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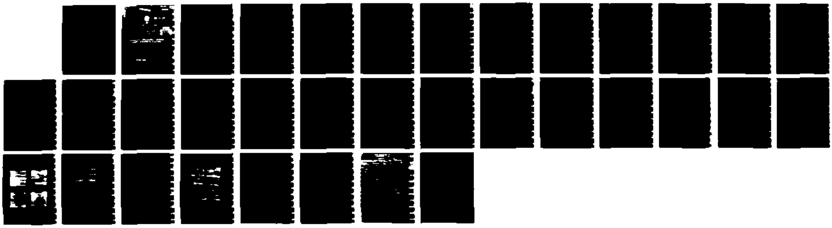

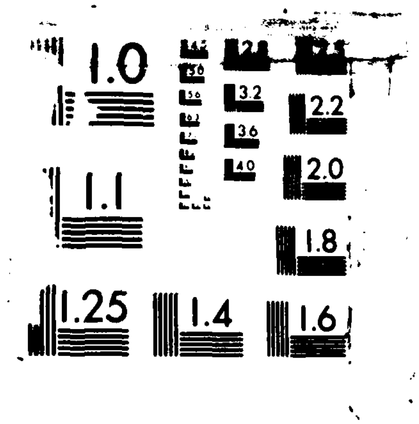

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April 1985

AUTOCLAVE MOULDING OF CARBON FIBRE REINFORCED PLASTICS

by

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SUMMARY

The numerous facets of autoclave moulding have been investigated in order to provide an improved understanding of empirically derived cure cycles. Such features as partial-cure, dwell-time, dual vacuum, and dual pressure have been examined and their significance in relation to laminate quality explained.

(keywords: carbon reinforced composites, carbon fibers, plastics)

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1 INTRODUCTION

Whilst the use of carbon fibre reinforced plastics (CFRP) in aerospace structures is now widespread and its performance as a material increasingly well understood, the technique of producing components of satisfactory quality remains more of an art than a science. Aerospace components of high and consistent quality can only be currently produced by empirically determining the appropriate processing parameters and then maintaining strict control over these parameters during production. This Report describes investigations into the various facets of autoclave moulding and the parameters affecting material quality in order to elucidate some scientific bases for the current empirical techniques.

The base material for the investigations was carbon fibre-epoxy preimpregnate (prepreg). Prepreg consists of thin sheets of uni- or multi-directional carbon fibres with an exact proportion of resin which has been partially cured. This partial cure provides some measure of physical integrity at room temperature to facilitate handling. Material fabrication is by laminating a number of prepreg sheets on a mould and compacting the laminae while the resin cures at elevated temperature. Heat is required both to reduce the resin viscosity, thus allowing compaction under pressure, and to cure the resin to form a solid laminate. The essential feature of an autoclave is that the material is both heated and compacted by the gas enclosed within the vessel. However, this simplicity of method gives rise to complex thermal considerations due to the interaction of convective heat transfer rate and vessel gas pressure and the effect of the thermal mass of material and mould on the material temperature cycle.

The viscosity of the matrix varies enormously during the material temperature cycle and it is shown that for a given resin system there is a specific combination of temperature cycle and pressure cycle which will produce a laminate without excess loss of resin. This particular combination of temperature and pressure cycles, which is usually derived empirically, is referred to as the cure cycle.

In addition to the laminate itself there are a number of other materials in the autoclave, in particular the absorbent layer(s) inserted to take up excess resin, and it is demonstrated that these also can have critical effects on the quality of the laminate.

Although the precise figures given in this Report are derived mainly for one specific CF-epoxy system, the general arguments concerning the interrelationships between the various autoclave parameters are valid for a wide range of fibres and matrices.

2 THE CURE CYCLE

For each resin system and for each laminate and its associated tooling there will be a particular combination of autoclave conditions which will produce a satisfactory laminate - there is no 'universal' cure cycle. In addition, it will be shown that the range over which any parameter may vary whilst still producing laminates of high quality is often extremely small. A typical cure cycle is illustrated in Fig 1. The vessel gas temperature is raised linearly from ambient A to the resin cure temperature T in time t_1 and is controlled at this temperature until the completion of the resin cure at t_3, when it is force-cooled to ambient at t_4. The laminate temperature follows the gas temperature asymptotically to the cure temperature at B where it is held until t_3 . It is then lowered to ambient by the cooling gas. The pressure is increased at time t_2 , thus increasing the heat transfer rate and producing a discontinuity in the laminate temperature curve.

The resin will initially behave as a liquid and with increasing temperature its viscosity will fall (PQ Fig 1). The resin passes through a stage known as gelation, which is associated with the change of phase as it tends towards the solid state. A precise definition of gelation is beyond the scope of this Report. For comparison purposes the gel time is here considered to be the time at which the resin viscosity rises to 700 poise since the steepness of the curve at this stage is such that the exact level is not critical.

3 AUTOCLAVE PROCEDURES AND ASSOCIATED MATERIALS

A typical assembly of laminate and associated materials for autoclave moulding is shown in Fig 2. The region of space containing the laminate is separated from the interior of the autoclave by a membrane, known as the 'bag', which is closed around its periphery by a 'sealant'. This closed space is connected to the outside of the autoclave by the 'bag pressure line'. On the inside of the bag is a 'porous membrane' which ensures a continuous gas path over the whole area. The 'prepreg laminae' lie on the moulding 'tool' and are covered by a porous 'release layer'. Excess resin exuded from the laminate is taken up by the 'absorber'. In many cases, release layers and absorber occur on both sides of the laminate and the laminate is surrounded by a close-fitting 'dam'. The 'dam' is normally used to prevent the edges of the laminate being rounded off due to the tension of the bag. For flat laminates, the outer, non-tool surface may have a 'top plate' to provide a more even finish. Two of these items are of particular importance in determining the quality of the final laminate. One, of course, is the prepreg and the other is the absorber.

3.1 Prepreg

Prepreg consists of thin sheets of evenly distributed uni- or multi-directional carbon fibres uniformly impregnated with an exact proportion of epoxy resin which has been partially cured. There are several prepreg variables which may affect the quality of a laminate, three of which are significant in autoclave techniques. These are the resin content, the degree of partial (B-staging) cure and fibre alignment, the latter being discussed in a later section.

3.1.1 Resin content

Subject to there not being less resin in the prepreg than required for final laminate, it might be thought that the amount of excess resin present is not critical. Indeed, for two very different resin systems Fig 3 shows that the fibre and void contents may not vary even with large changes in resin content given careful processing. However, excess resin takes time to exude and be absorbed thus reducing the tolerance of the cure cycle as will be shown later. Removal of relatively large volumes of resin also entails significant movement of the fibres and of the overall outer surface, both of which cause deterioration of laminate quality. For these reasons the prepreg normally used contains little excess resin, the process being referred to as 'zero-bleed'.

3.1.2 Partial cure

Prepreg materials are usually subjected to heat treatment during production both to remove solvents and to give the sheet some physical integrity. This heat treatment (often referred to as B-staging) partially reacts the resin constituents and thereby raises the viscosity of the matrix. This increase in viscosity carries right through the subsequent viscosity profile during cure. At room temperature the viscosity is increased from high to very high and the prepreg is stiff and coherent enough to handle; during the cure cycle the viscosity does not vary in the same manner as it would without partial cure. Such effects may be demonstrated experimentally using a resin cure-cycle simulator[1]. A number of resin samples were partially cured at 94°C for periods from 1 to 5 hours and then subjected to a simulated autoclave cycle. This cycle is shown in Fig 4 together with the simulator viscosity output curves for the 1 hour at 94°C and 5 hours at 94°C combinations. For the two curves, the differences in time to reach some selected levels of outputs A and B will give a measure of the effect of partial cure on the viscosity (A) and time of gelation (B). (Simulator output 50 = 700 poise, see previous section.) These variations of viscosity and gelation with the degree of partial cure are shown in Figs 5 and 6 respectively and illustrate how critical the partial cure process can be both in terms of temperature and time.

3.2 The absorber

Even in a nominally zero-bleed system there must be some excess resin in the prepreg and since the quantity of resin in both the prepreg and final laminate should be known, the amount of excess may be determined. This surplus resin will exude from the laminae during cure and therefore the correct amount of absorbent material must be incorporated. The absorbency of a material can only be determined experimentally and for a given material will depend on the operating pressure and the thickness of absorber as shown in Figs 7 and 8. The inverse relationship between absorbency and thickness is due in part to the finite time required for the resin to reach the outer material. This time may be a significant proportion of the time during which resin will flow and is one reason for keeping the excess resin to a minimum.

On account of its porosity the absorber can actively extract resin from the laminate and thus excess absorber can lead to high void contents. In this context it should be noted also that care must be taken to ensure that the porous membrane, Fig 2, does not contact the prepreg and act as an additional absorber.

4 THE TEMPERATURE CYCLE

A typical laminate temperature cycle was outlined earlier and illustrated in Fig 1. Autoclave cure cycles are typically optimised using a laminate heat-up rate of approximately 2°C/min. The rate of rise of applied gas temperature required to achieve this laminate response at atmospheric pressure may vary from 2°C/min to 10°C/min or more; the larger the thermal mass of the laminate and mould tool, the faster the gas temperature rise necessary to obtain a laminate temperature ramp of 2°C/min. It can be seen also from Fig 1 that it is during this laminate heat-up time that the changes in state of the resin occur. Most significant is the gel time, since after gelation no resin flow or forming of the laminate can occur. Some examples of the effects on gel time of variations in the temperature cycle are given as follows.

4.1 Spatial temperature variations and heat-up rate

Spatial temperature variations arise from differences in the thermal masses being processed and temperature gradients within the autoclave. These temperature differences are at a maximum during the initial stages of the temperature cycle and have a similar influence on the gel time. Fig 9 shows gelation to occur about 6 minutes earlier due to a quite modest temperature increase at gelation of 10°C. Curves such as those of Fig 9 arise equally from changes in heat-up rate; that is, the figure can be interpreted as the effect of increasing the heat-up rate such that, for example, the laminate reaches 130°C after 30 instead of 40 minutes.

4.2 Dwells

Where large thermal masses are involved, it is common-place to reach the final temperature in two stages. An intermediate temperature is held constant for a period of time so that the laminate can reach a uniform temperature before pressurization, this period being known as a dwell. Even though the dwell is introduced when the temperature is relatively low (*eg* 130°C) compared with the cure temperature (*eg* 180°C), it will none-the-less alter the gel time. Fig 10 illustrates the effect on the time of gelation of introducing dwells of indefinite length at 125°C and 135°C. It can be seen that this apparently trivial temperature difference, which may merely reflect a tolerance of ±5°C on 130°C, has a significant effect; an increase of 10°C reduces the time to gelation from 90 minutes to 60 minutes.

