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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART I: THERMAL AND MOISTURE DIFFUSION IN COMPOSITE MATERIALS

by

J. P. Komorowski

National Aeronautical Establishment

OTTAWA
JANUARY 1983

AERONAUTICAL NOTE
NAE-AN-4
NRC NO. 20974

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EFFETS HYGROTHERMIQUES DANS LES COMPOSITES À RENFORT DE FIBRE CONTINU

PARTIE I — DIFFUSION DE L'HUMIDITÉ ET DE LA CHALEUR DANS LES MATÉRIAUX COMPOSITES

by/par

J.P. Komorowski

National Aeronautical Establishment

OTTAWA
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AERONAUTICAL NOTE
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Director/Directeur

SUMMARY

This report is the first in a series of literature reviews in which hygrothermal effects on aerospace composite materials (CM) are examined. This first report (Part I) deals primarily with fundamental aspects of the diffusion of moisture into, and from, composite materials. The effects of temperature under both steady state and transient conditions are also examined.

Subsequent reports in this series will deal with the following topics:

Part II:	Physical Properties
Part III:	Mechanical Properties 1
Part IV:	Mechanical Properties 2
Part V:	Composite Structures and Joints
Part VI:	Numerical and Analytical Solutions
Part VII:	Summary of Conclusions and Recommendations

A bibliography has also been prepared to serve as a source of further information. It will also serve as a reference list for the various reports in this series, and therefore it is included as an appendix.

RÉSUMÉ

Le présent rapport est le premier d'une série d'études documentaires traitant des effets hygrothermiques sur le matériaux composites de l'industrie aérospatiale. Le premier rapport (Partie I) porte principalement sur les aspects fondamentaux de la diffusion de l'humidité entrant et sortant des matériaux composites. Les effets de la température à l'état stable et dans des conditions de transition sont également étudiés.

Les rapports subséquents de cette série traiteront des sujets suivants:

Partie II:	Propriétés physiques
Partie III:	Propriétés mécaniques 1
Partie IV:	Propriétés mécaniques 2
Partie V:	Structures et joints composites
Partie VI:	Solutions numériques et analytiques
Partie VII:	Résumé des conclusions et recommandations

Une bibliographie a de plus été préparée pour servir de source de renseignements supplémentaires. Elle servira également de liste de références pour les différents rapports de cette série, elle a donc été placée en annexe.

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I hope that you will find the reports informative and useful.

Yours faithfully,

L'Établissement aéronautique national (ÉAN) est heureux d'annoncer la publication d'une nouvelle série intitulée "Cahiers de l'aéronautique". Ces derniers complèteront les rapports d'aeronautique que publie déjà l'ÉAN. Quoique de champ plus restreint que les rapports, les notes fourniront des données techniques d'aéronautique fondamentale et appliquée propres à une grande diffusion et susceptibles de contribuer à l'avancement des connaissances.

Dans l'espoir que vous trouverez les notes intéressantes, je vous prie d'agréer, l'expression de mes sentiments les meilleurs.

G.M. Lindberg

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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART I: THERMAL AND MOISTURE DIFFUSION IN COMPOSITE MATERIALS

1.0 INTRODUCTION

As metals have seemed to reach their engineering limits in structural applications the aerospace community, in its search for higher performance, has turned to composite materials (CM). With rapid maturing of the technology, applications for composite materials have increased to include both secondary to primary structures. Materials of interest in structural applications are continuous fiber or fabric reinforced resins. Most of the materials used at present have been available for only a short period of time and new materials are constantly being developed. The aircraft structures designed currently are expected to maintain their integrity for up to 20 years of service under harsh environmental conditions. Appropriate coatings may provide protection against ultra violet radiation and rain erosion but varying loads, temperatures and absorbed moisture will continue to degrade the material properties.

Although a considerable amount of composite secondary structure has been designed in Canada, the inevitable move towards composite primary structure is being hampered by the lack of information on the environmental stability of these materials. The testing techniques needed to develop design allowables are not well established, nor are the methods used for accelerated testing to demonstrate structural integrity and stability over the design life of the aircraft.

To meet this challenge the Structures and Materials Laboratory of NAE set out to review the literature on the subject and assess current state of knowledge in this field. The review should also indicate areas where more research is still needed.

The author is not aware of any similar review having been prepared in recent years, although Schutz and Gerhart in 1979 published their "literature research on the mechanical properties" [109]* which included some of the papers reviewed here. More recently (1981) Delmonte published his book on carbon and graphite composites [84]. Both these references contain information on the state of the art. In this present work, environmental effects have been examined in greater detail and special emphasis has been placed on the latest publications.

All matrix resins presently used in advanced composite materials absorb moisture directly from the atmosphere. Since early 1970, significant changes in mechanical properties and dimensional stability have been observed in composites and have been correlated with moisture and the thermal environment. A considerable amount of research has been directed at understanding the mechanics of moisture and heat diffusion into composite materials.

Below a fairly detailed review of the problem is presented. The author believes that it is not possible to conduct environmental testing without a deep knowledge of the underlying processes. This fact has somehow been overlooked by some researchers. The aim of environmental testing is to develop means of predicting the behaviour of composite structures and materials under changing conditions. In laboratory investigations, moisture contents can be achieved far exceeding the amounts which can be expected in actual service. If estimates of degradation are based on these moisture gains, then overdesign would be unavoidable. However, in order to speed up processes higher humidities and temperatures are often used, and the researcher has to be aware of the consequences of such procedures. Finally it is not practical to wait 20 years to measure actual degradation and although such information is being collected and will be invaluable, degradation has to be estimated from accelerated tests. Again, the design of such tests requires a complete understanding of the underlying physical and chemical processes.

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* Numbers in the square brackets refer to the reference number on the list given in the Appendix.

2.0 ENVIRONMENT ENCOUNTERED BY COMPOSITE MATERIALS

Composite materials technology has advanced so far that it would be difficult to identify all its numerous applications. In this review aeronautical and space applications were of main concern.

During a subsonic flight mission of an aircraft, temperatures in the range of $-55°C$ to $60°C$ may be encountered. Supersonic flight is more demanding and skin temperatures as high as $150°C$ and rates of change of $15°C$/min. may be reached due to aerodynamic heating. On reduction of speed, the outer surface temperatures may drop at a rate up to $500°C$/min. [220]. This high temperature peak is often called a thermal spike [198], [201], [220], [267], (Fig. 1).

Relative humidities from a few percent to 100% are expected; additionally exterior surfaces are exposed to water from precipitation and condensation. Several authors have calculated moisture contents and profiles in composite material laminates after several years of service [274], [289], [293], [319]. It was shown that storage conditions are deterministic to the moisture level attained. Surface properties of the composite such as absorbance and emissivity have to be known as solar radiation is an important factor in overall moisture content. These calculations correlate well with results of long term service experience with composites in commercial aircraft [57], [88]. These indicate that complete weather data is required in predicting the long term behaviour of composite materials.

Some composite materials may be applied in cold parts of airplane engines, i.e. compressors-blades, discs, and nozzle flaps. A temperature range from $-40°$ to $350°C$ is typical for these applications.

Space environment poses yet another challenge to composite materials. The most important element is high energy radiation [192]. For many applications however, the effect of ultra hard vacuum resulting in loss of adsorbed and absorbed gases (mainly H_2O) and sublimation or evaporation of the more volatile constituents of matrix materials may be the primary factor [286]. Composite materials encounter extremes of temperature depending on whether they are in the sun or in the shade. Drying and temperature changes may cause loss of dimensional stability, particularly important in applications such as optical instruments and antennas [130].

Glass/epoxy composites are used for cryogenic service ($\sim 4°K$), where low thermal conductivity is exploited in conjunction with mechanical strength (thermal isolation structures for spacecraft hardware)[223].

Composite materials are often subjected to fatigue loads and should exhibit impact strength and shock resistance.

In the following chapters, some results of the investigations aimed at characterizing performance of composite materials in adverse environments are reviewed.

3.0 THE MECHANICS

3.1 Fourier and Fick Models

Moisture absorption can take place by the following mechanisms [262]:

1) through the fiber-matrix interface,
2) through cracks and voids in the composite,
3) through the resin.

The above is true for inorganic fiber composites such as boron, graphite or glass where the fibers do not seem to absorb moisture. However, for Kevlar CM it was found [9] that the fibres absorb moisture and reach equilibrium moisture contents of the same order as typical epoxy resins (5208 Narmco in this case). In-plane diffusion in Kevlar 49 fabric/epoxy laminate was measured to be two orders of magnitude ($6.1 \times 10^{-8} cm^2/s$ vs $1.7 \times 10^{-10} cm^2/s$) faster than through the thickness. Diffusivity in Narmco 5208 resin was $6.5 \times 10^{-10} cm^2/s$. Therefore for organic fiber composites, diffusion in the filament may be considered as the fourth mechanism of moisture absorption.

Several authors have applied Fourier theory of heat transfer to composite materials. Fourier's equation of heat transfer in tensor notation presented below was taken from [45].

$$\rho c \frac{\partial T}{\partial t} = \frac{\partial}{\partial x_i}\left[K_{ij} \frac{\partial T}{\partial x_j}\right] = (K_{ij}\, T_{,j})_{,i} \tag{1a}$$

where
- ρ — is material density
- c — is specific heat
- T — is temperature
- t — is time
- x_i — are material co-ordinates (i = 1,2,3)
- K_{ij} — is thermal conductivity tensor

If K_{ij} is not a function of temperature and position, (1a) simplifies to:

$$\rho c \frac{\partial T}{\partial t} = K_{ij} \frac{\partial^2 T}{\partial x_i\, \partial x_j} \tag{1b}$$

The rate of heat transfer per unit area per unit time is the heat flux vector:

$$-q_i = K_{ij} \frac{\partial T}{\partial x_j} \tag{2}$$

Equations (1) and (2) above are called second and first Fourier laws.

For most general material forms (triclinic) the conductivity tensor has the form:

$$\begin{bmatrix} K_{11} & K_{12} & K_{13} \\ K_{21} & K_{22} & K_{23} \\ K_{31} & K_{32} & K_{33} \end{bmatrix} \tag{3a}$$

However, materials of interest to this study are orthotropic, with conductivity tensor:

$$\begin{bmatrix} K_{11} & 0 & 0 \\ 0 & K_{22} & 0 \\ 0 & 0 & K_{33} \end{bmatrix} \tag{3b}$$

where K_{11}, K_{22}, K_{33} are conductivities in three perpendicular directions.

