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FRIEDEL-CRAFTS RESIN/CARBON FIBRE COMPOSITES: PART VI. MECHANIC--ETC(U)

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⑥ FRIEDEL-CRAFTS RESIN/CARBON FIBRE COMPOSITES:  
PART VI. MECHANICAL PROPERTIES.

by

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⑪ APR 76

⑫ 48p.

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SUMMARY

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ABSTRACT

→ The mechanical properties have been measured for composites of several types of carbon fibre with two different Friedel-Crafts resin matrices cured with two catalysts.

Composite void contents averaged 4% and a processing procedure for minimising voids is suggested. All composite mechanical properties were affected by the high void contents. However, calculations showed that strengths comparable with epoxy resin composites could potentially be obtained at zero void content.

Composites where the resin was cured with  $FeCl_3$  had excellent strength retention at  $200^{\circ}C$  after postcure.  $BF_3$ -cured resin composites had better interlaminar shear strengths than  $FeCl_3$ -cured but had poorer properties at elevated temperatures.

Aging at high temperatures indicated that, for  $FeCl_3$ -cured resin, the time taken to lose a quarter of the initial composite strength would be one year at  $200^{\circ}C$  and 10 years at  $150^{\circ}C$ .

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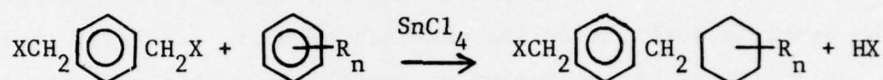
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## 1 INTRODUCTION

The original work of Friedel and Crafts in the 1870s described a number of reactions carried out in the presence of  $\text{AlCl}_3$  as catalyst. Since then the term 'Friedel-Crafts reaction' has come to embrace a very wide variety of alkylation and acylation reactions in the presence of Lewis acid-type acidic metal halides or Brønsted-Lowry proton acids.

Although a soluble polymer was produced as early as 1937<sup>1</sup> using stannic chloride as catalyst, the possibility of using the reaction for the production of a cross-linkable thermo-setting resin was not appreciated until the 1960s<sup>2</sup>.

The basic reaction for the preparation of such resins is



where  $\text{X} = -\text{Cl}$  or  $-\text{OCH}_3$ .  $\text{R}$  can be any group which is not electron-withdrawing and  $n$  has a maximum value of 4.

Chain growth proceeds by further condensation reactions involving abstraction of hydrogen atoms from the aromatic rings.

The first resins were made using *p*-xylylene dichloride ( $\text{X} = -\text{Cl}$ ). Asbestos laminates showed excellent heat resistance in comparison with phenolic resins but the production of gaseous  $\text{HCl}$  during lamination made their use unattractive.

An alternative system substituting *p*-xylylene glycol dimethyl ether [1,4-di (methoxy methyl) benzene] where the volatile product is methanol was chosen for the development of a resin system suitable for use at high temperatures as a matrix for carbon fibre reinforced composites.

Previous reports<sup>3-5</sup> have described the preparation of resins and some properties of their composites. In particular, the composites were evaluated for long term use at elevated temperatures ( $200^\circ\text{C}$  and above). The chemical resistance has also been studied<sup>6</sup>.

This Report gives the results of further work on the terphenyl based resin including mechanical properties and heat resistance of the carbon fibre composites. Consideration is given to the role of processing in determining the void content of a composite and the relation between void content and mechanical properties.

The results are compared with those previously obtained and with those for composites with epoxy resin matrices.

## 2 PREPARATION OF RESINS

When the condensation reaction is taken to maximum conversion, the product is a three dimensional insoluble gel. However the reaction can be stopped by cooling at any point almost up to the gel point to give a liquid resin of viscosity dependent on the extent of reaction. This resin is soluble in polar solvents such as 1,2-dichloroethane.

The aromatic reactants which have been studied in detail are toluene<sup>3</sup>, xylene<sup>4</sup>, terphenyl<sup>3</sup> and diphenyl oxide<sup>5</sup>.

Resins were prepared by heating together *p*-xylylene glycol dimethyl ether (DME) and the aromatic reactant in the presence of  $\text{SnCl}_4$  catalyst. The extent of reaction was monitored by the volume of distillate - mainly methanol. The resin resulting was characterised by its viscosity at 30°C.

The collected results are shown in Fig.1 where the points are differentiated only by the aromatic reactant used, the points for each reactant including resins made with a range of ratios of DME to reactant. Multiple ring reactants gave higher viscosity resins at the same extent of reaction than did single ring aromatic compounds. Since the typical preparation temperature for the resins (130°C upwards) was higher than the boiling points of the latter reactants, the difference was at least partially due to the loss of reactant in the distillate.

Details of the resins are given in Table 1. The ratio of DME to aromatic reactant selected was that giving carbon fibre composites with the highest retention at 200°C of room temperature flexural strength, after a standard postcure. Resins based on terphenyl and diphenyl oxide used in this study were synthesised by an outside firm.

The excellent shelf-life of the resins is illustrated in Fig.2 where the viscosity of a resin containing  $\text{FeCl}_3$  catalyst is shown to have increased only slightly during 10 months storage at room temperature.

## 3 COMPOSITE TEST METHODS

### 3.1 Mechanical properties

Longitudinal tensile strengths and moduli, flexural strengths and inter-laminar shear strengths were measured on test pieces and at rates recommended for carbon fibre reinforced composites<sup>7</sup>.

### 3.1.1 Composite tensile testing

Tensile strength and modulus were measured on the same specimen. Transverse tensile properties were measured on specimens of the recommended dimensions. However, as the specimens were very weak it was not possible to grind parallel waists. As a result, breaking points were not controlled. Longitudinal and transverse moduli were calculated from the output of a 10mm gauge length extensometer.

### 3.1.2 Resin tensile testing

Resin tensile testing was carried out on dumb-bell specimens about 2mm thick with parallel waists 10mm long by about 9mm wide. It was found necessary to bond aluminium alloy end plates<sup>7</sup> to the specimens in order to grip them satisfactorily. Tensile strength and modulus were measured using a crosshead speed of 0.05cm/min.

### 3.1.3 Flexural testing

Flexural modulus was measured either on a specimen of standard 220mm length over a 160mm span or on a flexural strength specimen over an 80mm span. The modulus was calculated from the output of a Preset Point Module linked with the crosshead. For fixed loads set on the module, a print-out was obtained giving counts proportional to the time elapsed from the start of crosshead movement. This allowed the calculation of crosshead travel between the fixed loads.

For comparison between composites of different fibres or fibre content, the flexural strengths are frequently used in the form of effective fibre strength - the actual flexural strength divided by the fibre content - and the efficiency of reinforcement - the effective fibre strength divided by the fibre tensile strength.

### 3.1.4 Interlaminar shear testing

Test pieces were usually cut from broken flexural test specimens.

### 3.1.5 Elevated temperatures

Tests were carried out using the same rigs as at room temperature inside an oven mounted on the test machine. The specimens were heat-soaked for up to 5 minutes before testing.

### 3.2 Fibre and void contents

Fibre contents by weight were determined by burning off the resin at temperatures up to 500°C, the temperature depending on fibre type. The volume fraction of voids ( $v_v$ ) was calculated from the formula

$$v_v = 1 - \rho_c \left[ \frac{w_f}{\rho_f} + \frac{1 - w_f}{\rho_r} \right]$$

where  $\rho_c$ ,  $\rho_f$  and  $\rho_r$  were densities of composite, fibre and resin respectively and  $w_f$  was the fibre fraction by weight.

Values used were  $\rho_r$  1.17,  $\rho_f$  1.91, 1.75 and 1.72 for fibre of types 1T, 2U and 2T respectively (manufacturers' data).  $\rho_c$  was calculated from the weights and measured dimensions of interlaminar shear test pieces. The measured void contents therefore included a contribution from surface irregularities. As this method of obtaining density is non-destructive, the void content of each separate specimen can be obtained. Taking estimates of possible errors in measurement of all the figures employed in the calculation gives an estimated error in void fraction of  $\pm 0.025$ , on values of the order of 0.050.

### 3.3 Heat aging

Specimens were cut to size and placed in an air-circulating oven at constant temperature for the required time. After cooling, they were tested at room temperature and/or at the aging temperature, with the exception of early experiments where specimens aged at 200°C were tested at 250°C.

Where the specimens used for a particular exposure were cut from more than one composite, aging times and specimen batches were arranged so that information could be obtained for the separate composites.

## 4 PREPARATION OF CARBON FIBRE COMPOSITES

### 4.1 Prepreg

Although the condensation reaction will go eventually to gelation on reheating the resin, it is necessary to add more catalyst for this to take place in a reasonable time. Two Friedel-Crafts catalysts were used in this work - ferric chloride, as a solution in isopropanol, and boron trifluoride, in the form of  $BF_3 \cdot 2MeOH$  and  $BF_3 \cdot OEt_2$  complexes, or as a 14% solution in propanol.

Carbon fibre preregs were prepared by cutting 10000 filament tows (long staple batch fibre) to length and laying them parallel on a sheet of release paper or film. (Details of the fibres are given in Table 1.)  $\text{BF}_3$  catalysts attacked the silicone-coated papers and could only be used with polymeric films such as fluorinated ethylene propylene copolymer.

A weight of resin equal to that of fibre was dissolved in 1,2-dichloroethane, catalyst solution added and the mixture applied evenly to the fibre. The amount of solvent used was such that the whole volume of mixture was taken up by the fibre.

Solvent was removed and the cure of the resin advanced by heating in an air-circulating oven. Typical conditions were 12 minutes at  $120^\circ\text{C}$  for preregs with 3 phr of  $\text{FeCl}_3$  catalyst and 25 minutes at  $120^\circ\text{C}$  where the same proportion of  $\text{BF}_3$  was used.

In some cases the gel content of the resin resulting from the precure was measured by Soxhlet extraction (with dichloroethane) of uncured resin from samples of prepreg, followed by burning off the insoluble resin residue from the fibre. Results illustrating the effects of time, catalyst and fibre concentration are given in Table 2 and Figs.3a-c. These show that, as expected, increasing time and catalyst content generally increase the gel content, but that the results are very variable.

Where the proportion of gel exceeded about 20%, the preregs were hard and difficult to handle.

#### 4.2 Lamination and postcure

Preregs were cut to size and laid up in an open-ended metal mould coated with PTFE release agent. The cold mould was placed in a preheated press, which was then closed so that the platens were just in contact with the mould faces without exerting pressure on the prepreg. Once the mould had been heated enough for the resin to start reacting (marked by bubbling) a pressure of not greater than  $0.3\text{MN/m}^2$  was applied gradually to maintain a slow flow of excess resin. When the resin appeared to be approaching the gel point (a noticeable increase in viscosity of the flowing resin), a pressure of  $1\text{-}2\text{MN/m}^2$  was applied to close the mould down to 2mm stops. The time between contacting the mould with the platens and the final application of pressure is referred to later as 'time to pressure'.

After the mould was closed, pressure and temperature were maintained for an hour after which the mould was cooled under pressure to below 60°C before the composite was removed.

Times to pressure with  $\text{BF}_3$  catalysed resins, where the rate of reaction of the resin was slower, were generally of the order of 20-40 minutes, whereas times for  $\text{FeCl}_3$  catalysed resins were between 5 and 15 minutes. For prepregs with high resin gel contents, very short times were used in order to achieve some consolidation of the prepreg before complete gelation took place.

With  $\text{FeCl}_3$  catalysed resins press temperatures as low as 120°C and as high as 200°C were used. At the lower temperatures reaction was slow and it was not easy to determine the gel point. The preferred temperature was 170°C, gelation occurring in less than 20 minutes.

With the slower-reacting  $\text{BF}_3$  catalysed resins, moulding at 170°C produced a composite which was obviously undercured and was difficult to release from the mould. Generally, the resin was gelled at 170°C and the temperature then raised to 200°C at the same time as the pressure was increased.

Composites were postcured at 200°C to increase their strength at elevated temperatures.

#### 4.3 Processing and composite properties

##### 4.3.1 Resin gel content

Resin gel content had no distinguishable effect on the properties of the final composite except in relation to the time before pressure was exerted during moulding.

##### 4.3.2 Composite void content

Composite void content was independent of the chemical nature of the resin, the concentration of catalyst, the moulding temperature and the gel content of the resin in the prepreg where this was low. The effect of time to pressure on the composite void content is shown in Fig.4.

For times greater than about 8 minutes, the void content increased with time. The level of void content was generally about 2% lower for  $\text{BF}_3$ -catalysed composites than for those with  $\text{FeCl}_3$ . At times to pressure less than 8 minutes, the void content increased rapidly from 3% at 8 minutes to 9% at 1 minute. The three  $\text{BF}_3$ -cured composites in this region were made from prepregs which were known to have a high gel content, although this was not measured. On the other

hand the measured gel contents of the  $\text{FeCl}_3$ -cured prepregs moulded at these short times were of the same order as those moulded at longer times. All these composites were made in the same open-ended aluminium alloy mould to the same nominal thickness.

