Comparing the effect of fillers on physical properties of epoxy resin

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Comparing the Effect of Fillers on Physical Properties of Epoxy Resin

May 1999
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1 Introduction

There are four basic categories of structural materials: metals, polymers, ceramics and composites. Composites consist of two or more distinct constituents, combined in a macroscopic structural unit. They are made of combinations of the other three materials. Composites are not new; they occur frequently in nature. Bone, which combines oriented and layered collagen fibrils with a protein-calcium phosphate matrix, is one of nature’s examples of composite material. Also, wood, consisting of fibrous cellulose, combined in a matrix of lignin, can be classified as a composite. One of the first man-made composites dates back to the time when bricks were made of mud, reinforced with straw.

Composites are generally used because they have desirable properties, which can exceed those achievable from any of the individual components used in isolation. They can be engineered to provide a wide range of tensile, flexural and impact strength properties. Composites are corrosion resistant to most chemicals, do not suffer from electrolysis and incorporate long term benefits such as weatherability and UV stability.

The most common type of composite is the fiber composite consisting of reinforcing fibers of high strength and stiffness embedded in a binder, the matrix. Fiber composites are a very promising construction material, designed to put strength where it is needed and reduce the weight of structures. Other classes of composites are:

- flake composites, composed of flat flakes with or without a matrix,
- particulate composites, composed of particles with or without a matrix,
- filled (or skeletal) composites, composed of a continuous skeletal matrix filled by a second material,
- laminar composites, composed of layer or laminar constituents.

[Schwartz, 1984]

Originally fiber composites were developed for the aerospace industry as they offered attractive properties of strength and stiffness compared to their weight. Today composites have found their way into a much wider range of applications such as transportation, sports and also civil engineering. This infiltration is mainly due to the development and more generalized use of inexpensive basic materials such as fiberglass and resins. Another factor is the energy content of plastics which can be half that of metals because of the greater material efficiency and tailorability of design and the low scrap factor. Compared to steel, composites provide lightweight structures that reduce the demands of cost on transport, installation and long term maintenance. To further the lower density, lightweight filler materials are mixed in with the matrix material. Fillers are not only used to reduce weight. They can also improve smoke and fire resistance, water resistance and surface smoothness, and they can reduce costs, since a part of the more expensive resin volume is replaced by a cheaper material.

For this project several filler materials will be mixed with resin in different volume percentages. The flexural and compression properties of the resulting composites will be compared. The next chapter will give a summary of the materials used for this project. After a description of the fabrication of the test specimens in chapter three, the test methods will be outlined in chapter four. In the last chapters the results of the test series will be discussed.
2 Materials

The interest of this project lies with the influence filler material has on the properties of the matrix material. The idea behind this project is to eventually incorporate the filler material in a matrix that will be reinforced with fibers, but for now no fibers will be added to the test materials.

§ 2.1 Matrix

The matrix holds the fibers together in a structural unit and protects them from external damage [Gibson, 1994]. It transfers and distributes the applied loads to the fibers, protects the fibers from environmental influences such as moisture and chemicals, and in many cases the matrix contributes some needed property such as ductility, toughness or electrical insulation. Polymers are the most widely used matrix materials. They fall into two categories: thermoset and thermoplastic. While both types consist of molecular chains, thermosetting plastics (also called ‘resins’) crosslink during the cure reaction (initiated by heat, catalysts, or both) and ‘set’ into a final rigid form [McConnel, 1998]. This process is not reversible and gives high thermal stability and good resistance to creep. Thermoplastic molecular chains are processed at higher temperatures and do not crosslink. As a result, thermoplastics will soften when reheated and can be reshaped more than once. Fiber reinforced composites are mostly made of thermosetting resins; only this resin type will be discussed here.

A few examples of thermosetting resins are:

- Polyester
  Polyester resins have excellent flexibility in terms of the material properties that are attainable. This versatility results from the fact that the resin is a combination of several components; by varying the ratios of constituents used in the manufacture of the resins they can be tailor-made for specific applications. Other advantages of polyester resins are low cost and good chemical resistance. Disadvantages are comparatively large shrinkage and styrene emission during processing. Polyester resins are generally used in low-tech applications.

- Vinyl esters
  Vinyl esters are, similarly to polyesters, available in different chemical compositions to allow them to be customized for specific applications. They offer improved mechanical and chemical properties, have good electrical properties, low peak exothermic temperature and display less shrinkage on cure compared to polyesters. Vinyl esters are, however, more expensive and the adhesive strengths are moderate.

- Epoxy
  Epoxy resin offers the best performance for fiber composite matrix material. Epoxy resins are available in a wide variety of types and display far superior properties to both polyesters and vinyl ester matrix materials. They exhibit extremely low shrinkage on curing, adhere to almost any surface, offer excellent resistance to chemical attack and release no by-products that have a negative effect on the composite. However, epoxy resin systems are relatively expensive and higher viscosity may limit processing. Also, exothermal reactions can cause spontaneous combustion during curing.
Phenolic
Phenol Formaldehyde resins are one of the oldest commercial polymeric resin systems. Their origin can be traced to the early 1900s. Due to their outstanding flame resistance, low smoke generation and low toxicity emission, phenolic resins are used in advanced composites related to aircraft interior and offshore oil platforms. Disadvantages are brittleness and the evolution of by-products during curing.

Other matrix materials
Other types of commercially available matrix materials are polyimide resins, polyurethane resins and silicones. In addition to thermosetting matrix materials discussed, there are a number of thermoplastic resins available, but these resin systems cannot provide the same performance as thermosetting resins.

Selection of matrix systems enables the designer to vary the service temperature capabilities, chemical resistance properties, weatherability, electrical properties, fire resistance, adhesive characteristics, etc. of the finished composite.

The resin system most commonly used in the fabrication of fiber composites is epoxy resin. This resin system will be used in this project.

§ 2.2 Fillers
An other way to influence the suitability of a composite for a specific application is to mix a filler in with the matrix material during fabrication. As the name implies, fillers have commonly been employed to extend the product or to lower the cost. Evident modification of properties of the unfilled materials such as hardness, viscosity or color is brought on by the incorporation of fillers. In addition to these apparent modifications, other, less obvious properties can be enhanced by selective use of fillers. Because of the wide range of particle variables, particles combined in a matrix can produce composites with many unusual properties. Filler materials are now employed to obtain any, or a combination, of the following effects:

- reduce cost
- reduce weight
- extend the resin content
- add rigidity, stiffness or hardness
- regulate thermal expansion and shrinkage
- improve heat resistance
- improve or regulate electrical characteristics
- increase strength and reduce creep
- modify rheological properties (flow, thixotropy)
- aid processability (lubrication, flow, mixing, dispersion, etc.)
- modify appearance (color, texture)

[Whisson, 1971]

The materials known in the plastics industry as fillers are defined by ASTM Standard's Committee D-20 as "relatively inert material added to a plastic to modify its strength, permanence, working properties, or other qualities, or to lower cost". This definition also includes fibrous materials and fabrics, which primarily account for any reference to the modification of strength or permanence. [Stieglitz, 1974]. These fillers will not be discussed here. The fillers that are of interest, however, are powder fillers.
The main types of powder filler are:

- mineral powders, such as carbonates, oxides, clays and aluminosilicates,
- organic powders, such as carbon blacks, graphite, and cellulose,
- metallic powders, such as aluminum, copper, iron, magnesium, silver and zinc,
- glass powders.

As mentioned before, the matrix material used in this project is epoxy resin. This resin type has a (relative) high viscosity, which could be a limiting factor in the production process. The addition of a filler increases the viscosity of the material even further. This viscosity build-up is controlled by filler particle size, size distribution, particle shape and surface characteristics. Minimization of the viscosity build-up can be established by the following filler characteristics:

- minimum surface area to volume ratio,
- maximum packing of particles,
- smooth uniform particle surfaces,
- low surface porosity.