For unidirectional laminate having transversly isotropic properties (square or hexagonal fiber packing) and x_1 parallel to fiber direction:

$$K_{22} = K_{33}$$

and

$$\begin{bmatrix} K_{11} & 0 & 0 \\ 0 & K_{22} & 0 \\ 0 & 0 & K_{22} \end{bmatrix} \tag{3c}$$

conductivity of a laminae with arbitrary fiber orientation (Fig. 2) can be calculated from Equation (4) (tensor notation)

$$K_{\alpha\beta} = a_{\lambda\alpha}\, a_{\gamma\beta}\, K^{1}_{\lambda\gamma} \tag{4}$$

where

$a_{\lambda\alpha}, a_{\gamma\beta}$ — are direction cosines

$K_{\alpha\beta}$ — conductivities according to specimen geometry

$K^{1}_{\lambda\gamma}$ — conductivities in principal material co-ordinates, x'_1 parallel to fiber axis.

usually

$$\gamma = 90^\circ \quad (\beta = 90^\circ - \alpha)$$

and

$$\left.\begin{aligned} K_{11} &= K'_{11}\cos^2\alpha + K'_{22}\sin^2\alpha \\ K_{12} &= (K'_{22} - K'_{11})\cos\alpha\sin\alpha \\ K_{22} &= K'_{11}\sin^2\alpha + K'_{22}\cos^2\alpha \\ K_{33} &= K'_{22} = K'_{33} \end{aligned}\right\} \tag{5}$$

K'_{11} — conductivity in CM along the fiber

K'_{22} — conductivity in CM transverse to the fiber

Springer and Tsai [272] approximated K'_{11} and K'_{22} by following equations:

$$K'_{11} = (1 - V_f)K_r + V_f K_f \tag{6}$$

$$K'_{22} = \left[1 - 2\sqrt{\frac{V_f}{\pi}}\right]K_r + \frac{K_r}{\beta_k}\left[\pi - \frac{4}{\sqrt{1 - \left(\beta_k^2 \dfrac{V_f}{\pi}\right)}}\tan^{-1}\frac{\sqrt{1 - \beta_k^2 \dfrac{V_f}{\pi}}}{1 + \beta_k\sqrt{\dfrac{V_f}{\pi}}}\right] \tag{7}$$

V_f — volume fraction of fiber

K_r — thermal conductivity — resin

K_f — thermal conductivity — fiber

$$\beta_k = 2\left(\frac{K_r}{K_f} - 1\right)$$

Transfer of heat by conduction is due to random molecular motion while diffusion is a process by which matter is transferred as a result of random molecular motion. This analogy was recognized by Fick who adopted the mathematical formulae of Fourier to diffusion. Fick's diffusion model has been applied frequently to CM, [22], [262], [272], [312], [313].

Fick's second law in tensor notation:

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial x_i}\left[D_{ij}\,\frac{\partial c}{\partial x_j}\right] = (D_{ij}\;c_{,j})_{,i} \tag{8a}$$

c — concentration of moisture

D_{ij} — diffusivity tensor

It has been observed (i.e. [22]) that diffusivity varies little with moisture content (hence x_i) for CM, therefore:

$$\frac{\partial c}{\partial t} = D_{ij}\,\frac{\partial^2 c}{\partial x_i\,\partial x_j} \tag{8b}$$

Fick's first law states that F, the rate of transfer (flux) of matter, by diffusion through a unit area of a section is proportional to the concentration gradient normal to the section:

$$-F_i = D_{ij}\,\frac{\partial c}{\partial x_j} \tag{9}$$

All arguments developed for the conductivity tensor apply to the diffusivity tensor so for laminae with fibers at an angle to the sample co-ordinates (Fig. 2)

$$\left.\begin{aligned} D_{11} &= D'_{11}\cos^2\alpha + D'_{22}\sin^2\alpha \\ D_{12} &= (D'_{22} - D'_{11})\cos\alpha\sin\alpha \\ D_{22} &= D'_{11}\sin^2\alpha + D'_{22}\cos^2\alpha \\ D_{33} &= D'_{22} = D'_{33} \end{aligned}\right\} \tag{10}$$

This analogy between heat transfer and diffusion was carried further by Springer and Tsai [272] who used Equations (6) and (7) for calculating D'_{11} and D'_{22} from diffusivities of fiber and resin (D_f and D_r) since for most fibers $D_f << D_r$.

$$D'_{11} = (1 - V_f)\,D_r \tag{11}$$

$$D'_{22} = \left(1 - 2\sqrt{V_f/\pi}\right)D_r \tag{12}$$

(D'_{22} for tetragonal packing)

Augl and Berger [22] calculated effective diffusion coefficients in the transverse direction (D'_{22}) by solving the Poisson equation for hexagonal and tetragonal packing using finite difference methods with appropriate boundary conditions. They compared their results with Springer and Tsai's heat transfer analogy and Rayleigh's electricity conduction analogy (Fig. 3). The thermal analogy underestimates the diffusion coefficient because it does not account for flow around fibers. The analogy of conduction of electricity gives excellent result up to $V_f = 0.7$.

$$D'_{22} = \left(1 - \frac{2\,V_f}{1 + V_f - 0.3058\,V_f^4 \ldots.}\right) D_r \tag{13}$$

For laminated plates Whitney [313] showed that effective diffusivity through the thickness is

$$D = \sum_{i=1}^{N} \frac{h^i}{D^i_{33}} \tag{14}$$

where N = number of laminae
h = thickness
i = superscript denoting i — the layer

while in-plane effective diffusivities are

$$(\bar{D}_{11}, \bar{D}_{12}, \bar{D}_{22}) = \frac{1}{h} \sum_{i=1}^{N} (D^i_{11}, D^i_{12}, D^i_{22})\, h^i \tag{15}$$

3.2 Solving Heat and Moisture Transport Equations

From Equations (1) and (8) it can be seen that $K_{ij}/\rho c$ and D_{ij} are measures of the "speed" by which temperature and moisture concentration change in a material. Since $[K_{ij}/\rho c]/D_{ij}$ is of the order of 10^6, most authors solve Equations (1) and (8) separately, ([272], [22], [38], [309], [312], [313]).

For most diffusion problems temperature is assumed to be equal to ambient and uniform inside CM and solutions to Fick's Equation (8) given by Jost and Cranck are used. For one-dimensional problem, infinite plate of thickness L, with constant boundary conditions such that [38]:

$$c = c_o \quad \text{at} \quad t = 0 \quad \text{and} \quad \text{all } x$$

$$c = c_\infty \quad \text{at} \quad x = 0 \quad \text{and} \quad x = L \quad \text{at} \quad t > 0$$

$$c = c_\infty \quad \text{at} \quad t = \infty \quad \text{and} \quad \text{all } x$$

then

$$\frac{c_t - c_o}{c_\infty - c_o} = 1 - \frac{4}{\pi} \sum_{n=0}^{n=\infty} (2n+1)^{-1} \sin\left[\frac{(2n+1)\,\pi x}{L}\right] \exp\left[\frac{-D(2n+1)^2\pi^2 t}{L^2}\right] \tag{16}$$

n — integer

the total amount of penetrant (water) is

$$m = \int_0^L c(x,t)\,dx \tag{17}$$

on substitution

$$\frac{m_t - m_o}{m_\infty - m_o} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{n=\infty} (2n+1)^{-2} \exp\left[\frac{-D(2n+1)^2\pi^2 t}{L^2}\right] \tag{18}$$

However for solutions where t is short ($10^{2}-10^{6}$ sec) Equation (16) converges very slowly and several hundred terms may be required. In such cases, a Laplace transformation solution is more suitable:

$$\frac{c_t - c_o}{c_\infty - c_o} = \sum_{n=1}^{\infty} (-1)^{n+1} \left\{ \mathrm{erfc}\left(\frac{2n-1-x/L}{2\sqrt{t^*}}\right) + \mathrm{erfc}\left(\frac{2n-1+x/L}{2\sqrt{t^*}}\right) \right\} \tag{19}$$

where

$$t^* = \frac{Dt}{L}$$

for $t^* < 10^{-2}$ only the first term needs to be considered [309]

$$\frac{c_t - c_o}{c_\infty - c_o} = \mathrm{erfc}\ \frac{1 - x/L}{2\sqrt{t^*}} \tag{20}$$

Shen and Springer used a correction factor to account for edge effects when applying 1-D solution to problems with finite laminated plates [272].

Whitney [312] [313] presented 3-D solutions to Fick's Equation (8). His trigonometric and Laplace solutions are both products of 1-D solutions. He also compared these solutions with experimental data and concluded that for thin laminates a one-dimensional solution can be used, while for moderately thick laminates, a one-dimensional approximation (with edge factor correction) gives good results (Figs. 4, 5, 6).

3.3 Moisture Content of CM under Transient Conditions

In [262], [311], [111], it has been found that in the case of moisture-absorption, the diffusion coefficient is sensitive to temperature and follows the relation of the activated transition state theory of diffusion:

$$D = D_o \exp[-E_d/RT] \tag{21}$$

D_o — permeability index [m^2/s]
E_d — energy of activation for diffusion [J/mol]
R — universal gas constant
T — temperature [°K]

Weitsman [309] has suggested a solution to Fick's second equation for the case when temperature changes with time. For such cases, the diffusion coefficient D becomes time varying (21). Weitsman used simple transformation and reduced the problem to the familiar case of time-independent solution.

In the same paper, he demonstrated that the decoupling of diffusion and heat transfer equations does not produce significant errors in absorption-desorption analysis as compared to coupled solutions. However, Sih and Shih in [268] [270] solved coupled equations for large gradients of moisture and temperature using finite difference methods. They found that for cases when transient stresses are of interest, coupled diffusion theory for an infinite plate gives stresses 20-80% higher as compared to the uncoupled theory.

Springer [274] [272] investigated diffusion of moisture into uncoated and coated plates made of fiber reinforced plastic. The plates were exposed on both sides to temperature and humidity variations in a cyclic manner. A computer program "W8GAIN" (for listing see [272]) was developed and used to determine moisture content (per cent weight gain) and distribution inside the material as a function of time. "W8GAIN" solves Fick's second 1-D Equation (8) uncoupled with the second Fourier Equation (1) for concentration independent diffusion. From the results Springer concluded that:

1) For Transient Ambient Conditions:

   a) After 10 years of exposure moisture content nearly reaches steady state. After that there are only slight fluctuations around this value.

   b) Moisture distribution never attains a steady state. It changes continuously and after about 6 years most changes are taking place in a narrow (0.05 mm) "boundary layer" near exposed surfaces.

2) Constant Ambient Conditions

   a) The actual variation of the moisture content and distribution with time cannot be duplicated in accelerated tests by simply replacing transient ambient conditions by constant conditions of temperature and humidity.

   b) The "steady state" moisture distribution inside the material (but outside the boundary layer) and "steady state" moisture content can be approximated by constant ambient conditions. However, the appropriate constant relative humidity to be used in the simulation cannot be guessed a priori, but must be determined by solving the entire transient problem.

3) Coated Composites

   All of the above conclusions are valid for coated composites. However, permeable coatings may reduce the amount of moisture absorbed (Figs. 6, 7, 8, 9).

Bohlmann and Derby [38] compared infinite trigonometric series solution, "Multicomp" (finite difference numerical method), and an empirical hyperbolic tangent solution with test data, for moisture predictions when ambient conditions changed. They concluded that when moisture predictions for transient conditions, with different relative humidities on each surface are necessary, a numerical method such as "Multicomp" must be used to account for the actual moisture profile. However, if only a quick estimate of moisture content is desired (15% accuracy) for equal relative humidity on both surfaces or for one insulated surface, both the series solution and hyperbolic tangent solution method can be used.

Augl and Berger [24] also studied long-term exposure to transient ambient conditions. They postulated that some kinetic average temperature (T_{avk}) and humidity (RH_{avk}) conditions exist that would give the same moisture profile and content as the actual varying environment. A rationale for calculating T_{avk} and RH_{avk} was given. Results are compared for trigonometric series solution using T_{avk} and RH_{avk} with finite difference method for actual weather data in 3 hourly steps and monthly steps where T_{avk} and RH_{avk} were calculated for any given month. If moisture content is to be determined, then kinetic averages give good results for thick (24-ply T300/5208) laminates. For initial moisture uptake using monthly averages in conjunction with finite difference method, the starting month maybe important. Moisture profiles calculated from monthly averages are accurate if the boundary layer is not considered. However, 3-year kinetic averages with trigonometric series solution for thin (6-ply) laminates cannot be used. This method gives satisfactory results for laminates of 36 plies.

