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The thermal expansion of carbon-reinforced plastics

Part 7 *Technological implications*

B. YATES
Department of Pure and Applied Physics, University of Salford, Salford, UK

K. F. ROGERS, D. M. KINGSTON-LEE, L. N. PHILLIPS
Materials Department, Royal Aircraft Establishment, Farnborough, UK

We compare the convenience offered by alternative ways of recording thermal expansion data. Limitations imposed on the values, for every day engineering, of the linear thermal expansion coefficients of laboratory produced carbon fibre reinforced plastics are assessed. It is concluded that the commonly encountered levels of moisture content, voids and effects due to the misalignment and non-uniform distribution of fibres do not provide cause for serious concern in most applications. The practical consequences of thermo-elastic stress are deemed to be more important. It is concluded that provided sensible account is taken of the experience accumulated over the years from the production and application of carbon fibre reinforced plastics, laminate theory may be confidently applied to laboratory data in order to gain a good idea of the behaviour expected from new constructions under normal operating conditions.

1. Introduction

Earlier papers in this series [1–5] have described the temperature dependence of the principal linear thermal expansion coefficients (α) of plates of epoxy resin reinforced with unwoven carbon fibres in various planar orientations, and of plates reinforced with woven carbon fibres, all of the plates being produced with the greatest care possible when working under laboratory conditions. The principal objects of the investigations were to produce data which were of direct relevance to technological applications and to assess the extent to which the thermal expansion characteristics of carbon fibre composites conformed with predictions based upon theoretical models.

With a view to identifying some of the practical implications of the results, it is proposed to review the progress achieved up to this point in relation to current technological requirements.

2. Representation of data

It was concluded earlier [4] that the design of carbon fibre reinforced plastics (CFRP) structures with given directional thermal expansion characteristics might best be achieved by using empirical knowledge, supplemented where possible by numerical analysis. Proceeding from this basis, it is appropriate to examine ways of displaying experimental data for plates reinforced with unwoven fibres. The most appropriate data for this purpose within the work covered so far are those based upon Code 69 resin*.

Adopting a form of representation employed in current engineering practice, Fig. 1a shows the in-plane linear thermal expansion coefficient, α, of a $0°$, $\pm 45°$, $90°$ angle-ply laminate of CFRP at room temperature, measured in the $0°$ direction. In displaying α as a function of the proportions of fibre which are oriented at $0°$, $\pm 45°$ and $90°$ to the direction of measurement, detailed corrections have not been applied for the influences of small differences of fibre volume fraction between one specimen and another when taking smoothed data from the earlier papers [3, 4]. The point near the

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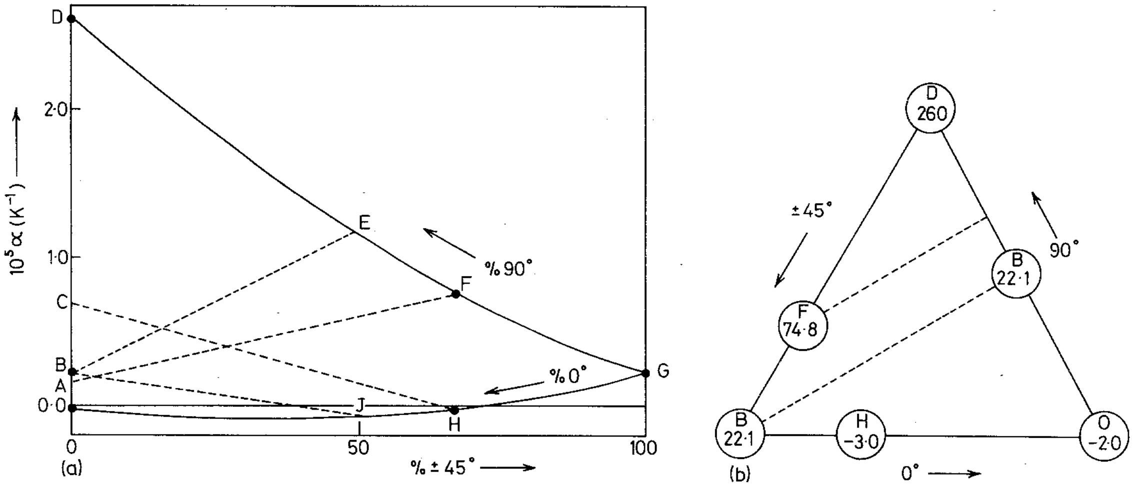