Of all the possible particle shapes, a sphere has minimum surface area to volume ratio and maximum packing of particles is possible. Therefore, microspheres are commonly used as fillers in plastics.

**Microspheres**

Microspheres are particulate fillers that work well in both thermosets and thermoplastics, and can be molded in a variety of processes. They can cut costs, lower density, or add strength. Spheres increase volume loading capacity (figure 2.1), reduce mix viscosity, and can be used to control warpage and reduce shrinkage since a sphere is isotropic (has no orientation) and provides uniform cure shrinkage without causing internal stresses.

![Figure 2.1 Volume loading capacity](image)

Solid spheres provide reinforcement and are tough enough to be extruded or injection molded. Hollow spheres, which can be 10 times lighter than solid spheres, are used for weight reduction because of their low density. They affect most mechanical properties adversely as collapse or breakage of hollow fillers may occur as a result of mixing, pumping, spraying, etc. in the production process. Therefore, hollow microspheres are not suitable for all production processes.
In the following a description of the filler materials used in this project will be given.

§ 2.2.1 E-spheres

The first two filler materials have been designed by Envirosphere Australia Pty.Ltd (Lindfield NSW, Australia). They can be classified as mineral powders. The following information comes from Envirosphere Australia.

E-spheres are white, hollow ceramic microspheres, which are used as versatile, economical and effective fillers in a host of applications and formulations. E-spheres are one of the strongest hollow microspheres available and provide:

- reduced density/weight
- reduced shrinkage
- improved impact and thermal shock resistance
- improved thermal insulation (higher exotherm)
- non-flammable/high melting point
- improved rheology (increased viscosity reduces spillage but may limit processing)
- increased stiffness-to-weight ratio
- high compressive strength
- lower costs (resin extension)
- improved thermal insulation (higher exotherm)
- improved rheology (increased viscosity reduces spillage but may limit processing)

Also, they have a very low moisture content and will not absorb moisture from the atmosphere. Lower moisture content results in a shorter cure time of the resin. E-spheres have an approximate specific gravity (or true density) of 0.7 g/cc and a bulk density of 0.4 g/cc. The shell is approximately 10% of the diameter of the microsphere and is remarkably strong, providing excellent resistance to planar shear as well as excellent isostatic crush resistance.

E-spheres have inherent refractory characteristics resulting in high heat and flame resistance. The ceramic outer shell of the sphere together with its hollow structure provides good thermal insulation properties. Such qualities are well suited to refractory applications and use in manufacture of building materials.

They are relatively chemically inert. Their spherical particle shape, thin surface wall and virtually nil absorption levels provide the benefits of high filler loading at an economical cost with high flow characteristics when compared with other common fillers.

E-spheres are a good filler and resin extender. They are a good replacement for heavy conventional fillers in thermoset, thermoplastic systems, polyesters, epoxies, adhesives etc. Significant reductions in weight and improved impact resistance are obtained, along with the economies resulting from resin reduction.

E-spheres have a particularly low iron oxide content and a significantly higher alumina content than other known ceramic microspheres. These characteristics contribute to the higher melting point (1600-1800 C), which provides their suitability in a wide range of refractory applications.

The concepts of E-sphere usage are:
1. Density and weight reduction
E-spheres used as a means of reducing density of a product provide many potential benefits, e.g. less sag, improved flow, lower handling and installation costs, lower freight costs, increased economies or efficiency, etc.
2. Reduced shrinkage
Shrinkage caused by absorption or hydration generally results in cracking. The surface of E-spheres is non-absorbent, which therefore results in lower shrinkage which is beneficial in cementitious applications, crack repair putties, sealants, etc.

3. Improved impact resistance
Because of the low 1:1 aspect ratio of microspheres, it does not assist in improving most physical parameters. Like most mineral fillers, an overload of filler will ultimately destroy the physics of any system. However, microspheres do enhance certain properties like no other filler can. Impact resistance is almost always improved, due to the microspheres acting as miniature shock absorbers within the resin matrix, i.e. their isotropic behaviour redirects force and energy in an omnidirectional manner.

4. Thermal insulation
E-spheres are non-flammable and the high alumina and low iron content of the shell gives the series an exceptionally high melting point. This together with the hollow center also provides excellent thermal insulation. These features are beneficial in refractory applications and a wide range of thermal insulation and fire retardant products, e.g. automotive components and construction materials.

5. Rheology
Due to the spherical shape and smooth surface of the microspheres, mix viscosity is reduced, giving an equal volume of filler loading.

6. Increased stiffness-to-weight ratio
Microspheres in polyurethane or syntactic foams (combination of microspheres and a polymeric resin) have a minimal effect on flexural strength, compressive modulus and compressive strength, but increase the flexural modulus and therefore the overall stiffness with little effect on weight.

7. Compressive strength
E-spheres possess extremely high isostatic compressive strengths compared with other types of hollow ceramic microspheres and non-ceramic hollow spheres. Tests on the series produced results of 700 kg/cm² with an attrition rate of only 60%

8. Reduced costs
E-spheres will reduce the resin requirements.

Two brands of E-sphere fillers were used: SLG and BLF. Some of their properties are given in the following section.

**SLG series:**
Description: Standard grade, full particle distribution.
Nominal particle size: 20-300 microns
Approximate particle mean: 130 microns
Bulk density: 0.39-0.42 g/cc
Relative density: 0.70-0.78 g/cc
Moisture content (max): 0.1%
Sediment % by volume (max): 3%
BLF series:
Approximate particle mean: 160 microns
Bulk density: 0.34-0.39 g/cc
Relative density: 0.6-0.7 g/cc
Moisture content (max): 3%
Floaters % by volume typically: 95-99%

<table>
<thead>
<tr>
<th>Micron range</th>
<th>% passing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top size range</td>
<td>250-300</td>
</tr>
<tr>
<td>Mid size range</td>
<td>106-212</td>
</tr>
<tr>
<td>Passing</td>
<td>106</td>
</tr>
</tbody>
</table>

Table 2.1 Particle size distribution for E-spheres BLF

<table>
<thead>
<tr>
<th></th>
<th>BLF</th>
<th>SLG</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>59.40%</td>
<td>55.00%</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>33.20%</td>
<td>43.30%</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>1.97%</td>
<td>0.50%</td>
</tr>
<tr>
<td>TiO₂</td>
<td>1.26%</td>
<td>1.40%</td>
</tr>
<tr>
<td>MnO</td>
<td>0.01%</td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>1.44%</td>
<td></td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.16%</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>1.48%</td>
<td></td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.15%</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>0.79%</td>
<td></td>
</tr>
<tr>
<td>SO₃</td>
<td>0.08%</td>
<td></td>
</tr>
<tr>
<td>BaO</td>
<td>0.03%</td>
<td></td>
</tr>
<tr>
<td>SrO</td>
<td>0.03%</td>
<td></td>
</tr>
<tr>
<td>ZnO</td>
<td>0.01%</td>
<td></td>
</tr>
</tbody>
</table>

Table 2.2 Typical chemical analysis of E-spheres BLF and SLG: Oxide % by weight

§ 2.2.2 3M Glass Bubbles K-series

The next two fillers are glass bubbles. They are produced by 3M Australia Pty. Ltd. (St. Marys NSW, Australia) and are part of the 3M Glass Bubbles K-series. The particles of this filler are white, hollow, unicellular glass microspheres, composed of a water resistant and chemically stable borosilicate glass. In general, the chemical properties of these glass bubbles resemble those of a soda-lime-borosilicate glass.