In the same work, the influence of the sun's radiation on diffusion was studied and it was found that the surface of the CM could be 22°C - 28°C warmer than the surrounding air, thus increasing the diffusion coefficient.

Very detailed parametric analytical studies of the influence of surface and environmental thermal properties on the moisture absorption in fiber-reinforced CM, subjected to convection and solar radiation have been reported by Tompkins, Tenney and Unnam [290], [289], [293]. They have also included in their analysis variations in diffusion coefficient due to cyclic wetting and drying and calculated moisture content and profiles for weather data from different bases and for different flight scenarios. The most significant finding was that a composite panel exposed to the sun (12-ply T300/5208) will pick up approximately 30% less moisture than a panel exposed in the shade. The average moisture content of CM panels is relatively insensitive to geographical location, but the large cyclic seasonal variations occurring in desert areas may be more detrimental to CM than the high moisture content associated with humid areas.

The results for commercial aircraft service scenarios indicate that equilibrium moisture content depends primarily on the ground relative humidity during non-flight hours, (Figs. 10, 11, 12, 13, 14).

NASA has sponsored a long-term project on Environmental Effects on CM, during which moisture pickup was registered world wide in samples exposed on the ground and in real in-flight service environment in commercial aircraft. The results agree very well with these obtained by Tompkins et al. [57], [88], [237].

Recently Weitsmann [307] has suggested an alternative numerical method of computing moisture distribution under time varying ambient relative humidities and temperatures. Moisture diffusion was assumed to follow Fick's laws. It was shown that by switching among various forms of analytic solutions, all involving infinite series, it is possible to attain extremely high accuracy by means of a small number of terms.

Bergman and Nitsch [34] have discussed factors affecting the accuracy of analytical estimates. These were:

1) The applicability of the classical theory of diffusion to fibrous composites.

2) The adequacy of mathematical models and their computational aspects.

3) The realistic definition of the environmental conditions.

4) The reliable determination of material properties affecting diffusion.

4.0 METHODS OF EXPERIMENTAL IDENTIFICATION OF DIFFUSION PARAMETERS

4.1 Methods for Determining Diffusion Coefficients in Polymers ([9], [22], [262], [272])

Before Fick's equation can be solved the diffusion coefficient D and moisture equilibrium content or saturation level m_∞ have to be established experimentally. m_∞ is usually found by monitoring weight pickup during exposure to constant humidity and temperature of initially dry samples. Often very small (thin) samples are used since rates of diffusion at room temperature are slow.

Diffusion coefficients are usually measured by two widely applicable methods. The first is based on steady state flow rate determinations through a membrane.

From (9)

$$F = -D(c,x)\frac{dc}{dx} \tag{22}$$

For steady state flow rate

$$F = \frac{1}{L} \int_{C_L}^{C_0} D(c)dc \tag{23}$$

The second method is based on absorption or desorption measurements of diffusant, mostly in plates. The ratio, $\frac{m_t}{m_\infty}$ is plotted vs $\sqrt{t}$ for initially dry plates held at constant temperature and humidity. For concentration independent diffusion, solutions to Equation (8) are used ([9], [22], [272],[311], and others) for determining 3-D diffusion coefficients, method from Reference [190] can be used.

The initial slope of the plot of $\frac{m_t}{m_\infty}$ vs $\sqrt{t}$ is measured, and provided that the diffusion is Fickian, D can be calculated. Shen and Springer [272] have improved this method by introducing a correction to account for edge effects. Carter and Kibler [50] have used an "incremental grinding method" for rapid measurement of m_∞ and D, which is based on the solution of Fick's equation. The method depends on the fact that, whereas the initial rate of moisture uptake depends only on the product $m_\infty\sqrt{D}$, the distribution of moisture near the surface depends on m_∞ and D separately. When slices of a few thousands of an inch are ground from one or both sides of a specimen exposed for short times (few days usually) to moisture, the remaining moisture content together with the initial weight gain, provide reasonable estimates of both parameters.

For concentration dependent diffusion coefficient method described by Tajima [282] can be used.

4.2 Determination of Moisture Distributions

Leung, Kaelble and Dynes have presented in a series of papers [190], [191], [152] a method of calculation of moisture profiles based on measurements of effusion rates, and on previously established diffusion coefficients. This method is effective even if non-Fickian diffusion is evidenced by bulk water penetration into open microcracks. For such cases, rates of desorption seem to be constant for subsequent absorption-desorption cycles. These constant rates are used in the Inverse Diffusion Model, together with measured rates of effusion as a function of time after the specimen was heated to a desired temperature. Statistical estimation theory is applied to obtain initial moisture profile from experimental data.

DeIasi and Schulte [82] devised experimental methods for the evaluation of localized moisture content. It involves conditioning of the specimen in D_2O, followed by measurement of the localized deuterium concentration by means of a nuclear reaction. Both equilibrium moisture levels and profiles can be obtained in this way.

Sandorff and Tajima [252] developed a simple and inexpensive method which involves splitting a small specimen of the laminate into thin slabs and drying them to determine their moisture content.

Singh, Holt and Mock [271] suggested that moisture profiles could be determined through the measurement of positron lifetime. They showed that this lifetime is a linearly decreasing function of the moisture content. If magnetically analyzed, positron beams should provide a map of the moisture depth distribution. This would be the first quick and non-destructive method of moisture profiling.

Before some of the results obtained in CM are presented, a paper by Edge [98] should be mentioned. He has pointed out the importance of drying specimens completely prior to exposure, since failure to do so may lead to serious errors when conducting moisture absorption experiments.

5.0 EXPERIMENTAL RESULTS

5.1 Moisture Absorption in CM

Several authors have monitored moisture absorption and desorption in CM and in non-reinforced resins. Shirrel and Halpin in their review [262] have presented the most typical results obtained, (Figs. 15, 16, 17, 18,19).

These results were for panels exposed to constant temperature and humidity conditions. More recently similar results were obtained by Loos and Springer [196] and Long [195].

Moisture equilibrium depends on relative humidity but is usually independent of temperature (Fig. 17). The following empirical relation is appropriate.

$$m_{\infty} = a(RH)^{b} \tag{24}$$

where a and b are material constants (this is known as Henry's Law).

Eckstein [96] studied moisture absorption by epoxy resins of different composition used in laminates. Kourtides [178] published weight gains of epoxy and bismaleimides A and B and their composites immersed in water. Bismaleimides absorb less water. Crossman et al., [70], [69] measured equilibrium moisture contents in laminates of HMF 330C/934, T300/5209 and GY70/339 as function of temperature and humidity. From Figure 20 it can be seen that for 95% RH there is a marked dependence of m_{∞} on temperature.

The temperature dependence of diffusion coefficient seems to closely follow Equation (21). Loos and Springer [196] concluded that transverse diffusivity of a composite can be estimated from diffusivity of the resin, provided that the resin and the composite were cured in the same manner (they used the Springer-Tsai thermal analogue). However Augl and Berger [22] measured diffusivities of neat resins and their composites using two types of fibers (HMS and T300). Results showed between 20% to 60% higher diffusivities for composites (same matrix and V_f difference less than 4%) with HMS fibers than with T300. This was because T300 was sized with epoxy for handleability, and the result points to the importance of interface on diffusion. Measured diffusion coefficients were 30% lower than those calculated by the finite difference method from neat resin diffusivities.

Because of the highly sensitive nature of diffusion parameters to resin composition, cure quality, void content and interface, Carter and Kibler [50] suggested that diffusion of moisture could be used as a screening test for consistency of mechanical properties.

Menges and Gitschner [216] introduced the "interface factor" for calculating equilibrium water absorption in laminates, from equilibrium for neat resin and fiber volume fractions. The interface factor is essentially a measure of the quality of a composite. As can be seen from Figure 21, for glass composites for higher interface factors, composite may absorb more moisture than the equilibrium for neat resin.

Hertz [130] observed weight gain of pseudo-isotropic GY-70/X-3 (graphite/epoxy) exposed first to room temperature/humidity and then to room temperature vacuum (10^{-6} torr). Results indicate that desorption is generally slower than absorption and highly dependent on the moisture distribution.

Published results on absorption experiments with organic fiber composites are few. Augl, [25] studied moisture absorption and diffusion in Kevlar 49. Moisture absorption equilibrium concentration as function of relative humidity and diffusion coefficient as function of concentration at 28°C and 50°C have been measured. This diffusion coefficient was considerably lower than for most other polymers. Thus, Augl concluded, in composites the effective diffusion coefficient is governed by the resin diffusion coefficient and therefore the fibers behave as if they do not contribute to moisture transport, although the total moisture uptake has to be taken into account. No significant difference in the absorption behaviour of yarns with and without sizing was observed. Allred and Lindrose [9] determined moisture diffusion coefficients for quasi-isotropic Kevlar 49 181-style fabric reinforced Narmco 5208 epoxy. Results were 6.1 X 10^{-8} cm^2/s in the laminate plane, 1.7 X 10^{-10} cm^2/s for the through the thickness diffusion and 6.5 X 10^{-10} cm^2/s for pure resin. This would indicate that the rapid in-plane diffusion is due to the preferential diffusion of moisture in the filament along its length, while Augl's conclusion would still be true for through the thickness diffusion.

Recently, jute fibers have been used in composites and some results on their diffusion properties will be found in [239].

Bohlmann and Derby, [38] found that surface finish has no effect on diffusion of moisture.

5.2 Effect of Thermal Spiking

McKague et al. [213] have found that exposing CM to thermal spiking, similar to that encountered during supersonic flight, caused permanent changes in the subsequent moisture diffusion behaviour of graphite/epoxy. Both the amount and rate of moisture absorption increased considerably. They have also found that exposure to sub-zero temperature did not cause changes in diffusion behaviour.

Bohlmann and Derby [38] studied the effect of the thermal spike encountered by the Shuttle Orbiter Aft Propulsion Subsystem which is characterized by much slower heating and cooling rates than the ones encountered during supersonic flight missions. This may explain why Bohlmann and Derby did not record any effect of thermal spiking on diffusion of moisture.

Browning [42], [44] reported that increased diffusion rates and higher absorptivity result from microcracks which are formed due to stresses caused by moisture and temperature gradients (during the thermal spike). These microcracks provide additional surface area for absorption/desorption processes. Due to the lowering of glass transition temperature (Tg), with higher moisture content, composite materials can more easily undergo viscous flow to accommodate the water.

McKague [211], [214] also gives evidence for microcracks being responsible for additional moisture absorption. He suggests that the relationship between moisture content and Tg forms a service envelope for CM.

Similar results were reported in [81], [128].

Loos and Springer [198] studied the relationship between material behaviour and the thermal spike variables: max. and min. temperature during the spike, rates of increase and decrease of temperature, duration of the spike and number of spikes. For the material chosen (T300/1034) thermal spiking seemed to have no effect on equilibrium moisture content and transverse diffusivity. This led the authors to conclude that the effect of thermal spiking depends on the composition of the material.

Recently Shyprykevich and Wolter [267] proposed a semi-empirical transport model based on Fickian diffusion to describe the changes in absorption characteristic as a function of Tg exceedences.

