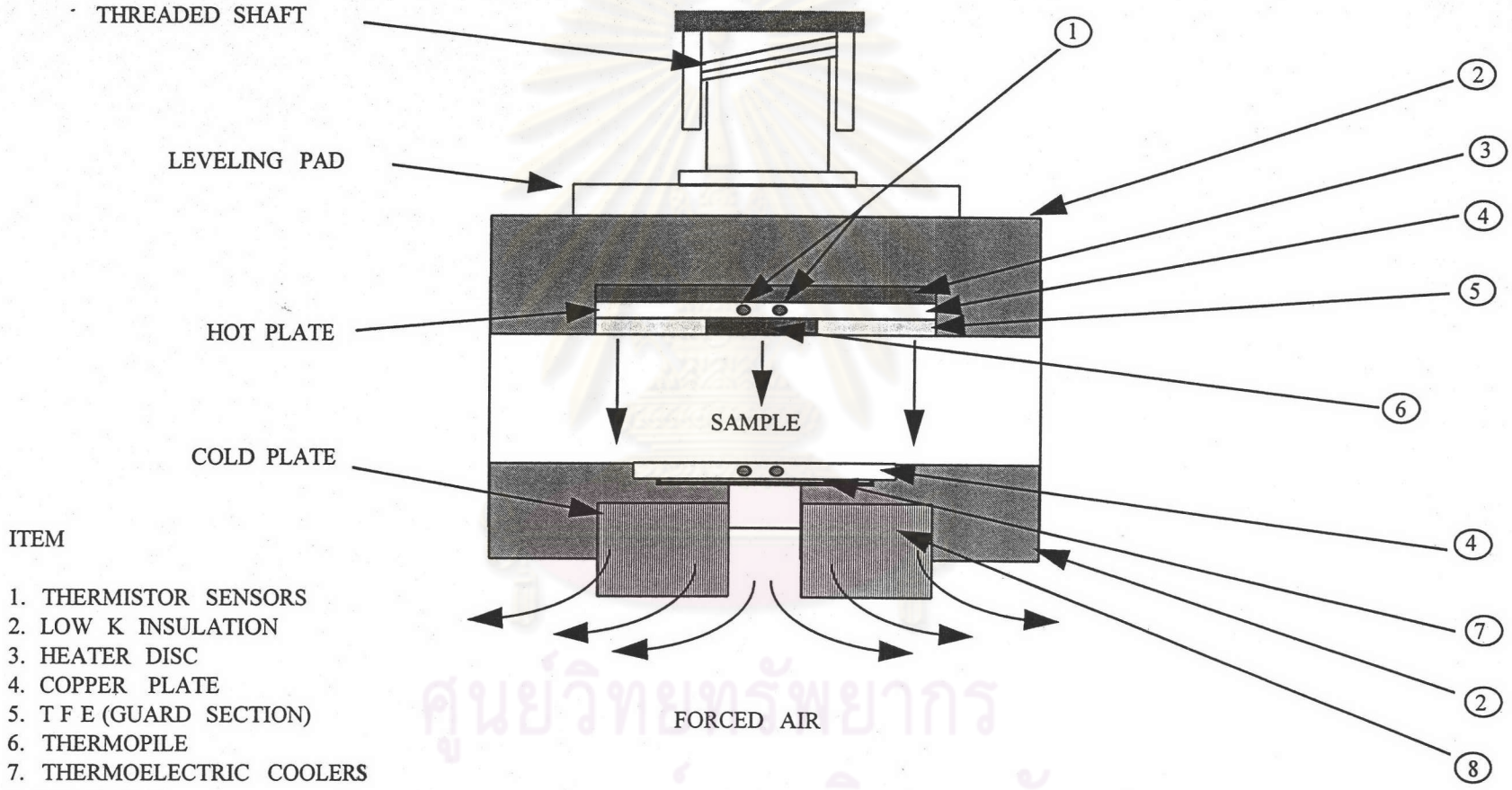
The signal to the display digital voltmeter is selected by a multi-push button selector switch and is displayed by the readout on the right side of front panel. The hot plate assembly consists of a 4" diameter copper plate 1/8" thick. Both thermistors sensors (for control and readout) are located in the aforementioned disc. see Figure 3.4 and Figure 3.5. The heater disc. type 4" in diameter is located on the top surface of the assembly. The heat flow sensor is located on the bottom surface of the hot plate. The complete assembly is insulated and all surfaces are sealed with thermal compound.