The strength of the hollow bubbles, which varies with density, is a function of wall thickness. Average wall thickness ranges from 0.5 μm, for bubbles having an isostatic strength of 250 psi, to 2 μm for the 10,000 psi microsphere.
Because glass spheres have less surface area than irregularly shaped fillers such as perlites, they permit higher loading in a resin without exceeding viscosity limitations. Loading is limited, however, by the amount that would reduce properties of the compound, although some properties are improved with glass microspheres. They increase stiffness-to-weight ratio, improve dimensional stability, lower dielectric constant, and improve sanding and machining rates. Furthermore, because glass is inert, it improves the corrosion resistance of a composite and decreases the flammability of resins.

Some of the benefits of 3M K fillers are:
- resin extension
- weight reduction
- high filler loading for improved flow
- abrasion resistance

Two filler materials from the 3M K-series were used; K1 and K15. Some of the physical properties of these fillers are listed below.

Typical Density:
K1: 0.125 g/cc
K15: 0.15 g/cc

<table>
<thead>
<tr>
<th>Product</th>
<th>test pressure [psi]</th>
<th>Typical % Survival</th>
<th>Minimal % Survival</th>
</tr>
</thead>
<tbody>
<tr>
<td>K1</td>
<td>250</td>
<td>90%</td>
<td>80%</td>
</tr>
<tr>
<td>K15</td>
<td>300</td>
<td>90%</td>
<td>80%</td>
</tr>
</tbody>
</table>

Table 2.3 Isostatic crush strength of 3M K1 and 3M K15

<table>
<thead>
<tr>
<th>Microns</th>
<th>% passing</th>
</tr>
</thead>
<tbody>
<tr>
<td>125</td>
<td>100</td>
</tr>
<tr>
<td>102</td>
<td>90</td>
</tr>
<tr>
<td>70</td>
<td>50</td>
</tr>
<tr>
<td>35</td>
<td>10</td>
</tr>
</tbody>
</table>

Table 2.4 Particle size distribution for K1 and K15 Glass Bubble

To obtain a good bond between the spheres and the resin it is recommended to put a coating on the spheres. The type of coupling agent depends upon the type of resin used.

§ 2.2.3 Fly ash

The last filler is an organic filler: fly ash. Fly ash is a by-product of burning finely ground coal in electricity generating power plants. Australian coal-fired power stations produce over 8 million tons of fly ash every year. It is captured from the exhaust gases of the plants by electrostatic precipitators or bag houses, which leaves relatively clean air to escape from the smokestacks. This accomplishes three objectives:
the fine fly ash is prevented from polluting the ambient air,
the collected material is diverted from the waste stream, and, once processed, becomes a valuable recycled resource for industry, and
the energy investment in processing virgin materials is reduced, and virgin materials are conserved.

The material is composed primarily of complex aluminosilicate glass, mullite, hematite, magnetic spinel and quartz. The proportion of quartz in fly ash varies depending on the quartz content of the coal. Two classifications of fly ash are produced, according to the type of coal used. Anthracite and bituminous coal produce fly ash classified as Class F. Class C fly ash is produced by burning lignite or subbituminous coal. Class C fly ash is the main type offered.

Fly ash particles carried out of the boiler by the exhaust gases are extremely variable but have some characteristics of interest. The particles are generally less than 250 micrometers in size, almost totally spherical in shape, have a high mechanical strength, a range of densities from about 3 to less than 0.6 g/cc, a melting point above 1000 °C, low thermal conductivity and are mostly chemically inert.

The cenospheres - literally ‘round hollow balls’ - offer the most exciting possibilities. These have a density of less than 1.0 g/cc and hence float on the surface of ash dams. They are light but very strong. They can be used in epoxy resins.

Fly ash is commonly used in concrete. Thirty percent of the fly ash in the U.S. is recycled into making concrete, which is used, in structural applications. It improves workability, performance and quality of concrete and reduces water demand and permeability. Use of fly ash in plastics can result in an increased stiffness-to-weight ratio. Using fly ash not only results in a stronger composite, it also increases the recycled content of the product, which is an important concern in today’s society.

The fly ash used in this project was provided by Pozzolanic Enterprises Pty.Ltd. (Milton, Brisbane, Australia).
§2.3 Interfacial phenomena

The reader should bear in mind that the strength of a composite not only depends on the material properties of the different constituents but that coherency strains between particle phase and matrix also affect strength. Contact between the filler and the polymer must be intimate and capable of remaining so even when the material is subjected to deformation [Wake, W.C., 1971]. The ease with which contact of polymer and filler is achieved, and the strength and nature of the union are interfacial phenomena occurring on the surface of the filler and leading to modifications in the behaviour of the bulk material. Low energy at the particle-matrix interface implies good coupling, which is necessary if the particles are to act as barriers to dislocation flow [Schwartz, 1984]. High interfacial energy, on the other hand, is equivalent to a hole surrounding a particle. This would not only be a poor barrier to dislocation motion (with a resulting lowering of strength), but would also act as a microcrack in the structure.
3 Fabrication of test specimens

In order to be able to determine the influence that the different filler materials have on the physical properties of the composite, two tests are done:

- test beams were subjected to a three point flexural test to ascertain the flexural strength, the flexural modulus and the flexural strain, and
- cylinders were subjected to a compression test to determine the compression strength, the compression modulus and the compression strain.

In the following, the fabrication of the test specimens will be described.

Materials

The following materials were used in the fabrication of the test specimens:

- Resin 1: Epoxy, ADR246
- Resin 2: Epoxy, ADR246 TX
- Hardener: Slow hardener, ADH160
- Filler 1: E-spheres, BLF grade
- Filler 2: E-spheres, SLG grade
- Filler 3: 3M Glass Bubbles, K1
- Filler 4: 3M Glass Bubbles, K15
- Filler 5: Fly ash

Sample identification

The flexure samples were identified with J and two numbers, and the compression samples with JC and two numbers. One mixture was made to make the flexure beams as well as the compression cylinders. Firstly, test specimens were made of pure resin as a reference. The J(C)01 series was made of resin 1 and for J(C)02 resin 2 was used. Of the two epoxy resin systems chosen, the second system (ADR246 TX plus ADH160) had the best properties, and this system was used for fabrication of the rest of the test specimens. For these specimens the 5 different filler materials were mixed in with the resin in four different volume percentages, namely 29, 40, 49 and 60 %. A mixture of 69 volume % was made with filler 3, but this was difficult to mix and too much air was entrapment in the composite. A review of the sample identification is given in table 3.1.

To determine the volume of filler material, a measuring cup was used. The filler and the resin were mixed by hand and poured into different molds (figure 3.1):

- The mold for the flexure specimens was a plastic container with the dimensions 300 x 120 x 75 mm. The container was partly filled, until a height of approximately 25 mm was reached.
- The mold for a compression specimen was a cylindrical photo film container, which was slightly tapered. The top diameter was 30 mm and the height was 50 mm. The mold was filled to the brim.

The curing process is an exothermic reaction. This means that heat is produced during curing of the composite. This heat is not wanted, since the molds were made of plastic that would deform when heated. Therefore, after filling the molds, the mixture was cooled with compressed air. When the mixture was completely hardened (this took about two days), the molds were removed and the composites were post cured in an oven for 9 hours at a temperature of 45 °C.
The test specimens were cut from the molded shapes. To determine the flexural properties, an ASTM test standard was followed. According to this standard, 5 flexure beams are needed. The span (this is the distance between the two support points in the flexural test) to depth ratio should be 16 to 1. In accordance with this, the beam dimensions were chosen as follows:

\[ \text{depth} \times \text{width} \times \text{length} = 15 \times 20 \times 270 \text{ mm.} \]

For the compression testing, six specimens were made for each of the material samples. The top and bottom were cut off of the molded shape to give the specimen level and parallel surfaces. Prior to testing, the flexural and compression specimens were conditioned at room temperature (23 ± 2°C) and 50 ± 5% relative humidity, for not less than 40 hours.