Morgan et al. [220], [221] have presented results on the effects of thermal environment and absorbed moisture on cured amine epoxies. The moisture induced swelling stresses together with the enhanced mobility of the water molecules within the epoxy-moisture system during thermal spiking produce free-volume increases that involve rotational-isometric configurational changes within the epoxy network. Such changes are fixed in the epoxy during the rapid cooling after the thermal spike. This additional free-volume allows water molecules access to previously inaccessible sites within the epoxy. Morgan et al. see this as a primary mechanism responsible for increased moisture absorption, while rupture of cross-links, crazing and/or cracking, and loss of unreacted material should be regarded as important factors. From Figure 22 it can be seen that constant tensile stresses over 38 MPa applied for one hour on initially dry epoxies enhanced moisture absorption by 0.5 wt%. Those studies indicate that the initial stages of failure, that involve dilatational craze propagation as well as subsequent crack propagation, enhance accessibility of moisture to absorption sites to a greater extent than in the later stages of failure which involve crack propagation alone.

5.3 Effect of Cycling Environments and Pretreatment

Several investigators have cycled either temperature or humidity (or both) during absorption experiments.

Sometimes cycling was due to the fact that while samples were exposed at higher temperatures they had to be brought to room temperature for weighing. Kaelble and Dynes [155] did not observe any changes in the diffusion kinetics for graphite/epoxy composites in this case.

Blaga [37] measured moisture absorption and desorption kinetics of weathered glass reinforced polyester-styrene (cross-linked). After three years, weathered samples absorbed more moisture, 20-26% for exposure from 30 to 80% RH and 13 to 15% more for higher RH exposure. All absorption-desorption was carried at a temperature of 23°C. The diffusion coefficients for absorption and desorption for weathered sheets were 47% and 30% higher respectively than in control sheets (at 80% RH) with an even greater difference for higher RH. A possible explanation for these changes was the UV-induced photo-oxidative degradation observed on the surface of weathered sheets.

Halloff [121] studied the effect of heat treatment, before exposure to humid conditions on graphite/epoxies (T300/5208, HT-S/3501 and Fibredux 914C). Two laminates of T300/5208 $[(\pm 45)_2/45]_{S10}$ and $[(0/\pm 45/90)_2]_{S16}$ with void contents of <0.1 and 0.9 respectively were tested. However, from the data presented, it seems that absorbed moisture weight gains for thinner laminates were inadvertently interchanged with weight gains for thicker laminates, (Figs. 23 and 24). Much higher absorption rates correspond to thinner laminates. The fact that equilibrium was not reached after 125 days of exposure also indicate a characteristic of thicker laminates with higher void contents (non-Fickian diffusion could be expected). However it can be seen that heat treatment prior to humidity exposure had a different effect depending on both the thickness of composite and the void content (a difference up to 50% in equilibrium moisture content). Fibredux 914C did not show any different effects after various heat treatments, and it can be concluded that heat treatment effects only some materials.

Shirrell [263] studied diffusion of water into T300/5208 laminates. He observed that apparent values of equilibrium solubility of moisture in laminates are affected by the degree of cure. Laminates subjected to postcure treatment absorbed more water, with a trend towards lower equilibrium moisture content with increasing temperature. Non-postcured specimens exhibited constant equilibrium moisture in relation to temperature.

Crossman et al. [69] subjected T300/5208 and T300/5209 laminates to 100 hygrothermal cycles between temperatures −54°C and 70° or 93°C. Specimens were held for 15 minutes at each temperature and switched between two chambers maintained at the two temperature extremes. After five cycles, specimens were held at constant humidity and temperature for two hours, then after every 15 cycles, specimens were held in constant humidity/temperature overnight. No significant changes in moisture content was found and neither surface or edge cracks, fiber matrix debonding nor transverse microcracks were detected.

Apicella and Nicolais [16] recently reported absorption data for neat epoxy resin (Epikote 828 cured with TETA curing agent) which showed a dependence on temperature and humidity histories. Samples exposed at 60°C and higher temperatures and higher RH, absorbed more moisture and displayed lower diffusion coefficients when dried and exposed for a second time. No significant change in either value was observed for additional drying and wetting cycles. This change was explained in terms of hypothesized induced microcavities that can be formed by solvent crazing in the plasticized system.

6.0 THEORIES ACCOUNTING FOR DEPARTURES FROM FICK'S MODEL

6.1 Non-Fickian and Concentration Dependent Diffusion

Numerous investigators have claimed that moisture absorption in graphite/epoxies is a concentration-independent Fickian diffusion process. Some of their results were presented in previous sections. No attempt was made at explaining observed effects using other than simple Fickian diffusion. However, Shirrell [263] pointed out that below the glass transition temperature, both filled and neat epoxy resins can also exhibit either:

1) concentration dependent diffusion
2) time-dependent diffusion anomalies
3) Case II — transport*
4) solvent crazing/stress cracking.

This author observed non-Fickian absorption anomalies in T300/5208 laminates (postcured and non-postcured) at higher temperature (82°C) and moisture levels above 34% RH. Non-Fickian diffusion anomalies were observed at both high and low moisture concentrations and concentration dependent diffusion could not be excluded. In [265] Shirrell presented similar results for AS/3501-5 and Boron/5505 laminates.

In [264] Shirrell et al. described a microscopic examination of T300/5208 laminates. Microcracks were observed after exposures to 82°C and different RH, while for lower temperatures at similar humidities microcracks were not found. At 82°C, the severity and frequency of cracks increased with humidity. Postcured specimens formed more severe microcracks than non-postcured. It is not clear whether cracks were formed due to hydrothermal exposure or due to the fact that considerable temperature cycling was introduced for weighing samples during moisture gain monitoring. Kaelble, Dynes and Leung [191], [152] have suggested using moisture diffusion analysis (MDA) to scan the area or length of a composite panel to locate regions of micro-structural degradation. In such regions non-Fickian diffusion is evidenced by bulk water penetration into open microcracks followed by accelerated molecular diffusion in the regions between cracks.

Whitney and Browning [311] presented moisture diffusion data on 3501-5 neat resin and AS/3501-5 graphite/epoxy composites which indicate a departure from classical Fickian diffusion behaviours. If moisture percent weight gains are plotted against $\sqrt{t^*}$ $\left(t^* = \frac{Dt}{L} \text{ see Eq. (17)}\right)$ then one master curve can be plotted for different RH and temperatures. This plot can then be checked for compliance with Fickian diffusion Equation (16) or (17). At higher RH and temperatures neat resins and their composites exhibit two-stage diffusion. An initial equilibrium is reached and remains constant for some time. Later, additional amounts of water are absorbed. At this stage, cracks can usually be found in the matrix. Whitney and Browning observed the largest departure in bidirectional laminates. The through-the-thickness diffusion coefficient was considerably higher than that for unidirectional composites but when a time decreasing diffusion coefficient was used, improved correlation with theory was achieved. This decrease in diffusion is coincident with decrease of tensile transverse stresses (significant for bidirectional laminates).

Hahn and Kim [118] noted that for subsequent immersion in water at 82°C and desorption in vacuum of AS/3501-5 (graphite/epoxy), initial absorption in virgin specimens seems to produce microcracks. The residual-swelling stresses appeared to be responsible for absorption being initially slower than desorption, because the boundary layer is in compression during absorption whereas it is in tension during desorption.

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* A case where a sharp boundary, advancing with constant velocity, separates the inner glossy state from outer solvent swollen, rubbery shell [6].

Gillat and Broutman [111] and Kim and Broutman [170] studied the effect of external stresses on moisture diffusion. Graphite/epoxy (SP-313) specimens were loaded in tension and then immersed in water at 25°, 40° and 60°C. Even at 0.25 of the ultimate tensile stress (UTS), when no cracks could be detected in the composite, the diffusion coefficient was about 80-90% higher than the unloaded case, and equilibrium moisture increased slightly. More significant changes were observed for loads exceeding 0.45 UTS. However, for temperatures not exceeding 60°C Fickian diffusion gave good correlation with experiments, if the diffusion coefficient for stressed material was used.

Marom and Broutman [207] found that the rate of water uptake by unidirectional glass and graphite reinforced epoxy composites was an increasing function of the loading angle with respect to the fibre direction. This suggests some dependence on the increase of the matrix volume, which is influenced by the local strain and the material Poisson ratio.

Apicella and Nicolais [16] observed synergistic effects of absorbed moisture, temperature and applied stress. Two samples were immersed in water at 40°C, one of them under uniaxial tension, (7% of yielding stresses), and the other stress free. After drying and subsequent soaking with both samples unloaded, the previously loaded specimen gained 16% more moisture in the equilibrium condition. These authors support the theory which explains this absorption behaviour in terms of crazing. It is a process of plastic deformation in the tensile stress direction without lateral contraction involving significant cavitation and localized fibrillation. Stress field is induced by mechanical load, temperature or swelling due to absorption. A crazing criterion (after Sternstein and Ongchin [275]) can be used to determine whether the type of stress field induced increased the crazing tendency.

An interesting observation was made by Apicella and Nicolais [16] and by Adamson [4], (Fig. 25). Specimens, both neat resins and their composites, were exposed to moisture at 75°C. After the equilibrium or near equilibrium moisture content had been achieved, the temperature was dropped while humidity was kept at the same level. The samples absorbed additional moisture and a 15% higher equilibrium was reached. Apicella and Nicolais explain this effect in terms of additional moisture trapped in the formed voids. Adamson called this effect the reversed thermal effect, and explained it in terms of free volume theory, which accounts for the fact that the network structure of cross-linked epoxy resins is not homogeneous. Rather, it is a mixture of highly cross-linked microgel particles (micelles) embedded in a less highly cross-linked matrix (i.e. two phase network). Adamson simultaneously monitored both weight gain and swelling. In Figure 26 the rate of swelling is divided into three regions. First to Region I, in which resin swelling is far less than the volume of the water absorbed. This region includes absorption into free volume and the bonding of some water molecules (causing swelling). The rate of absorption is rapid in this region. In Region II, swelling is equal to the volume of absorbed water, an indication that all free volume is occupied and water can be absorbed at the rate at which it is bounded to the resin. Finally in Region III, swelling is again less than the volume of water absorbed which is attributed to the free volume in the micelles being occupied. As free volume increases with decreasing temperature, more water can be absorbed into the resin. The most significant difference between the two given explanations for the "reversed thermal effect" is the fact that defects are not necessary in Adamson's model, i.e. the process is reversible. However, both models see resins as multiphase mediums, in which case Fick's model cannot adequately describe diffusion.

The reported "reverse thermal effect" questions the validity of most diffusion experiments. The majority of investigators, while monitoring the weight gains of their samples exposed in higher temperatures, cooled them prior to weighing at room temperature while simultaneously maintaining constant humidity. Thus higher moisture contents could have been reached than should be expected for given exposure temperature. To avoid this error, specimens should be weighted at the same temperature as was used during exposure.

Tajima and Wanamaker [283] studied absorption properties of T300/5209 laminates and 5209 neat resins. The desorption rate was greater than the rate of absorption and some moisture was retained irreversibly (samples were dried in air). For both absorption and desorption diffusion was concentration dependent and it was observed that activation energies for diffusion in resins and composites were not equal which is contrary to the theory of diffusion in filled polymers. The results indicate a difference between the molecular structure of a 5209 neat resin and a 5209 composite matrix.

For T300/5208 laminates Tajima [282] found that absorption in unaged laminates is initially simple Fickian (constant diffusion coefficient). The first desorption is Fickian with a concentration-dependent diffusion coefficient. The diffusion process continues to change with hygrothermal conditioning and may become class II diffusion; that is, diffusion which is rate-controlled by polymer relaxation resulting in distributions as shown in Figure 27 (for first absorption, distributions are shown in Fig. 28). However, Tajima stated that his results do not rule out alternative explanations such as crazing and/or formation of a connected network of microcracks which would enable fluid flow with simultaneous Fickian or strain-dependent diffusion.

