

# Hygrothermal characteristics of carbon fibre reinforced plastics

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The following account describes investigations of the moisture absorption characteristics of specimens prepared from a unidirectionally reinforced sheet of carbon fibre reinforced plastic (CFRP), Fibredux 914C, which were subjected to a variety of environmental conditions. Experiments were undertaken over the approximate temperature range  $-70$  to  $+180^{\circ}\text{C}$ , employing specimens containing moisture at levels between approximately 0 and 2 wt%. A scheme has been developed to assess the fractional length changes which might be anticipated in specimens containing different moisture contents resulting from a change of temperature, assuming no loss of moisture during the temperature change. Application of the analytical scheme to the experimental data has revealed the detailed dependence of specimen dimensions upon moisture content and temperature. The investigation has served to underline the importance of knowing the humidity of the environment in which a CFRP component has to operate, as well as its temperature, in order that it might be possible to predict its subsequent dimensional behaviour.

## 1. Introduction

The temperature dependence of the dimensions of composite structures consisting of a resin matrix reinforced with carbon fibres has been studied in some detail in earlier investigations conducted in this laboratory [1-6]. The dimensions of composite components are also liable to alter because of water absorbed from the atmosphere by the resin matrix. The dimensional strains resulting from the swelling of the constituents of a fibre reinforced composite have been analysed in some detail by Halpin and Pagano [7], whose results have been extended to cover thermally induced dilatation. However experimental investigations of the absorption characteristics of fibre reinforced plastics have concentrated largely upon changes of the mass of the specimen concerned, rather than its dimensions [e.g. 8-11].

Carbon fibre reinforced plastics (CFRP) are finding an increasingly wide range of applications in aerospace activities and it is in this connection that the present project arose. In real life situations a modern high performance aircraft may be required to operate in an environment having a humidity at almost any level between 0 and 100% and structural components may be subjected to temperatures ranging between approximately  $-50$  and  $+150^{\circ}\text{C}$ . The response of their dimensions to such changes is a matter of concern to designers and the anticipation of behaviour is complicated by the fact that humidity and temperature usually change together, in a manner which may vary from day to day. The present project was undertaken in an attempt to determine the influences of moisture content and temperature upon the

dimensions of a series of CFRP specimens which might be regarded as constituting the structural elements from which complex assemblies are built up.

## 2. The specimens

The specimens consisted of a series of strips measuring  $50\text{ mm} \times 20\text{ mm}$ , cut from a plate of CFRP having a thickness of 2 mm. The plate itself consisted of a matrix of Ciba-Geigy resin Fibredux 914 reinforced unidirectionally with Courtaulds XAS fibre, present at nominally 60% by volume. The plate was prepared and supplied by British Aerospace plc, Warton Division and the specimens were cut such that the fibres ran parallel to their 20 mm long sides, employing a diamond wheel saw. The preparation of the edges of the specimens was completed by hand grinding.

## 3. Experimental details

### 3.1. Outline of the investigation

The procedure adopted to establish the temperature dependence of the length of each of a series of ostensibly identical unidirectionally reinforced specimens of CFRP which contained different but fixed contents of water, uniformly dispersed throughout the matrix, in a direction perpendicular to the fibres (henceforth described as the lateral direction), was as follows:

- (i) The moisture contents of the specimens were raised to the chosen levels. The lateral dimensions of the specimens were measured at intervals during this conditioning process with the aid of a micrometer screw gauge reading to  $\pm 5 \times 10^{-6}\text{ m}$ .
- (ii) The temperature of a conditioned specimen was

raised at a rate of approximately  $6^{\circ}\text{C min}^{-1}$  to some elevated value  $T$ , at which it was held constant. Its lateral dimension  $L$  was monitored continuously throughout this process.

(iii) The weight of an exactly similar specimen was monitored throughout a temperature excursion which was identical to that employed in Stage (ii).