4.3 Pressure changes

It has already been noted that because laminates in an autoclave are heated by forced convection, the heating rate will depend on the vessel pressure. Consequently the gel time will vary with both the magnitude of the pressure and its time of application. Fig 11 shows how the gel time varies when, for a representative temperature cycle, different pressures are applied after the same time interval. Similar curves are obtained due to changes in the time of pressure application and it should be noted that gel time is not linearly related to the time of pressure application as can be seen from Fig 12.

4.4 Partial curing

When discussing the effect of variation of partial cure by the prepreg manufacturers (section 3.1.2) it was seen that such variations had important effects on the resin viscosity cycle. For low viscosity resin systems in particular it has been shown[2] that increasing the degree of partial cure prior to pressurization in the autoclave may considerably improve the quality of a laminate by raising the minimum viscosity of the resin. Whilst incorporation of partial cure into the temperature cycle is identical in form to the introduction of a dwell, it must be emphasised that the purpose is completely different and the temperature, pressure and time more critical[2]. The state of cure of a resin is continually increasing with time; thus, where nominally identical batches of prepreg are supplied over a long period but used within a short time of each other, it may be desirable in later batches to introduce artificial ageing (partial cure) during the temperature cycle in order to provide constant batch-to-batch viscosity characteristics during the critical stages of the cure cycle.

5 FIBRE PACKING

So far the prime considerations have been related to the response of the resin. Before turning to the pressure cycle it is necessary to discuss the behaviour of the fibres under pressure. The carbon fibres in the prepreg material will have some packing arrangement in which the fibre volume may vary from the maximum hexagonal array for well-aligned fibres to less than half the maximum for misaligned fibres. In the precursor tows the fibres will be well-aligned, but at each processing stage up to and including final lamination this arrangement will be gradually degenerated.

Although in the prepreg the fibre tows and individual fibres may be widely spaced by excess resin, this degree of alignment will be preserved and this will result in an 'inherent packing fraction' for a particular material. When the prepreg is heated at the commencement of the cure cycle the spatial constraint placed upon the fibres by the resin will be reduced and, even under zero applied pressure, surface tension will act to draw them together. Application of pressure will cause some further compaction and therefore the magnitude of the pressure required affects not only the resin properties (section 4.4) but may also affect the fibre and void content of the final laminate. How much pressure to apply is not easily predicted and it is certainly not a case of a simple relationship between, say, desired fibre volume fraction V_f and pressure as can be seen from Fig 13 which shows the variation of V_f with compacting pressure. It can be seen for a typical prepreg quite small pressures can cause increases in V_f of as much as 10%, but above 150 kPa V_f is almost constant. Fig 14 shows cross sections of laminates, made of nominally identical material but supplied by two prepreg manufacturers, having fibre volume fractions of 62% and 75%. The 75% material can be seen to have well-aligned fibres resulting in large areas with closely spaced hexagonal arrays, whilst the less well-aligned fibres in the 62% material restrict the general proximity of the fibres. Thus the packing fractions achieved are in the main an inherent property of the particular prepreg material. In current material, fibre alignment within a prepreg sheet is usually good and most misalignment occurs between the plies of a laminate due to handling and slight misalignment between sheets.

When the compacting pressure is applied the resin is normally in a liquid state and the pressure is therefore, under static conditions, supported entirely by the fibres. Thus under increasing pressure the fibres will move together until enough are in contact to react the pressure. Thereafter, further increase of pressure serves only to distort the fibre tows or break those fibres whose misalignment is restricting compaction. Hence it would seem from Fig 13 that 150 kPa should be an optimum operating pressure. However, it will be shown in the next section that greater pressure has to be applied in order to reduce the void content of the laminate.

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The same concept of an inherent packing fraction eliminates the production of low fibre volume fraction laminates from prepreg by using a very low compacting pressure even if the excess resin can be prevented from exuding. Fig 15 shows a cross section of a material made in this way. It is of high inherent volume fraction (75%) but was cured under conditions leading to an average V_f of 60%. However, all the additional resin has been retained between the laminae and V_f varies locally between 0 and 75%. Such a material is unsatisfactory since the mechanical properties vary significantly and randomly throughout the laminate. Thus the final thickness and fibre volume fraction of a good quality laminate are little affected by the autoclave cycle but are determined by other manufacturing processes up to and including prepreg lay-up.

6 THE PRESSURE CYCLE

During the autoclave process there will usually be a single synchronous pressure change of both bag and vessel as shown schematically at time T , Fig 16. Before T it is necessary to maintain pressure within the bag (P_b) below one atmosphere both to hold the bag contents in place and to enhance removal of any gases, including air, a technique known as 'vacuum stripping'. The simplest cycle requires the vessel to be at amospheric pressure (P_v). Under these circumstances the laminate is subjected to a compaction pressure of one atmosphere. If this pressure is shown to be too great at this early stage in the cycle, for instance causing too much resin loss, it is necessary to reduce the vessel pressure to some lower value P'_v , less than P but greater than P_b . This method is known as 'dual vacuum'. The reduced pressure will also lower the heat-up rate and thus the resin viscosity will not fall as rapidly. After application of pressure at time T , a similar complication arises. The simplest and most economical condition is to increase the vessel pressure to its higher level Q_v and vent the bag to atmosphere, Q_b . A larger pressure can be applied to the laminate by maintaining the bag under vacuum Q'_b . In the case of some systems, large absolute pressures may retain any volatiles dissolved in the resin and thus it may be considered beneficial to provide a positive bag pressure Q''_b at this stage to avoid over-compaction. This technique is referred to as 'dual pressure'. The additional pressure will, of course, increase the heat transfer rate and consequently advance the gel time, thus in itself restricting resin flow and reducing the likelihood of voids.

It was shown earlier (section 4.3) that the time of application of pressure T can greatly influence the gel time of the resin and thus T , normally the time when the viscosity is low as will be seen later, is determined by the resin characteristics and the particular temperature cycle being used. Thus for a given resin system and temperature cycle the time of alteration of pressure and its magnitude are so interlinked as to produce a unique cycle within which it is very difficult to adjust just one parameter.

7 VOIDS AND THE VISCOSITY CYCLE

Conventionally voids are considered to be a consequence of either gaseous emanation of solvents retained in the prepreg or entrapped air. Although volatile voids are usually held to quite low values by the prepreg manufacturers, large amounts of air are inevitably trapped even in the most careful handling and lay-up procedures. However, adequate void removal can be obtained by the use of a vacuum stripping stage early in the cure cycle (section 6). There is nevertheless a third source of voids which is more subtle and is a consequence of a mismatch between the interfibre spaces and the final resin content.

It has been shown earlier (section 5) that for each particular prepreg type and batch the fibres have some inherent packing density which is maintained almost irrespective of external conditions (see Fig 13). Fig 17 shows the variation in void content V_v with compacting pressure for the same laminates as used for Fig 13. The misaligned fibres determining the fibre volume fraction also create spaces from which resin is lost and only considerable pressure can reduce these spaces. Voids due to resin shortage are most likely to arise in resin rich areas and, as described earlier, these normally occur at laminae boundaries as illustrated in Fig 18.

A good quality laminate clearly requires that the spaces between the fibres should be completely filled with resin. However, it has been shown (section 5) that during the autoclave cycle resin either exudes or is forced out of the laminate and unless this flow is controlled an excessive amount may be removed and voids arise due simply to insufficient resin. Resin flow is naturally related to resin viscosity and thus the time of gelation is important in determining laminate quality (see section 4). Now it has been established (section 4.3) that due to heat transfer considerations a significant increase in pressure can precipitate resin gelation: hence flow can be terminated at a specific time by the application of pressure at that time. Since as has been seen, pressure is normally applied at some stage in the autoclave cycles to assist in laminate compaction, resin flow can be controlled by simply choosing the optimum time of pressurization.

The manner in which this control is achieved can be illustrated in general terms by discussing the viscosity of the resin as a function of time. Consider a family of temperature-time curves such as already seen in Fig 11. By combing these data with the particular resin viscosity - time curve (see Fig 1) a family of reciprocal viscosity (fluidity) v time can be constructed. If it is assumed that resin flow is proportional to the product of fluidity and time, then by summing the area under a fluidity - time plot a curve can be drawn of the total resin flow as a function of pressurization time as shown in Fig 19 (A). In this figure the axis has been scaled non-dimensionally so that a value of unity corresponds to the ideal laminate and values greater than 1, those shaded, represent laminates deficient in resin (*ie* voidy laminates). It can be seen that for a good quality laminate the time during which pressure should be applied is quite small, in this case just 10 minutes. For a typical CFRP prepreg a resin loss by weight 5% in excess of the ideal will cause a void content of about 0.75% by volume and even for this relatively high void content the time bandwidth during which pressure should be applied is but 14 minutes. If, for this laminate, pressure is applied after only 20 minutes the consequent increase in heat-up rate and fall in viscosity will cause excess resin to be lost before even the advanced gel-time is reached; if pressure is applied after 40 minutes, excess resin will have already been exuded by natural flow before pressure is applied.

Because of the assumptions made in the derivation of Fig 19, two experiments were carried out to confirm the deductions. By the use of non-reactive additives, one resin system was modified to give minimum viscosities during a given cure cycle of between 0.6 and 60 (simulator output). One batch of fibres was employed for all the modified resin prepregs and laminates autoclaved using identical cure cycles. The void content of each laminate was determined and the results shown in Table 1. It can be seen that with the very low viscosity resins the laminate quality is poor due to excess resin loss before application of pressure; area M Fig 19. The second experiment involved removing identical laminates from the autoclave at various times before pressurization and measuring the weight loss. Fig 20 shows the weight of resin loss, expressed as a percentage of the original laminate weight, plotted as a function of time. Since the optimum resin loss for this material and cure cycle was known to be 11%, it can be seen that gelation should be precipitated after about 30 minutes to avoid excess resin loss. If, for instance, gelation was delayed until 60 minutes the excess resin loss would lead to a void content greater than 2%, area N Fig 19.