6.2 Lagumir Type-Two Phase Diffusion Model in Composites

Carter and Kibler [49] proposed a linear model which involves sources and sinks of diffusing moisture molecules. With respect to bound and unbound particles, it is similar to the Lagumir theory of adsorption isotherms. An approximation of their exact solution of coupled differential equations was used to fit data for mildly anomalous moisture uptake curves for 5208 resin for over two years. Since the same parameters gave equally good fits to the data at all humidities, it appears that the absorption anomaly does not result from nonlinear (concentration or stress dependent) effects. A very similar model was presented by Gurtin and Yatomi [117]. In both papers ([49], [117]) the model formulation and solution is presented. The unbound or free phase molecules follow the concentration independent Fick's diffusion model where molecules are being bound with probability per unit time γ and released with probability per unit time β. A method for calculating γ, β, M_∞ — moisture gain at saturation and D-diffusivity for this model is given by Bonniau and Bunsell [39], who made a comparative study of the two models in glass/epoxy composites. The three materials used were the same except for the type of hardener used. (E glass with Bisphenol A Resin). The Diamine cured material exhibited simple Fickian diffusion. Dicyandiamide hardener gave results which fit better to Lagumir type of diffusion model. Probabilities γ and β increased with temperature while the total fraction of water in free phase remained constant $\left(\frac{\gamma}{\gamma+\beta} = 0.7\right)$. For single and two phase diffusion, diffusivity was only a function of temperature and followed a Arrhenius-type relationship. The saturation limit was a linear function of relative humidity. For both materials no damage was noticed except under most severe conditions. For the third hardener (anhydride) considerable loss of material was evident at 40°C and above excluding any possibility of general description of water absorption by diffusion.

Bunsell et al. [8], [68] examined the limitations of the laws of diffusion with the aid of dielectric measurements on the same set of materials as in previous papers. Three mechanisms of absorption were observed. The first corresponded to a simple Fickian mechanism and was not accompanied by an irreversible change in properties. The second was observed at levels of saturation greater than 0.6 to 0.7% when materials were exposed to water vapours. A large increase in dielectric losses was absorbed together with electrical conduction. The third mechanism, only seen during immersion, was the transport of water by capillary action along microcracks in the matrix.

6.3 Constitutive Theory for Anisotropic Hygrothermoelasticity

Chung and Prater [63] developed a constitutive theory for hygrothermoelasticity in anisotropic media, from the first and second law of thermodynamics with moisture diffusion included. Fick's first law was modified to account for a dependency on strain gradient. Chung and Bradshaw [62] expanded this theory to include effects of Duffour (diffusion-thermal) and Soret (thermal-diffusion). The fact that moisture may be present in free and bound phases is also taken into account. The final forms of governing equations (momentum, heat conduction and two-phase diffusion), when solved simultaneously, provide a complete coupling of deformation field with heat and mass transfer of moisture. Processes analyzed may be irreversible or reversible with viscoelastic effects present. Finite element solutions are presented for slightly simplified cases. Further investigations and experiments are necessary before this model is practical.

7.0 CONCLUSIONS AND RECOMMENDATIONS

In the previous sections numerous conclusions drawn by researchers were cited. Here the most significant and general conclusions and recommendations are listed:

1) The only general theory which addresses all possible modes of moisture diffusion is still in its development stage (see 6.3).

2) At present, the researcher must first identify the model of diffusion which is most suitable for the material and environmental conditions of interest.

3) The model of diffusion may change with time due to material degradation.

4) There is no general model for degradation of composite materials.

5) The accuracy of analytical estimates depends on the adequacy of the mathematical models and their computational aspects, and on the realistic definition of environmental conditions.

6) Material properties affecting diffusion have to be reliably determined.

7) Very stringent quality assurance procedures must be followed during production of composite structures as slight variations in composition and/or cure may result in very different diffusion properties which influence the long term effect of the environment on mechanical properties.

8) A limited amount of research was devoted to transient effects — the performance of composites subjected to high temperature and humidity gradients.

The literature reviewed above and the conclusions drawn represent the state of the art in moisture and heat absorption into advanced composite materials. These studies were primarily directed at providing the answers to the question of how much moisture or what temperature will be expected to be found at a given point of a composite material given the conditions.

In the following parts of the review the effect of moisture and temperature on physical and especially mechanical properties will be described and further conclusions and recommendations will be drawn.

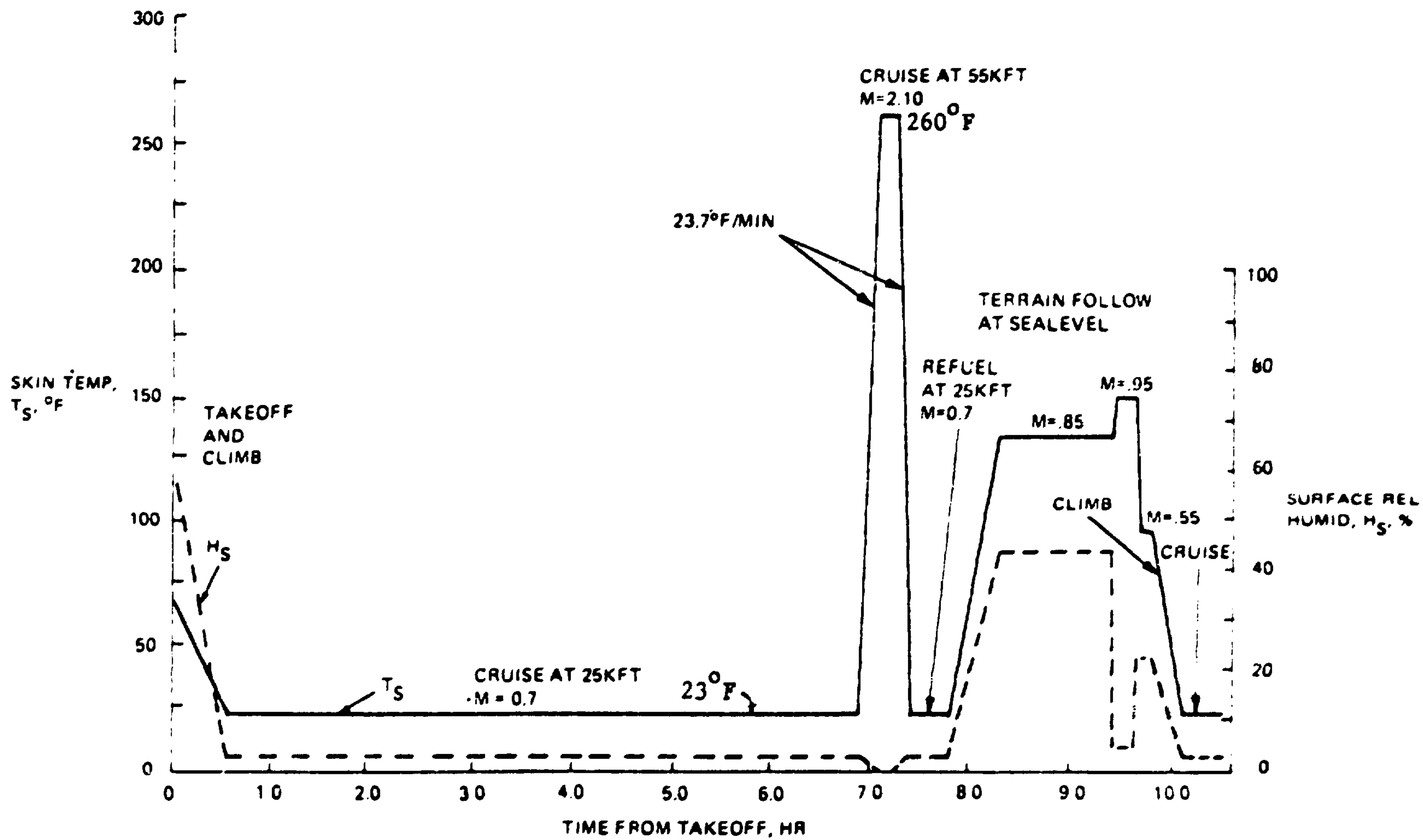

FIG. 1: FLIGHT THERMAL PROFILE[26]