The time of 8 minutes for minimum void content may well represent the time taken for the whole of the mould and prepreg to reach the reaction temperature of the resin. Where pressure is applied earlier, most of the resin flow needed to achieve the required volume of composite takes place before the reaction starts. During subsequent reaction and elimination of volatiles, voids remain in the composite. On the other hand, if an over-cured composite is used, consolidation of the composite does not take place since the flow of resin between prepreg layers is reduced. Similarly, a long dwell-time in the mould before the pressure is applied is equivalent to extra precure.

For the mould used, 8-10 minutes seems to give a balance between the two effects which, for a prepreg of low gel content, results in minimum void content.

#### 4.3.3 Composite flexural strength

The results for the percentage retention at  $200^\circ\text{C}$ , of room temperature flexural strength, are given in Table 3 for composites prepared under different conditions and with varying lengths of postcure at  $200^\circ\text{C}$ .

Fig.5a illustrates the effect that varying press temperature had on the strength retention before and after 48 hours postcure. The results shown are those for a series of composites made with Ter resin and 3phr  $\text{FeCl}_3$  catalyst. The initial strength retention was dependent on the cure temperature, increasing from about 10% at  $120^\circ\text{C}$  to 30% at  $200^\circ\text{C}$ .

Postcure increased the strength retention. After 48 hours only the composite moulded at  $120^\circ\text{C}$  had not reached a high retention (over 80%).

Fig.5b shows how the strength retention was affected by the concentration of catalyst used. The strength retention of Ter resin with  $\text{FeCl}_3$  catalyst was relatively independent of the catalyst concentration but was increased by postcure. With  $\text{BF}_3$ -catalysed resins there was a strong dependence of retention on catalyst concentration even after 65 hours postcure. Composites with DPO resin ( $\text{BF}_3$  catalyst only) cured much faster reaching about 60% of the room temperature flexural strength after only 24 hours postcure.

#### 4.3.4 Postcure

The effect of postcure on composite flexural strength at  $200^\circ\text{C}$  is further demonstrated in Fig.6 where the efficiency of reinforcement (i.e. the ratio of

composite strength to fibre tensile strength, for 100% fibre content) at 200°C is plotted as a function of postcure time, press temperature and catalyst concentration for Ter resin catalysed with FeCl<sub>3</sub>. The initial efficiency of reinforcement depended on the press temperature at constant catalyst concentration, but for moulding at a constant temperature of 200°C was independent of catalyst concentration. The composite prepared at the lowest cure temperature reached a plateau value of 40% efficiency within 32 hours postcure. All the other composites reached a plateau efficiency of 55-60% within 32 hours except for the fastest reacting, W47, with the highest catalyst content, which attained this value within 16 hours.

The results of a similar study on changes in flexural modulus are given in Table 4. The room temperature flexural modulus efficiency of reinforcement was independent of cure temperature, catalyst concentration and postcure time. The initial percentage of the modulus retained at 200°C, depended on catalyst content for 200°C cured composites. All composites reached maximum retention within 16 hours, the 200°C modulus being at least equal to that at room temperature.

As has been discussed under section 4.3.3, BF<sub>3</sub>-cured composites took very much longer to reach high 200°C strengths on postcure than did FeCl<sub>3</sub>-cured ones.

#### 4.4 Variability

##### 4.4.1 Fibre content

The average value of the coefficient of variation of the fibre content within each composite was 5%.

Composites were moulded to the same nominal thickness from prepregs made using the same proportions of fibre and resin. The average fibre contents achieved in the composites varied both with the resin and the catalyst, the following values being obtained:

Ter resin/FeCl <sub>3</sub> catalyst	0.50
BF <sub>3</sub> catalyst	0.56
DPO resin/FeCl <sub>3</sub> catalyst	0.53
BF <sub>3</sub> catalyst	0.61.

The coefficient of variation for all four sets of results was between 6 and 7%.

##### 4.4.2 Void content

It has already been shown (section 4.3.2) how the void content depended on the moulding conditions. The variation of void content within the composites

was of the same order as the estimated error in void content measurement (section 3.2).

#### 4.4.3 Flexural properties

Efficiencies of reinforcement at room temperature calculated from the composite flexural strengths are given in Table 3. The average variations found between composites over all lengths of postcure, were

Type 1T; Ter resin 67 ±6%, DPO resin 54 ±5%

Type 2U; Ter resin about 50%

Type 2T; Ter resin 77 ±5%, DPO resin about 70%.

The number of specimens tested from each composite was too low to give an estimate of the variation within composites. The coefficient of variation between composites prepared under the same conditions, W20-W40, was 6%. For composites prepared under different conditions, the coefficient of variation increased to 10%.

The results for efficiency of translation of fibre modulus, given in Table 4, showed a very low variation.

### 5 MECHANICAL PROPERTIES OF RESIN AND COMPOSITES

#### 5.1 Tensile properties of cast resin

A block of resin about 3mm thick was prepared by curing DPO resin catalysed with  $\text{BF}_3$  very slowly. The resin solution was placed in a glass dish coated with PTFE release agent. When a flexible gel had been formed at 65°C (up to two days), it was removed from the dish and the cure completed by raising the temperature of the oven slowly (about 10°C per day) to 150°C. The block was then postcured at 200°C. Strips about 15mm wide and 120mm long were cut from the block and the faces abraded parallel. Dumb-bell specimens were shaped and tested as described in section 3.1.2. Tensile modulus was measured over the first 0.1% extension, on the linear part of the stress-strain curve.

Attempts to cast void-free samples with ferric chloride catalyst were unsuccessful.

The properties found are given in Table 5 in comparison with those for some cast epoxy resins<sup>8</sup>.

#### 5.2 Composite tensile properties

Longitudinal and transverse tensile strengths and moduli were measured on composites of Ter resin cured with  $\text{BF}_3$  reinforced with carbon fibres of Types 1T, 2U and 2T. The results are given in Table 6.

Only 50-60% of the fibre strength was realised in the longitudinal strength tests. Efficiencies of reinforcement for epoxy resin composites are reported<sup>11</sup> varying from 62-65% for Type 1T fibre and Epikote 828/DDM/BF<sub>3</sub>400 to 84-90% with the same resin and 92% with ERLA 4617/DDM, both with Type 2T fibre.

Retention of fibre modulus with Friedel-Crafts resin was about 70%. Although the low results were certainly connected with the high composite void contents, there was no definite trend in properties with void content.

In the transverse tensile tests, the composite moduli were very close to the value found for the matrix alone.

### 5.3 Flexural strength and modulus

Flexural strengths and moduli for composites of Ter resin catalysed with BF<sub>3</sub> and fibres of Types 1T, 2U, 2T and A are given in Table 7. Other efficiency of reinforcement and strength retention results are given in Tables 3, 4 and 10.

As can be seen from Fig.7, the efficiency of reinforcement for Type 1T composites was dependent on the nature of the resin. Composites containing Type 2U fibre were similar in properties to those of Type 1T fibre while Type 2T composites showed much better translation of fibre strength than for other fibre types.

In Fig.8 strength retentions are plotted against efficiencies of reinforcement for the composites of three resins based on terphenyl. 1 Ter and 2 Ter resins are those reported on in Ref.3. All the composites were of Type 1T fibre cured with 3 phr of FeCl<sub>3</sub> at 170-180°C. In each case there was a definite trend for the retention of flexural strength at elevated temperature (200°C for Ter composites, otherwise 250°C) to increase as the efficiency of reinforcement decreased. In other words, the more highly crosslinked was the resin, the poorer was its adhesion to the carbon fibre. The average level of retention varied with the resin used, the higher the initial resin viscosity, the higher the retention at any one fibre efficiency.

### 5.4 Interlaminar shear strength

Interlaminar shear strength results are given in Table 8(a) (Type 1T fibre, FeCl<sub>3</sub> catalyst), Table 8(b) (Type 1T fibre, BF<sub>3</sub> catalyst) and Table 8(c) (other fibres). It has previously been shown<sup>5</sup> that the shear strength of Friedel-Crafts resin composites is highly dependent on the void content. With DPO resin it was also shown<sup>5</sup> that BF<sub>3</sub>-cured composites had higher shear strengths than those cured with FeCl<sub>3</sub>. Fig.9 confirms that this is true also for Ter resin. The average

shear strengths from Tables 8(b) and (c) are plotted in Fig.10. The linear regression line for the results in Table 8(a) is also shown. This confirms that composites containing Friedel-Crafts resin cured with  $\text{BF}_3$  have, in general, shear strengths some 20% higher than those cured with  $\text{FeCl}_3$  at the same void content. The composites containing Type 2U fibre had very poor shear strengths.

The interlaminar shear strength increased as the mode of failure<sup>9</sup> changed from buckling on the compression face, through shear failure to cracking on the tension face. Compression buckling occurred with composites of Type 2U fibre, while tensile cracking, in association with shear failure, was noted only with Type 2T fibre composites.

Ideally, comparisons between these composites and those of other resins, should be made at low or zero void contents. The interlaminar shear strengths calculated by linear regression using the average values of strength and void content are given in Table 9 along with results for some nominally void-free epoxy resin composites. The 'zero-void' shear strengths for Friedel-Crafts resin were: for Type 1T fibre with  $\text{BF}_3$  catalyst  $45\text{MN/m}^2$ , and for Type 2T fibre  $57\text{MN/m}^2$ , with no real difference between the two catalysts.

In comparison with the epoxy resin composites, the results for Friedel-Crafts resin were potentially somewhat lower.

## 6 PROPERTIES AT ELEVATED TEMPERATURES

### 6.1 Strength retention and postcure

The changes in retention of room temperature flexural and interlaminar shear strengths with increasing temperature are given in Tables 10 and 11. The results are plotted in Fig.11, the difference in the form of the two curves being the result of postcuring the interlaminar shear strength specimens. These had received 65 hours postcure at  $200^\circ\text{C}$  whereas the flexural strength specimens had been given none.

The effects of processing temperature and catalyst concentration on the changes in strength during postcure have been discussed in section 4.3.4.

### 6.2 Thermal aging

In Table 12, all the results of aging at  $200^\circ\text{C}$  from this and previous<sup>3</sup> studies have been collected together. The figures from the earlier work have been recalculated on the same basis as the present results to allow direct comparison. The results are shown in Fig.12.

During the initial 50-100 hours aging, the strength retention increased to a high value. Thereafter the retention varied about a 'plateau' value for 3-4000 hours before loss of strength became noticeable.

The initial strengths varied directly with the resin viscosity but the maximum strengths were in inverse order of resin viscosity being 81% for 1 Ter, the most viscous resin, and 103% for Ter. This order held even allowing for the 1 Ter and 2 Ter composites being tested at 250°C rather than 200°C.

A further estimate of the reproducibility of thermal resistance can be gained from the results from Table 12 for the separate composites, which are plotted in Fig.13. The band drawn is for one standard deviation on either side of the average value over all the composites at each time. Differences in behaviour between the composites are apparent.

The thermal resistance of the composites can also be compared by calculating the time at which a certain reduction in the 'plateau' strength retention has occurred. It was found convenient to take a strength loss of 25%, giving a ' $\frac{3}{4}$  life'. The  $\frac{3}{4}$  lives were calculated for the separate composites W20 to W28 from the data in Table 14, and are shown in Figs.14 as functions of the initial interlaminar shear strengths and the initial percentages of flexural strength retained at 200°C.

The variations in the strengths and retentions were such that the linear regression lines shown only indicate possible trends. From the shear strength plot, there is an indication that less voidy composites had better thermal resistance. There is also the possibility that composites with low initial hot strengths have better thermal resistance. This could be linked with the longer time taken to reach maximum strength, i.e. greater initial undercure.

Results for aging temperatures greater than 200°C are given in Table 15 and Fig.12. Included are the flexural strength changes for the three terphenyl resins cured with  $\text{FeCl}_3$  and aged at a range of temperatures, and also changes in interlaminar shear strength for a  $\text{BF}_3$ -cured resin. The results for the two different strength tests were comparable. The actual changes in shear strength at both ambient and elevated temperatures are plotted in Fig.15. This shows that the hot strength passed through a maximum at a time dependent on the aging temperature while the ambient strength fell continuously.

The  $\frac{3}{4}$  lives for all the runs in Fig.12 are given in Table 16 and are plotted as log (time) against inverse absolute temperature in Fig.16.

Extrapolation of the results for Ter resin flexural strength retention at 300<sup>o</sup>, 270<sup>o</sup> and 240<sup>o</sup>C suggested that, at 200<sup>o</sup>C, it would take over 5 years for a quarter of the strength to be lost. This was not borne out in practice, the  $\frac{3}{4}$  lifetime at 200<sup>o</sup>C being only slightly greater for Ter resin than had previously been found for the other terphenyl resins.