More detailed specimen fabrication records can be found in appendix A.

<table>
<thead>
<tr>
<th>Test specimen</th>
<th>Filler type</th>
<th>Volume %</th>
</tr>
</thead>
<tbody>
<tr>
<td>J(C)01</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>J(C)02</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>J(C)03</td>
<td>BLF</td>
<td>49</td>
</tr>
<tr>
<td>J(C)04</td>
<td>SLG</td>
<td>49</td>
</tr>
<tr>
<td>J(C)05</td>
<td>K1</td>
<td>49</td>
</tr>
<tr>
<td>J(C)06</td>
<td>K15</td>
<td>49</td>
</tr>
<tr>
<td>J(C)07</td>
<td>BLF</td>
<td>29</td>
</tr>
<tr>
<td>J(C)08</td>
<td>SLG</td>
<td>29</td>
</tr>
<tr>
<td>J(C)09</td>
<td>K1</td>
<td>29</td>
</tr>
<tr>
<td>J(C)10</td>
<td>K15</td>
<td>29</td>
</tr>
<tr>
<td>J(C)12</td>
<td>SLG</td>
<td>60</td>
</tr>
<tr>
<td>J(C)13</td>
<td>K1</td>
<td>60</td>
</tr>
<tr>
<td>J(C)14</td>
<td>K15</td>
<td>60</td>
</tr>
<tr>
<td>J(C)16</td>
<td>SLG</td>
<td>40</td>
</tr>
<tr>
<td>J(C)17</td>
<td>K1</td>
<td>40</td>
</tr>
<tr>
<td>J(C)18</td>
<td>K15</td>
<td>40</td>
</tr>
<tr>
<td>J(C)21</td>
<td>K1</td>
<td>69</td>
</tr>
<tr>
<td>J(C)23</td>
<td>Fly ash</td>
<td>29</td>
</tr>
<tr>
<td>J(C)24</td>
<td>Fly ash</td>
<td>40</td>
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<tr>
<td>J(C)25</td>
<td>Fly ash</td>
<td>49</td>
</tr>
<tr>
<td>J(C)26</td>
<td>Fly ash</td>
<td>60</td>
</tr>
</tbody>
</table>

Table 3.1 Sample identification and filler volume percentage
Figure 3.1 The molds
4 Test methods

§ 4.1 Flexural tests

The flexural testing was done according to the ASTM D 790M-93 standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials. This test standard describes two test methods:

- Method I: a three-point loading system utilizing center loading on a simple supported beam.
- Method II: a four-point loading system utilizing two load points equally spaced from their adjacent support points, with a distance between load points of either one third or one half of the support span.

The basic difference between the two test methods is in the location of the maximum bending moment and maximum stress. The maximum stress occurs on a line under the loading nose in test method I and over the area between the loading noses in test method II. Since the maximum deflection is measured more easily in test method I, this method was chosen.

Apparatus
The flexural tests were carried out on a Shimadzu Universal Testing Machine, type: REH-10 T.V. (Seisakusho LTD). The cylindrical supports and the loading nose had a radius of 16 mm, and the support span was 240 mm.

![Figure 4.1 Flexural test configuration](image)

Measuring the load
The loading nose was connected to a load cell (Scale Components, 5 kLB). The load cell was sampled at a frequency of 5 scans per second. The load was numerically displayed using the software program System 5000, version 2.13 (Strain Smart Application Software). In order to plot a load-deflection curve, simultaneous load-deflection data were taken for every 0.5 mm deflection. The load, however, was difficult to read, since there was too much fluctuation in the displayed numbers. To solve this problem, the scan number was written down instead of the actual load, and after the test the load was found in the data file that was created by System 5000. This also made it possible to check the crosshead motion. The test standard required a crosshead motion of 6.4 mm/min ± 50 %.
Measuring the deflection
The deflection of the flexural beams was measured with a dial gage, by measurement of the motion of the loading nose relative to the supports. The loading nose had rotational freedom around the vertical axis. Therefore, placing the dial gage directly on the loading nose would give inaccurate measurement. Since the loading nose was connected to the load cell without translational freedom, a dial gage could be placed on the base of the load cell to measure the deflection of the test specimen.

The specimen is deflected until rupture occurs or until the maximum strain of 5% is reached, whichever occurs first.

§ 4.2 Compression tests

Apparatus
In order to determine the compression properties of the composites, cylinders were fabricated, using photo film containers as a mold. The compression cylinders were tested on an Avery testing machine, type: 7110 DCJ. Figure 4.2 shows the test configuration.

![Figure 4.2 Compression test configuration](image)

Measuring the compression distance
The compression distance was measured with a dial gage. Three readings were done: the first two at relative small loads, in order to calculate the compression modulus, and the third reading was taken at maximum occurring load to determine the maximum strain.
5 Calculations

Particulate composites are usually isotropic because particles are randomly distributed. These composite materials exhibit quasi-linear elastic behaviour with relatively low ultimate strains. In general, particulate composites are modeled as linear elastic materials, and equations of elasticity are used to calculate stresses, strains and deflections.

§ 5.1 Flexure

Maximum flexural stress
When a beam of homogeneous, elastic material is tested in flexure as a simple beam supported at two points and loaded at the midpoint, the maximum stress occurs at midspan. This stress may be calculated for any point on the load-deflection curve by the following equation:

\[ \sigma = \frac{3FL}{2bd^2} \quad [\text{MPa}] \]

where:
- \( \sigma \) = stress at midspan, MPa,
- \( F \) = load at a given point on the load-deflection curve, N,
- \( L \) = support span, mm,
- \( b \) = width of beam, mm,
- \( d \) = depth of beam, mm.

Note: This equation applies strictly to materials for which the stress is linearly proportional to strain up to the point of rupture and for which strains are small. Since this is not the case, a slight error will be introduced in the use of this equation. This equation will, however, be valid for comparison data and specification values up to the maximum strain of 5%.

Maximum strain
The maximum strain occurs at midspan, and may be calculated as follows:

\[ \varepsilon_{\text{max}} = 6 \frac{D_{\text{max}}d}{L^2} \quad [-] \]

where:
- \( \varepsilon_{\text{max}} \) = maximum strain,
- \( D_{\text{max}} \) = maximum deflection of the center of the beam, mm,
- \( L \) = support span, mm,
- \( d \) = depth of beam, mm.

Tangent modulus of elasticity
The tangent modulus of elasticity, often called the modulus of elasticity, is the ratio, within the elastic limit of stress to corresponding strain and shall be expressed in megapascals. It is calculated by drawing a tangent to the steepest initial straight-line portion of the load-deflection curve and using the following equation:

\[ E_t = \frac{L^3m}{4bd^3} \quad [\text{MPa}] \]
where: \( E_B \) = modulus of elasticity in bending, MPa,
\( L \) = support span, mm,
\( b \) = width of beam, mm,
\( d \) = depth of beam, mm,
\( m \) = slope of the tangent to the initial straight-line portion of the load-deflection curve, N/mm.

Naturally, this equation can be derived by substituting equations (1) and (2) in Hooke's Law \( (\sigma = E \cdot e) \).

For the determination of the slope of the tangent to the initial straight-line portion of the load-deflection curve, two points on the curve are needed. These points are taken at \( \varepsilon = 0.0005 \) and \( \varepsilon = 0.0025 \). Using equation (2), the deflection at these strain levels are \( D = 0.32 \) mm and \( D = 1.60 \) mm.

§ 5.2 Compression

**Maximum compression stress**
The maximum compression stress was calculated as follows:

\[
\sigma_{\text{max}} = \frac{F_{\text{max}}}{\frac{1}{4} \pi d^2} \quad [\text{MPa}]
\]

where: \( \sigma_{\text{max}} \) = maximum compression stress, MPa,
\( F_{\text{max}} \) = maximum occurring load, N,
\( d \) = diameter of cylinder, mm.