Figure 1 Alternative representations of the in-plane linear thermal expansion coefficients of uni-, bi- and 0°/± 45°/90° specimens of CFRP expressed as functions of the volume percentage concentration of fibres oriented at 0°, ± 45° and 90° to the direction of measurement. (a) • = smoothed experimental data at 300 K, — — — = approximate locations of α corresponding to nominally 33.3% 0° fibre (CH), 33.3% 90° fibre (AF), 50% 90° fibre (BE). (b) — — — = approximate locations of contours of equal α. The letters, B, D, F and H correspond to those employed in (a) and the numbers are values of α in units of $10^{-7}\ K^{-1}$.

origin corresponds to Specimens 26 [3], D corresponds to Specimens 27 [3], B and G correspond to Specimens 28 [3], F corresponds to Specimens 36 [4] and H corresponds to Specimens 35 [4]. Of the three bounding axes, that corresponding to a zero content of ±45° fibre is clearly defined, being a straight line. DG, which corresponds to a zero content of 0° fibre, is less well defined, as also is the axis between G and the point near the origin, which corresponds to a zero content of 90° fibre. Clearly, more points are required in order to define the positions of the axes more precisely. With the aid of such points, however, together with additional points within the area defined by the axes, it should be possible to locate the positions of subsidiary axes corresponding to other fixed proportions of individual components of the fibre content, producing a figure which takes the form of a grid. In Fig. 1a, for example, CH, BJ, AF and BE show the approximate locations of the lines giving the α values of material containing nominally 33.3% of 0° fibre, nominally 50% of 0° fibre, nominally 33.3% of 90° fibre and nominally 50% of 90° fibre, all percentages being by volume.

An alternative form of representation is depicted in Fig. 1b, in which the same data points are plotted in a manner appropriate to a ternary phase diagram and the values of α are given in units of $10^{-7}\,K^{-1}$ within the circles. The presence of more data points would permit the locations of lines of constant α, crude approximations to two of which are shown in the figure.

In addition to examining the influence of fibre type, resin type and fibre volume fraction, the investigations from which the present data points were taken produced results for composites based upon the present resin system which cannot be represented on diagrams such as these, e.g., in-plane results for laminates with an inter-ply angle of 63° and out-of-plane results. In spite of these limitations, the results suffice to illustrate the potential value of representations of this kind, for the development of which a more extensive programme of measurements would be appropriate. Although the representations have been limited to room temperature data, comparable graphs may be set up employing data taken at higher or lower temperatures. Similarly, the out-of-plane linear thermal expansion coefficient could equally form the subject of similar representations, corresponding to such higher or lower temperatures.

3. Practical consequences of thermoelastic stress

3.1. Curing stresses

The response of the shape and dimensions of a CFRP structure to a change in its temperature is governed principally by the elastic constants and thermal expansion characteristics of its constituents. Recognizing the effect on its subsequent performance of stresses which arise in a CFRP structure during cure, measurements of the volume changes which occurred during the curing of a representative series of resins have been monitored [6]. Interpretation of the volume–time curve for each resin led to the approximate location of the temperature, T_c, below which further cure was unlikely to occur. Although for many resins over-cure presents no problems, some resins become brittle if they are cured for too long and/or at too high a temperature. Assuming that the resin shrinkage observed in these investigations provided a direct indication of the progress of polymerization and crosslinking, and that the absence of volume changes could be taken to indicate that polymerization/crosslinking had ceased, it is clear that if a component were to be made which incorporated such a resin but which had been slightly under-cured, it would probably perform perfectly well at temperatures below T_c. However, if during the course of its service life it should rise to temperatures above T_c, further cure would occur and this might influence its subsequent behaviour. Pursuing investigations of this kind and employing the results obtained in association with other physical data for matrix resins and reinforcing fibres should lead to the eventual identification of the temperature below which a resin might be supposed to "lock on" to the fibres of a composite.

A knowledge of this temperature, the residual stress-free temperature, employed in association with knowledge of the temperature dependences of the elastic constants and linear thermal expansion coefficients of the resin and fibres, should permit the calculation of the thermally induced stresses within a composite at any temperature to which it was subsequently heated or cooled. This problem has been considered in some detail by Hahn and Pagano [7], who developed a method of curing stress analysis based upon total stress–strain temperature relations. These workers concluded that their method had advantages over an approach based upon incremental stress–strain temperature relations, since the former method required the thermal strains and the stress–strain relations only at the final temperature of interest. In a further study of the subject, Hahn [8] concluded that the residual stress-free temperature could be lower than the curing temperature and that it depended upon the curing process, a result with which the authors of later work [6] would concur.