(iv) Fractional reductions of length were plotted against fractional reductions of weight, at corresponding times beyond that at which temperature  $T$  was reached. Extrapolating the graph so produced to the moisture content produced in Stage (i) gave an estimate of the increase in the lateral dimension that would have resulted from raising the temperature to  $T$  if moisture had not been driven out as the temperature was raised in the experiment described in Stage (ii).

(v) Repeating Stages (ii) to (iv) for a second pair of specimens, which contained the same moisture content as those employed previously, but this time raising the temperature to a new level  $T'$ , produced a second pair of data points, similar to those described in (iv) above. This time however the incremental change of length so determined was an estimate of the fractional increase which would have occurred if no moisture had been lost as the temperature was raised to  $T'$ .

(vi) Repeating Stage (v) for a series of different temperatures produced a series of pairs of results from which a graph of fractional length increase could be plotted against temperature rise. This graph referred to a set of specimens conditioned to one of the moisture levels achieved in Stage (i).

(vii) Repeating the above procedure with sets of specimens conditioned to different levels of moisture content produced a series of graphs resembling that described in Stage (vi).

(viii) The fractional increases in lateral dimensions at room temperature which resulted from the increases in moisture content, as measured during the course of the experiments described in Stage (i), defined the separations of the graphs described in Stage (vii). Combining these two sets of information led to the generation of a family of curves, from which the separate influences of temperature and moisture content upon the lateral dimensions of the subject of the investigation could be deduced.

### 3.2. Conditioning arrangements

With the exception of specimens subjected to complete immersion in water, the moisture content of a batch of specimens was raised to the required level by exposing the specimens to the atmosphere above a saturated solution of an appropriate salt. The weights and dimensions of specimens were recorded before exposure and the progress of the absorption process was monitored by weighing at intervals measured into the conditioning period. By way of a safeguard against the influence of minor structural or compositional variations between specimens, these were studied in batches of five, the average behaviour of the specimens within each batch being taken as representative of the batch. By way of a further precaution the difference of averages of three determinations of the lateral dimension of each specimen, made before and after

conditioning, was employed in the final presentation of their behaviour.

In order to speed up the conditioning process, this was undertaken in a cabinet maintained at  $60^{\circ}\text{C}$ . This temperature was considered to be sufficiently high to reduce the conditioning times to acceptable levels without being high enough to induce chemical changes in the resin matrix. Before being weighed at intervals, employing a microanalytical balance which gave results accurate to  $\pm 2 \times 10^{-4}\text{g}$ , specimens were wiped with absorbent paper and allowed to dry in the air for about 5 min.

### 3.3. Incremental length change determinations

It was anticipated at the outset that raising the temperature of a moist specimen would produce two conflicting effects upon its length. A length increase was expected to result from the thermal expansion of the composite [1, 12], but a decrease in length was expected to result from the loss of water from the resin [13]. In order to monitor the lateral dimensions of specimens throughout the temperature sequence of Stage (ii), use was made of a quartz pushrod system resembling that described earlier [14], upgraded so as to incorporate a transducer, obtained from Automatic Systems Laboratories, interfaced to a digital display unit reading to  $\pm 10^{-3}\text{mm}$ . Temperatures above ambient were achieved using a temperature controlled oven. Temperatures between ambient and  $-70^{\circ}\text{C}$  were achieved with the aid of a Townson and Mercer constant temperature bath employing a suspension of crushed solid carbon dioxide in silicone fluid MS 200/1 supplied by BDH Ltd. The incremental length change sequence was repeated to cover a total of three separate specimens from each of the batches listed in Table I. Means were taken from these results.

