CHAPTER 4

EXPERIMENTAL STUDIES
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4.1 Experimental study plan – The flow diagram

REPEATED DROP TESTS ON POLYMER COMPOSITES (GFRP AND CFRP) WITH VARIED INCIDENT IMPACT ENERGIES (Einc)

ASSESSMENT OF THE MATERIAL BEHAVIOUR DURING REPEATED DROPS BY THE USE OF LOAD-TIME-ENERGY DATA AND CORRESPONDING DERIVED PLOTS

DEFINING OF NUMBER OF DROPS TO FAILURE (Nf)

ARRIVAL AT THE INTERRELATIONSHIPS BETWEEN Einc AND Nf

USE OF Nf TO STUDY THE IMPACTOR MASS EFFECTS

UNDERSTANDING AND INTERPRETATION OF THE CURVES

THEORETICAL INPUTS

EXPERIMENTAL VERIFICATIONS AND COMPARATIVE EVALUATION

ANALYSIS AND CONCLUSIONS

VISUAL INSPECTION DURING IMPACT

FINAL DELAMINATION AREA MAPS DURING REPEATED DROPS
In the experimental part, extensive studies were conducted on carbon and glass-epoxy composites as they represent the ductile and brittle class of materials. Limited studies were conducted on some commercial grade kevlar-epoxy composite, results of which, has been included in Annexure–3.

4.2 Test specimen preparation

4.2.1 Reinforcements

The reinforcements used for these experimental works are 2X2 twill woven E-glass fabric (obtained from a local supplier; M/s Arun fabrics Pvt. Ltd.,) 2x2 twill woven carbon fabric (obtained from M/s Hexcel Fabrics). The areal density of the fabrics were 280 GSM. Nine layers of fabric was used in the fabrication of test laminates to achieve a thickness of 2.1±0.1 mm and 0.60 ± 0.02 fibre weight fraction.

4.2.2 Mix ratios and cure schedules for different matrix systems

Table 4.1 shows the mix ratios and process cure details for the three structural grade epoxy matrix systems used in the preparation of composites. Some of the matrix systems mentioned require pre-heating and ingestion at pre-defined temperatures. In such cases, care is taken to pre-heat the mould as well, so that the whole set-up is maintained at the desired temperature.

4.2.3 Mould preparation

The mould plates and the spacers used for laminate fabrication are cleaned well and wax is applied on them. The application of wax is to ensure closure of small pits and smoothening of the mould surface. After few
minutes the excess wax is wiped off the surface and a suitable release agent usually PVA (Poly Vinyl Alcohol) is applied onto the mould plates and allowed to form a thin layer of release film. The requisite number of layers of the reinforcement are packed into the mould cavity (as per the intended fibre fraction, taking into account the cavity volume) and the mould plates are tightened all round with bolts and nuts. Also suitable grade sealant is applied all-round during assembly of the mould to assist in vacuum evacuation during resin injection. The assembled mould set-up is attached to a vacuum source and the air inside is completely evacuated.

Table 4.1: Mix ratios, cure schedule details of different matrix systems used in test laminate fabrication

<table>
<thead>
<tr>
<th>Matrix system</th>
<th>Mix Ratios Resin : hardener</th>
<th>Fabrication process</th>
<th>Cure schedule</th>
<th>Post cure schedule</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy (LY 556; HY 951)</td>
<td>100:11</td>
<td>Resin and hardener are mixed in the defined ratio, 10% diluent Methyl Ethyl Ketone is added and matrix system is ingressed as described in 4.2.4</td>
<td>Overnight room temperature cure and demould</td>
<td>50°C for ½ hour; 70°C for 1 hour; 85°C for 2 hours</td>
</tr>
<tr>
<td>Epoxy (LY556-HT 972)</td>
<td>100:28</td>
<td>Preheat mould set-up and resin at 80°C for 3 hours. Melt the hardener at 90°C. Mix and pour the matrix system as described in 4.2.4 maintaining the temperature at 80°C</td>
<td>120°C for 2 hours</td>
<td>150°C for 2 hours and then demould</td>
</tr>
<tr>
<td>Epoxy (C14 : K68: K112)</td>
<td>100:95:2</td>
<td>Preheat mould set-up, resin and hardener at 75°C for 6 hours, Mix resin, hardener and catalyst in the defined ratios. Mix and pour the matrix system as described in 4.2.4 maintaining the temperature at 75°C.</td>
<td>80°C for 12 hours; 100°C for 4 hours and demould</td>
<td>150°C for 3 hours and 180°C for 15 hours</td>
</tr>
</tbody>
</table>
4.2.4 The Resin Ingression Technique* for laminate fabrication

All the test laminates for this work has been prepared using a new and improved process viz., Resin Ingression Technique for which patent grant has been awaited. The process consists of a cavity mould with an outlet at the top end and an inlet at the bottom end (Refer figure 4.1). Several layers of the reinforcement are placed, such that, there is a cavity at the bottom end (resin inlet portion) for resin ingression. The bottom inlet is attached to a resin ingression source in such a manner as to provide a resin column above the top of the outlet in the mould. In accordance with the mix ratios of the respective matrix systems, the matrix system is poured into the resin ingression source. Care is taken to minimise the entry of air bubbles into the mould cavity. The outlet is connected to a vacuum source via a resin collector as shown in the diagram. The vacuum is switched on from the time of ingression till a surplus quantity of the resin system overflows into the resin collector. Once the resin ingression technique is complete, the mould set-up is not disturbed till the laminate is taken to its complete cure schedule. Subsequently, the laminate is demoulded, post-cured and other procedures such as trimming, cutting, dimensioning, fibre weight fraction measurements and specimen preparations (90mm x 90mm) are undertaken.