Alternatively the effect of decreasing the overall resin viscosity can be illustrated as curve B Fig 19 under which conditions it would not be possible to produce a void free laminate. Similarly, increasing the resin viscosity, C , Fig 19, reduces the likelihood of voids due to excess resin loss, but at very high viscosities some voids are likely to arise due to the difficulty in compacting relatively stiff laminae. This, of course, accounts for the tolerance and popularity of some relatively high viscosity resin systems. It may be noted here that the capillary action of the absorber (section 3) may act in the same way as reduced viscosity in removing more resin than required. Hence for some systems it may be desirable to use a barrier of limited porosity - the restricted bleed system - between the absorber and the laminate to reduce the rate of resin flow, thereby in effect increasing the resin viscosity.

8 CONCLUDING REMARKS

A typical autoclave cycle consists of an initial heat-up under vacuum and application of pressure at a predetermined time and as such would appear a simple procedure. However, numerous experiments and measurements taken during routine processing have shown that ancillary materials and apparently trivial modification of cure cycle parameters frequently exert an influence on the quality of the final laminate out of all proportion to their expected significance. Indeed, frequently the effect is vastly different from the one intended.

Whilst the temperature cycle may be generally accepted as complex, the pressure cycle is often regarded only as an aid to compaction. It has been shown that both cycles interact in such a complicated manner that it is virtually impossible to consider either in isolation. For example, a change in the magnitude of the pressure applied can have a totally unexpected effect due entirely to its alteration of the temperature cycle and thereby the time of gelation. Such a minor change could easily ruin a perfectly valid cure cycle and could not be explained satisfactorily in terms of pressure effects only.

It has been argued that the packing of the fibres is determined by processes other than, and remote from, the autoclave. Thus the final thickness and fibre volume fraction are not under the autoclave operator's control. The cure cycle must be tuned to give, not a specific thickness for it cannot, but that thickness which is inherent in the prepreg laminae used. The common factor behind all these phenomena has been shown to be the relationship of viscosity to gel time in that resin can flow only until gelation effectively stops all flow. This combination, either partner of which is affected in different ways by each change of cycle parameter, must be correct for each laminate and each resin system and the operating tolerance is frequently only small. Indeed, significant cure cycle variations are only possible with high viscosity resins and this fact clearly separates tolerant from intolerant systems. Epoxy resin manufacturers are endeavouring to produce more tolerant matrix systems with simpler cure characteristics; for example, with pressure applied from cold and a simple heating cycle. However, existing systems and many derivations will require consideration of the arguments presented here for some time to come in order to produce acceptable autoclave mouldings.

In view of the complex interaction of the many variables it is not surprising that empirically derived cure cycles are not well understood and that the diagnosis and rectification of defects relies almost entirely on the experience of the autoclave operator. It is hoped that this Report will inject a modicum of understanding in to what has for many years remained a black art.

Acknowledgment

The authors wish to acknowledge the contribution of Mr F. Cox whose willing and conscientous assistance made this Report possible.

Table 1

VARIATION OF VOID CONTENT WITH RESIN VISCOSITY

Sample	Minimum viscosity (simulator output)	Void content volume %
AR 3	0.6	>2
AR 6	1.5	<1.5
AR 5	5	<0.5
AR 7	40	<0.5
AR 5	60	<0.5

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No.	Author	Title, etc
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3	D.E.W. Stone B. Clarke	Non-destructive determination of the void content in carbon fibre reinforced plastics by measurement of ultrasonic attenuation. RAE Technical Report 74162 (1974)