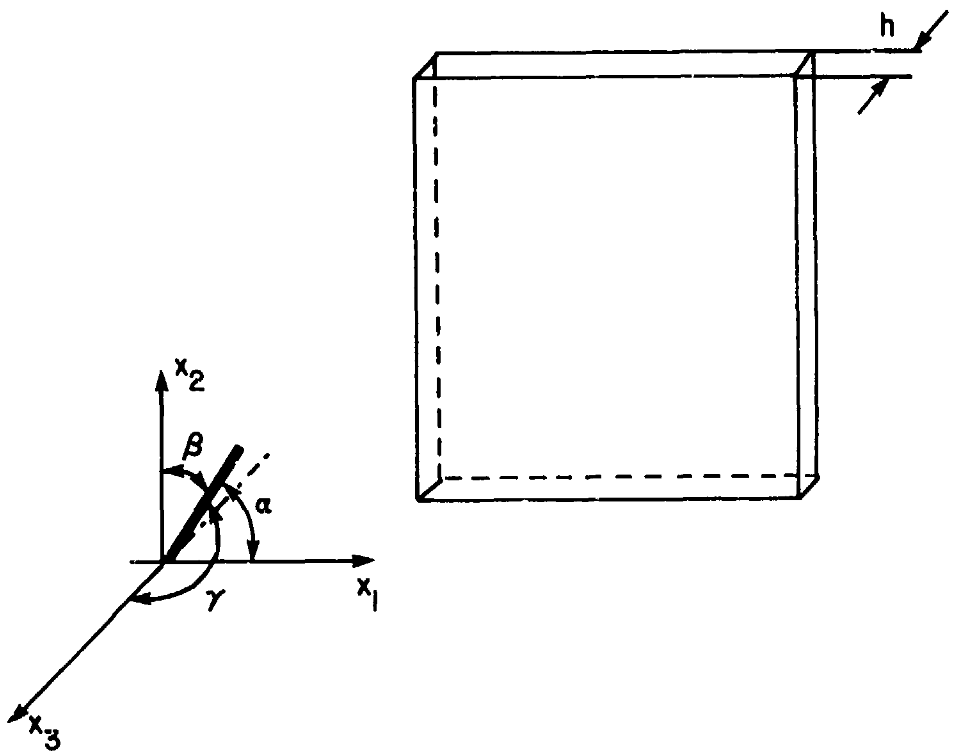

FIG. 2: CO-ORDINATE SYSTEM

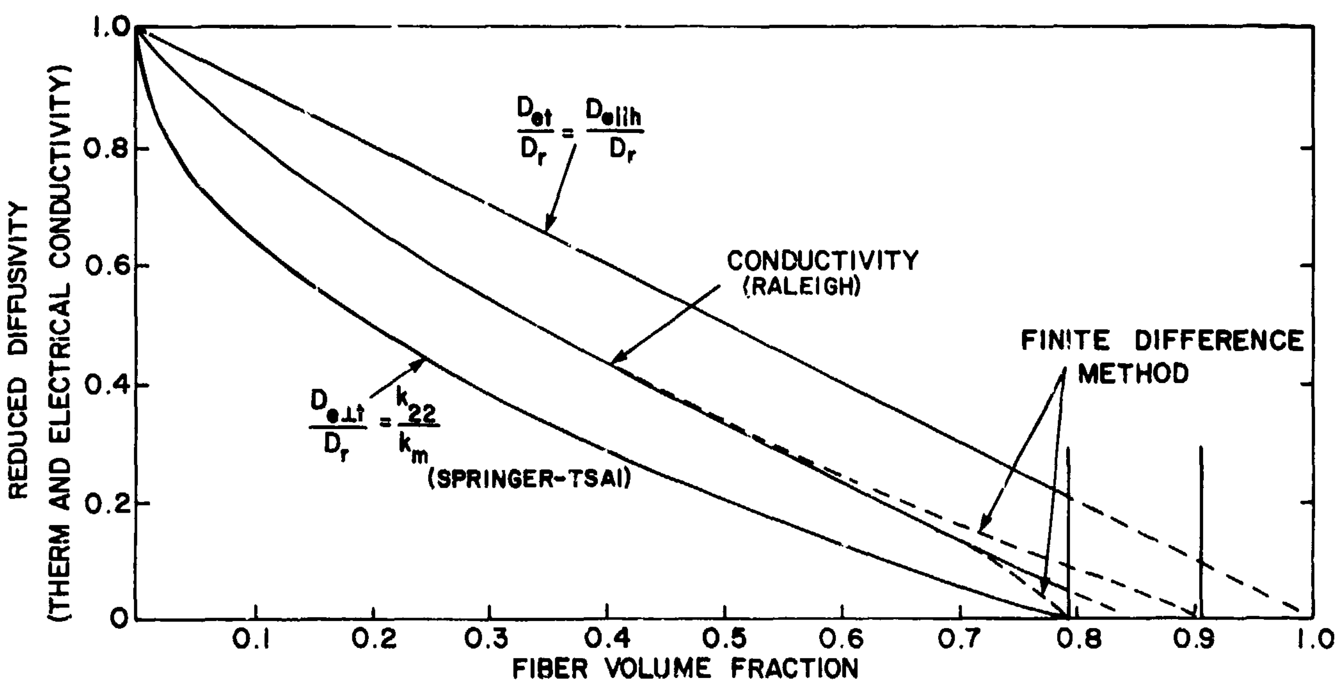

FIG. 3: A COMPARISON OF THE REDUCED DIFFUSIVITY VERTICAL TO THE FIBER DIRECTION WITH THERMAL AND ELECTRICAL ANALOGUE[22]

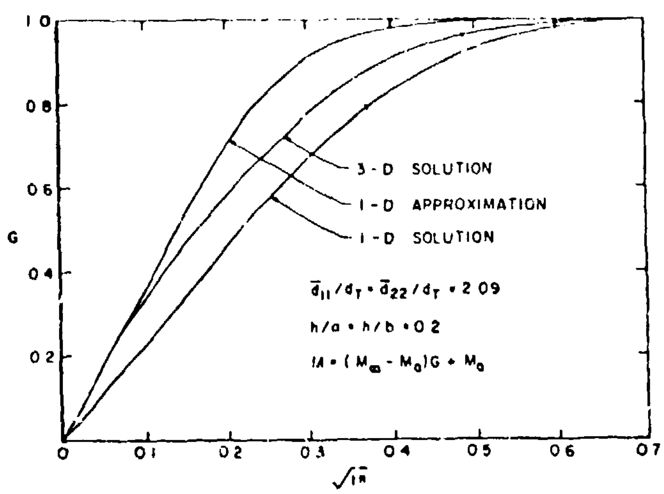

FIG. 4: TOTAL WEIGHT GAIN FOR THREE-DIMENSIONAL DIFFUSION IN A THICK COMPOSITE LAMINATE[312]

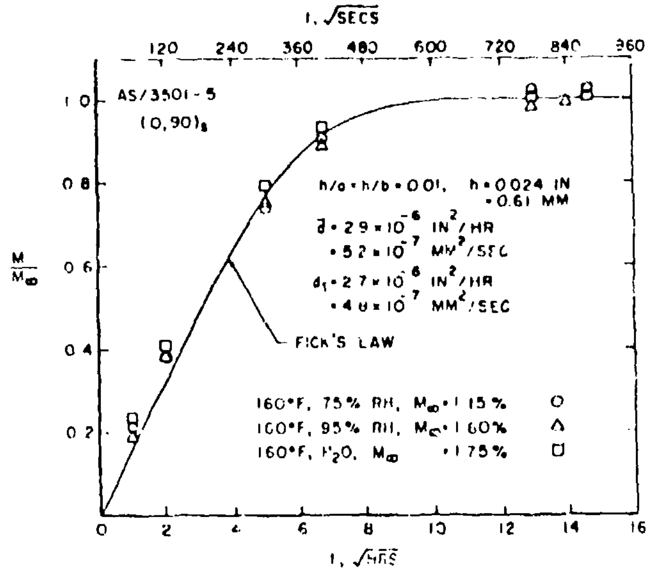

FIG. 5: COMPARISON OF THEORY AND EXPERIMENT FOR A THIN GRAPHITE/EPOXY COMPOSITE LAMINATE[312]

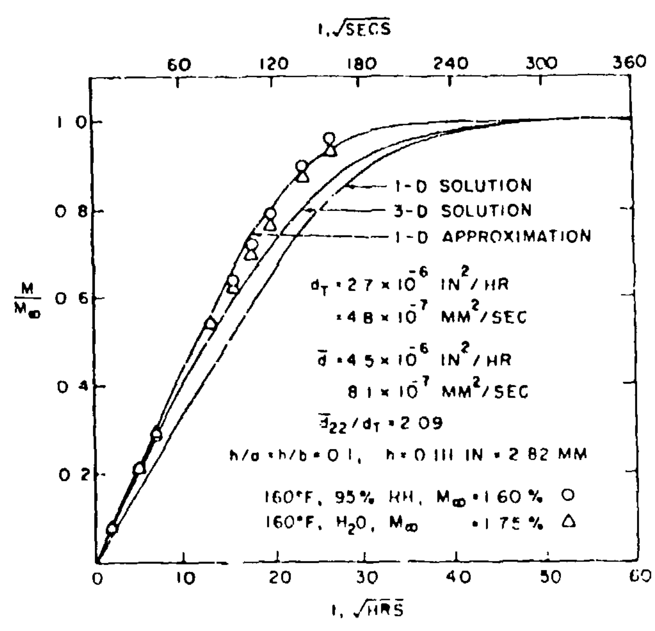

FIG. 6: COMPARISON OF THEORY AND EXPERIMENT FOR A MODERATELY THICK GRAPHITE/EPOXY LAMINATE[312]

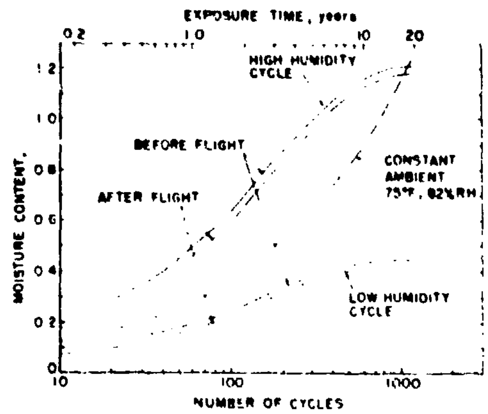

FIG. 7: THE VARIATION OF MOISTURE CONTENT WITH TIME[274]

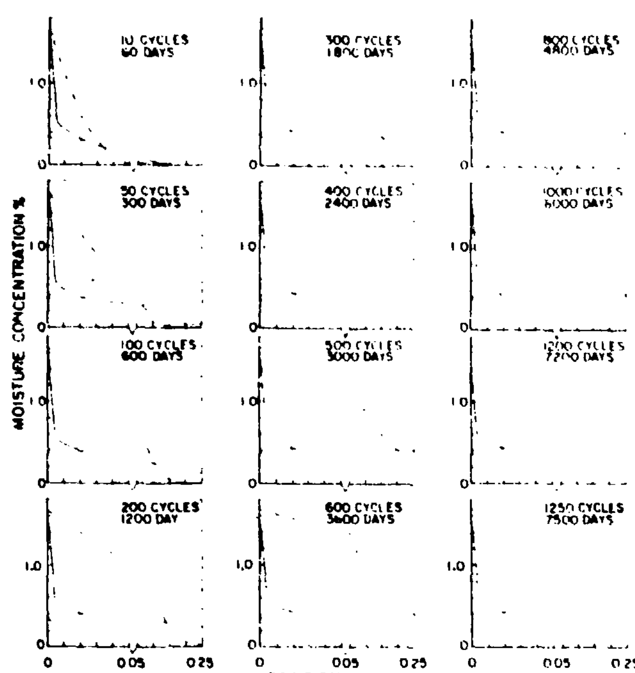

FIG. 8: THE VARIATION OF THE MOISTURE DISTRIBUTION WITH TIME[274]

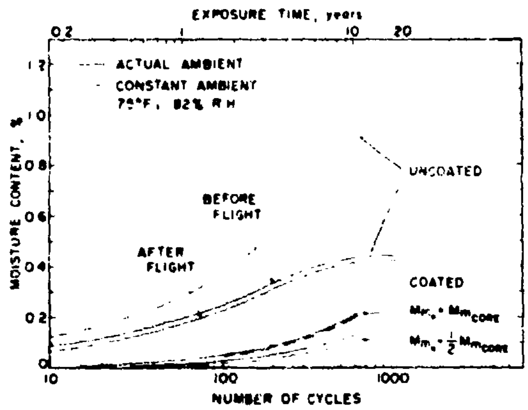

FIG. 9: THE VARIATION OF MOISTURE CONTENT WITH TIME. COATED AND UNCOATED COMPOSITE[274]

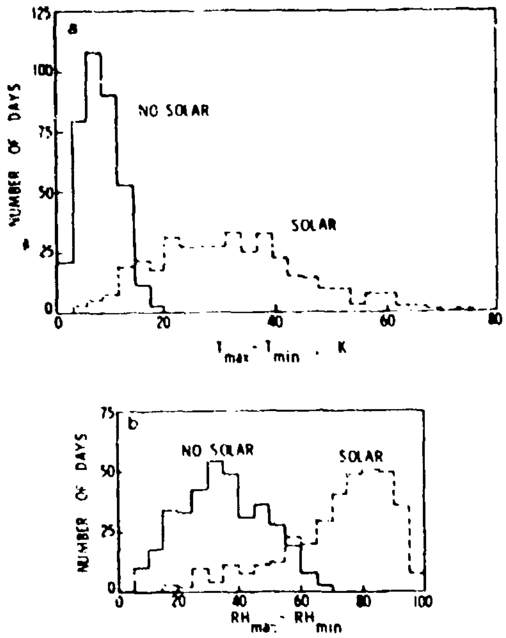

FIG. 10: DAILY TEMPERATURE AND RELATIVE HUMIDITY CHANGES FOR A PANEL WITH AND WITHOUT CONVECTION AND SOLAR RADIATION. (a) DAILY TEMPERATURE CHANGE, AND (b) DAILY RELATIVE HUMDITY CHANGE[289]