The linear regression line for all the results gives 1 year at 200<sup>o</sup>C and 10 years at 150<sup>o</sup>C for quarter-strength loss.

## 7 CONCLUSIONS

Mechanical properties have been measured for composites of three types of carbon fibre made with two Friedel-Crafts resins based on mixed terphenyl isomers (Ter resin) or on diphenyl oxide (DPO resin). Two cure catalysts, FeCl<sub>3</sub> and BF<sub>3</sub> were used.

As was to be expected with matrix resins which give off volatiles during cure, the composites tended to have high void contents, averaging 4% and ranging from 1-20%. Analysis of the variation of void content with the moulding conditions indicated that the minimum void content should be obtained by applying pressure to the prepreg in the mould 8-10 minutes after placing the cold mould in a hot press at 120-200<sup>o</sup>C.

The properties measured on the cast resin were similar to those of epoxy resins used for carbon fibre composites.

All the mechanical properties were adversely affected by the high void contents. In longitudinal tension 50-60% of the fibre strength and 70% of the fibre modulus were realised in the composite. The transverse tensile strength was low while the transverse modulus was almost the same as for the resin itself.

The interlaminar shear strength was also noticeably affected by void content. However extrapolation of the results to zero void contents showed that potentially the shear strength could approach those of typical epoxy resin composites.

Very little difference was noticed between DPO and Ter resin composites except that the former resin was faster curing.

Composites of resins cured with FeCl<sub>3</sub> had excellent retention of strength at elevated temperatures after postcure. When BF<sub>3</sub> was used as catalyst, the composites had better interlaminar shear strengths than with FeCl<sub>3</sub> but were poorer in strength retention at high temperature.

Aging tests were carried out at a number of temperatures between 200°C and 300°C. Times at which a quarter of the average maximum strength at the aging temperature had been lost were calculated, and a plot of log (time) against inverse temperature was found to be linear. From the line, '½ lives' were calculated as 1 year at 200°C and 10 years at 150°C.

Table 1

## RESINS AND FIBRES USED

Resin	Aromatic reactant	Mole ratio DME:aromatic	Viscosity at 30°C (N s/m <sup>2</sup> )
1 Ter	Terphenyl	3:1	100
2 Ter	Terphenyl	3:1	7.5
Ter*	Terphenyl	3:1	0.63
DPO*	Diphenyl oxide	7:2	1.8

\* Resins prepared commercially on 5 litre scale

Fibre type	Ultimate tensile strength (MN/m <sup>2</sup> )	Tensile modulus (GN/m <sup>2</sup> )	Fibre density (g/cc)
Type I treated (1T)	1510	432	1.91
	1790	393	
	2170	382	
Type II untreated (2U)	2610	261	1.75
Type II treated (2T)	2680	284	1.75

Table 2

## GEL CONTENTS OF PRECURED PREPREGS

Resin	Catalyst	Catalyst concentration (phr)	Precure		Number of samples	Average resin gel content (%)	Nominal fibre content (N/W)
			Time (min)	Temperature (°C)			
Ter	FeCl <sub>3</sub>	3.0	10	120	3	0.5 ± 0.3	50
			11		1	3.4	
			12		15	2.3 ± 1.3	
			13		3	3.5 ± 1.6	
			15*		6		
			15		1	2.6	
			12½		1	3.6	
15	1	0.9					
	BF <sub>3</sub>	3.0	20	120	3	1.9 ± 0.2	
			23		1	4.6	
			25		4	6.9 ± 6.9	
DPO	BF <sub>3</sub>	0.7	30	120	1	3.5	
			20		1	1.7	
			30		1	0.6	
			30		1	0.2	

\* Extrapolated from Fig. 3a

Table 3  
COMPOSITE FLEXURAL STRENGTHS, BEFORE AND AFTER POSTCURE AT 200°C

Composite number	Fibre type	Resin	Catalyst	Catalyst concentration (phr)	Press temperature (°C)	Efficiency of reinforcement* (%)					Retention of ambient strength at 200°C (%)									
						0	24 hours	48 hours	65 hours	88 hours	0**	24 hours	48 hours	65 hours	88 hours					
W47	1T	Ter	FeCl <sub>3</sub>	4.5	200			70												
W42				3.0	120															
W45					140															
W40					170															
W46				2.0	200			72												
W48					200			78												
B2			BF <sub>3</sub>	1.6	170 + 200				53									62		
H		DPO	FeCl <sub>3</sub>	2.0	200															
J-L	0.9			+200			58													
M-P	0.45			-200			50			53										
G			BF <sub>3</sub>	2.1	170 + 200															
IA		1.4		170			55													
IB				170			55													
D				170			45													
IF				170 + 200			51													
E				0.35	170			64												
B7	2U	Ter	BF <sub>3</sub>	2.7	170 + 200															
W43	2T	Ter	FeCl <sub>3</sub> BF <sub>3</sub>	3.0	120															
B3				2.3	170 + 200															
B4				2.1	170 + 200															
F		DPO	FeCl <sub>3</sub> BF <sub>3</sub>	2.3	200															
V					0.3	170 + 200														

\* Efficiency of reinforcement = flexural strength/fibre volume fraction × fibre UTS  
 \*\* Figures in brackets: initial 200°C strength as % of room temperature after 48 hours postcure

Table 4

## EFFECT OF POSTCURE ON FLEXURAL PROPERTIES

Ter resin with  $\text{FeCl}_3$  catalyst  
 Postcure at 200°C  
 Fibres: Type 1T UTS 1510 MN/m<sup>2</sup>, modulus 432 GN/m<sup>2</sup>  
 Type 2T UTS 2680 MN/m<sup>2</sup>, modulus 284 GN/m<sup>2</sup>

Composite number	Fibre type	Catalyst concentration (phr)	Cure temperature (°C)	Flexural modulus			Flexural strength								
				Efficiency at ambient (%)		Retention at 200°C (%)		Efficiency at 200°C (%)		Postcure (hours)					
				0	Postcure (hours)	0	Postcure (hours)	0	Postcure (hours)						
W48	1T	2.0	200	77	76	77	76	56	95	99	100	37	22	55	54
W43	2T	3.0	120	77	77	77	73	64	95	95	114	10	28	42	40
W45	1T	3.0	140	69	69	73	73	52	101	103	103	21	49	55	55
W46	1T	3.0	200	79	75	73	75	74	97	98	98	32	53	60	58
W47	1T	4.5	200	77	72	74	71	80	103	102	112	39	62	58	58

Table 5

## CAST RESIN PROPERTIES

Resin system	Tensile strength (MN/m <sup>2</sup> )	Modulus (GN/m <sup>2</sup> )	Elongation at break (%)	Density (g/cc)
DFO/BF <sub>3</sub>	34	2.1	1.1	1.17
Epikote 828/DDS	59	3.1	3.3	1.59
DX210/DX137	62	3.6	2.8	1.21
DX210/BF <sub>3</sub> 400	49	3.4	2.3	1.48
DLS300/BF <sub>3</sub> 400	34	2.5	1.6	1.40
ERLA4617/MPD	90	6.3	4.5	1.27
Code 69	41	3.7	1.1	1.27

Table 6

## COMPOSITE TENSILE PROPERTIES

Ter resin, catalyst BF<sub>3</sub> at 2-3 phr  
 Fibres: Type 1T UTS 2225 MN/m<sup>2</sup>, modulus 382 GN/m<sup>2</sup>  
 Type 2U UTS 2610 MN/m<sup>2</sup>, modulus 261 GN/m<sup>2</sup>  
 Type 2T UTS 2680 MN/m<sup>2</sup>, modulus 284 GN/m<sup>2</sup>

Composite number	Fibre type	Composite void content (%)	Fibre volume fraction	Longitudinal tensile properties			Transverse tensile properties		
				Strength (MN/m <sup>2</sup> )	Efficiency (%)	Modulus (GN/m <sup>2</sup> )	Efficiency (%)	Strength (MN/m <sup>2</sup> )	Modulus (GN/m <sup>2</sup> )
B14	1T	6.2	0.533 ± 0.027	591 ± 93	50	148 ± 26	72	1.6	3.4
B8	2U	18.5	0.557 ± 0.058	720 ± 65	50	82 ± 54	56	-	0.04
B15		3.4	0.528 ± 0.020	754 ± 97	55	115 ± 37	84	0.6	0.2
B9	2T	9.8	0.548 ± 0.021	629 ± 86	43	142 ± 40	91	1.9	0.2
B10		9.6	0.521 ± 0.031	874 ± 86	61	105 ± 6	69	1.9	2.0
B11		5	0.597 ± 0.015	882 ± 64	58	102 ± 7	63	-	-
B12		5.7	0.572 ± 0.028	741 ± 83	50	108 ± 13	68	1.4 ± 0.8	2.4
B13		9.5	0.537 ± 0.021	835 ± 94	58	92	60	0.7	3.0
Resin				Average	53 ± 5		70 ± 11	1.3 ± 1.1	2.1 ± 1.5
								34	2.6

Table 7

## COMPOSITE FLEXURAL PROPERTIES

Ter resin, catalyst BF<sub>3</sub> at 2-3 phr, 65 hours postcure at 200°C  
 Fibres: Type 1T UTS 2225 MN/m<sup>2</sup>, modulus 382 GN/m<sup>2</sup>  
 Type 2U UTS 2610 MN/m<sup>2</sup>, modulus 261 GN/m<sup>2</sup>  
 Type 2T UTS 2680 MN/m<sup>2</sup>, modulus 284 GN/m<sup>2</sup>

Composite number	Fibre type	Composite void content (%)	Fibre volume fraction	Flexural strength		Flexural modulus	
				Strength (MN/m <sup>2</sup> )	Efficiency (%)	Modulus (GN/m <sup>2</sup> )	Efficiency (%)
B14	1T	6.2	0.533 ± 0.027	737 ± 32	63	135 ± 15	66
B7	2U	6.3	0.545	637 ± 83	44	107 ± 3	75
B8		18.5	0.557 ± 0.020	732 ± 65	51	113 ± 2	78
B15		3.4	0.528 ± 0.020	767 ± 65	56	101 ± 4	73
B3	2T	7.1	0.611 ± 0.017	1161 ± 297	72	113 ± 22	65
B4		-	0.543 ± 0.036	1034 ± 148	70	105 ± 2	68
B9		9.8	0.548 ± 0.021	960 ± 172	65	111 ± 5	71
B13		9.5	0.537 ± 0.021	1175 ± 81	81	115 ± 8	75

Table 8

INTERLAMINAR SHEAR STRENGTH(a) Ferric chloride catalysed resins  
Fibre: Type 1T

Composite number	Resin	Catalyst concentration (phr)	Void content (%)	Fibre volume fraction	Interlaminar shear strength (MN/m <sup>2</sup> )
W47	Ter	4.5	4.7 ±1.4	0.508 ±0.010	23.3 ±0.7
W42		3.0	3.7 ±0.9	0.482 ±0.018	20.4 ±0.5
W29			3.7 ±1.4	0.475 ±0.013	24.4 ±1.5
W45			5.0 ±0.6	0.474 ±0.014	21.6 ±1.4
W20			8.7 ±1.1	0.498 ±0.011	24.0 ±0.6
W26			5.6 ±1.3	0.461 ±0.014	26.1 ±2.0
W27			8.5 ±1.3	0.475 ±0.022	23.1 ±0.8
W28			4.8 ±1.2	0.537 ±0.016	24.1 ±0.4
W30			5.1 ±1.0	0.483 ±0.019	25.0 ±1.2
W31			5.7 ±0.7	0.498 ±0.014	26.4 ±1.1
W32			3.4 ±0.9	0.479 ±0.019	29.0 ±2.6
W33			5.8 ±1.0	0.494 ±0.005	26.4 ±1.4
W34			3.3 ±0.7	0.490 ±0.019	25.5 ±0.4
W37			3.8 ±0.8	0.461 ±0.014	26.4 ±0.8
W38			4.3 ±1.3	0.466 ±0.027	26.2 ±0.8
W46		4.1 ±1.5	0.513 ±0.040	26.0 ±1.2	
T6		3.8 ±0.7	0.550 ±0.025	36.3 ±4.7	
W48		2.0	3.7 ±1.3	0.498 ±0.019	28.9 ±1.0
T5			1.9	0.510 ±0.015	28.6 ±2.7
T2		0.8	5.4 ±2.2	0.549 ±0.030	42.8 ±2.6
T3	1.4 ±0.8		0.526 ±0.031	47.0 ±3.9	
T4	2.5 ±2.2		0.529 ±0.025	43.3 ±5.6	
H	DPO	2.0	4.5 ±1.6	0.476 ±0.023	35.7 ±2.1
J		0.9	4.0 ±1.6	0.591 ±0.029	28.8 ±3.1
K			4.4 ±1.8	0.553 ±0.031	28.4 ±3.0
L			3.7 ±1.1	0.561 ±0.025	28.3 ±2.7
M		0.45	1.8 ±1.1	0.508 ±0.023	29.4 ±2.8
N			2.3 ±1.9	0.550 ±0.033	33.0 ±3.1
P			3.6 ±3.9	0.523 ±0.033	34.8 ±6.3