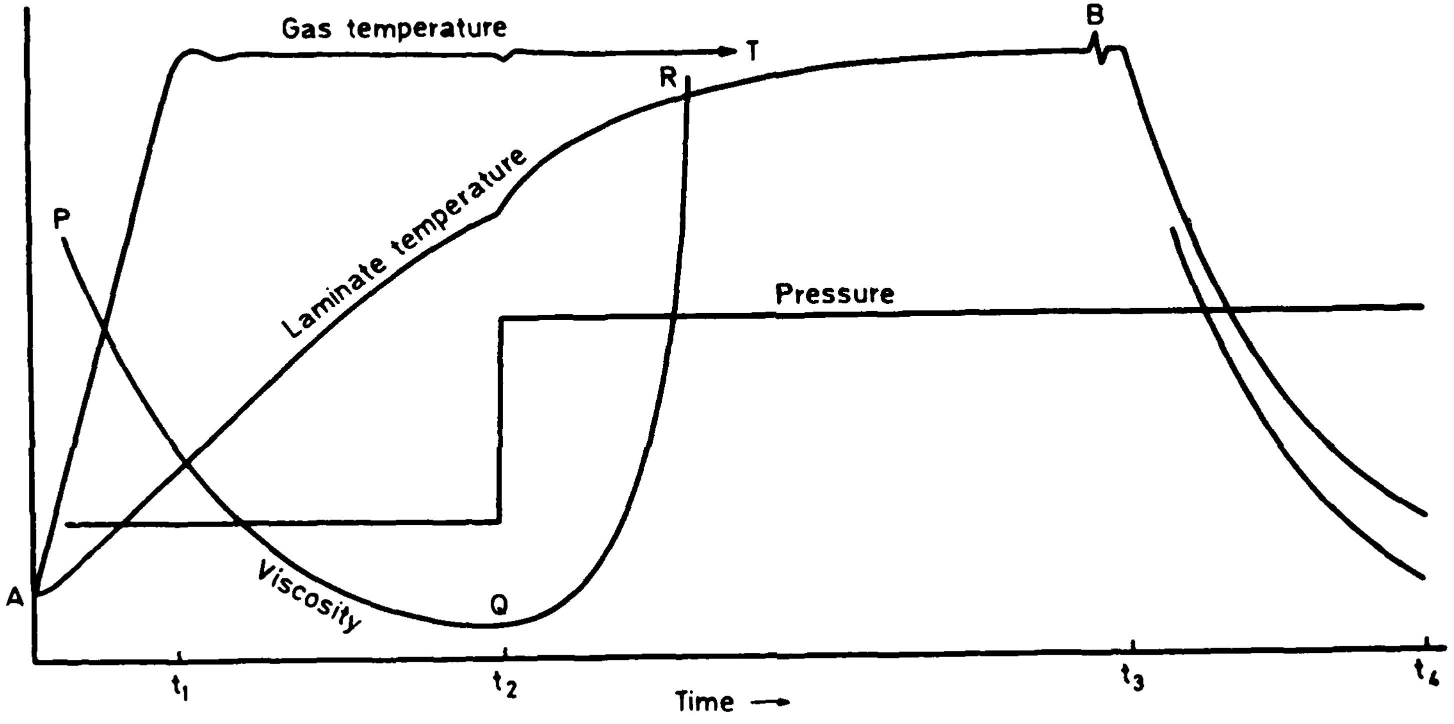

Fig 1 Typical autoclave cure cycle for a fibre reinforced epoxy laminate

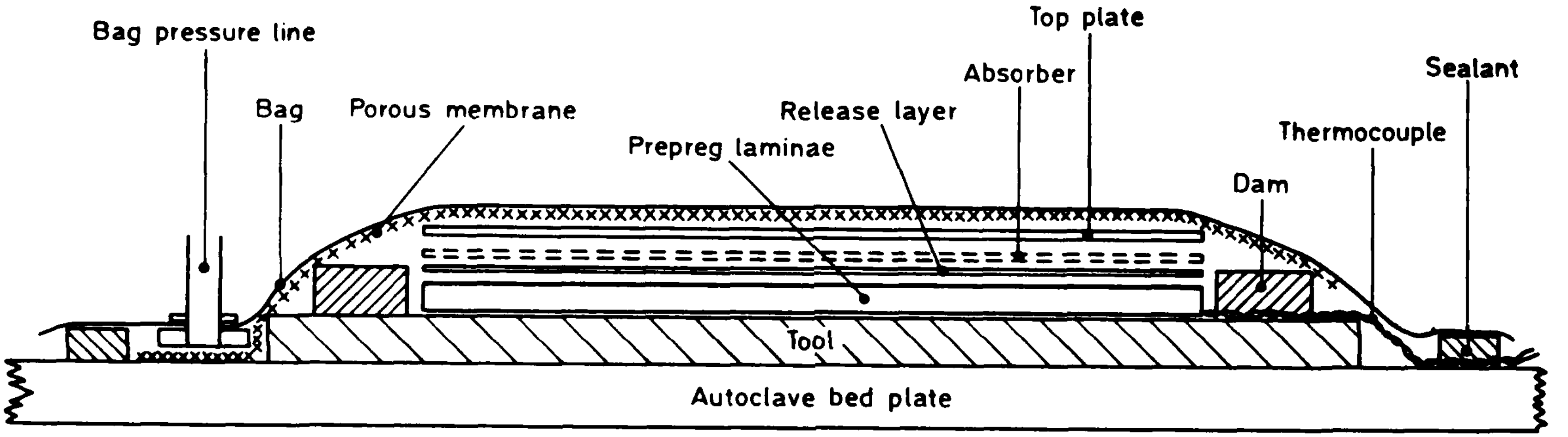

Fig 2 Autoclave moulding assembly

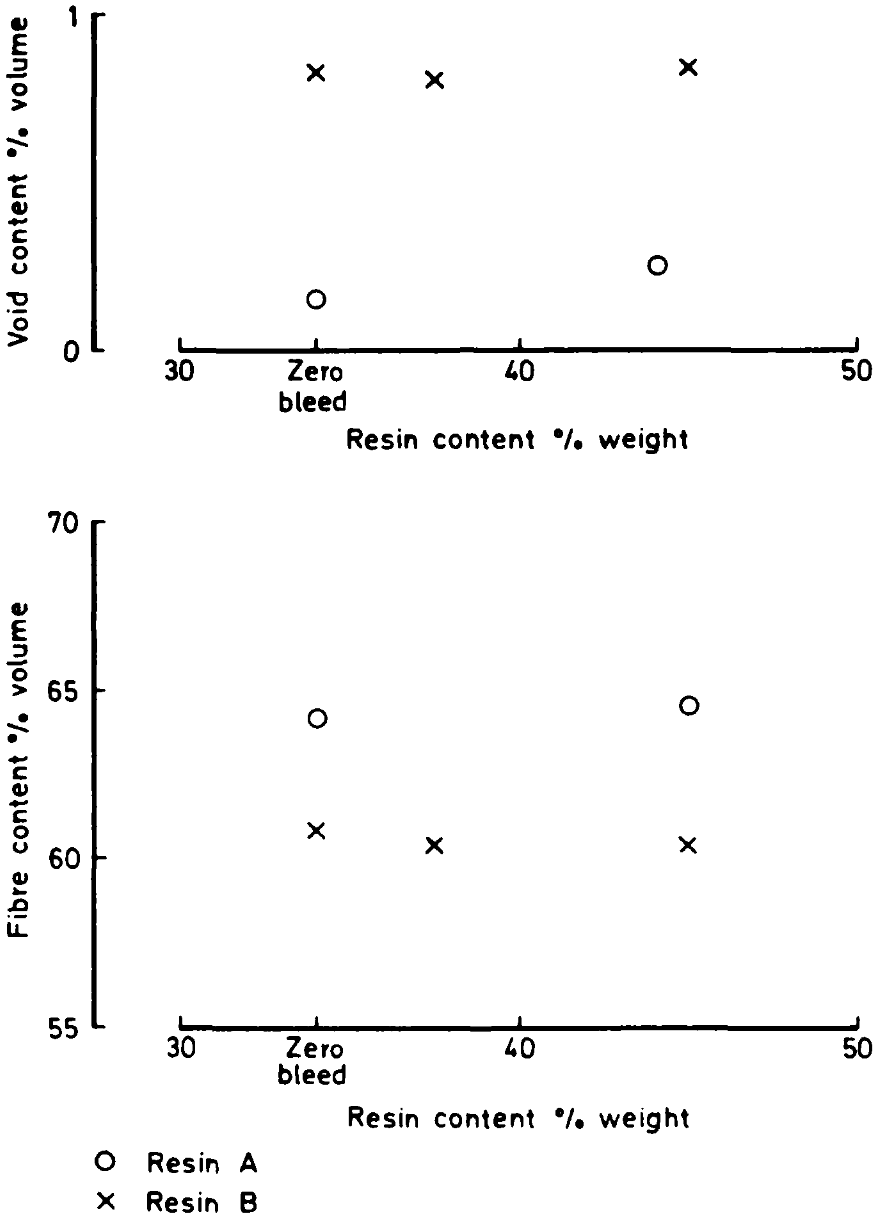

Fig 3 Effect of pre-preg resin content on composite quality

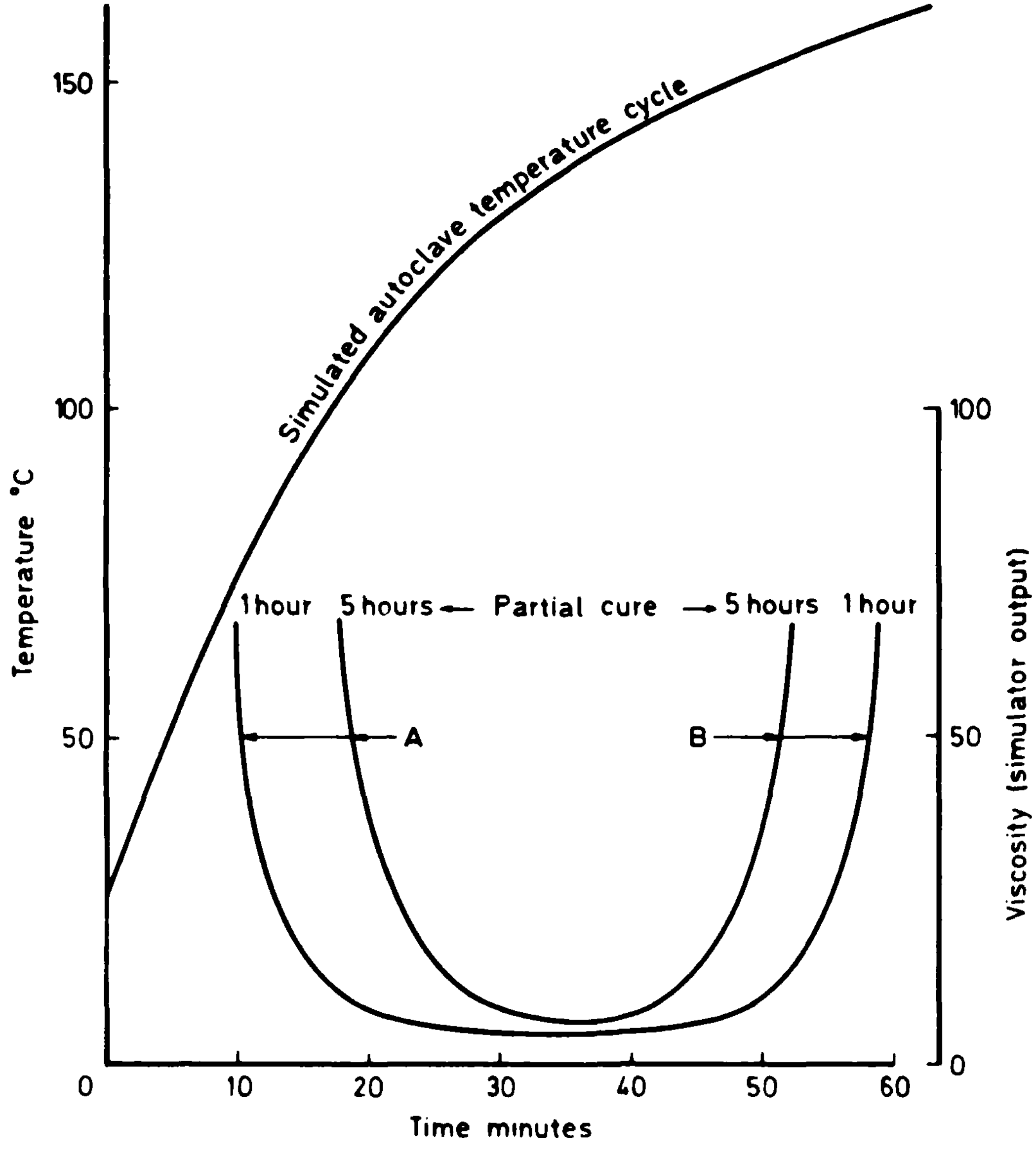

Fig 4 The effect of length of partial cure on the time to viscosity A and gelation B for an epoxy resin system

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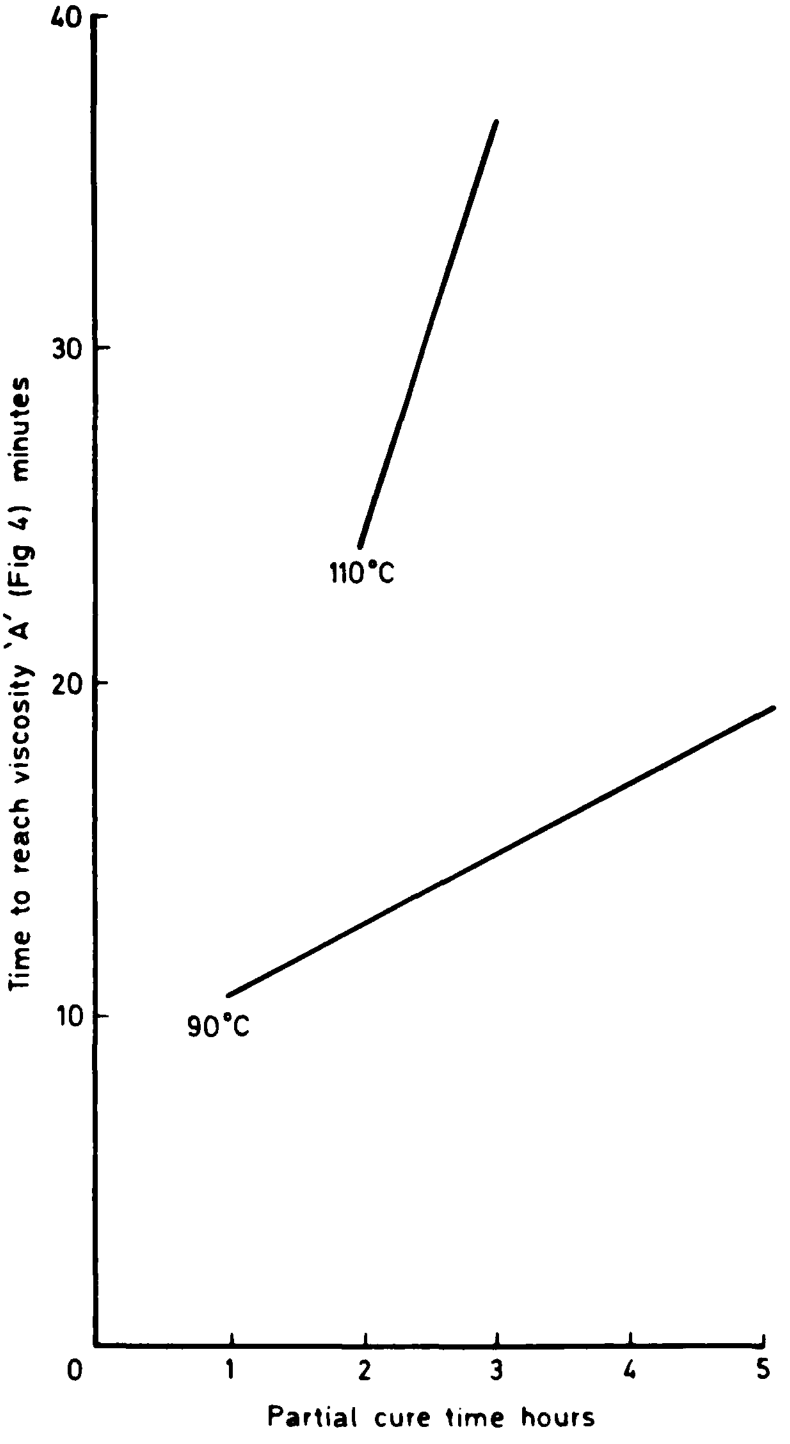