### 3.4. Incremental weight change determinations

In order to monitor the changes of weight encountered in Stage (iii), use was made of a thermobalance equipped with a potentiometric recorder and giving results which were accurate to  $\pm 5 \times 10^{-4}\text{g}$ . Specimens were located at the centre of an oven, the temperature of which was raised at the same rate and to the same temperature as in Stage (ii). As in Section 3.3, the experiment was repeated twice, using a second and third specimen, and averaged results for the three

TABLE I The specimens

Specimen batch number	Relative humidity of conditioning environment (%)	Saturation moisture content of specimens (%)
1	Specimens as produced (i.e. unconditioned)	
2	30	0.47
3	43	0.70
4	57	0.95
5	80	1.57
6	(Specimens immersed in water)	2.05

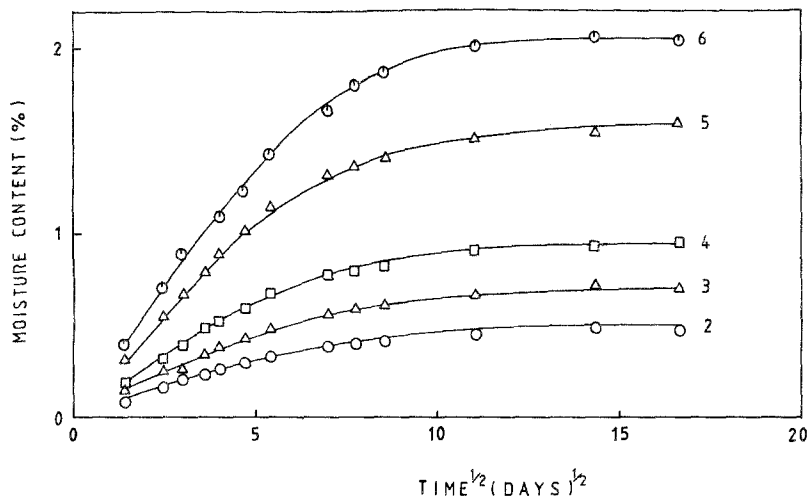


Figure 1 The absorption of water by the specimens, during the conditioning process, in which the numbers identify the specimen batches summarized in Table I.

specimens were taken to be representative of the batch from which they came.

#### 4. Presentation and discussion of results

The progress of the absorption process is illustrated in Fig. 1, from which the saturation levels summarized in Table I were obtained. Figs. 2 and 3 illustrate typical sets of results corresponding to the procedures adopted in Stages (ii) and (iii), respectively. Fig. 4 illustrates the association of reductions of length and weight beyond the steady temperature  $T$ , described in Stage (iv) of Section 3.1, in which results for the same batch of specimens have been employed as in the earlier figures. The caption to Fig. 4 explains the manner of assessment of the length that would have been attained by the chosen specimen in the absence of moisture loss. This deduction must be qualified by noting that the observations recorded in Figs. 2 and 3, upon which the location of point F in Fig. 4 was deduced, were taken as the specimen was drying out. The moisture distribution within the specimen could not be expected to be uniform during this period and the location of F by this procedure must therefore be influenced to some extent by the geometry of the specimen. However, because the specimens were thin it was not considered that the dynamic nature of the experiment would seriously influence the results.

At this juncture it was convenient to commence the association of the increase of length, deduced above, with the temperature to which the specimen had been

raised. Fig. 5 illustrates the result of this procedure, which constitutes Stage (iv) of Section 3.1. Point F in this figure corresponds to point F in Fig. 4, which corresponds to the moisture content of specimen batch 4, i.e. 0.95%. Repeating Stages (i) to (iii) of Section 3.1 for specimens from the same batch as that employed so far, but raising the temperature to a series of different levels in Stages (ii) and (iii), led to the generation of the curve displayed in Fig. 5, which gives the anticipated length changes of a notional specimen containing 0.95% water, which would be achieved by changing its temperature from ambient if its moisture content remained constant during the change of temperature. The analysis so far has dealt with only one moisture content, i.e. 0.95%. Going through the same sequence of operations with the results for the other batches of specimens listed in Table I produced four more curves, of the type shown in Fig. 5.