Table 8 (continued)

(b) Boron trifluoride catalysed resins  
Fibre: Type 1T

Composite number	Resin	Catalyst concentration (phr)	Void content (%)	Fibre volume fraction	Interlaminar shear strength (MN/m <sup>2</sup> )
B14	Ter	3.0	6.2 ± 1.9	0.533 ± 0.027	34.6 ± 1.7
B2		1.6	8.2 ± 2.1	0.602 ± 0.041	28.9 ± 2.0
G	DPO	2.1	4.3 ± 1.7	0.586 ± 0.037	39.7 ± 4.6
1A		1.4	7.6 ± 2.6	0.504 ± 0.019	35.2 ± 7.6
1B		0.7	8.0 ± 4.0	0.495 ± 0.029	36.3 ± 2.4
D			2.4 ± 1.6	0.601 ± 0.021	41.9 ± 3.6
1F			1.7 ± 0.8	0.560 ± 0.021	39.5 ± 5.1
1G			2.7 ± 1.3	0.542 ± 0.035	44.0 ± 5.3
1H			0.4 ± 1.4	0.525 ± 0.020	44.9 ± 5.8
1J			4.0 ± 2.4	0.482 ± 0.020	43.0 ± 5.7
1K			7.9 ± 1.8	0.494 ± 0.024	43.0 ± 5.7
C			0.35	7.3 ± 1.1	0.572 ± 0.029
E	5.2 ± 2.4	0.662 ± 0.017		42.3 ± 5.1	

Table 8 (concluded)

(c) Fibre: Types 2U and 2T

Composite number	Fibre type	Resin	Catalyst	Catalyst concentration (phr)	Void content (%)	Fibre volume fraction	Interlaminar shear strength (MN/m <sup>2</sup> )
B15	2U	Ter	BF <sub>3</sub>	3.0	3.4 ± 1.3	0.528 ± 0.020	22.7 ± 2.8
B7				2.7	6.3 ± 3.0	0.545 ± 0.032	21.8 ± 0.2
B8					18.5 ± 1.4	0.557 ± 0.058	16.2 ± 1.0
W43	2T	Ter	FeCl <sub>3</sub>	3.0	4.4 ± 0.9	0.530 ± 0.013	41.2 ± 9.8
T1				0.8	5.9 ± 3.0	0.555 ± 0.046	58.1 ± 9.0
B9				3.4	9.8 ± 2.9	0.548 ± 0.026	49.7 ± 3.2
B10				3.0	9.6 ± 3.0	0.521 ± 0.031	45.0 ± 4.0
B11					5.0 ± 2.0	0.597 ± 0.015	46.1 ± 1.7
B12					5.7 ± 2.4	0.572 ± 0.028	44.5 ± 2.3
B13					9.5 ± 5.3	0.537 ± 0.021	42.3 ± 4.8
B3				2.3	7.1 ± 1.5	0.611 ± 0.017	49.2 ± 2.9
W				0.3	4.7 ± 4.5	0.617 ± 0.048	58.8 ± 11.3
A				2.3	4.1 ± 2.5	0.529 ± 0.046	56.1 ± 5.6
B					3.5 ± 1.2	0.534 ± 0.023	54.5 ± 6.4
F					4.3 ± 1.7	0.451 ± 0.029	49.6 ± 4.8
V				0.3	6.1 ± 2.8	0.616 ± 0.023	56.6 ± 2.0

Table 9

COMPARISON OF INTERLAMINAR SHEAR STRENGTHS  
WITH EPOXY RESIN COMPOSITES

Fibre type	Resin system	Reference number	Interlaminar shear strength (MN/m <sup>2</sup> )
1T	Friedel-Crafts/FeCl <sub>3</sub>		29 ±6
	Friedel-Crafts/BF <sub>3</sub>		39 ±5 (45)*
	ERLA 4617/MPD	10	42
	ERLA 4617/DDM	11	47
	Epikote 828/MNA/BDMA	11	58
	"	12	50
	"	13	59
2T	Friedel-Crafts		50 ±6 (57)*
	ERLA 4617/MPD	10	84
	ERLA 4617/DDM	11	60
	Epikote 828/MNA/BDMA	11	85
	"	12	62
	"	13	82
2U	Friedel-Crafts		20 (24)*
	ERLA 4617/MPD	10	29
	ERLA 4617/MPD	11	26
	Epikote 828/MNA/BDMA	11	34
	"	13	49

\* Calculated on average values. Figures in brackets are values for zero void content calculated on average strengths and void contents.

Table 10

VARIATION WITH TEMPERATURE OF FLEXURAL STRENGTH

Type II fibre, UTS 1510MN/m<sup>2</sup>. Ter resin with 3 phr FeCl<sub>3</sub> catalyst  
Cure temperature 170°C, no postcure

Composite number	Effective fibre strength at elevated temperature (MPa)							Retention of strength (%)								
	120°	140°	160°	180°	200°	240°	270°	300°	120°	140°	160°	180°	200°	240°	270°	300°
W30	1038 ±73				574 ±86			255 ±17					55			24
W31	974 ±34				421 ±43			297 ±28					43			30
W32	1038 ±118				350 ±95		303 ±43						33		27	
W33	923 ±50				347		253 ±15						38		27	
W34	976 ±90				360 ±35		237 ±11						37		24	
W37	1048 ±19				217 ±32		207 ±64						21		20	
W38	1121 ±88				153 ±31		218 ±27						14		19	
W40	1088 ±9				161 ±3		205 ±11						15		19	
W41	828 ±77				249 ±37								33			
									70	48	38	33	32 ±13	19 ±1	26 ±1	27
									Average							
									70	48	38	33	32 ±13	19 ±1	26 ±1	27

Table 11

VARIATION WITH TEMPERATURE OF INTERLAMINAR SHEAR STRENGTH

Ter resin with 3 phr BF<sub>3</sub> catalyst  
Fibre: Type 2T  
65 hours postcure at 200°C

Composite number	Test temperature (°C)	Interlaminar shear strength (MN/m <sup>2</sup> )	Retention (%)
B9	Room temperature	49.7 ±3.2	
B11		46.1 ±1.7	
B9	100	44.1 ±2.4	89
B9	150	42.3 ±2.5	85
B9	200	37.2 ±1.6	75
B11		38.9 ±1.6	84
B9	250	28.9 ±0.8	58
B11		28.3 ±1.1	61
B9	300	22.9 ±1.2	46
B11		19.9 ±1.0	43



**Table 13**  
**AGING OF COMPOSITES AT TEMPERATURES ABOVE 200°C**

Terphenyl resins with 3 phr catalyst  
 Flexural strength: Type 1T fibre, FeCl<sub>3</sub> catalyst  
 Shear strength: Type 2T fibre, BF<sub>3</sub> catalyst

Resin	Fibre UTS (MN/m <sup>2</sup> )	Aging temperature (°C)	hours	Effective fibre strength at aging temperature (Z)									
				Initial ambient effective fibre strength		on flexural strength		Length of aging (hours)					
Ter	1510	240	hours	0	8	16	24	48	72	120	265	476	1008
			hours	21, 14	64	44	51	78	60	78	90	73	82
			hours	2000	3000	62							
1 Ter	1790	250	hours	0	49	96	237	502	999				
			hours	69	84	71	75	51	45				
2 Ter	1790	250	hours	0	49	96	237	502	999				
			hours	43	78	67	77	71	36				
Ter	1510	270	hours	0	8	16	24	48	96	144	240	360	480
			hours	27, 27, 24	71	69	75	83	87	77	73	70	24
Ter	1510	300	hours	0	7	18	22	40	64	71	96	120	120
			hours	24, 30	50	71	67	68	58	73	37	46	
				Interlaminar shear strength at aging temperature (Z)									
Ter	1510	250	hours	0	23.5	48	73.5	120	240	549	862	1341	
			hours	61	67	71	67	76	70	61	51	36	
Ter	1510	300	hours	0	23.5	48	72	120	168	216			
			hours	44	49	55	51	49	41	31			

**Table 14**  
**COMPARISON OF THERMAL AGING RESULTS**

Resin	Aging temperature (°C)	'Plateau' strength retention (%)	Life (hours)	Flexural strength
Ter	200	96	7200	Flexural strength
	240	83	2900	
	270	80	390	
	300	68	90	
	250	70	740	Shear strength
	300	52	170	
1 Ter	200	75	5600	Flexural strength
	250	75	800	
2 Ter	200	71	6100	Flexural strength
	250	73	800	

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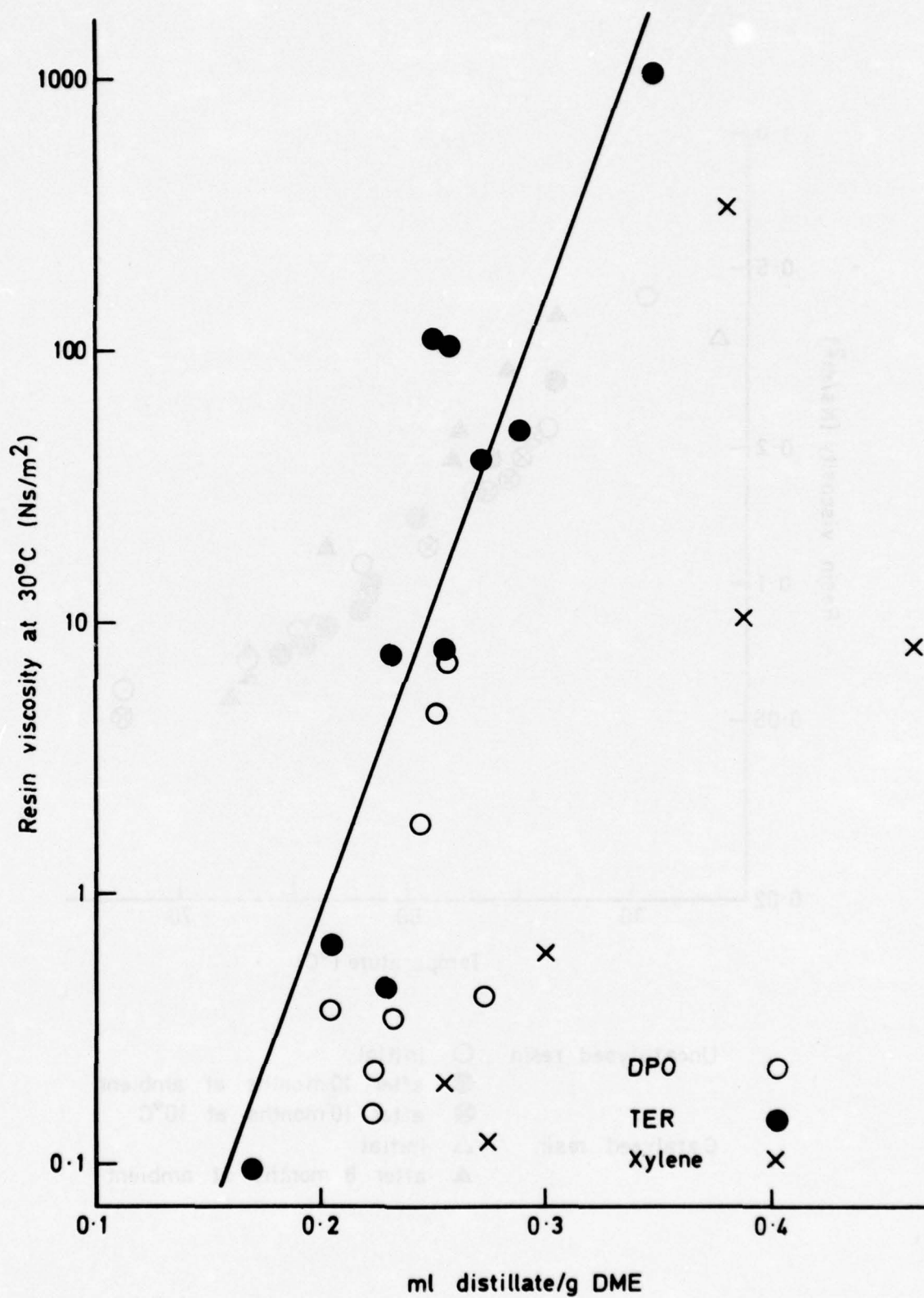
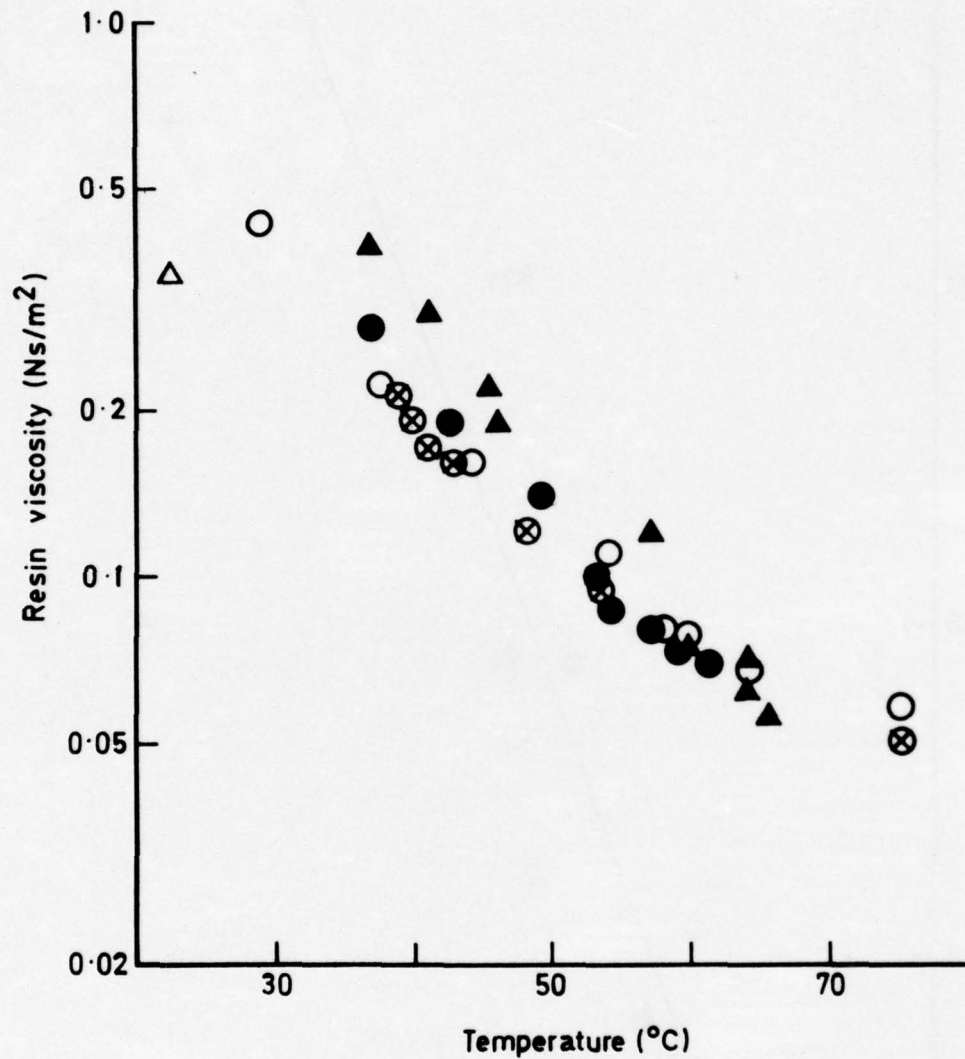