Fig 5 Effect of partial cure time on the viscosity of an epoxy resin

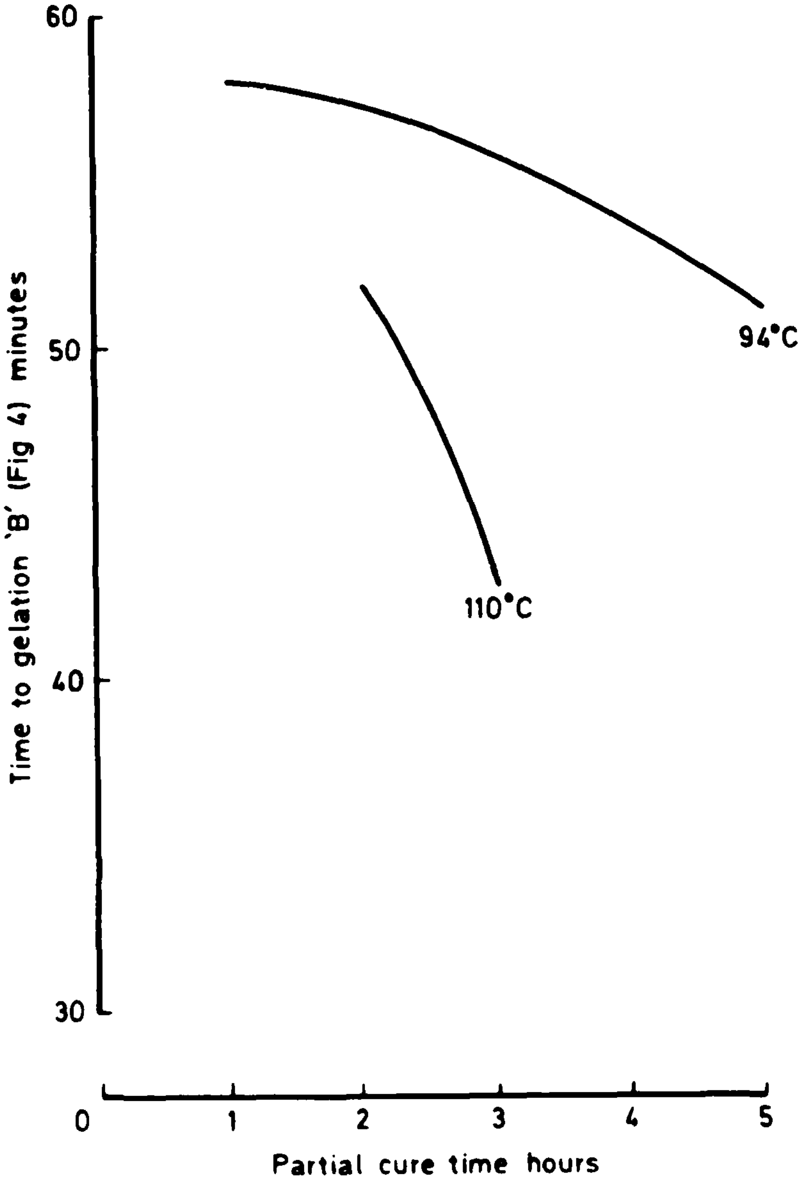


Fig 6 Effect of length of partial cure on the time to gelation for an epoxy resin system