4.3 The Drop Weight Instrumented Impact Tester

Figure 4.2 shows the schematic illustration of the drop weight impact tester (also refer 1.5.3.2 of chapter 1) used in these studies.

An instrumented impact test machine incorporates a tup in a tower enclosure. The outputs of the test machine include a trigger signal used for starting data acquisition (triggered by a flag moving past a detector on the impact test machine) and determining the impact velocity as well as the load signal generated by the tup.

Two separate signal processing elements are used; a signal conditioning (SC) unit and an analog-to-digital (A/D) board. The SC unit receives the trigger and tup impact signals from the impact test machine.
The unit filters and amplifies the tup (impact) signal and calculates impact velocity. It then passes these signals to the A/D board. The PC based A/D board installed in the computer acquires and digitises the impact data under software control. The software uses a high speed (1 MHz) A/D board. The test software then acquires, analyses, stores impact test data and provides graphic displays of load, energy, velocity, time or deflection within seconds after the test. The software has provisions to save the test results for later review and analysis.

The 8250 drop tower has provisions for changing the impactor mass. In all our tests, three impactor weights have been used viz., 2.74Kgs, 5.42 Kgs and 12.28 Kgs. In conjunction with the desired energy, the drop height is calculated. The instrument has provisions to test both in the gravity mode and also pneumatic mode. All our experimental works were carried out in the gravity mode. The instrument has some self incorporated safety devices. The power supply to the pendant is cut off when any or all of the doors are open. There is also a backup alarm during firing to warn the user that the impact testing is taking place. The stop blocks/rebound break (to prevent multiple impacts during the same drop) connected pneumatically are adjusted such that, when the tup touches the specimen the distance between the top of the stop blocks and the bottom of the impactor mass is about $\frac{3}{4}$th of an inch. In this position the velocity detector is adjusted such that the top portion of the double pronged flag is just above the line passing between the two photoelectric devices. The movement of the entire crosshead for these settings is carried out in the manual mode using the UP and DOWN buttons after switching on to the manual mode. Once the settings are made, the switch on the pendant is turned onto the automatic mode at which point the
cross-head automatically moves on to the pre-defined position. Now the equipment is ready for testing.

**Fig 4.2: System illustration for drop weight impact testing**

### 4.4 Experimental work plan for the studies on $E_{in}$ versus $N_f$ interrelationships

Table 4.2 shows the specimen details and the incident energies that they are subjected to, for deriving this interrelationship. It is to be noted that the incident energies chosen are arbitrary more dependent on the flexibility of the hardware part of the equipment. As previously described, at each
energy level the specimen is impacted until failure as previously described in para 3.1 of chapter 3.

Table 4.2: Test specimen details and incident energies used for deriving and verifying the $E_{in}$ versus $N_f$ interrelationship

<table>
<thead>
<tr>
<th>Material details</th>
<th>Thickness (mm)</th>
<th>Fibre weight fraction</th>
<th>Incident energies (joules) $E_{in}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Twill woven glass epoxy (LY556-HT972) laminate (120°C cure)</td>
<td>2.2</td>
<td>0.62</td>
<td>3.4; 4.95; 5.5; 7.6; 8.4; 10.1; 11.0; 14.7</td>
</tr>
<tr>
<td>Twill woven glass epoxy (LY556-HY951) laminate (Room temperature cure)</td>
<td>2.03</td>
<td>0.69</td>
<td>3.2; 5.4; 6.5; 7.0; 8.2; 12.5</td>
</tr>
<tr>
<td>Twill woven glass epoxy (C14-K68-K112) laminate (180°C cure)</td>
<td>2.2</td>
<td>0.60</td>
<td>5.3; 8.4; 14.5</td>
</tr>
<tr>
<td>Twill woven carbon epoxy (LY556-HT972) laminate (120°C cure)</td>
<td>2</td>
<td>0.59</td>
<td>3.7; 5.0; 7.0; 10.1; 14.7</td>
</tr>
</tbody>
</table>

$E_{in}$, mentioned against each type of composite are those for which the composites failed in a finite number of drops. The composites could not be tested exactly at the same energy levels due to practical reasons. Moreover, this dissertation does not highlight the matrix effects or other such parameters (fabrication process, fibre weight fraction etc.) since its primary objective is to establish the usefulness of repeated drop tests, as an entity by itself, in characterising the impact behaviour of polymer composites.
4.5 Experimental work plan for the studies on impactor mass effects

Table 4.3 shows the specimen details and incident energies used for the studies of impactor mass effects

Table 4.3: Experimental details used to study the impactor mass effects

<table>
<thead>
<tr>
<th>Material details</th>
<th>Fabrication process</th>
<th>Incident energies (joules)</th>
<th>Drop height (mtrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>2.74 Kg</td>
<td>5.42 Kg</td>
</tr>
<tr>
<td>Twill woven glass epoxy (LY556 HY951) laminate (RT cure)</td>
<td>Compression moulding</td>
<td>3.2</td>
<td>0.119</td>
</tr>
<tr>
<td>Twill woven glass epoxy (LY556-HT972) laminate (120°C cure)</td>
<td>Resin Ingression Technique</td>
<td>3.4</td>
<td>0.127</td>
</tr>
<tr>
<td>Twill woven glass epoxy Resin (C14-K68-K112) laminate (180°C cure)</td>
<td>Resin Ingression Technique</td>
<td>3.3</td>
<td>0.127</td>
</tr>
</tbody>
</table>

Summary

This chapter starts with the experimental study plan, gives details about the raw materials, fabrication process and the instrument used for impact testing. Further, the chapter gives details of the experimental work plan for studies on $E_{in}$ $N_f$ interrelationships and impactor mass effects.