Fig. 2



Uncatalysed resin    ○ initial  
                             ● after 10 months at ambient  
                             ⊗ after 10 months at 10°C  
Catalysed resin      △ initial  
                             ▲ after 8 months at ambient

Fig. 2 Shelf life of Friedel-Crafts resins

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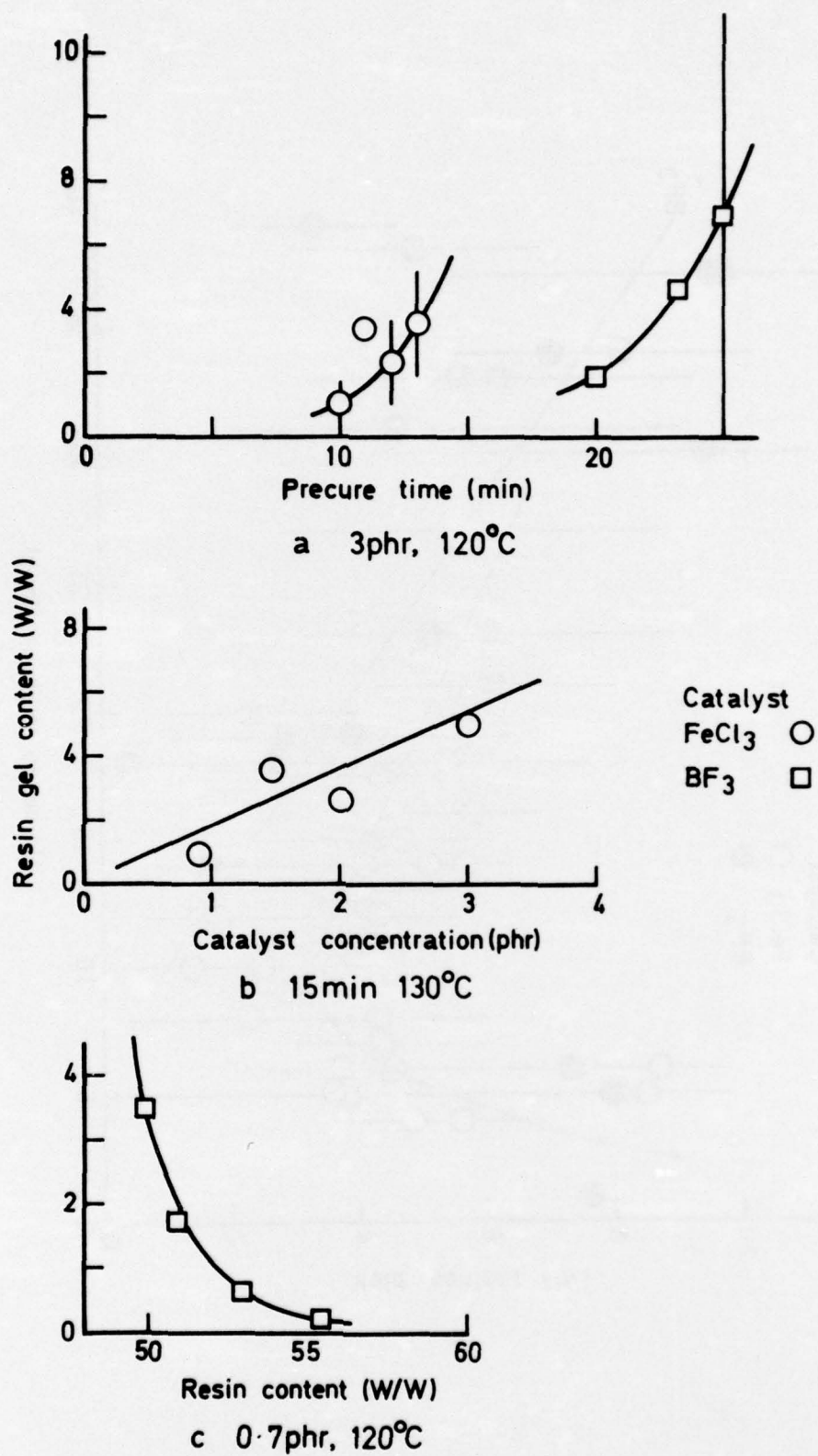


Fig.3a-c Precure of Friedel-Crafts resin prepregs

Fig. 4

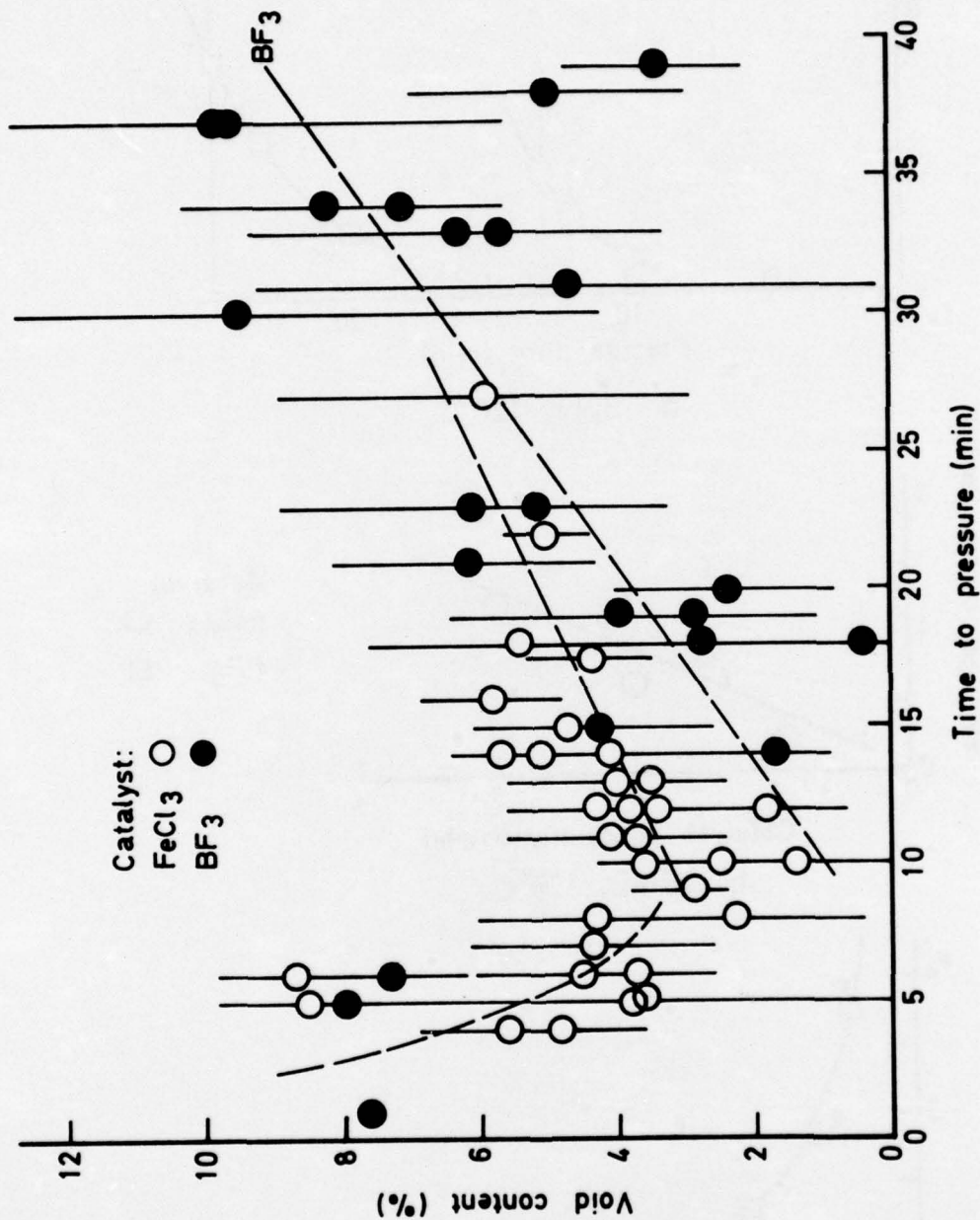
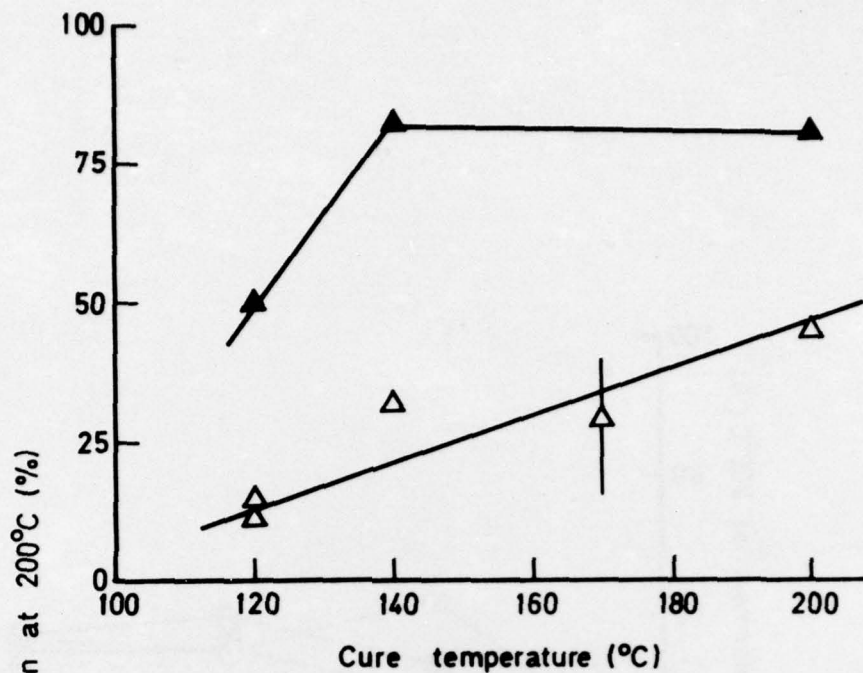
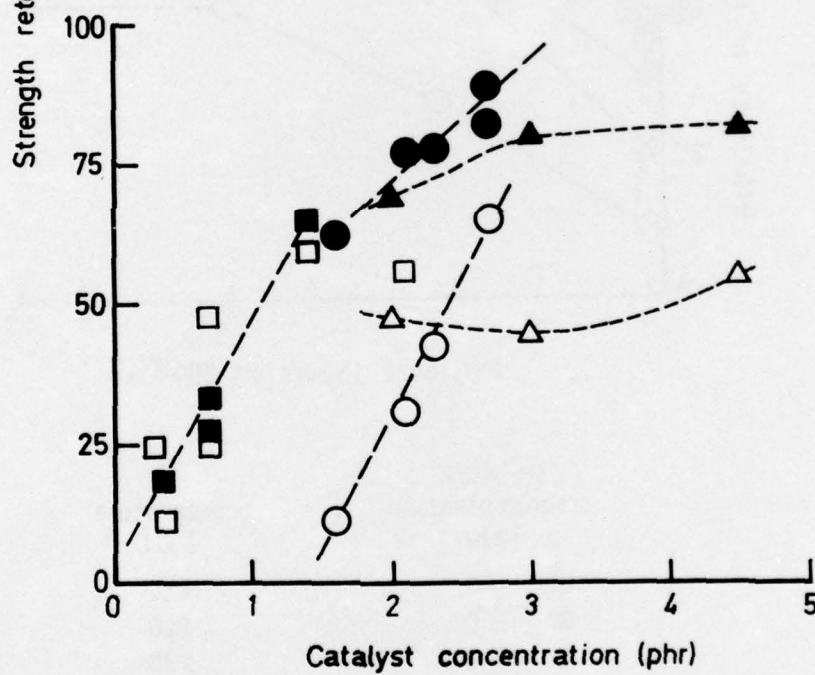