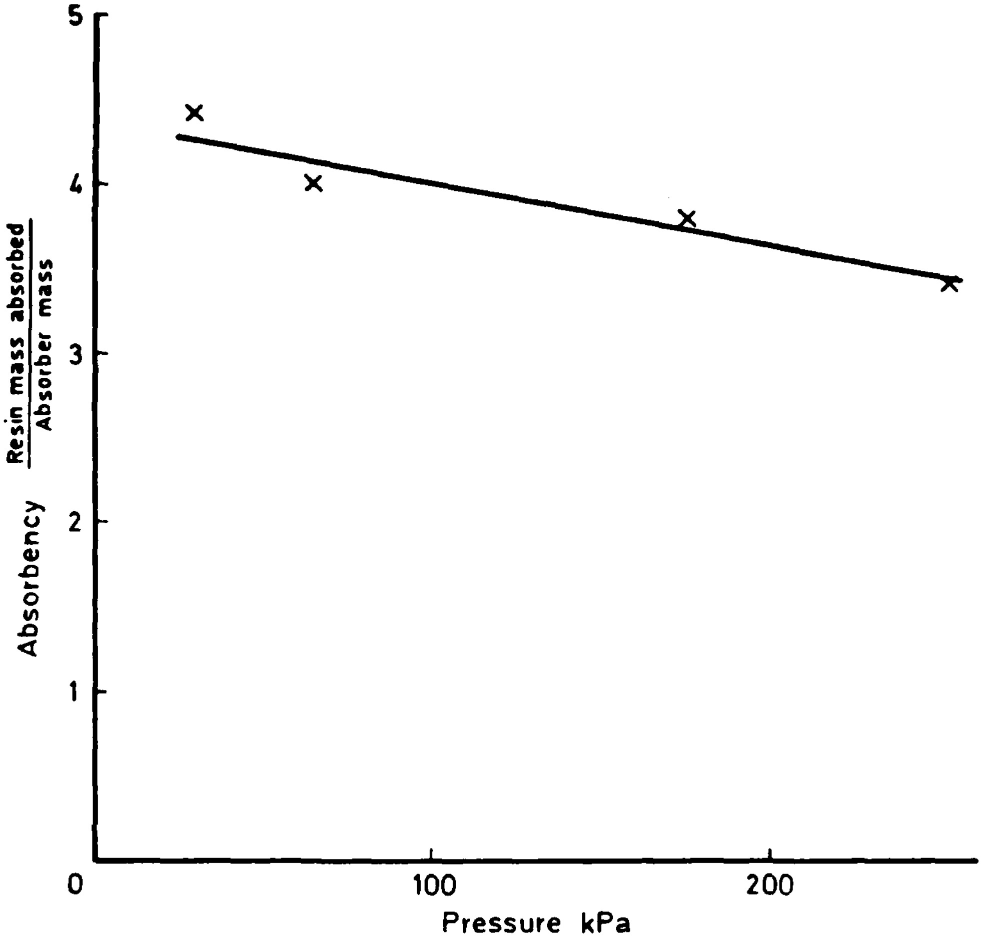


Fig 7 Variation of resin absorption with autoclave pressure

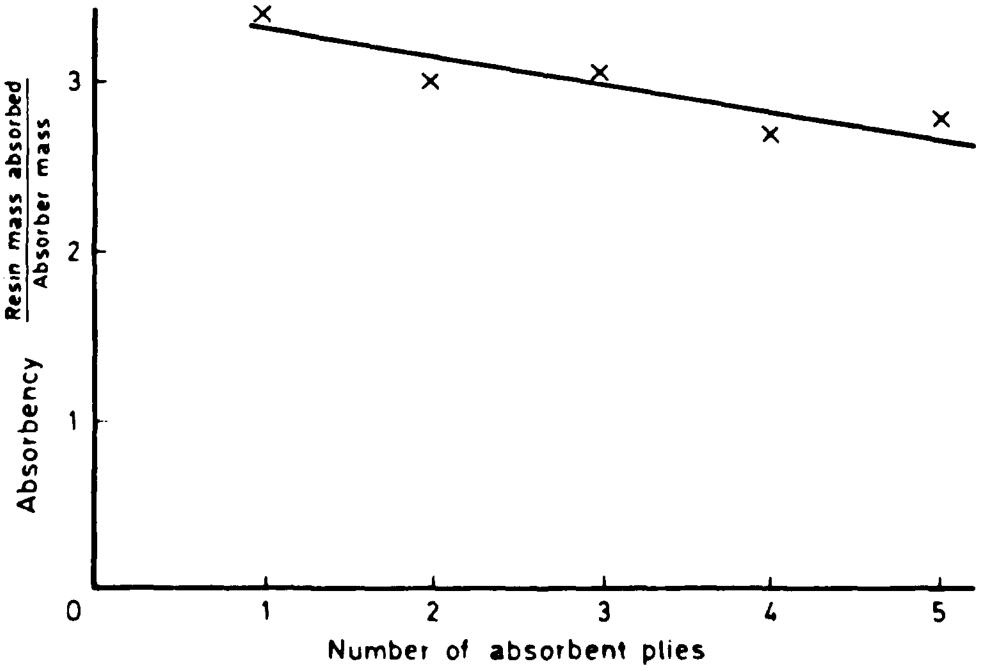


Fig 8 Variation of resin absorption with quantity of absorber

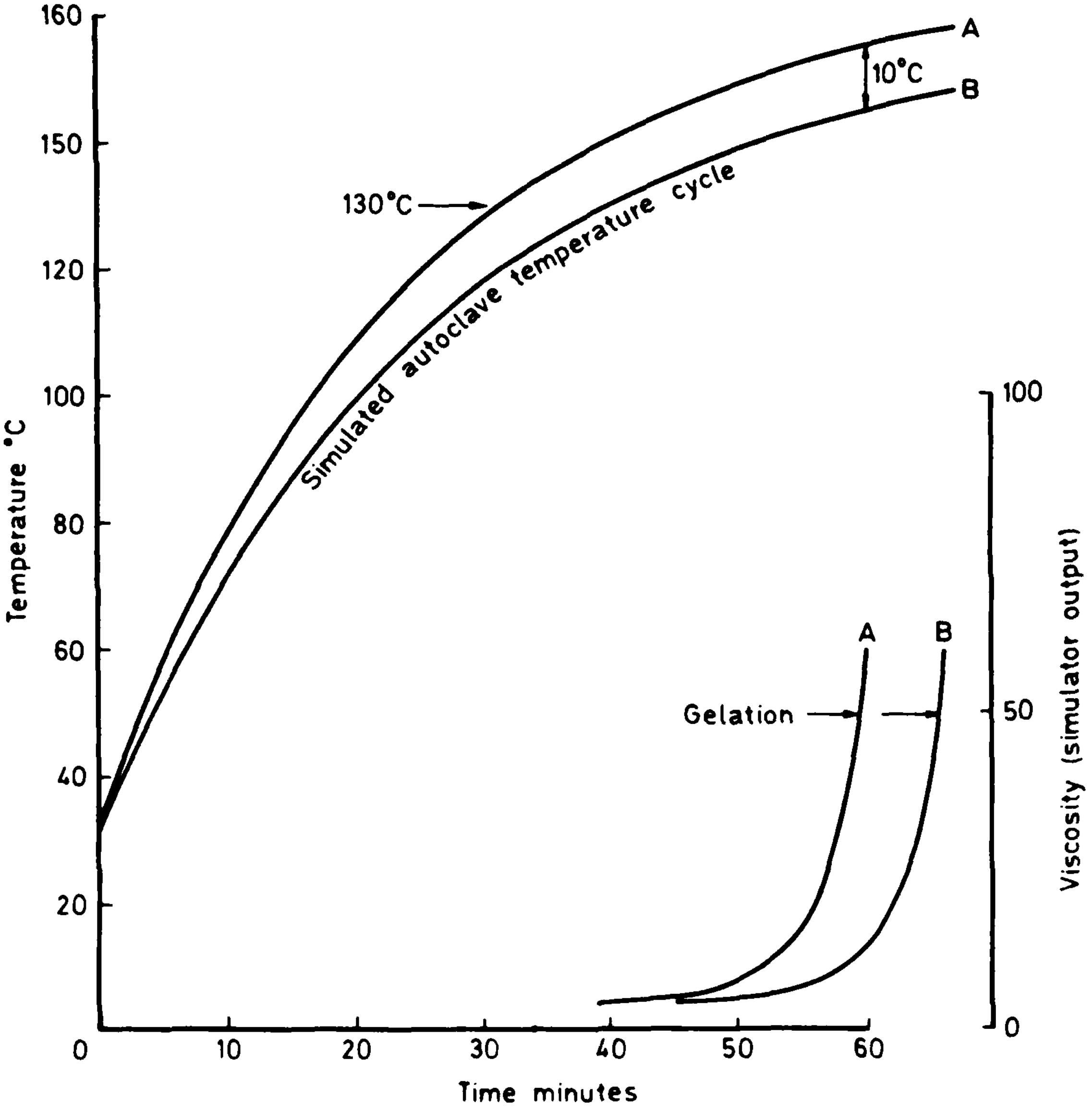


Fig 9 Effect of temperature cycle on gel time of epoxy resin

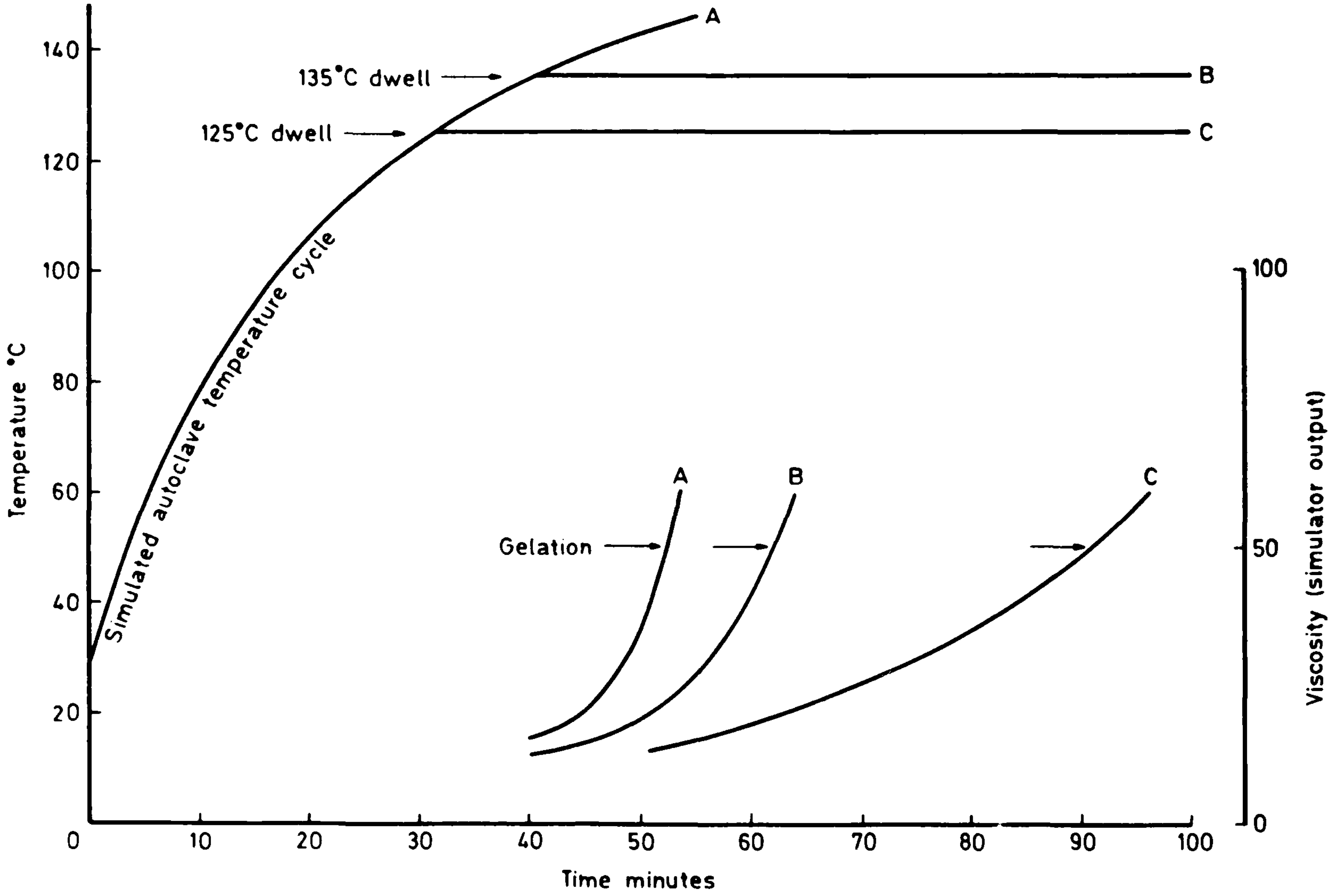


Fig 10 Effect of dwells on gel time of epoxy resin

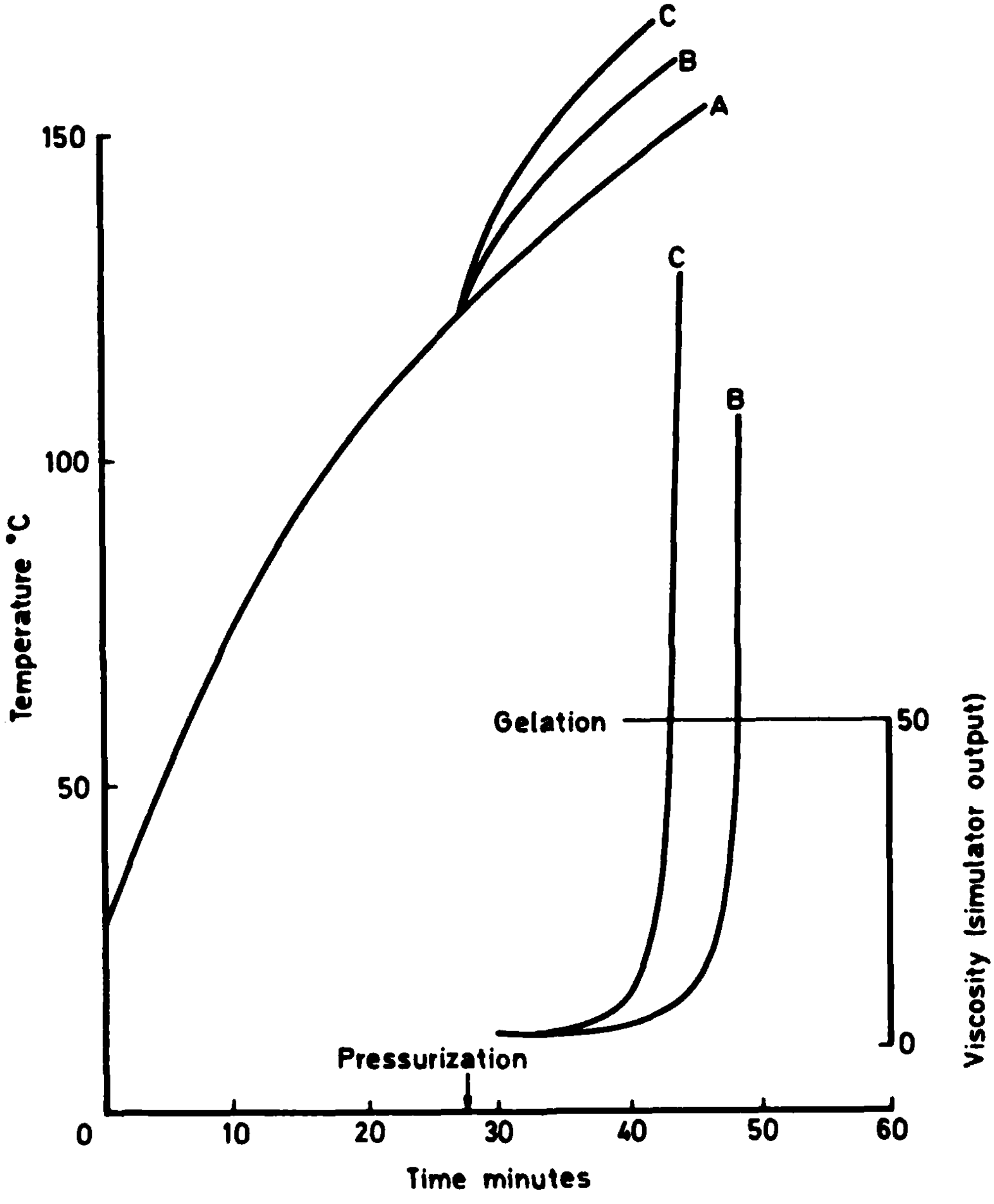