**Strain at maximum load**
In order to determine the strain that occurs at maximum load, the following equation can be used:

\[
\varepsilon = \frac{L_{\text{max}}}{H} \quad [-]
\]

where: \( \varepsilon \) = strain,
\( L_{\text{max}} \) = compression distance at maximum load, mm,
\( H \) = original height of compression cylinder, mm.

**Compression modulus**
The compression modulus is calculated according to Hooke's law:

\[
E_C = \frac{(F_2 - F_1) \cdot H}{(L_2 - L_1) \cdot A} \quad [\text{MPa}]
\]

where: \( E_C \) = compression modulus, MPa,
\( F_1 = 3 \cdot 10^5 \) N,
\( F_2 = 6 \cdot 10^5 \) N,
\( L_1 \) = compression distance at \( F_1 \), mm,
\( L_2 \) = compression distance at \( F_2 \), mm,
\( A \) = average section area, mm²,
\( H \) = original height of compression cylinder, mm.
6 Results

§ 6.1 Flexural tests

Among the five test specimens of the J01 sample, there was one beam that did not break within the maximum strain level of 5%. This was also the case with the J02 sample. These two samples were not included in the calculation of the average sample properties, which are listed in table 6.1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$S_{\text{MAX}}$ [Mpa]</th>
<th>$E_B$ [Mpa]</th>
<th>Strain [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>J01</td>
<td>69</td>
<td>2947</td>
<td>0.024</td>
</tr>
<tr>
<td>J02</td>
<td>71</td>
<td>3032</td>
<td>0.025</td>
</tr>
</tbody>
</table>

Table 6.1 Flexural tests results of two resin types

Appendix B contains some of the stress-strain curves. For the J01 and J02 samples, each specimen’s curve is given, for the other samples only one curve is shown.

Figure 6.1 shows the average maximum flexural stress, or the average flexural strength, for all test samples. This property decreases with increasing filler percentage. The fly ash samples have the highest strength, but do not reach the strength of the pure resin.

The average flexural modulus is given in figure 6.2. Composites with E-spheres BLF, E-spheres SLG and fly ash have a higher stiffness with increasing filler percentage, while the opposite is true for the 3M K1 and K15 filled materials. For all samples the flexural modulus is approximately proportional to the filler percentage.
Figure 6.2 Average flexural modulus

Figure 6.3 shows that the flexural strains decrease with increasing filler percentage. This can be caused by the fact that the filler particles deform less due to the applied load than the resin does. Furthermore, it can be seen that the flexural strain is lower for materials with higher flexural stress.

Figure 6.3 Average flexural strain
§ 6.2 Compression tests

The results of the compression tests on the pure resin samples are listed in table 6.2.

<table>
<thead>
<tr>
<th>S_{\text{MAX}} [\text{Mpa}]</th>
<th>E_{\text{C}} [\text{Mpa}]</th>
<th>Strain [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>JC01</td>
<td>101</td>
<td>1930</td>
</tr>
<tr>
<td>JC02</td>
<td>105</td>
<td>2221</td>
</tr>
</tbody>
</table>

Table 6.2 Compression tests results of two resin types

The average compression stress is approximately proportional to filler percentage, as can be seen in figure 6.4. There is a decrease for 3M K1, K15 and E-spheres BLF and SLG composites and an increase for fly ash composites. The fly ash composites with a filler percentage higher than 40% have a compression stress higher than that of the pure resin, the other composites have a lower compression stress.

![Average Maximum Compression Stress](image)

Figure 6.4 Average compression strength

The resulting compression moduli are given in figure 6.5. For 3M K1 and K15 composites the compression modulus is lower than that of the pure resin, whereas for the E-spheres BLF and SLG and for the fly ash composites it is higher than that of the pure resin. $E_{\text{C}}$ increases with increasing E-spheres BLF, E-spheres SLG and fly ash filler percentage and decreases with increasing 3M K1 and K15 filler percentage. It can be seen that the compression modulus has a step for the composites filled with 49 V% E-spheres BLF, E-spheres SLG and 3M K1. This is caused by a difference in calculating the compression modulus. For these three samples, and in the case of the pure resin samples (thus, for the samples J01-J05), $F_1$ and $F_2$ in equation (6) were 5 kN and 10 kN respectively in stead of 3 kN and 6kN. Obviously, this alteration has a big influence on the value of the compression modulus, and therefore figure 6.5 gives a distorted impression.
Figure 6.5 Average compression modulus

Figure 6.6 shows that the compression strains decrease with increasing filler percentage, as did the flexural strains. It should be said that the compression strains were difficult to determine and thus some error may be present. The difficulty lay with reading the compression distance at maximum load, since the maximum load appeared to be constant (or yield) during a few seconds while the compression distance increased.

Figure 6.6 Average compression strain
Standard deviations have been calculated and are listed in appendix C. Significant lower standard deviation of one composite compared with another composite suggests more uniform properties and better filler dispersion.
7 Conclusions and recommendations

The object of this project is to compare the influences of several filler materials on the physical properties of a composite. The filler materials that were chosen for this project were hollow spheres. The spherical geometry of the filler particles was chosen for two reasons:

1. A sphere has minimum surface area to volume ratio, which can minimize the viscosity build-up that is caused by the addition of filler material.
2. A sphere can reduce shrinkage, since it is isotropic and provides uniform cure shrinkage without causing internal stresses.

The reason behind the choice of hollow instead of solid particles lies in the fact that hollow particles reduce the density of the final product. The advantage of this weight reduction is that lightweight products can be manufactured that are manageable and reduce the cost on transportation, installation and long-term maintenance.

Three different types of filler were used: mineral, glass, and organic powders. The effects that the 3M K1 glass bubbles have on the physical properties of the composite resemble those of the 3M K15 filler. This is logical, because the two fillers are made of the same material and their particle size distribution is the same. The composite with 3M K15 filler is slightly stronger, because the K15 bubbles are stronger than the K1 bubbles. The tests on composites filled with 3M glass bubbles teach us that these two fillers do not assist in improving any of the tested physical parameters. It should be noted, however, that the filler particles were not coated. It is likely that the physical properties will improve when a suitable coating is used, because this will result in better coupling between filler and matrix.

Even though only two samples were made with E-spheres BLF, this filler appears to exhibit results that are similar to those of composites filled with E-spheres SLG. This too can be explained by the fact that these two fillers are alike. The SLG spheres are slightly stronger and this reflects on the composite properties. The composites filled with the mineral E-spheres perform better than the composites filled with the 3M glass bubbles. Increased E-sphere percentage results in increased flexural and compression modulus, but decreased strengths and strains.

All in all, fly ash seems to be the best filler material when it comes to strength and stiffness, but it is also the filler with the highest density and therefore causes the least weight reduction.

During the project several problems and questions were encountered and some points of attention are in place:

- Further investigation on filler volume percentages less than 30 % is recommended. It could very well be that for instance a 10 % filler volume still results in an interesting weight reduction and at the same time keeps strength losses to a minimum.

- This project concentrated on the hollow spheres, but there is a lot of diversity in filler materials. Filler materials come in all shapes and sizes, and the combinations with resin types are legion.

- The compression modulus increased when the load at which it was calculated increased. Apparently there is a toe region in the load-compression distance curve that does not represent a property of the material. It is an artefact caused by a take up of slack, and
alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus and strain, this artifact must be compensated for to give the correct zero point on the strain axis.

- It is advisable to determine the filler percentage more accurately, possibly by weight instead of volume. It should also be noted that the production process introduces variable shear forces and pressures which can change the effect a filler has on a volume. The effective density of a filler may differ from its true density in air because of resin absorption, air entrapment or release, and collapse or breakage of hollow low density fillers as a result of mixing in the production process.