The cold plate dimensions are identical to those given for the hot plate. The only difference between the two assemblies is the substitution of thermoelectric heat pumps and accessories for the disc. heater. Two thermoelectric modules are thermally close coupled with analytical control surfaces and the heat exchanger. The compound is used to further assure a good thermal bonding to electric modules are held between the cold plate and the heat exchange.



1 HIGH GAIN CONTROLLERS
2 HEATER
3 THERMISTORS
4 THERMOPILE
5 AMPLIFIER
6 COOLERS
7 CALIBRATE
8 THICKNESS COMPENSATION

Figure 3.4 Electronics block diagram of thermal conductivity analyser standard model 88



ITEM

1. THERMISTOR SENSORS
2. LOW K INSULATION
3. HEATER DISC
4. COPPER PLATE
5. T F E (GUARD SECTION)
6. THERMOPILE
7. THERMOELECTRIC COOLERS
8. HEAT SINK

Figure 3.5 Measuring assembly block diagram of thermal conductivity analyser

3.2 Material used

3.2.1 Epoxy adhesive

a) Epoxy adhesive A [24] manufactured by Somar Corporation, Japan

Constituents : -

Epoxy	:	BPA-epichlorohydrin
		m-phenylenediamine (MPDA)
Dye	:	Red type

Properties : -

Appearance	:	Dark Red Plaste
Density , 25°C	:	1.15 g/cm^3
Viscosity, 25°C	:	75,000 mPa s
Shelf life, 5-10°C	:	6 months
Curing performance	:	exposure to heat more than 120°C
		(typically 40 seconds at 150°C)
		(typically 60 seconds at 140°C)
		(typically 90 seconds at 130°C)
Thermal conductivity	:	0.13 W/mK (ASTM C177)

b) epoxy adhesive B [25] manufactured by Loctite Corporation., USA

Constituents : -

Epoxy	:	BPA-epichlorhydrin
		Beta phthalamide cyclic dimer
Dye	:	Rocket red dye
		[(2,2'-(2,5-thiophenediyl)Bis(5-t-
		Butylbenzoxazole)]

Properties : -

Appearance	:	Dark Red
Density , 25°C	:	1.2 g/cm^3
Viscosity, 25°C	:	125,000 mPa s
Shelf life, 5°C	:	6 months
Curing performance	:	exposure to heat above 100°C
		(typically 90-120 seconds at 150°C)
Thermal conductivity	:	0.2 W/mK (ASTM : C177)

3.2.2 Fillers (thermal conductive materials)

a) Aluminium metal powder

spherical 5-10 μm , 99 %

density = 2.702 g/cm^3

b) Berylium metal powder

spherical 10 μm , 98.5 %

density = 1.85 g/cm^3

c) Copper metal powder

spherical 5-10 μm , 99 %

density = 8.933 g/cm^3

d) Berylium oxide

particle size 5 μm , 98 %

density = 3.0 g/cm^3

e) Silicon carbide

particle size 15 μm , 99 %

density = 3.160 g/cm^3

3.3 Procedure

3.3.1 Adhesive sample preparation

The different composite samples with weight percentage of fillers from 0 to 20% were prepared from each of the two adhesives (epoxy adhesive A and epoxy adhesive B). Additive powder and adhesive were mixed together by weight and then stirred by the swing-out stirrer with spiral agitator until a homogeneous mixture was obtained. The appropriate speed of stirrer is 200 rpm.

Before curing, the prepared adhesive was placed into a 6" long, 6" wide and 0.25" deep tray made from a sheet of aluminium. The tray acts as a mould for

making cured epoxy adhesive specimen. In order to prevent cured epoxy adhesive from sticking with this tray, a thin layer of wax was coated on tray surface as a mould releasing agent. The adhesive was then cured in the convection oven. Epoxy adhesive A was cured at 150°C for 3 min. and epoxy adhesive B was cured at 150 °C for 5 min.