Fig. 4 Processing of Friedel-Crafts resin composites



a 3phr



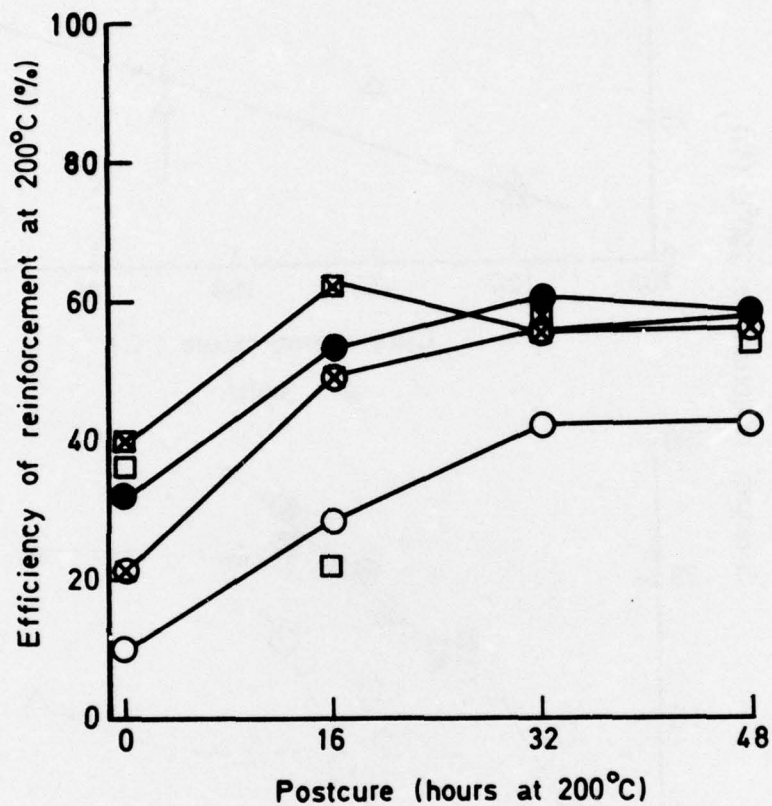
b 200°C cure

	Ter Resin		DPO Resin
BF <sub>3</sub> Catalyst	No postcure	○	24 hours
	65 hours	●	48 hours
FeCl <sub>3</sub> Catalyst	No postcure	△	
	48 hours	▲	

Fig. 5a & b Factors affecting composite strength retention

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Fig. 6



Catalyst concentration (phr)	Cure temperature (°C)
○ 3.0	120
⊗ 3.0	140
● 3.0	200
□ 2.0	200
⊠ 4.5	200

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Fig.6 Postcure of Friedel-Crafts resin composites

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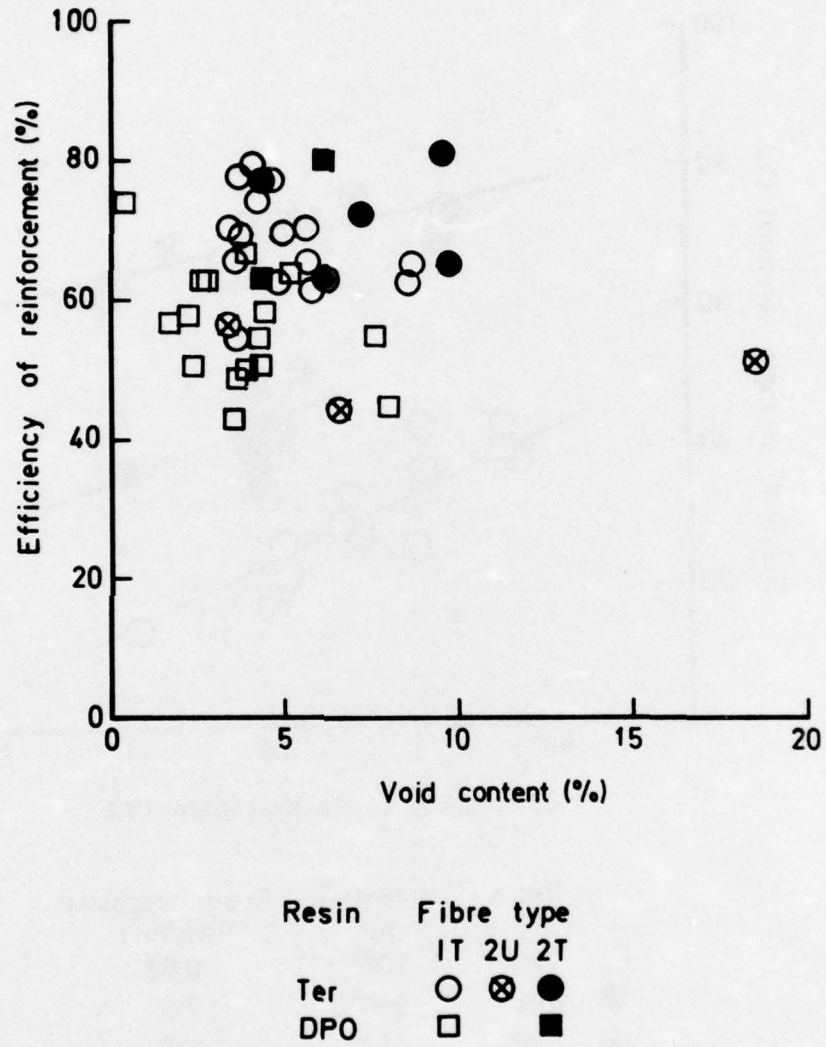
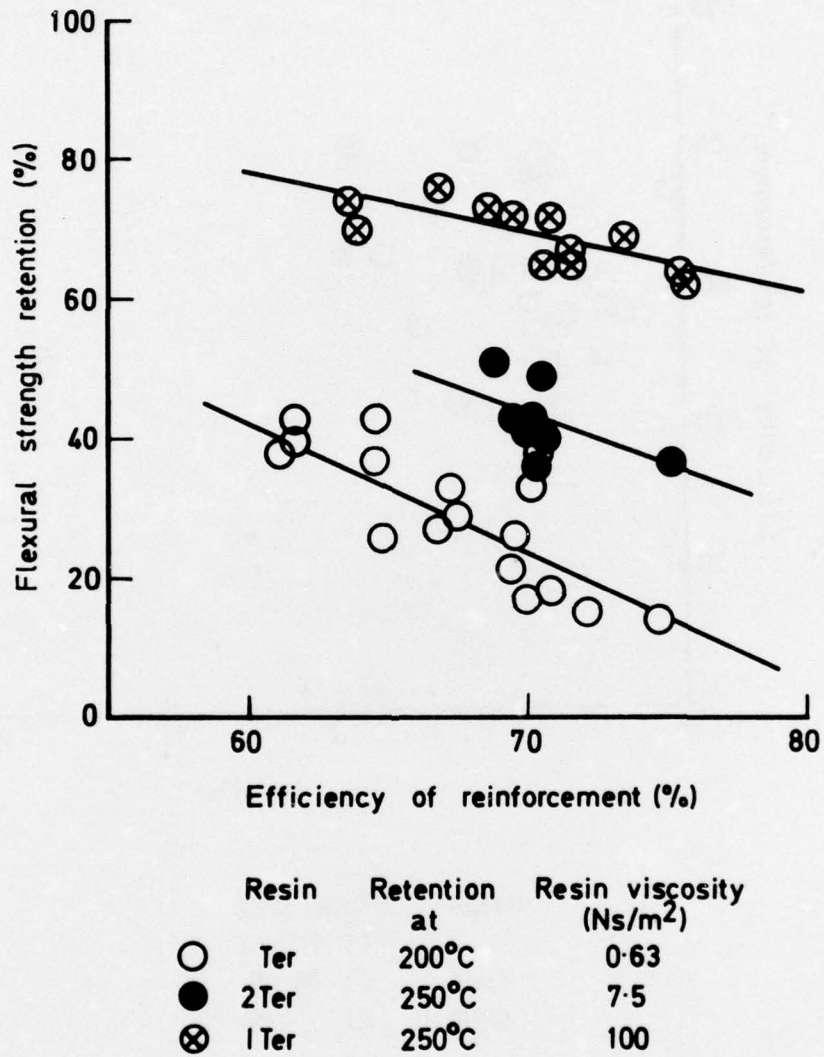


Fig.7 Factors affecting efficiency of reinforcement

Fig. 8



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Fig.8 Factors affecting strength retention