It was mentioned earlier that the lateral dimension of each of the specimens was monitored during the conditioning process. The results of such a sequence of measurements are shown in Fig. 6, from which it is clear that by drawing a smooth line through the results the influence of experimental scatter in deciding upon the value to take for the strain produced by the maximum moisture content attained by a particular batch of specimens can be reduced. Employing this information in association with the curve for each batch of specimens produced according to the procedure leading up to the construction of Fig. 5 allowed the results for

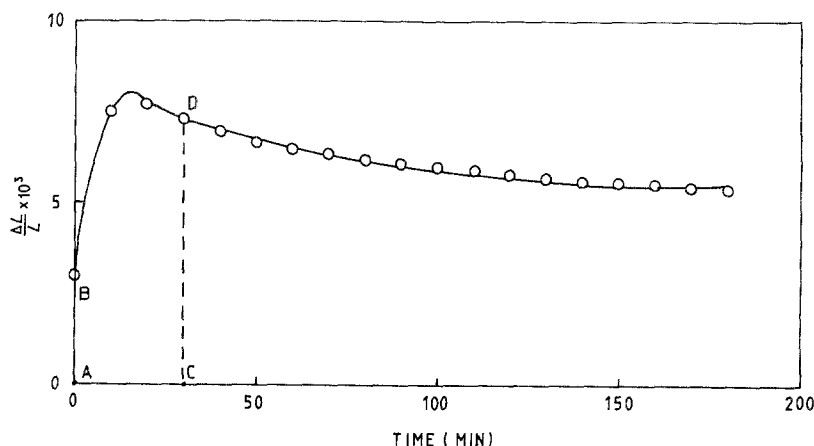


Figure 2 The dependence of fractional length changes  $\Delta L/L$  of a typical specimen (batch 4) upon time during the execution of Stage (ii), in which  $L$  is the length of the dry specimen corresponding to point A and B represents the conditioned specimen, both at room temperature. The temperature was raised at a constant rate from ambient to  $150^{\circ}\text{C}$  between AB and CD, beyond which it was maintained constant.

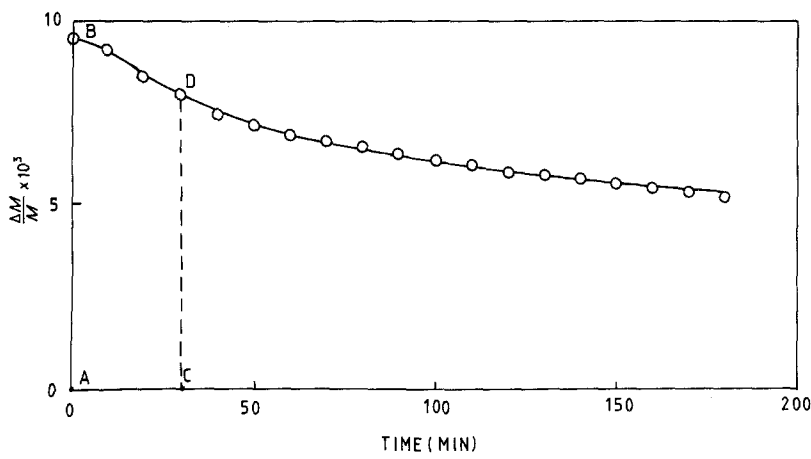


Figure 3 The dependence of fractional mass changes  $\Delta M/M$  of a typical specimen (batch 4) upon time during the execution of Stage (iii), in which  $M$  is the mass of the dry specimen corresponding to point A and B represents the conditioned specimen, both at room temperature. The temperature was raised at the same constant rate from AB to CD as in the experiment corresponding to Fig. 2, beyond which it was maintained constant.

Figure 4 The relationship between the fractional length changes  $\Delta L/L$  of a typical specimen (batch 4) and fractional changes of mass  $\Delta M/M$  resulting from loss of moisture, taken from the results of experiments illustrated in Figs. 2 and 3. Points D in Figs. 2, 3 and 4 correspond to the same condition of the specimen at which temperature  $T$  of Stages (ii) and (iii) was reached. Point E of Fig. 4 corresponds to point B of Fig. 3, from which the location of point F, which gives the expected fractional length changes in the conditioned specimen from which no moisture was lost as the temperature was raised to  $T$ , may be deduced.