- Is it possible to explain and predict the influence of filler materials by investigating interface phenomena?
Bibliography


http://org.et.byu.edu/sample/JAM.html

http://flyash-354918.livebuilder.com/Index.htm

http://www.flyash.com/pozzolanic/html

Appendix A: Specimen Fabrication Records

**General**
- Specimen ID Number: JO1 & JC01
- Specimen Description: Flexure Beams & Compression Cylinders
- Fabrication Date: 08-10-1998

**Material Details**

<table>
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<th>Matrix</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin Type</td>
<td>Epoxy</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Designation</td>
<td>ADR 246</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cure Agent</td>
<td>ADH 160</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Manufacturer</td>
<td>ATL Composites pty. ltd.</td>
<td></td>
<td></td>
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<tr>
<td>Manufacturer Batch Number</td>
<td>980326</td>
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<tr>
<td>Source</td>
<td>ATL Composites pty. ltd.</td>
<td></td>
<td></td>
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<tr>
<td>Certification Date</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Expiration Date</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Filler**
- Type: -
- Designation: :
- Manufacturer: :
- Manufacturer Batch Number: :
- Source: :

**Production Details**

| Material Preparation Method | Hand Lay-up |          |          |          |
| Fabrication Date | 08-10-1998 |          |          |          |
| Fabrication Start Time | 10.15 am | Fabrication End Time | 10.35 am |          |
| Fabrication Location | FCDD Laboratory |          |          |          |
| Cure Date | 15-10-1998 | Cure Cycle | 9 h at 42 °C |          |

- Approximate Mass Percentage Filler: 0
- Volume Percentage Filler: 0

**Environmental Conditions**

| Temperature (°C) | 16 | Relative Humidity (%) | 43 |

**Wet Resin Pot Weight**

| Resin: 960 g | Cure Agent: 240 g | Total: 1200 g | Left over: 14 g |

**Filler Details**

| Volume: 0 liter | Weight: 0 g |
**General**
Specimen ID Number: J02 & JC02  
Specimen Description: Flexure Beams & Compression Cylinders  
Fabrication Date: 08-10-1998

**Material Details**
Matrix
- Resin Type: Epoxy  
- Designation: ADR 246 TX  
- Cure Agent: ADH 160  
- Manufacturer: ATL Composites pty. ltd.  
- Manufacturer Batch Number: 980812  
- Source: ATL Composites pty. ltd.

**Filler**
- Type:  
- Designation:  
- Manufacturer:  
- Manufacturer Batch Number:  
- Source: 

**Production Details**
- Material Preparation Method: Hand Lay-up  
- Fabrication Date: 08-10-1998  
- Fabrication Start Time: 10.40 am  
- Fabrication Location: FCDD Laboratory  
- Cure Date: 15-10-1998  
- Cure Cycle: 9 h at 42 °C

- Fabrication End Time: 11.00 am  
- Approximate Mass Percentage Filler: 0  
- Volume Percentage Filler: 0

**Environmental Conditions**
- Temperature (°C): 16  
- Relative Humidity (%): 43

**Wet Resin Pot Weight**
- Resin: 1000 g  
- Cure Agent: 250 g  
- Total: 1250 g  
- Left over: 15 g

**Filler Details**
- Volume: 0 liter  
- Weight: 0 g
General
Specimen ID Number : J03 & JC03
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 22-10-1998

Material Details
Matrix
- Resin Type : Epoxy
- Designation : ADR 246 TX
- Cure Agent : ADH 160
- Manufacturer : ATL Composites pty. ltd.
- Manufacturer Batch Number : 980812
- Source : ATL Composites pty. ltd.
- Certification Date :
- Expiration Date :

Filler
- Type : E-Spheres
- Designation : BLF grade
- Manufacturer : Envirospheres Pty Ltd
- Manufacturer Batch Number :
- Source : Envirospheres Pty Ltd

Production Details
- Material Preparation Method : Hand Lay-up
- Fabrication Date : 22-10-1998
- Fabrication Start Time : 10.35 am
- Fabrication Location : FCDD Laboratory
- Cure Date : 26-10-1998
- Cure Cycle : 9 h at 42 °C
- Fabrication End Time : 11.00 am

Environmental Conditions
- Temperature (°C) : 21
- Relative Humidity (%) : 49

Wet Resin Pot Weight
- Resin : 580 g
- Cure Agent : 145 g
- Total : 725 g
- Left over : 17 g

Filler Details
- Volume : 0.63 liter
- Weight : 274 g
General
Specimen ID Number : J04 & JC04
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 22-10-1998

Material Details
Matrix
Resin Type : Epoxy
Designation : ADR 246 TX
Cure Agent : ADH 160
Manufacturer : ATL Composites pty. ltd.
Manufacturer Batch Number: 980812
Source : ATL Composites pty. ltd.
Certification Date : 
Expiration Date : 

Filler
Type : E-Spheres
Designation : SLG grade
Manufacturer : Envirospheres Pty Ltd
Manufacturer Batch Number: 
Source : Envirospheres Pty Ltd

Production Details
Material Preparation Method : Hand Lay-up
Fabrication Date : 22-10-1998
Fabrication Start Time : 10.10 am Fabrication End Time : 10.33 am
Fabrication Location : FCDD Laboratory
Cure Date : 26-10-1998 Cure Cycle : 9 h at 42 °C

Approximate Mass Percentage Filler : 26
Volume Percentage Filler : 49

Environmental Conditions
Temperature (°C) : 21
Relative Humidity (%) : 50

Wet Resin Pot Weight
Resin : 580 g Cure Agent : 145 g Total : 725 g Left over : 18 g

Filler Details
Volume: 0.63 liter Weight: 256 g
General
Specimen ID Number: J05 & JC05
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 20-10-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: 980812
Source: ATL Composites pty. ltd.
Certification Date:
Expiration Date:

Filler
Type: Glass Bubbles
Designation: K1
Manufacturer: 3M Australia Pty Limited
Manufacturer Batch Number:
Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 20-10-1998
Fabrication Start Time: 1.05 pm Fabrication End Time: 1.40 pm
Fabrication Location: FCDD Laboratory
Cure Date: 26-10 1998 Cure Cycle: 9 h at 42 °C

Approximate Mass Percentage Filler: 5
Volume Percentage Filler: 49

Environmental Conditions
Temperature (°C): 17
Relative Humidity (%): 48

Wet Resin Pot Weight
Resin: 580 g Cure Agent: 145 g Total: 725 g Left over: 15 g

Filler Details
Volume: 0.63 liter Weight: 42 g
**General**

Specimen ID Number : J06 & JC06
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 20-10-1998

**Material Details**

**Matrix**
Resin Type : Epoxy
Designation : ADR 246 TX
Cure Agent : ADH 160
Manufacturer : ATL Composites pty. ltd.
Manufacturer Batch Number: 980812
Source : ATL Composites pty. ltd.
Certification Date :
Expiration Date :

**Filler**
Type : Glass Bubbles
Designation : K15
Manufacturer : 3M Australia Pty Limited
Manufacturer Batch Number:
Source : 3M Australia Pty Limited

**Production Details**
Material Preparation Method : Hand Lay-up
Fabrication Date : 20-10-1998
Fabrication Start Time : 1.45 pm  Fabrication End Time : 2.15 pm
Fabrication Location : FCDD Laboratory
Cure Date : 26-10 1998
Cure Cycle : 9 h at 42 °C

Approximate Mass Percentage Filler : 6
Volume Percentage Filler : 49

**Environmental Conditions**
Temperature (°C) : 17
Relative Humidity (%) : 48

**Wet Resin Pot Weight**
Resin : 580 g  Cure Agent : 145 g  Total : 725 g  Left over : 12 g

**Filler Details**
Volume: 0.63 liter  Weight: 46 g
General
Specimen ID Number : J07 & JC07
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 12-11-1998