The work of Arridge and Speake [9] is relevant. They showed how the elastic properties of epoxy resins depended upon the time and temperature of cure and upon deviations from stoichiometry in the hardener. Johnson and Owston [10] went a stage further in showing how such variations in curing programmes manifested themselves in corresponding variations in the elastic properties of unidirectional CFRP laminae. It may be mentioned that advantage is sometimes taken of the effects of thermally induced stresses at the resin–fibre interface of unidirectional composites in order to produce improved mechanical characteristics. The present authors are not aware of any reports of the influence of thermal ageing upon the thermal expansion characteristics of CFRP, but the possibility of such effects arising from thermally induced stress at the resin–fibre interface should not be dismissed. Very significant changes in the thermal conductivity of a unidirectional glass fibre reinforced epoxy resin are reported to have been induced by repeated thermal cycling [11] and it is to be expected that associated changes in the thermal expansion characteristics of such a system could also occur.

3.2. Performance stresses

The above works refer largely to stresses which may be associated with the production of the composite. Themoelastic stresses also arise during the subsequent performance of the composite and these may have serious practical consequences. In this connection Kedward [12] has drawn attention to the buckling of an unbalanced laminate which will result from the application of a uniform temperature gradient and the displacement of the boundaries of a hoop-wound ring which will result from a rise in its temperature. The magnitude of the last effect becomes apparent when the rapid increase with temperature of the out-of-plane linear thermal expansion coefficients of typical CFRP cross-plies are examined [1]. Temperature-dependent length change reversals have been observed in carbon fibre fabric-reinforced plastics [5]. The origin of this effect appears to be a difference between the thermally induced stresses in the two principal fibre directions, though whether it arises from differences of fibre density or crimp in the warp and weft directions is not clear. Careful account should be taken of physical

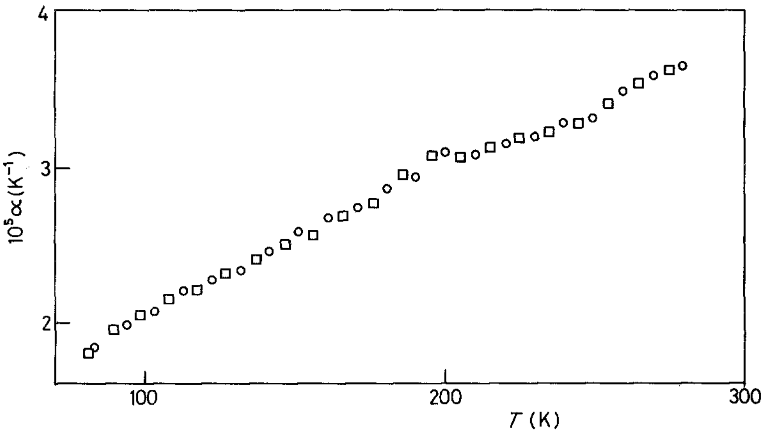

Figure 2 The linear thermal expansion coefficient, α, of specimens prepared from Bar 4 of Rogers *et al.* [1], i.e. a bidirectional lay-up of CFRP with an inter-ply angle of 63°, consisting of Courtaulds HTS fibre arranged in 25 alternate and equally populated layers in a matrix of resin ERLA 4617/mPDA, containing internal cracks. The direction of measurement was perpendicular to the plane of the laminate; ○ = run 1, □ = run 2.

characteristics such as these when designing structures which are subjected to temperature changes in which dimensional tolerances are particularly critical, e.g. microwave filters, waveguides and aerials employed in satellite communications systems [13], bushes supporting rotating shafts, cylinders containing moving pistons, etc.