Bring cured adhesive sample to measure thermal conductivity value by using thermal conductivity analyzer, standard model 88 of Ancon, Inc. USA.

3.3.2 Measurement procedure

3.3.2.1 Thermal conductivity analyzer calibration

The unit should be left at ***ON*** button all the time if possible as this will ensure stability and eliminate warm-up time before measurements. The instrument should be allowed to warm up during initial start-up for at least 30 minutes. During this time, the panel lights labelled HOT and COLD will be dim and become barely visible when the unit has stabilized.

The hot and cold plate temperatures have already adjusted. The checkout requires an accurate temperature potentiometer / thermocouple combination.

The following adjustments can be made at the back of the instrument as shown in Figure 3.6.

To adjust the hot plate, insert a sample of insulation into the unit with a thermocouple sandwiched between the sample and the center of the heat flow disc. Apply just enough torque to the hot plate assembly to ensure good contact of the thermocouple on the heat flow disc. Measure the temperature.

If the temperature is low, turn the HOT ADJ control on the back plate slightly clockwise. Wait five minutes for stabilization. Readjust as necessary allowing for the time lag until the steady state temperature condition is read on the potentiometer.

To adjust the cold plate, follow the same procedure outlined above only with the thermocouple between the sample and the center of the cold plate and adjusting the COLD ADJ on the back plate.

The thickness measurement is adjusted internally. A block of known thickness must be inserted into the instrument. The thickness trimpot is then adjusted while the meter switch is in THK. position until the correct thickness is shown. This calibration has already done and is not necessary calibrate each time used in the experiment.