Material Details
Matrix
Resin Type : Epoxy
Designation : ADR 246 TX
Cure Agent : ADH 160
Manufacturer : ATL Composites pty. ltd.
Manufacturer Batch Number : 980812
Source : ATL Composites pty. ltd.
Certification Date :
Expiration Date :

Filler
Type : E-Spheres
Designation : BLF grade
Manufacturer : Envirospheres Pty Ltd
Manufacturer Batch Number :
Source : Envirospheres Pty Ltd

Production Details
Material Preparation Method : Hand Lay-up
Fabrication Date : 12-11-1998
Fabrication Start Time : 11.45 am Fabrication End Time : 12.20 am
Fabrication Location : FCDD Laboratory
Cure Date : 15-11 1998 Cure Cycle : 13.5 h at 42 °C
Approximate Mass Percentage Filler : 13
Volume Percentage Filler : 29

Environmental Conditions
Temperature (°C) : 21
Relative Humidity (%) : 76

Wet Resin Pot Weight
Resin : 924 g Cure Agent : 231 g Total : 1155 g Left over : ? g

Filler Details
Volume: 0.417 liter Weight: 171 g
General
Specimen ID Number : J08 & JC08
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 12-11-1998

Material Details
Matrix
Resin Type : Epoxy
Designation : ADR 246 TX
Cure Agent : ADH 160
Manufacturer : ATL Composites pty. ltd.
Manufacturer Batch Number: 980812
Source : ATL Composites pty. ltd.
Certification Date :
Expiration Date :

Filler
Type : E-Spheres
Designation : SLG grade
Manufacturer : Envirospheres Pty Ltd
Manufacturer Batch Number:
Source : Envirospheres Pty Ltd

Production Details
Material Preparation Method : Hand Lay-up
Fabrication Date : 12-11-1998
Fabrication Start Time : 11.10 am Fabrication End Time : 11.40 am
Fabrication Location : FCDD Laboratory
Cure Date : 15-11 1998 Cure Cycle : 13.5 h at 42 °C

Approximate Mass Percentage Filler : 14
Volume Percentage Filler : 29

Environmental Conditions
Temperature (°C) : 21
Relative Humidity (%) : 76

Wet Resin Pot Weight
Resin : 924 g Cure Agent : 231 g Total : 1155 g Left over : 48 g

Filler Details
Volume: 0.417 liter Weight: 183 g
General
Specimen ID Number: J09 & JC09
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 12-11-1998

Material Details
Matrix
  Resin Type: Epoxy
  Designation: ADR 246 TX
  Cure Agent: ADH 160
  Manufacturer: ATL Composites pty. ltd.
  Manufacturer Batch Number: 980812
  Source: ATL Composites pty. ltd.
  Certification Date:
  Expiration Date:

Filler
  Type: Glass Bubbles
  Designation: K1
  Manufacturer: 3M Australia Pty Limited
  Manufacturer Batch Number:
  Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 12-11-1998
Fabrication Start Time: 9.36 am  Fabrication End Time: 10.15 am
Fabrication Location: FCDD Laboratory
Cure Date: 15-11-1998  Cure Cycle: 13.5 h at 42 °C

Approximate Mass Percentage Filler: 3
Volume Percentage Filler: 29

Environmental Conditions
  Temperature (°C): 20
  Relative Humidity (%): 76

Wet Resin Pot Weight
  Resin: 924 g  Cure Agent: 231 g  Total: 1155 g  Left over: 38 g

Filler Details
  Volume: 0.417 liter  Weight: 31 g
General
Specimen ID Number: J10 & JC10
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 12-11-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: 980812
Source: ATL Composites pty. ltd.
Certification Date:
Expiration Date:

Filler
Type: Glass Bubbles
Designation: K15
Manufacturer: 3M Australia Pty Limited
Manufacturer Batch Number:
Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 12-11-1998
Fabrication Start Time: 10.25 am Fabrication End Time: 11.00 am
Fabrication Location: FCDD Laboratory
Cure Date: 15-11 1998 Cure Cycle: 13.5 h at 42 °C

Approximate Mass Percentage Filler: 3
Volume Percentage Filler: 29

Environmental Conditions
Temperature (°C): 20
Relative Humidity (%): 76

Wet Resin Pot Weight
Resin: 924 g Cure Agent: 231 g Total: 1155 g Left over: 44 g

Filler Details
Volume: 0.417 liter Weight: 34 g
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<td>Volume: 1 liter</td>
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General
Specimen ID Number: J13 & JC13
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 26-11-1998

Material Details
Matrix
- Resin Type: Epoxy
- Designation: ADR 246 TX
- Cure Agent: ADH 160
- Manufacturer: ATL Composites pty. ltd.
- Manufacturer Batch Number: D981117
- Source: ATL Composites pty. ltd.
- Certification Date:
- Expiration Date:

Filler
- Type: Glass Bubbles
- Designation: K1
- Manufacturer: 3M Australia Pty Limited
- Manufacturer Batch Number: 3M Australia Pty Limited

Production Details
- Material Preparation Method: Hand Lay-up
- Fabrication Date: 26-11-1998
- Fabrication Start Time: 9.05 am
- Fabrication End Time: 9.58 am
- Fabrication Location: FCDD Laboratory
- Cure Date: 29-11 1998
- Cure Cycle: 9 h at 43 °C
- Approximate Mass Percentage Filler: 8
- Volume Percentage Filler: 60

Environmental Conditions
- Temperature (°C): 20
- Relative Humidity (%): 76

Wet Resin Pot Weight
- Resin: 600 g
- Cure Agent: 150 g
- Total: 750 g
- Left over: 23 g

Filler Details
- Volume: 1 liter
- Weight: 66 g
General
Specimen ID Number: J14 & JC14
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 25-11-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: D98117
Source: ATL Composites pty. ltd.
Certification Date: 
Expiration Date: 

Filler
Type: Glass Bubbles
Designation: K15
Manufacturer: 3M Australia Pty Limited
Manufacturer Batch Number: 
Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 25-11-1998
Fabrication Start Time: 3.03 pm Fabrication End Time: 3.50 pm
Fabrication Location: FCDD Laboratory
Cure Date: 29-11 1998 Cure Cycle: 9 h at 43 °C

Approximate Mass Percentage Filler: 9
Volume Percentage Filler: 60

Environmental Conditions
Temperature (°C): 23
Relative Humidity (%): 70

Wet Resin Pot Weight
Resin: 600 g Cure Agent: 150 g Total: 750 g Left over: 22 g

Filler Details
Volume: 1 liter Weight: 78 g
General
Specimen ID Number : J16 & JC16
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 26-11-1998

Material Details
Matrix
  Resin Type : Epoxy
  Designation : ADR 246 TX
  Cure Agent : ADH 160
  Manufacturer : ATL Composites pty. ltd.
  Manufacturer Batch Number : D981117
  Source : ATL Composites pty. ltd.
  Certification Date : 
  Expiration Date : 
Filler
  Type : E-Spheres
  Designation : SLG grade
  Manufacturer : Envirospheres Pty Ltd
  Manufacturer Batch Number : 
  Source : Envirospheres Pty Ltd

Production Details
Material Preparation Method : Hand Lay-up
Fabrication Date : 26-11-1998
Fabrication Start Time : 3.42 pm Fabrication End Time : 4.10 pm
Fabrication Location : FCDD Laboratory
Cure Date : 29-11 1998 Cure Cycle : 9 h at 43 °C
Approximate Mass Percentage Filler : 20
Volume Percentage Filler : 40

Environmental Conditions
  Temperature (°C) : 24
  Relative Humidity (%) : 76

Wet Resin Pot Weight
  Resin : 852 g Cure Agent : 213 g Total : 1065 g Left over : 102 g