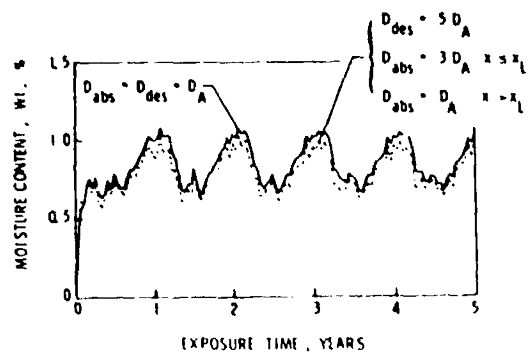

FIG. 11: EFFECT OF VARIATION IN DIFFUSIVITY ON MOISTURE CONTENT HISTORY OF A 12-PLY GRAPHITE EPOXY PANEL[289]

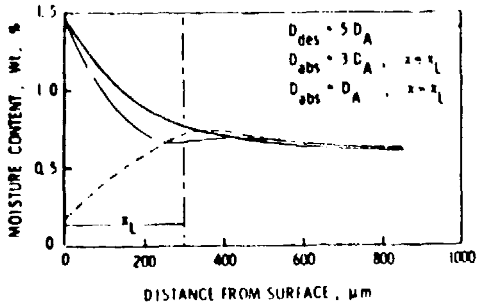

FIG. 12: TYPICAL CHANGES IN MOISTURE CONCENTRATION PROFILES DURING A SUMMER DAY USING A VARIABLE DIFFUSIVITY (D_{des} = $5D_A$, D_{abs} = $3D_A$, FOR $x < x_L$ AND D_{abs} = D_A, $x > x_L$)[289]

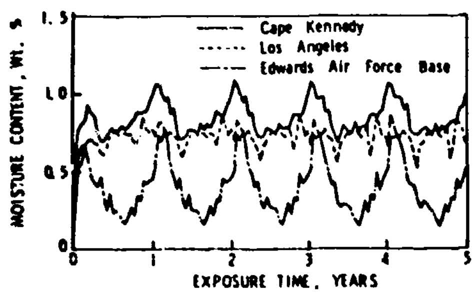

FIG. 13: INFLUENCE OF GEOGRAPHICAL LOCATION ON THE MOISTURE CONTENT HISTORY OF A 12-PLY GRAPHITE EPOXY PANEL[289]

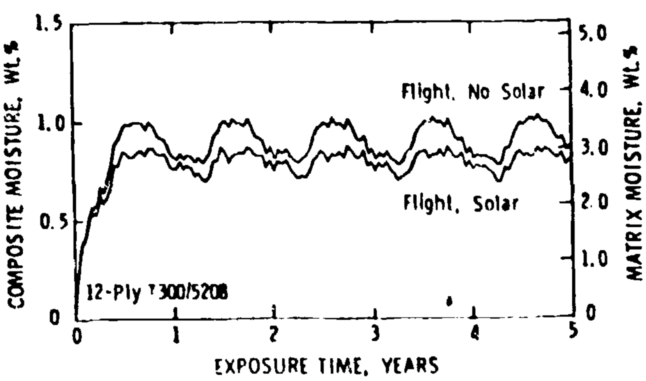

FIG. 14: COMPARISON OF MOISTURE CONTENTS FOR FLIGHT SERVICE WITH AND WITHOUT A CORRECTION FOR SOLAR HEATING DURING PERIODS OF GROUND EXPOSURE[293]

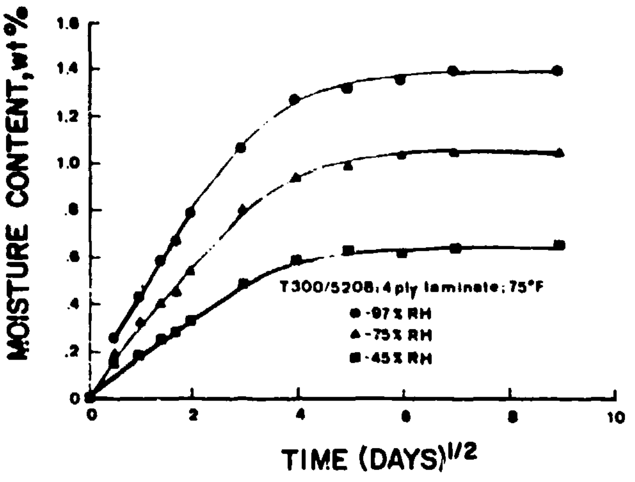

FIG. 15: EQUILIBRIUM SATURATION VARIES WITH HUMIDITY[262]

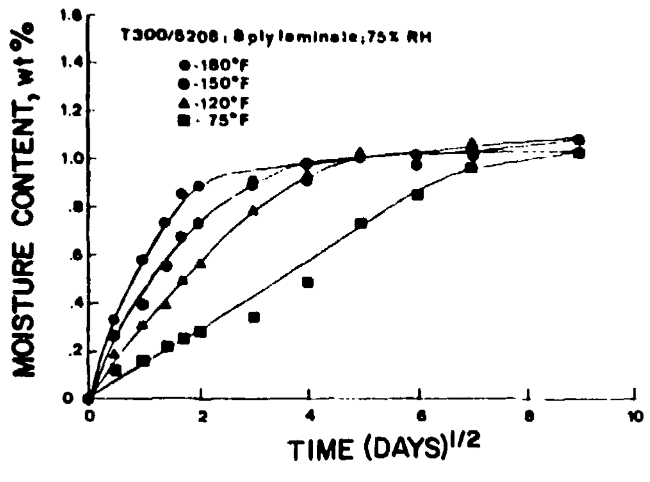

FIG. 16: TEMPERATURE ACCELERATES THE APPROACH TO EQUILIBRIUM SATURATION[262]

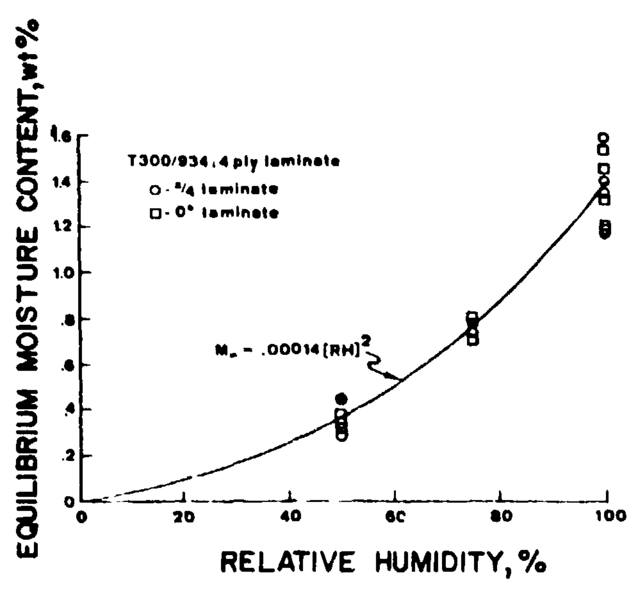

FIG. 17: RELATIONSHIP BETWEEN EQUILIBRIUM AND RELATIVE HUMIDITY[262]

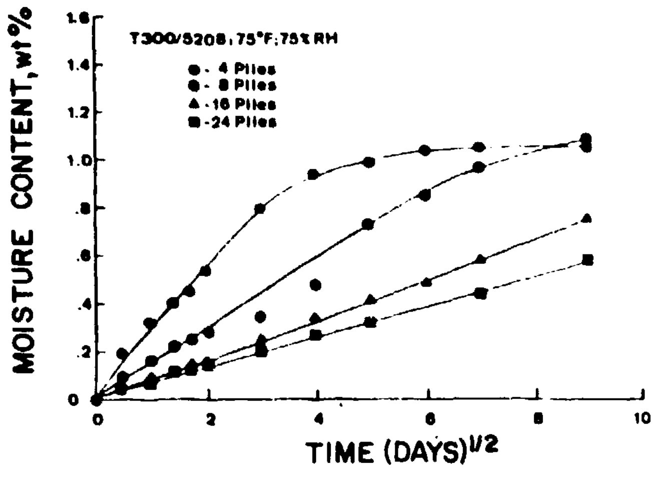

FIG. 18: MOISTURE ABSORPTION VARIES WITH THICKNESS[262]

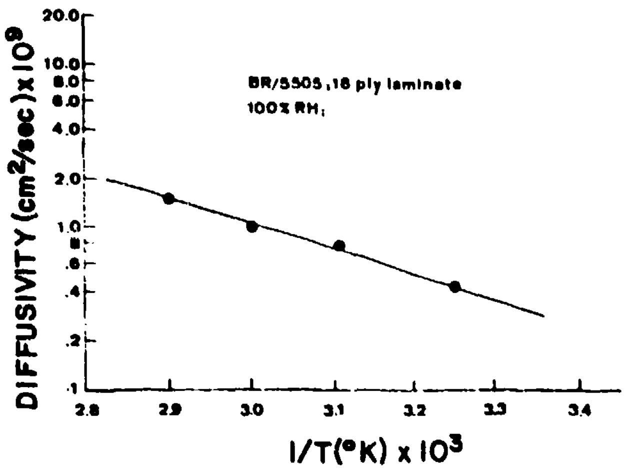

FIG. 19: DIFFUSIVITY AS A FUNCTION OF TEMPERATURE FOR MOISTURE ABSORPTION[262]

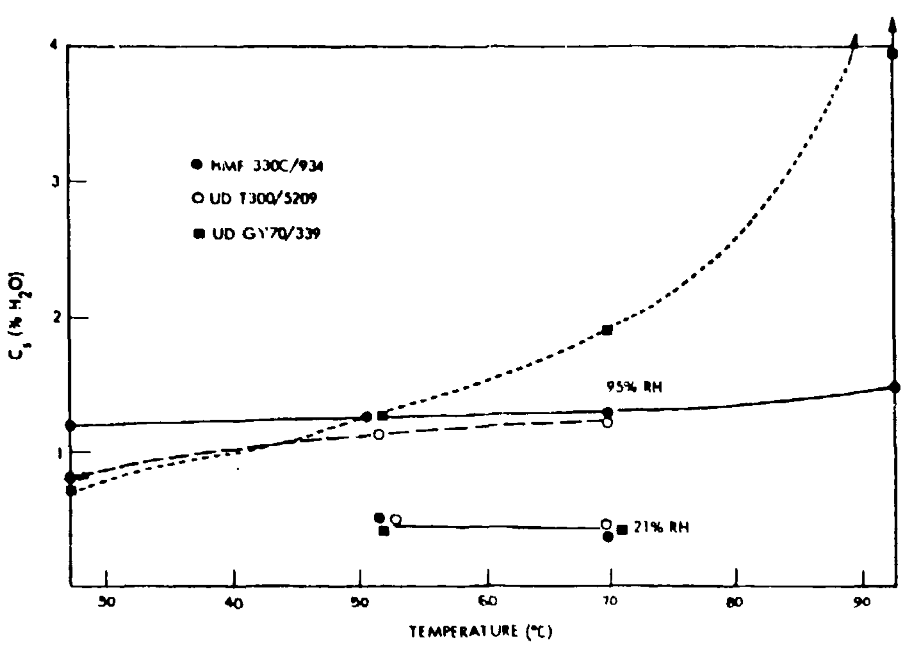

FIG. 20: EQUILIBRIUM ABSORBED MOISTURE CONTENT C_S AS A FUNCTION OF TEMPERATURE AND HUMIDITY[70]