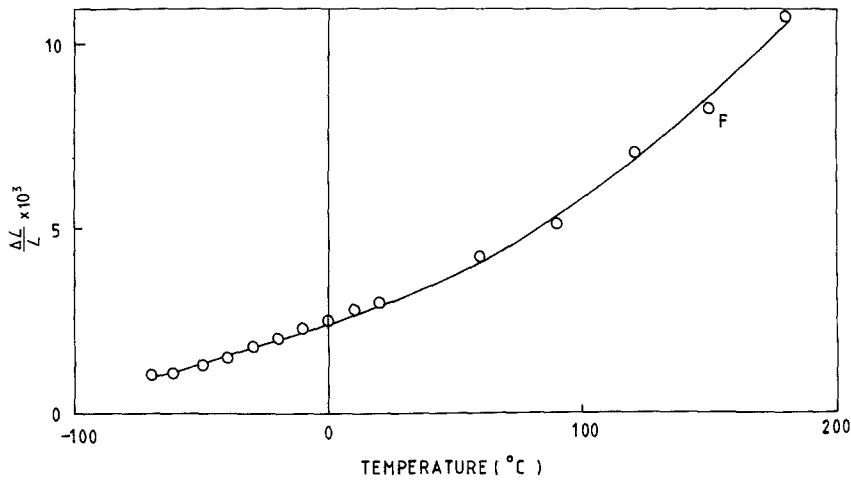
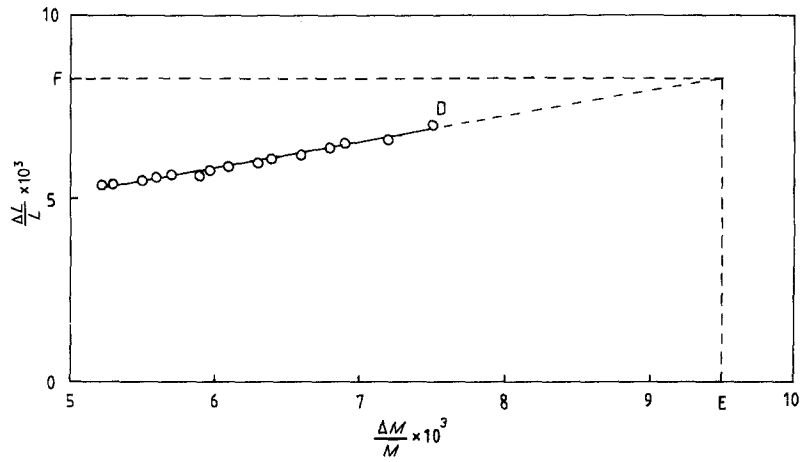
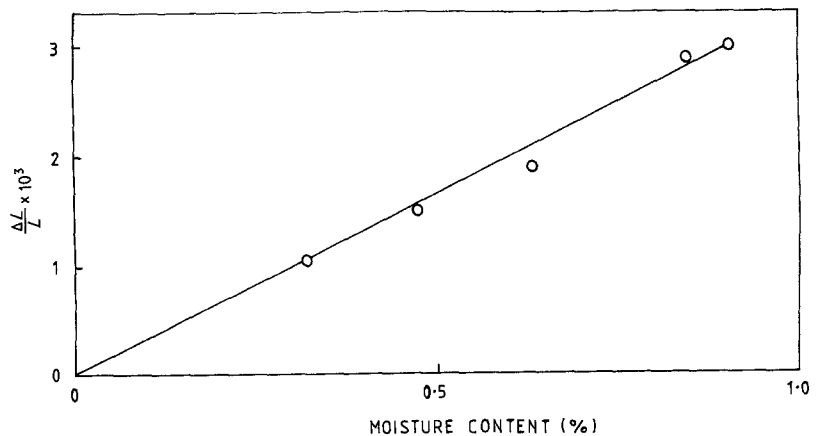


Figure 5 The fractional change of length  $\Delta L/L$  anticipated in specimens from batch 4 resulting from a change of temperature, assuming no loss of moisture during the temperature change. (Point F in this figure corresponds to point F in Fig. 4.)

Figure 6 The strain, measured at room temperature, produced in specimens from batch 4 during the conditioning process.