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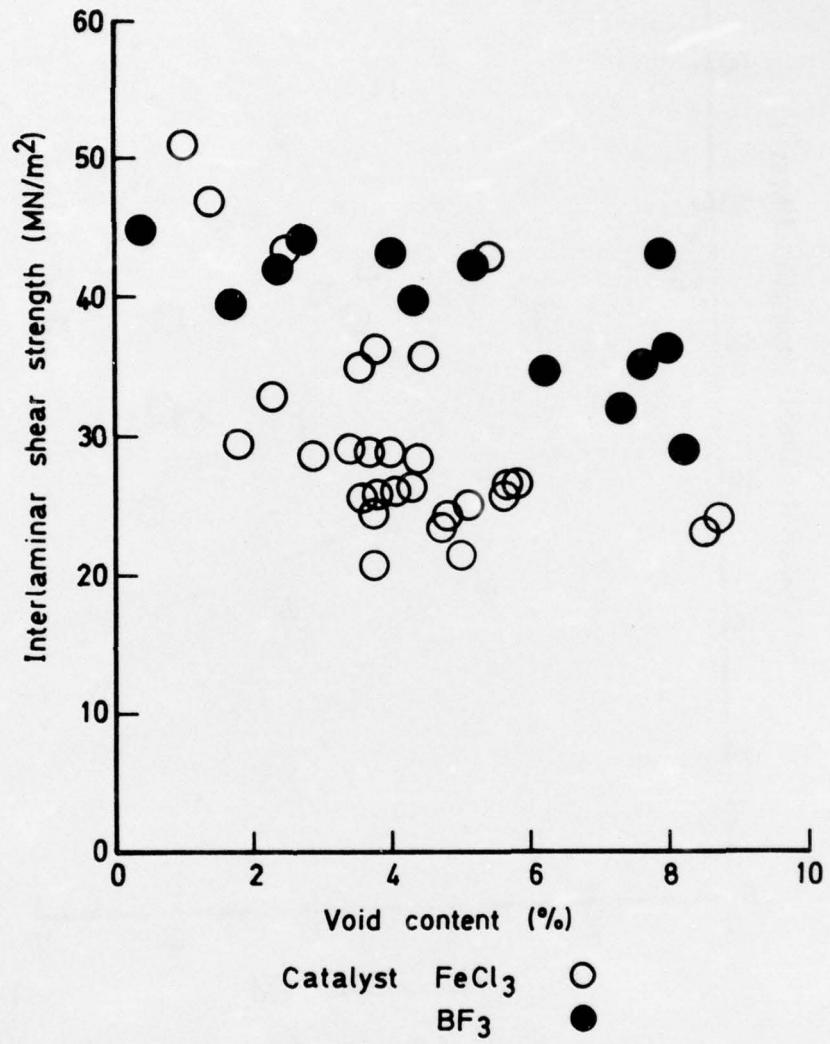
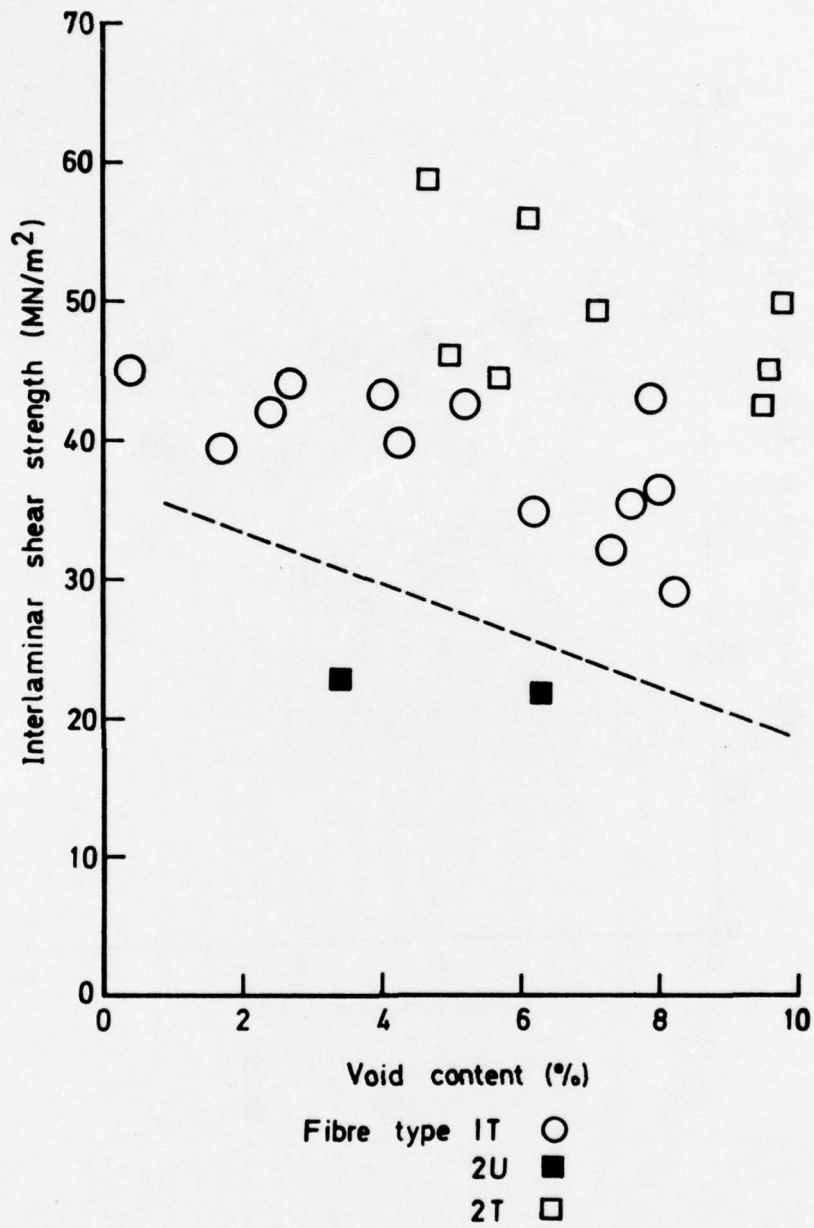


Fig.9 Factors affecting interlaminar shear strength:  
Type IT composites

Fig 10



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Fig.10 Factors affecting interlaminar shear strength: all fibres

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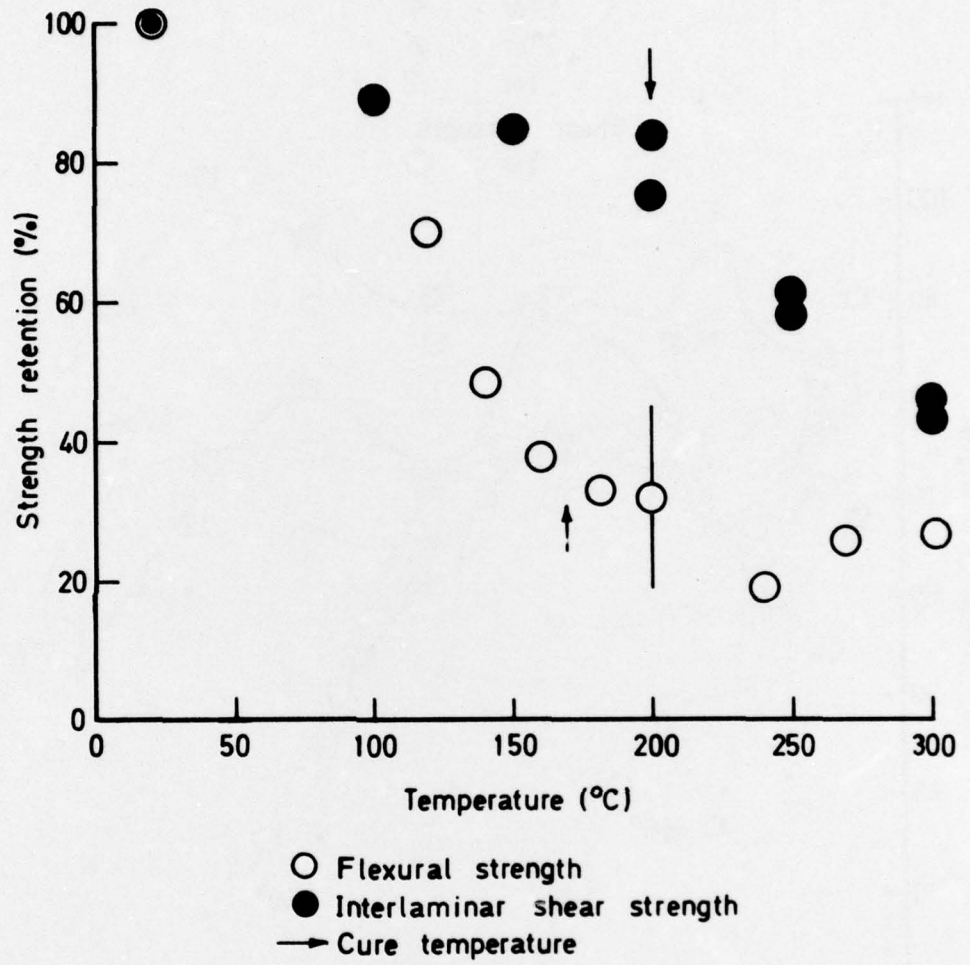
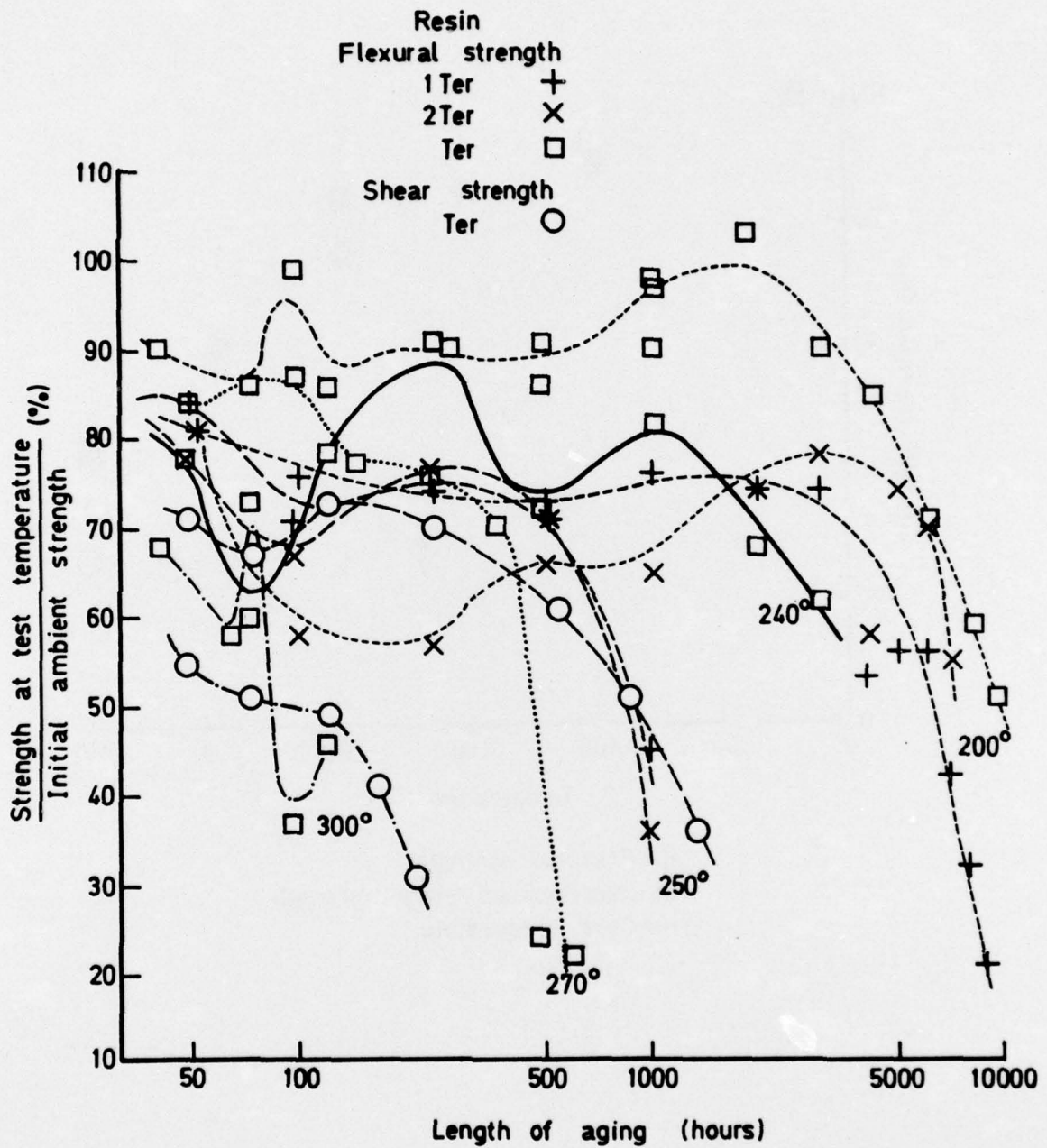


Fig.11 Effect of temperature on composite properties

Fig.12



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Fig.12 Heat aging of Friedel-Crafts resin composites