Fig 11 Effect of pressure on gel time of epoxy resin

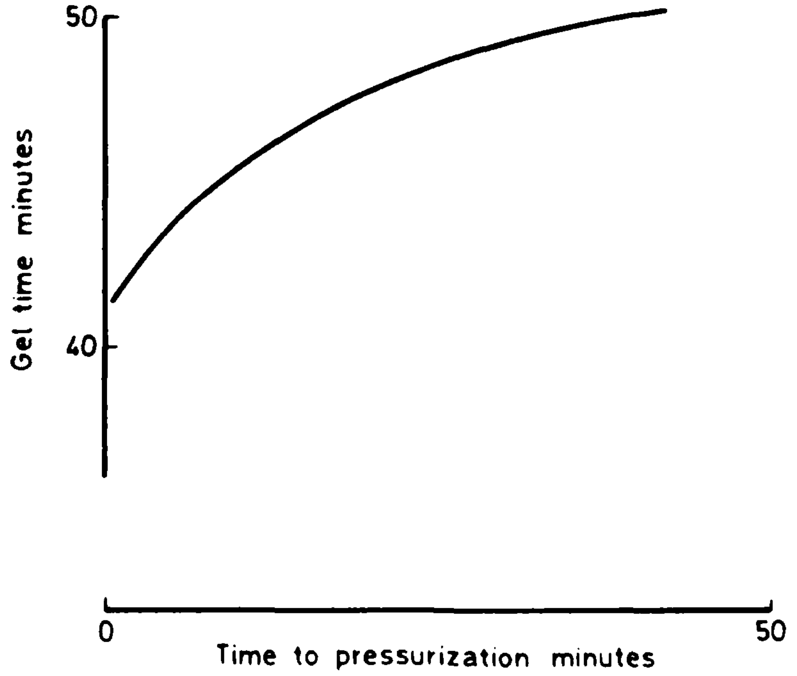


Fig 12 Variation of gel time with time to pressurisation

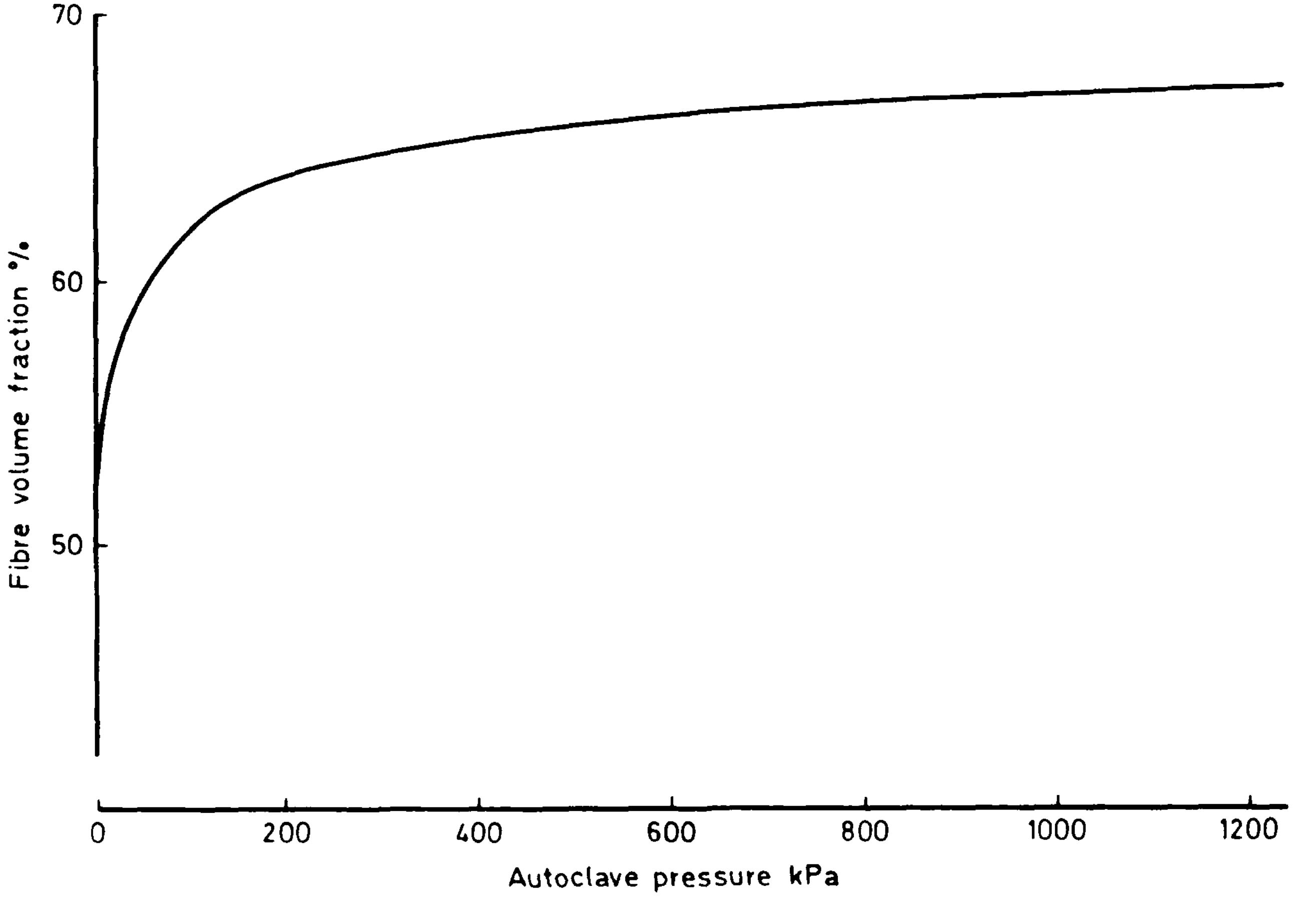
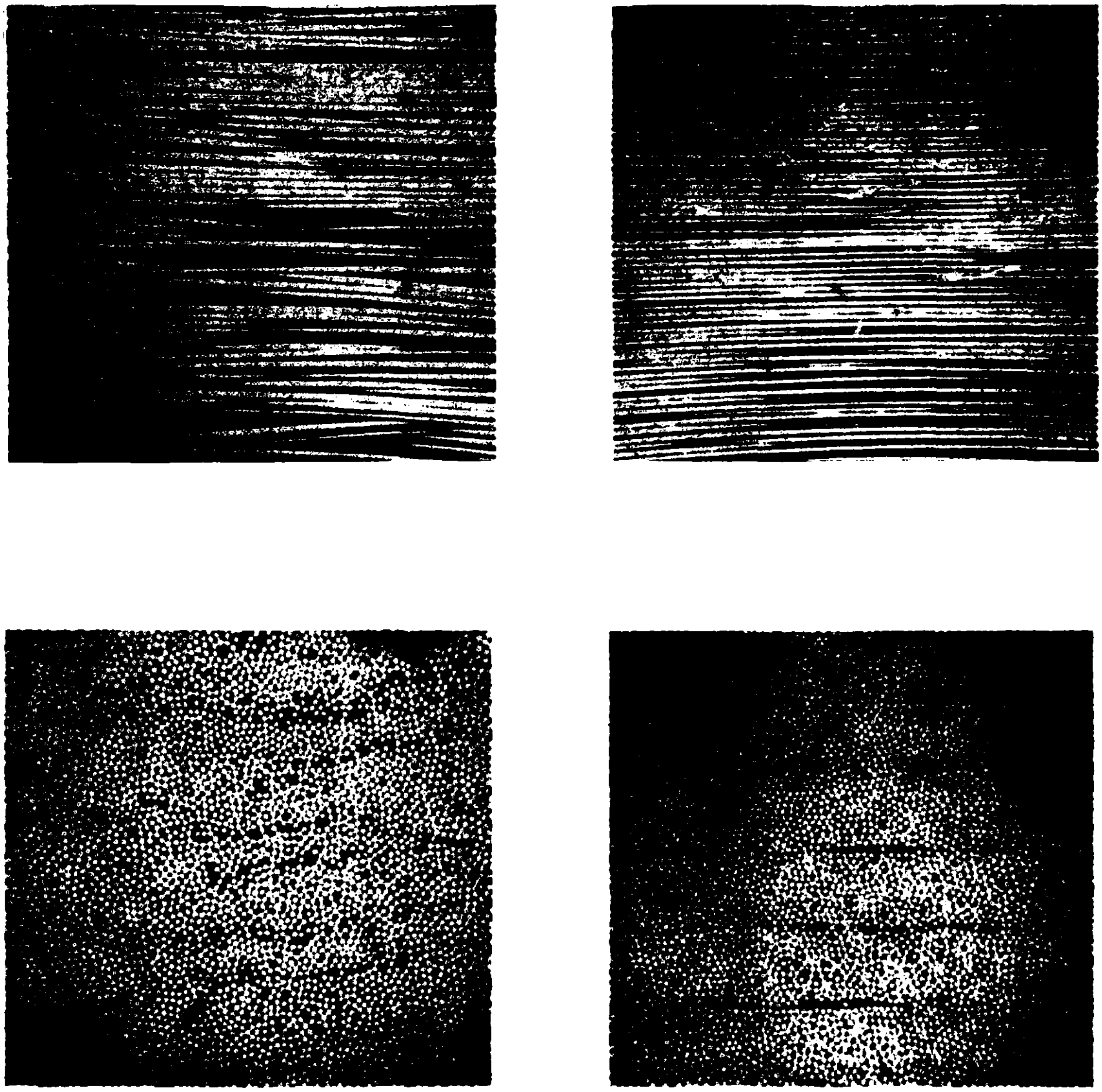


Fig 13 Variation of fibre volume fraction with laminating pressure for unidirectional CFRP



(a) (b)

Fig 14a&b Fibre distribution in CFRP laminates with fibre volume fractions
(a) 62% (b) 75%