Filler Details
  Volume: 0.63 liter Weight: 272 g
General
Specimen ID Number: J17 & JC17
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 26-11-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: D981117
Source: ATL Composites pty. ltd.
Certification Date:
Expiration Date:

Filler
Type: Glass Bubbles
Designation: K1
Manufacturer: 3M Australia Pty Limited
Manufacturer Batch Number:
Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 26-11-1998
Fabrication Start Time: 10.35 am Fabrication End Time: 11.07 am
Fabrication Location: FCDD Laboratory
Cure Date: 29-11 1998 Cure Cycle: 9 h at 43 °C

Approximate Mass Percentage Filler: 4
Volume Percentage Filler: 40

Environmental Conditions
Temperature (°C): 22
Relative Humidity (%): 76

Wet Resin Pot Weight
Resin: 852 g Cure Agent: 213 g Total: 1065 g Left over: 94 g

Filler Details
Volume: 0.63 liter Weight: 45 g
General
Specimen ID Number: J18 & JC18
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 26-11-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: D981117
Source: ATL Composites pty. ltd.
Certification Date:
Expiration Date:

Filler
Type: Glass Bubbles
Designation: K15
Manufacturer: 3M Australia Pty Limited
Manufacturer Batch Number:
Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 26-11-1998
Fabrication Start Time: 3.05 pm Fabrication End Time: 3.35 pm
Fabrication Location: FCDD Laboratory
Cure Date: 29-11 1998 Cure Cycle: 9 h at 43 °C

Approximate Mass Percentage Filler: 5
Volume Percentage Filler: 40

Environmental Conditions
Temperature (°C): 24
Relative Humidity (%): 76

Wet Resin Pot Weight
Resin: 852 g Cure Agent: 213 g Total: 1065 g Left over: 109 g

Filler Details
Volume: 0.63 liter Weight: 51 g
General
Specimen ID Number: J21 & JC21
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 25-11-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: D981117
Source: ATL Composites pty. ltd.
Certification Date: 
Expiration Date: 

Filler
Type: Glass Bubbles
Designation: K1
Manufacturer: 3M Australia Pty Limited
Manufacturer Batch Number: 
Source: 3M Australia Pty Limited

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 25-11-1998
Fabrication Start Time: 12.05 am  Fabrication End Time: 1.05 am
Fabrication Location: FCDD Laboratory
Cure Date: 29-11 1998  Cure Cycle: 9 h at 43 °C

Approximate Mass Percentage Filler: 13
Volume Percentage Filler: 69

Environmental Conditions
Temperature (°C): 22
Relative Humidity (%): 78

Wet Resin Pot Weight
Resin: 408 g  Cure Agent: 102 g  Total: 510 g  Left over: 20 g

Filler Details
Volume: 1 liter  Weight: 78 g
General
Specimen ID Number : J23 & JC23
Specimen Description : Flexure Beams & Compression Cylinders
Fabrication Date : 03-12-1998

Material Details
Matrix
Resin Type : Epoxy
Designation : ADR 246 TX
Cure Agent : ADH 160
Manufacturer : ATL Composites pty. ltd.
Manufacturer Batch Number : D981117
Source : ATL Composites pty. ltd.
Certification Date : 
Expiration Date : 

Filler
Type : Flyash
Designation : 
Manufacturer : Pozzolanic Enterprises pty. ltd.
Manufacturer Batch Number : 
Source : Pozzolanic Enterprises pty. ltd.

Production Details
Material Preparation Method : Hand Lay-up
Fabrication Date : 03-12-1998
Fabrication Start Time : 10.30 am Fabrication End Time : 11.10 am
Fabrication Location : FCDD Laboratory
Cure Date : 06-12 1998 Cure Cycle : 9 h at 42 °C

Approximate Mass Percentage Filler : 19
Volume Percentage Filler : 29

Environmental Conditions
Temperature (°C) : 22
Relative Humidity (%) : 49

Wet Resin Pot Weight
Resin : 924 g Cure Agent : 231 g Total : 1155 g Left over : 38 g

Filler Details
Volume: 0.417 liter Weight: 265 g
General
Specimen ID Number: J24 & JC24
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 03-12-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: D981117
Source: ATL Composites pty. ltd.
Certification Date:
Expiration Date:

Filler
Type: Flyash
Designation:
Manufacturer: Pozzolanic Enterprises pty. ltd.
Manufacturer Batch Number:
Source: Pozzolanic Enterprises pty. ltd.

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 03-12-1998
Fabrication Start Time: 9.30 am Fabrication End Time: 10.25 am
Fabrication Location: FCDD Laboratory
Cure Date: 06-12 1998 Cure Cycle: 9 h at 42 °C

Approximate Mass Percentage Filler: 29
Volume Percentage Filler: 40

Environmental Conditions
Temperature (°C): 21
Relative Humidity (%): 60

Wet Resin Pot Weight
Resin: 852 g Cure Agent: 213 g Total: 1065 g Left over: 46 g

Filler Details
Volume: 0.63 liter Weight: 439 g
### General

**Specimen ID Number**: J25 & JC25  
**Specimen Description**: Flexure Beams & Compression Cylinders  
**Fabrication Date**: 02-12-1998

### Material Details

**Matrix**

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### Production Details

**Material Preparation Method**: Hand Lay-up  
**Fabrication Date**: 02-12-1998  
**Fabrication Start Time**: 12.25 am  
**Fabrication End Time**: 1.10 pm  
**Fabrication Location**: FCDD Laboratory  
**Cure Date**: 06-12 1998  
**Cure Cycle**: 9 h at 42 °C

**Approximate Mass Percentage Filler** : 38  
**Volume Percentage Filler** : 49

### Environmental Conditions

**Temperature (°C)** : 23  
**Relative Humidity (%)** : 48

### Wet Resin Pot Weight

**Resin**: 580 g  
**Cure Agent**: 145 g  
**Total**: 725 g  
**Left over**: 25 g

### Filler Details

**Volume**: 0.63 liter  
**Weight**: 439 g
General
Specimen ID Number: J26 & JC26
Specimen Description: Flexure Beams & Compression Cylinders
Fabrication Date: 02-12-1998

Material Details
Matrix
Resin Type: Epoxy
Designation: ADR 246 TX
Cure Agent: ADH 160
Manufacturer: ATL Composites pty. ltd.
Manufacturer Batch Number: D981117
Source: ATL Composites pty. ltd.
Certification Date: 
Expiration Date: 

Filler
Type: Flyash
Designation: 
Manufacturer: Pozzolanic Enterprises pty. ltd.
Manufacturer Batch Number: 
Source: Pozzolanic Enterprises pty. ltd.

Production Details
Material Preparation Method: Hand Lay-up
Fabrication Date: 02-12-1998
Fabrication Start Time: 11.10 am
Fabrication End Time: 12.20 am
Fabrication Location: FCDD Laboratory
Cure Date: 06-12 1998
Cure Cycle: 9 h at 42 °C

Approximate Mass Percentage Filler: 45
Volume Percentage Filler: 60

Environmental Conditions
Temperature (°C): 21
Relative Humidity (%): 57

Wet Resin Pot Weight
Resin: 600 g
Cure Agent: 150 g
Total: 750 g
Left over: 39 g

Filler Details
Volume: 1 liter
Weight: 623 g
Appendix B: Stress-Strain Curves

Stress-Strain Curve for J01-1

Stress-Strain Curve for J01-2
Stress-Strain Curve for J09-5

Stress-Strain Curve for J10-4

Stress-Strain Curve for J12-2
Stress-Strain Curve for J13-1

Stress-Strain Curve for J14-5

Stress-Strain Curve for J16-3
## Appendix C: Standard Deviations

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