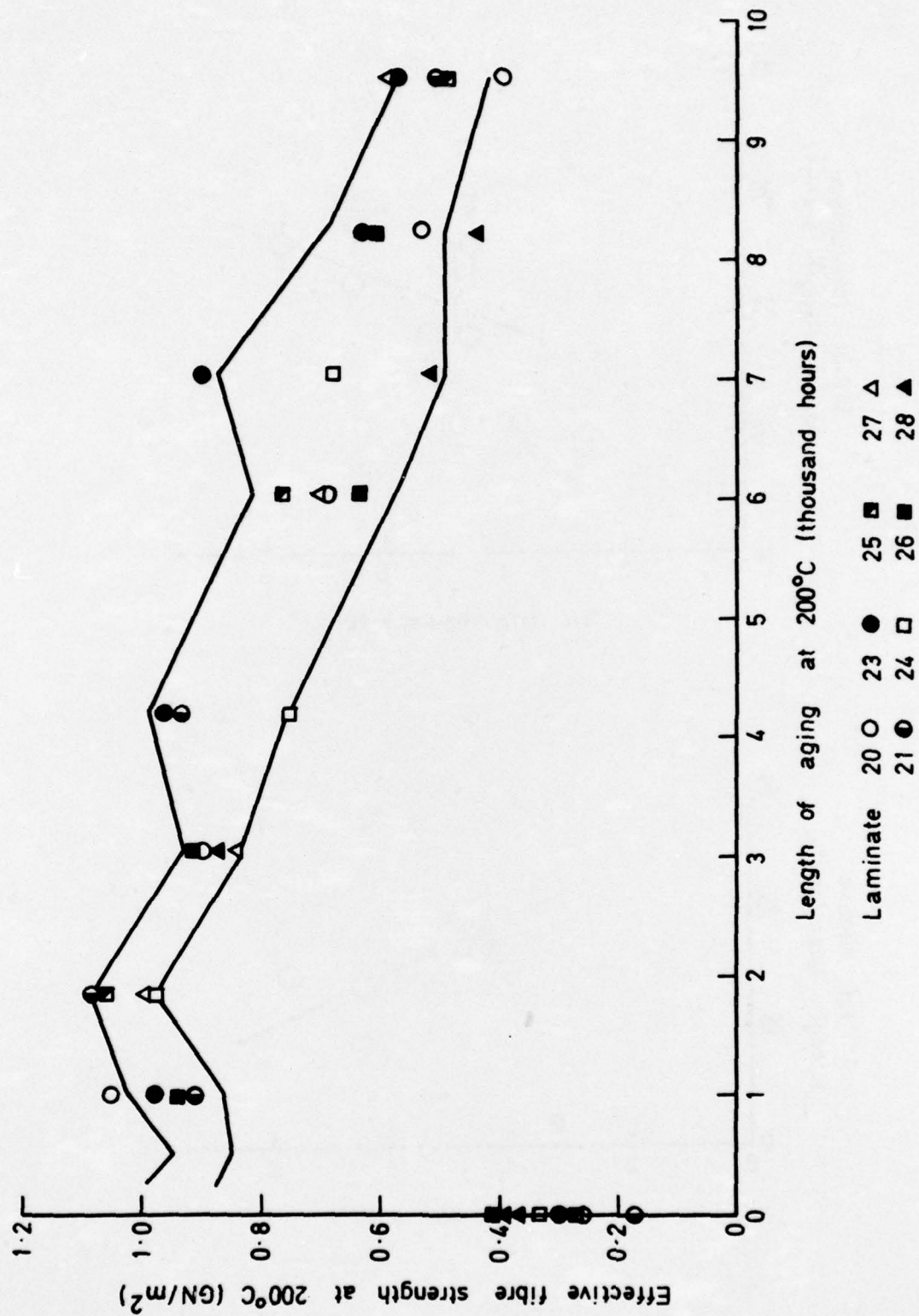
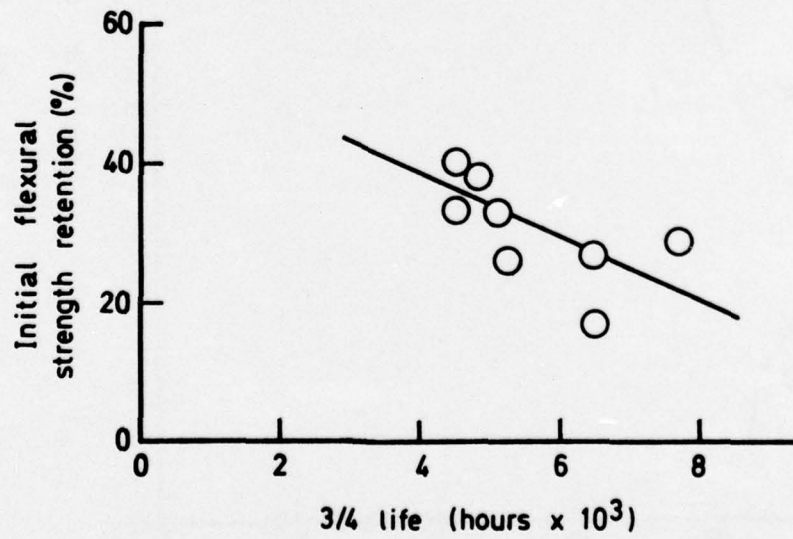
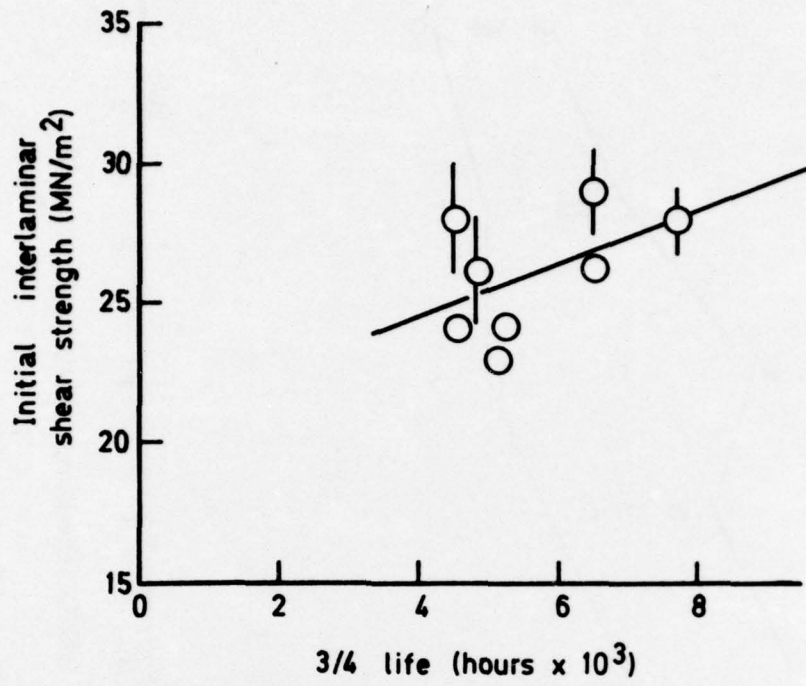


Fig. 13 Aging of composites at 200°C: variation between composites

Fig. 14



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Fig. 14 Factors affecting thermal resistance of composites

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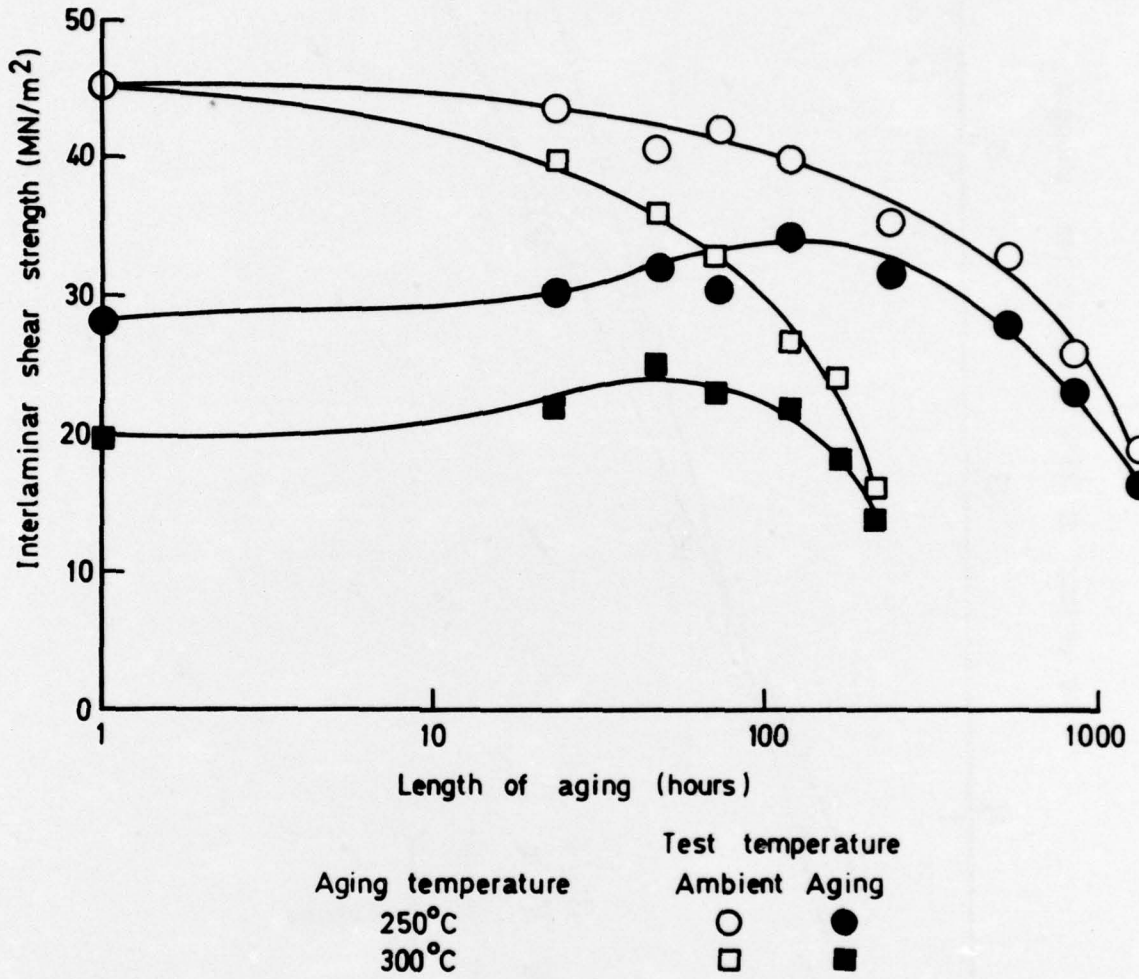
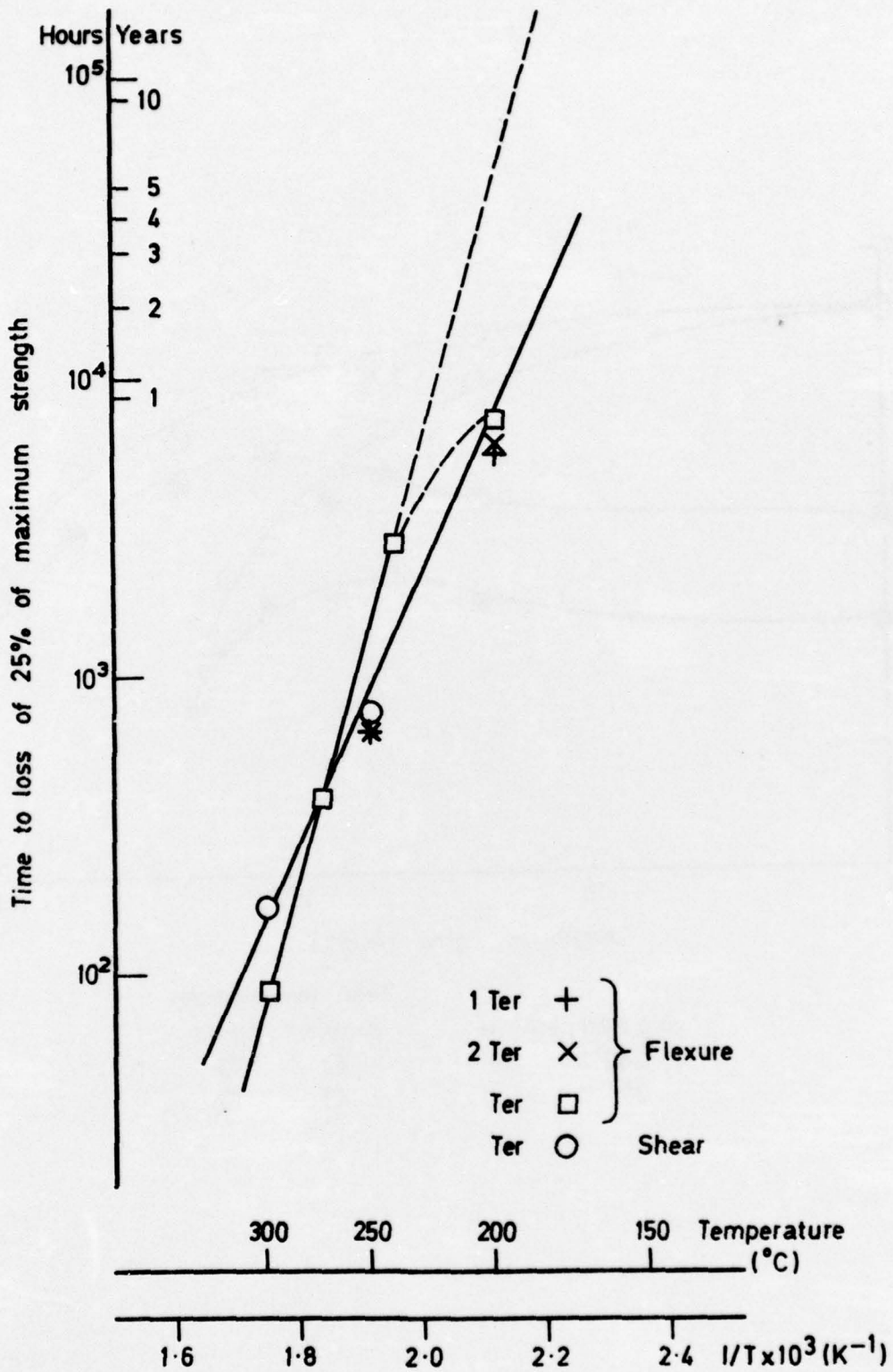


Fig.15 Aging of shear strength specimens

Fig. 16



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Fig.16 Thermal resistance of Friedel-Crafts resin composites