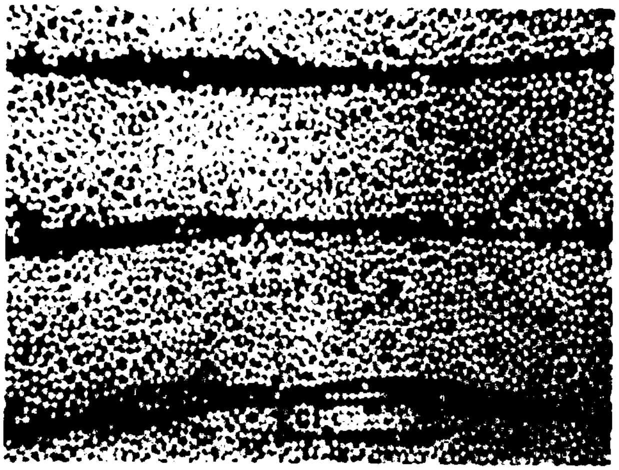


Fig 15 Resin rich areas in CFRP laminate due to low compacting pressure

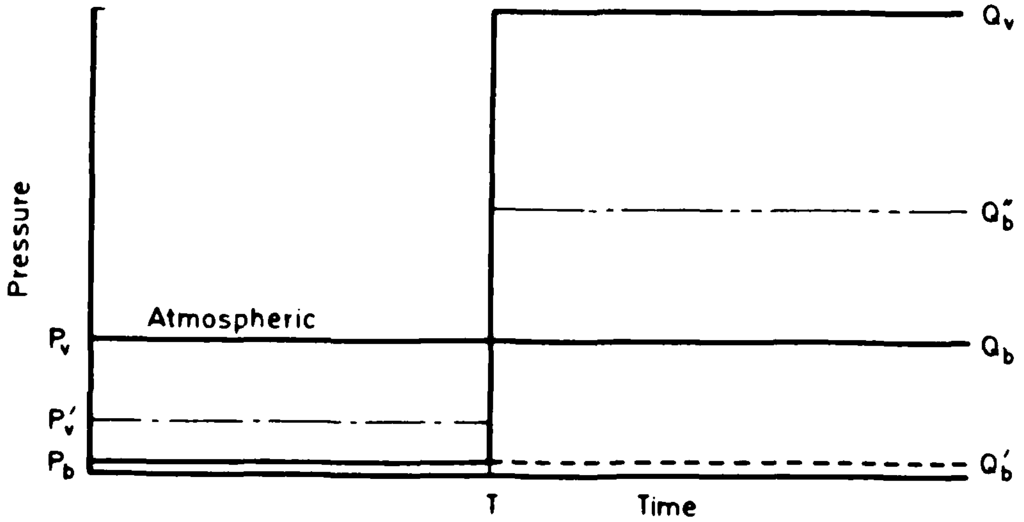


Fig 16 Pressure cycle variants

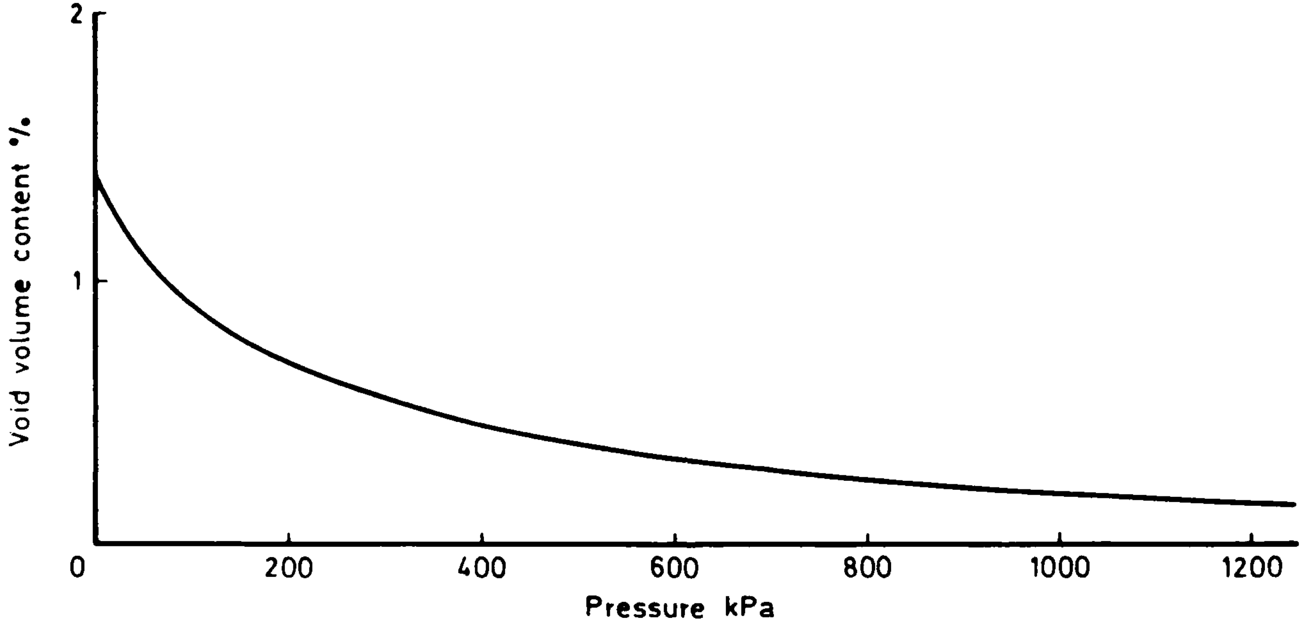


Fig 17 Variation of void content with laminating pressure for unidirectional CFRP

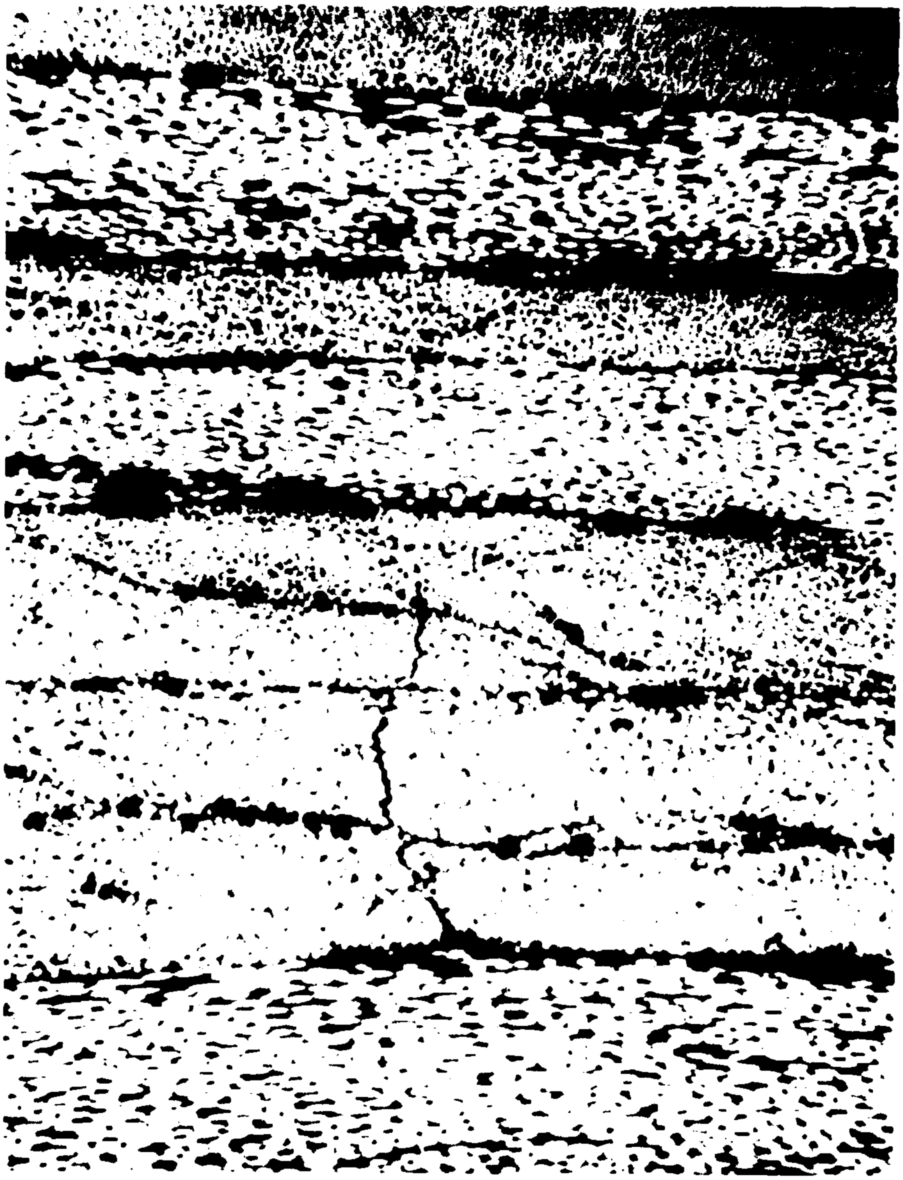


Fig 18 Void occurrence at laminae boundaries in a CFRP laminate

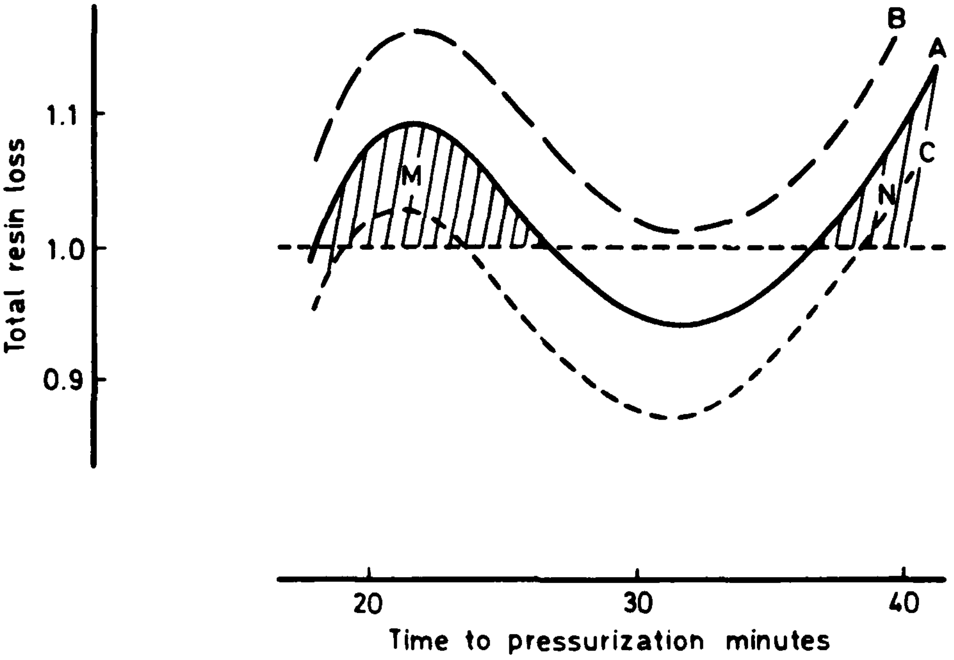


Fig 19 Variation of resin loss with time to pressurisation

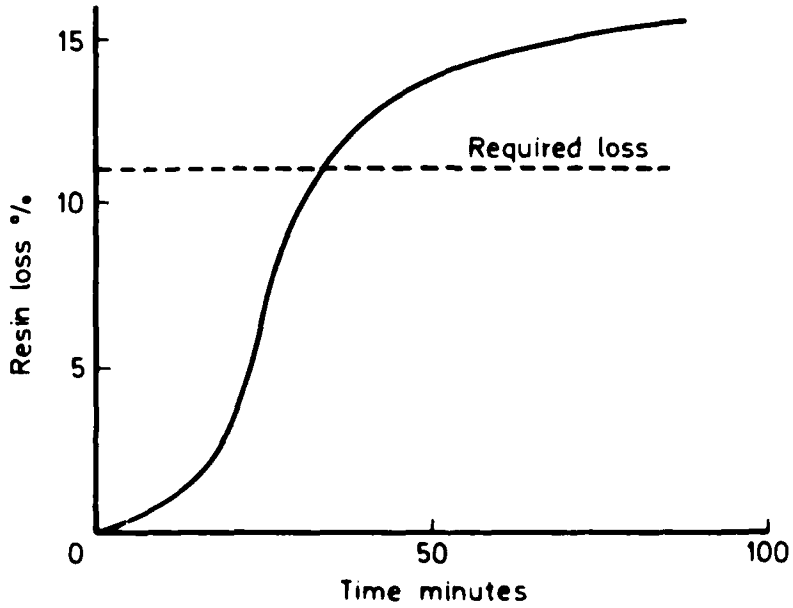


Fig 20 Variation of resin loss with time before pressurisation

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