Operating stresses between the constituent laminae of a laminate may be sufficient to cause partial or complete delamination in an imperfectly bonded structure. Some idea of the comparative influences of partial delamination upon the in-plane and out-of-plane thermal expansion characteristics of a bidirectional laminate may be gained from an examination of results for such a laminate obtained during the course of the present programme. The bar in question was a bidirectional laminate having an inter-ply angle of 63°, which formed Bar 4 in the work of Rogers *et al.* [1]. From the manner in which the in-plane results for this laminate fell into place within the pattern describing the dependence of the in-plane linear thermal expansion coefficient upon inter-ply angle in biaxial laminates, (Fig. 20 of [1]), it is clear that, in the presence of a limited amount of delamination, the different regions of a biaxial specimen may be sufficiently well balanced to exhibit the behaviour expected of a structurally perfect bar. Extrapolating the out-of-plane results for this laminate to room temperature (as shown in Fig. 2), and plotting this extrapolated result on the graph displaying a comparison of the measured and calculated dependence of the out-of-plane linear thermal expansion coefficient of bidirectional laminates upon inter-ply angle, (Fig. 22 of [1]) produces the result shown in Fig. 3. The observation that this result is considerably lower than the expected value, coupled with the good agreement obtained in the corresponding case of a structurally sound bar containing high modulus fibre (Fig. 23 of [1]), leaves little doubt that the inter-ply cracks accommodate a good deal of the thermal expansion in the out-of-plane direction of the partially delaminated biaxial bar.

In contrast to partial delamination between the constituent laminae, Marom and Gershon [14] examined the influence of the interfacial bond strength upon the thermal expansivities of epoxy resins reinforced unidirectionally by carbon and glass fibres. Degrading the bond strength by exposing the specimens to boiling water reduced the constraint upon the resin in the fibre direction and reduced the extent to which the resin was squeezed into the transverse direction upon subsequent heating. As expected, the result was to increase the longitudinal thermal expansion and to diminish that in the transverse direction.

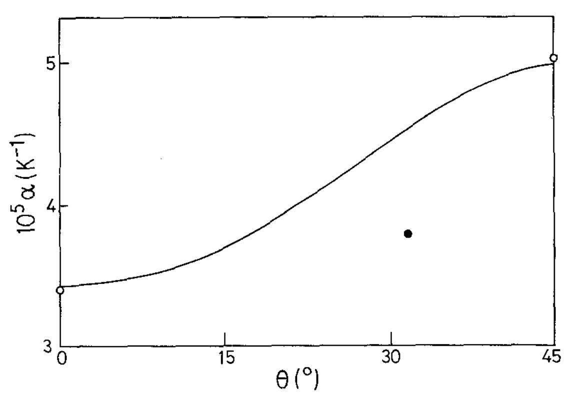

Figure 3 The out-of-plane linear thermal expansion coefficient α of a bidirectional laminate consisting of resin ERLA 4617/mPDA reinforced with HTS fibre, in which θ is half the inter-ply angle. The line was calculated as described earlier [1]; ○ = experimental results for structurally sound specimens: ● = experimental result for partially delaminated specimens.

3.3. Origins of potential failure

The origin of the imperfect bonding between resin and fibres in the unidirectional case, or between the constituent laminae of multidirectional laminates, may arise from a variety of causes. Theocaris *et al.* [15] reported the formation and growth of microcracks in an epoxy resin when this was being processed at moderately high temperatures, and attributed this to the rejection of the plasticizing agent by the surface layers of the material. Thermal cycling or mechanical shock might well cause such cracks to spread in composites employing such resins as matrix. Occasional mistakes have occurred over the years, during the curing programmes employed in the present work, arising from both equipment failure and human error. It was concluded from this experience that curing at too high a temperature or for too long a time can result in the development of interlaminar cracks. The stresses which result from imperfect bonding in fibre-reinforced composites (and the debonding which can result) have been examined analytically by Erdogan and Ozbek [16] and by Conway *et al.* [17]. The value of such treatments is clearly enhanced when the origin of the cause of the original failure can be identified.

3.4. The influence of thermal history

In formulating equations for the linear thermal expansion coefficients of a composite in terms of the linear thermal expansion coefficients and elastic constants of its constituents, it is usually assumed that the constituents are behaving in an elastic manner. For many applications this is a good approximation, but when working over wide ranges of temperature or when applying heavy loads the matrix may be sufficiently deformed to pass from the elastic to the plastic state, while the reinforcing fibres remain elastic. Karpinos *et al.* [18] have indicated how one of the usual expressions for the principal linear thermal expansion coefficient of a unidirectional composite might be expected to be modified in such a case, in order to incorporate the influence of the yield stress of the matrix. Situations such as this result in hysteresis effects, examples of which are to be found in the literature [19, 20], while Garmong [21, 22] has shown how the elastic–plastic deformation induced by thermal stresses associated with temperature cycling can influence the temperature-dependent dimensional behaviour of composites. In addition to examining various factors which are associated with the formation of cracks in CFRP, which result from thermal cycling, Spain [23] has examined the effects of adding plasticizing agents. Associated with this attempt to reduce cracking, a reduction of elastic constants was observed; and the author concluded that further progress might be possible by reducing the thermal expansion coefficient of the resin with the aid of an appropriate particulate additive or filler. In the context of practical applications, the phenomenon of microcreep [24] should be mentioned. The importance of this effect warrants its investigation in specific cases where dimensional tolerances are particularly critical over long periods. Shifts of the linear thermal expansion coefficients of carbon fibre reinforced plastics accompanying successive temperature cycles, which have been observed over a period of years and which have been attributed to changes of water content, are described in Section 5.