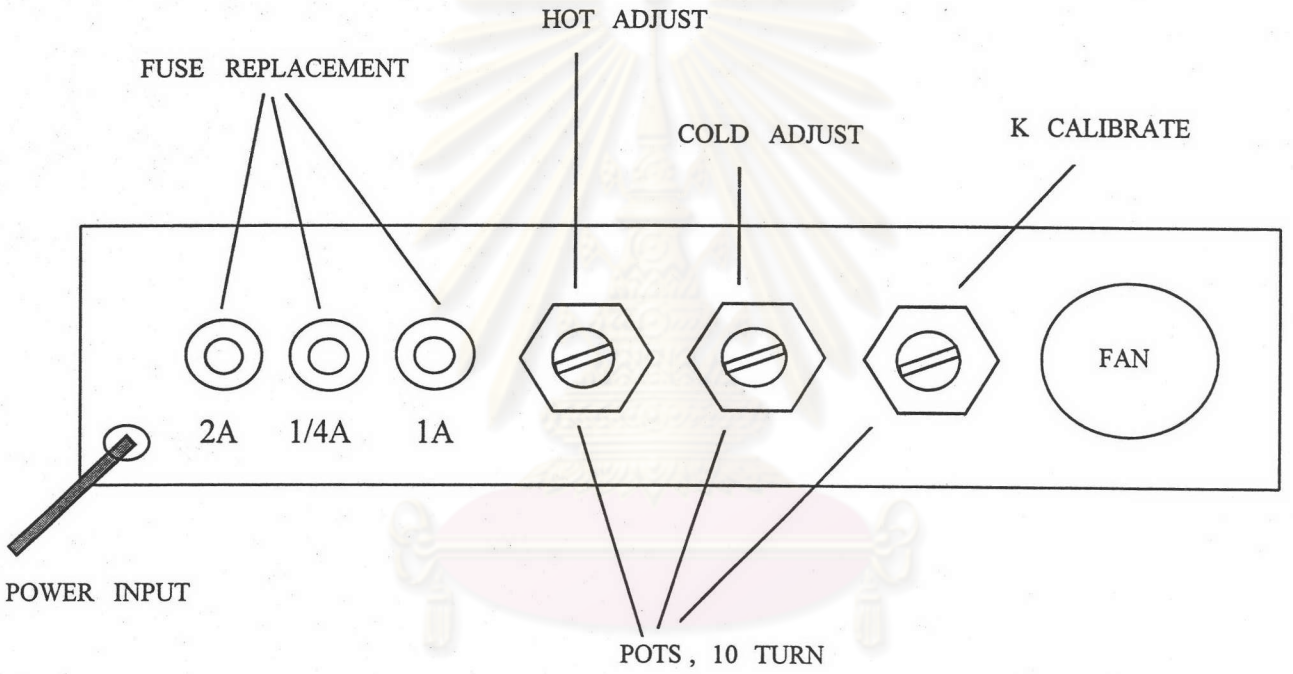


Figure 3.6 Chasis , back view of thermal conductivity analyser

The calibration of the "K" reading is performed with a known fiberglass standard. The standard must be oven dried before use. When the standard is inserted, push the THK. button and turn the torque knob until the meter reads the thickness of the standard. It can then be turned down an additional 2 to 4 thousandths to provide light compression of the standard. Allow 1/2 hour to stabilize. The control marked K CAL. on the back plate is then adjusted while reading "K" on the meter until it reads the correct value. The instrument will now correctly read the conductivity over the specified range and accuracy of the instrument.

Readout unit : W / mK

Power requirements : 115 V / 220 V AC, 50 / 60 Hz.

3.3.2.2 The operation of thermal conductivity analyzer

Remove packing sample from the instrument by turning the torque knob counter clockwise to release pressure on the sample - depress eject button to release sample. The packing sample should always be placed in position when the instrument is not in use or is being moved from one location to another.

a) Turn torque knob counter clockwise to raise hot plate and insert sample into the slot.

b) Turn torque knob clockwise until the two marks on the knob are aligned, then release.

c) Push the selector switch to "K" position and then allow reading to stabilize. The stabilization time is an average of 5-10 min. dependent on size and type of sample.

d) When the reading has stabilized it will be indicating the direct value of thermal conductivity in W / mK.

e) Check positions for the temperature of hot and cold plates together with thickness indication are provided on the push button selector switch.

f) To release the sample, turn torque knob counter clockwise and depress eject button.

g) Measure again when the sample cool down to room temperature.

3.3.2.3 Shear strength testing system

The tensile testing machine is used to determine adhesion strength properties of prepared epoxy composites. The ASTM : D 1002, standard test method for strength properties of adhesives covers the determination of the comparative shear strengths of metal-to-metal adhesive bonding when tested on a standard specimen and under specified conditions of preparation and test.

Standard test specimens shall conform to the form and dimensions as shown in Figure 3.7.

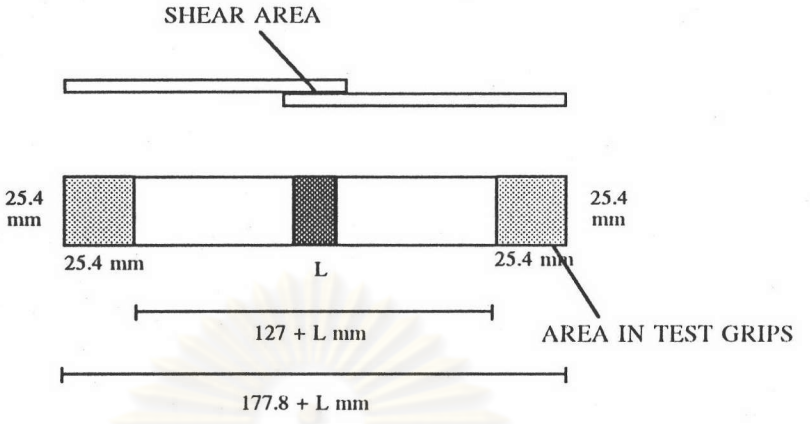


Figure 3.7 Form and dimensions of test specimen

The recommended thickness of the sheets is 1.62 + 0.125 mm (0.064 + 0.005 in.) and the recommended length of overlap for most metals is 12.7 + 0.25 mm (0.5 + 0.01 in.).

The grade of metal is recommended for the test specimens is aluminium B 209. Clean and dry the sheets carefully and then apply the adhesive to a sufficient length in the area across the end of one or both metal sheets so that the adhesive will cover overlap space. Assemble the sheets so that they will be held rigidly and the adhesive allowed to cure in the convection oven at 150° C for 3 min.