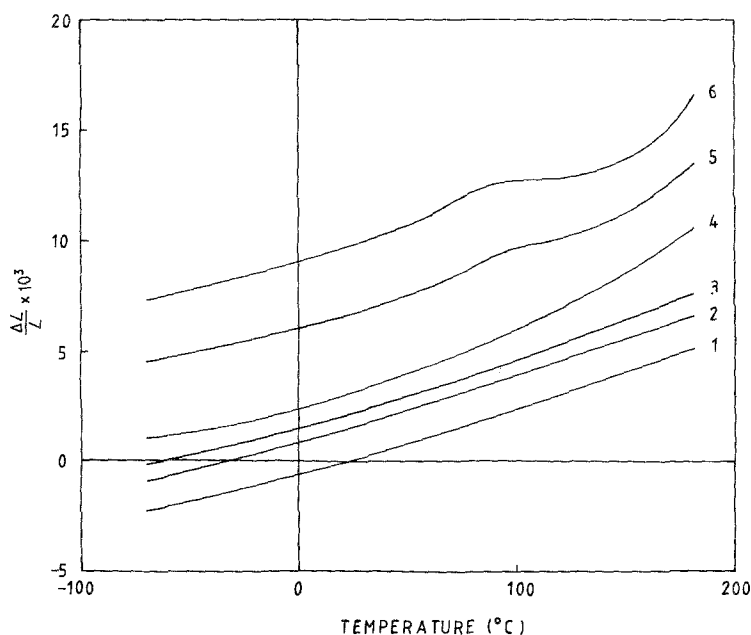


Figure 7 The fractional changes of length  $\Delta L/L$  anticipated in specimens containing different moisture contents resulting from a change of temperature, assuming no loss of moisture during the temperature change. 1: batch 1 (moisture content zero); 2: batch 2 (moisture content 0.47%); 3: batch 3 (moisture content 0.70%); 4: batch 4 (moisture content 0.95%); 5: batch 5 (moisture content 1.57%); 6: batch 6 (moisture content 2.05%).

all the batches of specimens to be presented in one figure, Fig. 7. For the dry specimens the data were obtained from dilatometer runs without any other experiments being necessary, the difference of averages of three determinations being employed in assembling representative data.

## 5. Conclusions

It is quite clear from the results of this investigation that the moisture content of CFRP exerts a very significant influence over its dimensions and the response of these dimensions to change of temperature. From time to time, irreproducible effects have been reported in accounts of the thermal expansion behaviour of CFRP coming out of this and other laboratories. While some of these effects (see e.g. [15]) probably originated predominantly from incompletely cured resin, it now seems likely that others (see e.g. [16, 17]) probably resulted from the resin matrix drying out during the course of the investigation.

It is known that water acts as a plasticizing agent and as such lowers the glass transition temperature of an epoxy resin [18]. Earlier measurements (e.g. [1]) revealed a marked increase in slope in the linear thermal expansion coefficient of unidirectionally reinforced carbon fibre composites as the temperature was raised and this has been associated with passage through the glass transition region of the resin matrix. Examination of curves 5 and 6 of Fig. 7 reveals an increase of slope with increasing temperature, corresponding to which the linear thermal expansion coefficient must increase quite rapidly. Comparing the positions in temperature of a small hump which appears to separate the regions of markedly differing slope in curves 5 and 6 with the absence of such a hump or a marked change in slope in the lower curves leads one to speculate: (i) that the change of slope is to be associated with passage through the glass transition temperature and (ii) that the glass transition temperature is reduced as the water content increases.

The results of this investigation are very conclusive and should be of value in predicting the behaviour of

the constituent elements from which composite structures in aircraft are built up, in a variety of environmental conditions. They serve to show that the designer should take account of the moisture content of CFRP as well as its thermal expansion characteristics when attempting to anticipate responses to real life service conditions. It would clearly be of considerable interest to extend the range of this investigation to cover composites containing fibre lay-ups, which are directly related to the composition and structure of commercial products, and to humidity-temperature cycles which are representative of the environmental conditions in which aircraft operate.

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