4. Structural imperfections

4.1. Voids

Although every effort is usually made to produce composites which are structurally perfect, it is sometimes difficult to avoid the inclusion of voids in a CFRP structure. The influence of voids upon the physical properties of a composite will depend upon: (a) the overall volume fraction which they occupy, (b) their distribution and (c) their shape and orientation. Petker [25] reported that the influence of void content on the interlaminar shear strength of epoxy resins reinforced with glass and boron fibres grew serious above a volume fraction of about 3%. In a series of investigations of the influences of flaws and voids upon the shear properties of CFRP rods and tubes, Hancox [26] concluded that whereas rods and tubes containing certain flaws were amenable to the application of fracture mechanics, materials containing voids did not lend themselves to comparable analytical treatments. However he did observe that the shear properties of CFRP were reduced by 30% of those for void-free CFRP by the presence of 5 vol% voids. Materials containing hypothetical void contents and distributions may be treated mathematically. Although such systems may not be physically realistic, analyses of this kind serve to give some idea of the magnitude of the influence of voids on the physical properties of a material. Employing a matrix representation for the determination of the stiffness coefficients of a composite, Kavanagh

[27] has shown that the theoretical minimum number of two experiments which are required to characterize an orthotropic material in plane stress is, in general, insufficient. The reason is that variations in void content of real specimens can lead to material constants which violate the symmetry conditions assumed.

The similarity between mathematical formulations of mechanical and thermal stress in composites permits direct adaptations of these ideas concerning the quantitative influence of voids to the phenomenon of thermal expansion. Until the uniformity of the void distribution has been established, however, the technological value of such work would be questionable and a first step in the appraisal of appropriate experimental data might attempt to employ a ternary phase diagram form of representation, as suggested by Price and Nelson [28]. Here the voids form one of the phases of a nominally two-phase composite. It is difficult to assess the level at which the influence of voids upon the thermal expansion of a typical composite is likely to assume significant proportions. An examination of the extent to which results obtained during the present series of experiments conform with expectations based upon theoretical models, leads to the belief that a void content of 2 to 3% is not particularly serious for most practical purposes. Having said this, the lack of a detailed understanding of the influence of voids upon the thermal expansion and other physical characteristics of CFRP structures, together with the knowledge that voids prevent the full realization of composite capabilities, leads to the conclusion that the void content should always be kept to a minimum.

4.2. Fibre misalignment

A further factor which may prevent a composite from realizing its full potential is the presence of misaligned fibres. Misalignment may arise from a variety of causes and may take two basic forms: (a) a distribution in which the fibres remain linear but which contains an angular scatter, (b) a lay-up in which the fibres are slightly buckled. The influences of these effects upon the elastic moduli of composites have been considered in some detail [29–31]. No rigorous treatment of their influence upon the thermal expansion of such materials appears to have been undertaken, though one approach would be to employ a modified version of the theory employed when considering multidirectional effects [4] in laminates. The effects will be most marked when the expansion parallel to the fibres of a unidirectional system is influenced by a small proportion of the much larger thermal expansions at right angles. Generally speaking, with constantly improving commercial techniques for achieving better fibre alignment, it seems unlikely that fibre misalignment will constitute a serious problem in future years.