Place the specimens in the grips of the testing machine (Figure 3.8) so that the outer 25 mm of each end are in contact with the jaws and so that the long axis

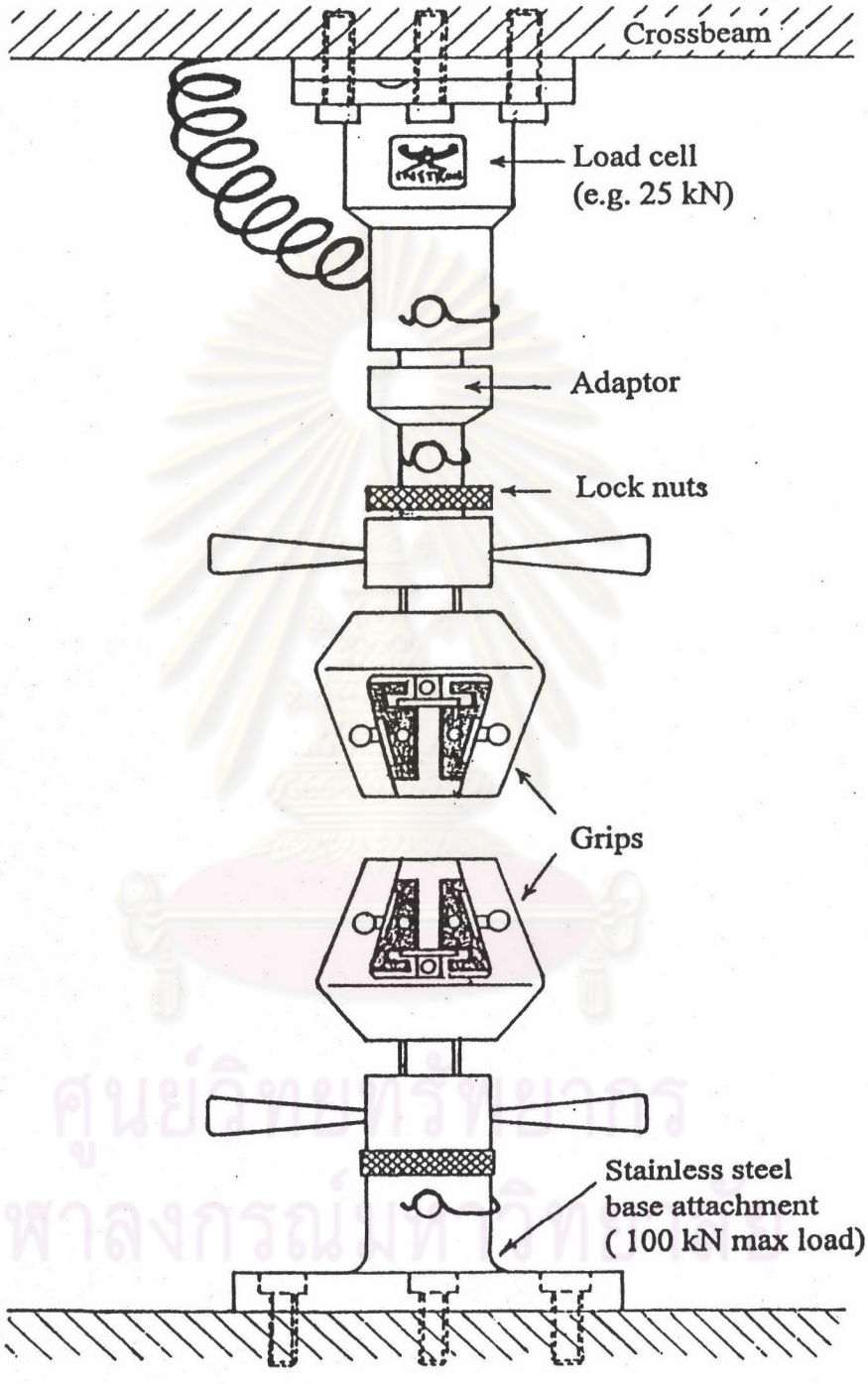


Figure 3.8 Tensile testing machine

of the test specimen coincides with the direction of applied pull through the center line of the grip assembly. Apply the loading to the specimen to go to break point of joint. It is recommended that test specimens be made up in multiples to confirm testing.

A variation in thickness of the metal, and the length of overlap, will likely influence the test values.