4.3. Fibre distribution

If the fibres of a composite are not distributed uniformly there will be regions which are resin-rich and regions which are fibre-rich. If these compositional variations conform to a regular 2- or 3-dimensional pattern, then the problem presented is that of a regular composite within another regular composite. Although many of the specimens employed in the later stages of the present investigations were constructed from commercial fabrics, the "building-blocks" of the earlier specimens took the form of discrete, unwoven, linear tows of fibre impregnated with resin [1]. Optical examination of these fabric-based bars had revealed that the moulding pressure caused the cross-sections of the tows to become oval, and resin-rich regions were clearly visible between them. The importance of any non-uniformity of a fibre distribution obviously depends upon its extent, but an example will serve to give some idea of the size of the effect in typical practice. Fig. 4 shows the linear thermal expansion coefficients of two sets of specimens cut from a bar of unidirectionally reinforced CFRP (Bar 5 of [1]). The principal axis of one set of specimens lay perpendicular to the fibre direction and perpendicular to the pressing plane while that of the other set lay perpendicular to the fibre direction but parallel to the pressing plane. Any difference between the two sets of results lies within the limits of experimental uncertainty.

A full appraisal of this phenomenon would constitute a major undertaking but it seems reasonable to conclude that the level of non-uniformity commonly encountered is not sufficiently serious to influence the thermal expansion characteristics.

5. Moisture absorption

Many CFRP structures will be exposed to environmental variations of humidity under normal operating conditions. In aerospace applications these variations are likely to be extreme and

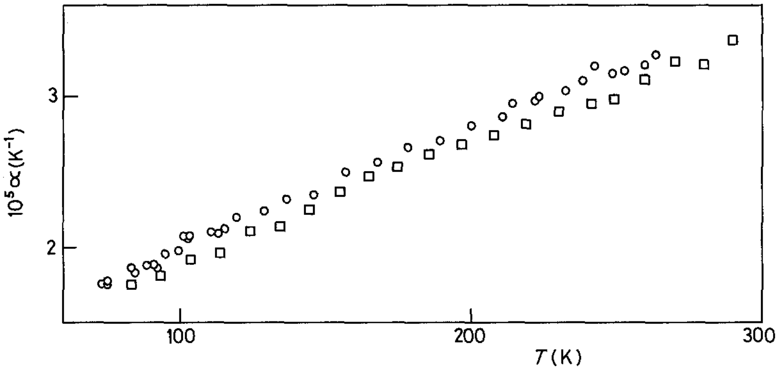

Figure 4 The linear thermal expansion coefficient, α, of unidirectional specimens of CFRP in directions perpendicular to the fibres; ○ = perpendicular to the pressing plane, □ = parallel to the pressing plane. The bar from which the specimens were cut is Bar 5 of [1], built from tows of resin-impregnated fibres.

the influence on the performance of a typical component is a matter of considerable practical importance. It is well known that epoxy resins absorb water. As a matter of interest the change in weight of a piece of DLS 351/$BF_3$400 resin was recorded during the course of the present investigations, as this was exposed first to a drying atmosphere, followed by immersion in water. The results of these weighings are reproduced in Fig. 5, from which it seems that a composite consisting of this resin reinforced by carbon fibres will exhibit mass changes amounting to a few per cent.

The nature of the absorption process and the degradation of the mechanical properties of composites based upon resin matrices have been studied in some detail [32–36]. Adamson [37] proposed a model to explain results for the absorption of water by Hercules 3501 epoxy resin, in

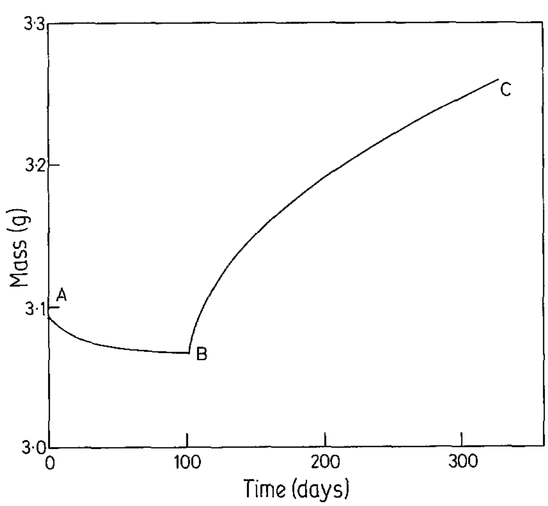

Figure 5 The time dependence of the mass of a piece of DLS 351/$BF_3$400 resin subjected to different environments at ambient temperature. Between A and B the specimen was surrounded by an atmosphere dried by anhydrous calcium chloride; between B and C the specimen was immersed in water.

which he supposed that packing irregularities between molecular domains resulted in the formation of voids. It was supposed that some of the water molecules which first entered this "free volume" caused no swelling, while others disrupted interchain hydrogen bonds and caused swelling by bonding with the resin. The linear thermal expansion coefficient of resin between 274 and 347 K, which had first been saturated with water to equilibrium at 347 K, was almost two and a half times that of the dry resin. The value for a specimen which had been similarly saturated, then temperature cycled several times before being dried out prior to investigation, was intermediate between these limiting values. The results were explained in terms of water molecules breaking away from the polymer molecules to which they had become attached at 347 K, as the temperature was reduced. These water molecules were believed to enter additional free volume made available by the reduction in vibrational amplitudes accompanying the fall of temperature. Adding this contraction to the "normal" contraction accompanying a fall of temperature of dry resin was believed to give rise to the abnormally large thermal contraction observed. The influence of the resin upon the thermal expansion coefficients of a composite, of which it forms the matrix, is made through its thermal expansion coefficient and elastic constants. The increased influence of the linear thermal expansion coefficient of the hydrated resin would be offset by a reduction in its elastic moduli. The resultant effect of water absorption by the resin matrix upon the linear thermal expansion coefficients of the composite is likely to be less marked than it was in the case of the pure resin. The authors are not aware of any studies of the influence of water absorbed by *carbon fibres* upon their thermal or mechanical properties, but the influence of any such absorption upon the temperature dependent dimensional behaviour of carbon fibre reinforced plastics is likely to be very small compared with effects associated with water absorption by the resin matrix.

The influence of absorbed water upon the linear thermal expansion coefficients of carbon fibre reinforced plastics does not appear to have been studied directly. However Parker *et al.* [38] compared observations made during the course of their investigations of the thermal expansion characteristics of some carbon fibre reinforced plastics, with earlier observations of Dootson *et al.* [39], Yates *et al.* [6], Freeman and Cambell [40] and Tennyson *et al.* [41] to draw tentative conclusions. Coupling these with observations of dimensional reductions resulting from temperature cycling observed recently [5], it seems reasonable to conclude that the presence of water in carbon fibre reinforced plastics is likely to cause the linear thermal expansion coefficient to be different from that of the corresponding dry composite. The effect seems to be to reduce the linear thermal expansion coefficient, α, the percentage reduction in α depending upon its absolute magnitude and the amount of water present. Time-dependent increases observed in the linear thermal expansion coefficients of CFRP at constant elevated temperatures [39] are also likely to have their origin in the presence of water, while it appears that the removal of water from CFRP results in a reduction of dimensions, at a fixed temperature [5].

6. The influence of weaving fibrous reinforcement

Increasing use is being made of carbon fibre reinforced plastics in which the reinforcing fibres are woven. An account of a detailed investigation of the influence of crimp and fibre tow density in the warp and weft directions of a series of such laminates upon their principal linear thermal expansion coefficients has been given elsewhere [5]. One of the more striking observations to emerge from this investigation, that of temperature-dependent length change reversals, has already been mentioned in Section 3.2. In the present context, it may be noted that this behaviour can be prevented by arranging for warps and wefts to alternate in direction from one layer to the next, so as to equate thermally induced stresses in the two principal fibre directions of the laminate. Failure to observe this precaution also results in temperature-induced length changes differing in the principal fibre directions, by anything up to 100%.

7. Summary

The object of the present study was to examine the extent to which practical considerations influenced earlier conclusions [1–4] concerning the applicability of theoretical models to the prediction of the thermal expansion characteristics of resins reinforced by carbon fibres. A final assessment is prevented by the absence of knowledge of some of the physical properties of both resins and fibres, but some provisional conclusions are possible.

It is probably true to say that the least important of the factors considered in this brief review are structural imperfections associated with the misalignment and non-uniform distribution of fibres. Also, the level of voids commonly achieved is usually sufficiently low to cause no serious problems. The importance of moisture clearly depends upon the level at which it is present, but evidence suggests the influence of the moisture encountered under normal atmospheric conditions upon the thermal expansion characteristics of CFRP components is not sufficient to cause concern in most applications. Consequences of thermoelastic stress can be sufficiently serious to result in failure in extreme cases, if due regard is not paid to experience which has been accumulated over the years. Much work remains to be done in order to clarify the influence of details of the past history of a composite upon its subsequent thermal behaviour, but the balance of evidence gives confidence for the employment of laminate theory in providing a good indication of the expected thermal expansion characteristics of CFRP under normal working conditions.

Acknowledgement

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