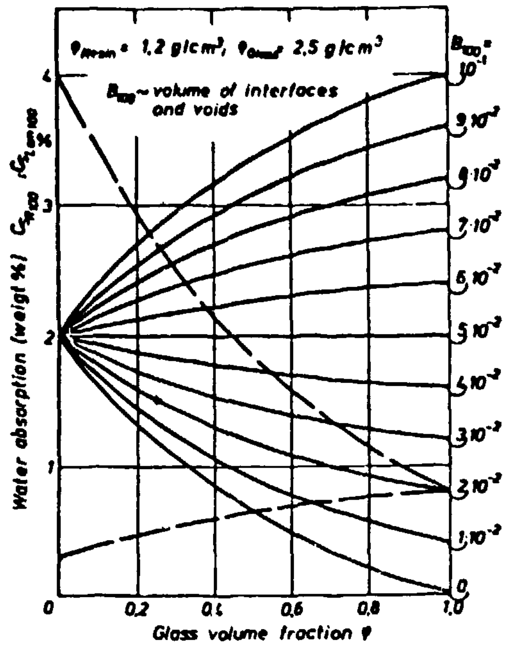

FIG. 21: WATER ABSORPTION IN COMPOSITES DEPENDENT ON THE ABSORPTION BEHAVIOUR OF THE RESIN AND THE VOLUME OF INTERFACES AND FLAWS ($\sim B_{100}$)[216]

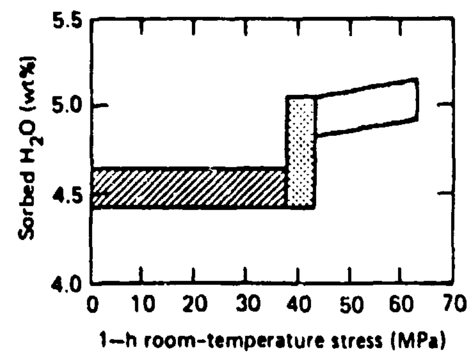

FIG. 22: EQUILIBRIUM wt% MOISTURE ABSORBED BY TGDDM-DDS (27-wt%-DDS) EPOXIES AT 100% RELATIVE HUMIDITY 23°C, AS A FUNCTION OF 1-h CONSTANT STRESS LEVELS APPLIED PRIOR TO MOISTURE EXPOSURE[220]

Material	Interface factor B_{100}
Glass-fabric reinforced epoxy resin, hot-setting	$1 \cdot 10^{-2}$
Glass-fibre mat reinforced UP-resins (room temperature)	$1.5 \div 2.5 \cdot 10^{2}$
Unidirectional glass-fibre reinforced UP-resin layers (room temperature)	$2.0 \div 3.0 \cdot 10^{-2}$
Glass-fibre reinforced UP at high temperature	$B_{100} \neq \text{const} > 5 \cdot 10^{-2}$

TABLE 1 - INTERFACE FACTORS B_{100} FOR WATER ABSORPTION[216]

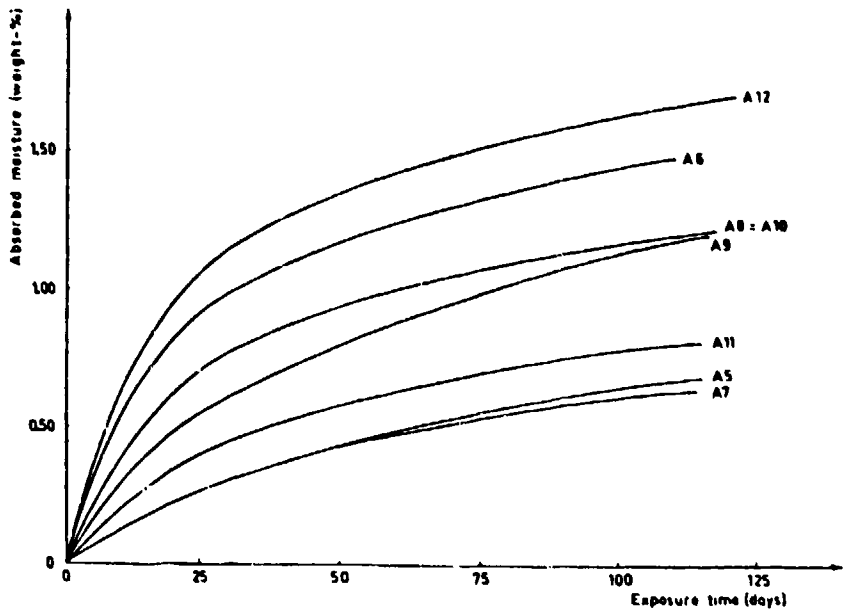

FIG. 23: MOISTURE ABSORPTION OF NARMCO T300/5208 WITH THE FIBRE ORIENTATION $[(\pm 45)_2/45]_{S10}$ AS A FUNCTION OF EXPOSURE TIME[121]

Ai – VARIOUS PRETREATMENTS

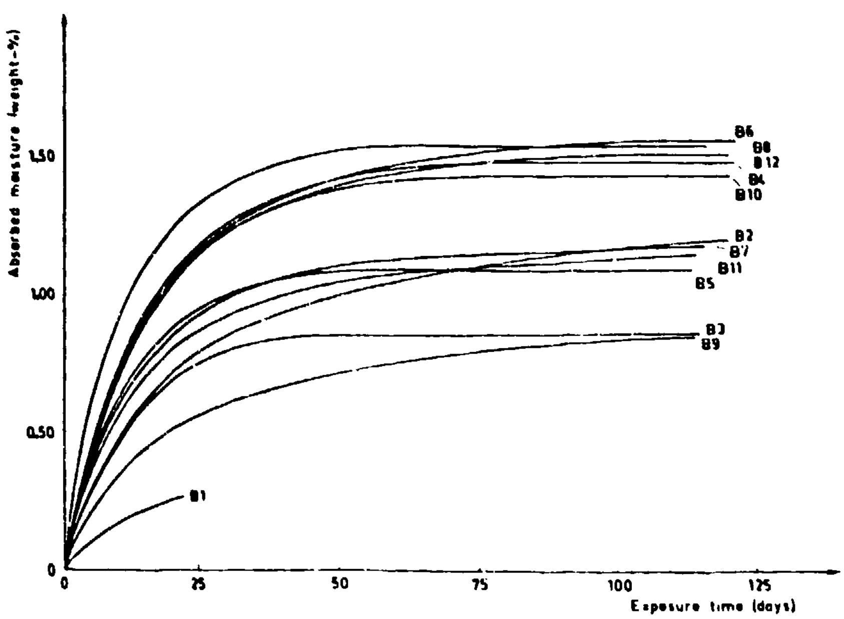

FIG. 24: MOISTURE ABSORPTION OF NARMCO T300/5208 WITH THE FIBRE ORIENTATION $[(0/\pm 45/90)_2]_{S16}$ AS A FUNCTION OF EXPOSURE TIME[121]

Bi – VARIOUS PRETREATMENTS

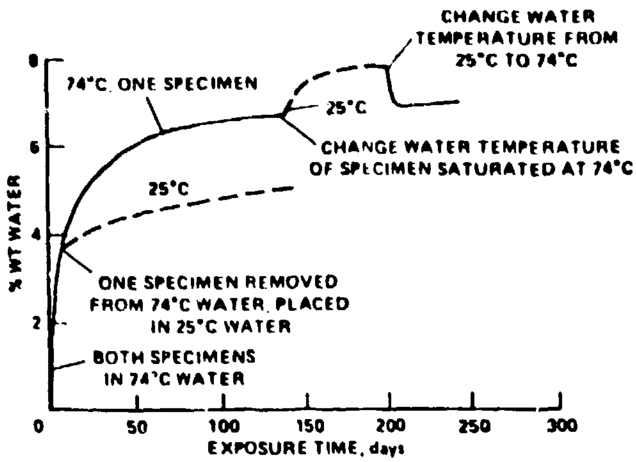

FIG. 25: REVERSE THERMAL EFFECT. HERCULES 3501 RESIN, TWO SPECIMENS, FULLY IMMERSED. SOLID LINE REPRESENTS WATER SOAK AT 74°C, BROKEN LINE REPRESENTS WATER SOAK AT 25°C. BOTH SPECIMENS INITIALLY IMMERSED IN 74°C WATER[4]

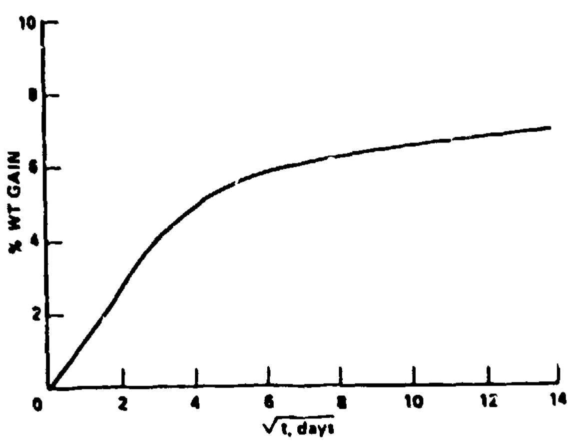

FIG. 26(a): ABSORPTION CURVE, HERCULES 3501 RESIN SPECIMEN FULLY IMMERSED IN 74°C WATER[4]

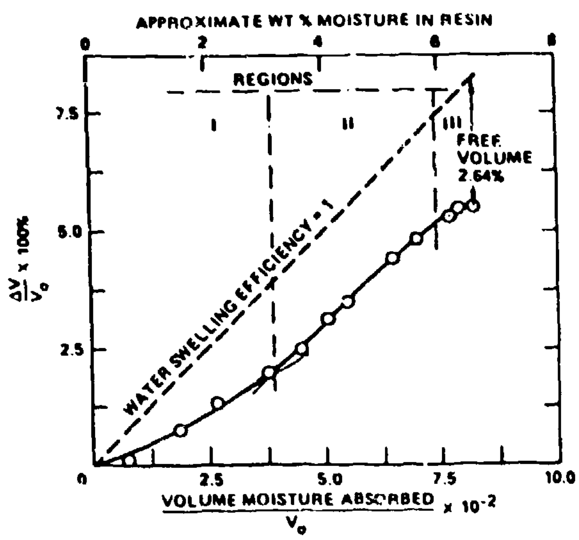

FIG. 26(b): SWELLING EFFICIENCY OF HERCULES 3501 RESIN IMMERSED IN 74°C WATER[4]

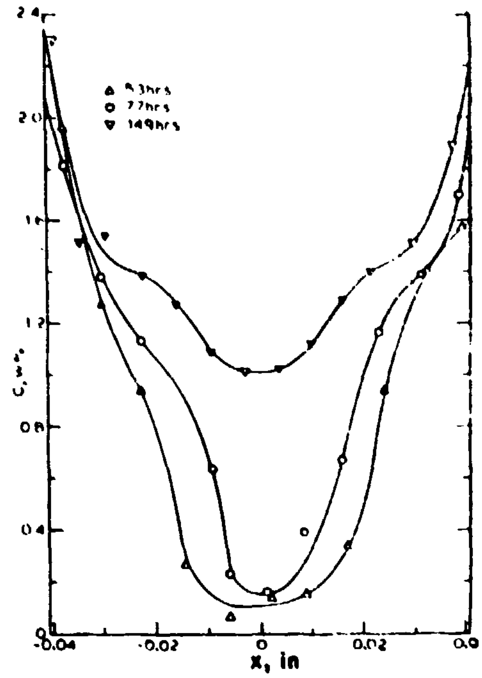

FIG. 27: MOISTURE DISTRIBUTIONS IN UNI-DIRECTIONAL COUPONS DURING SECOND ABSORPTION OF FIGURE 5. THE DISTRIBUTIONS ARE CHARACTERISTIC OF CRANK'S MODIFIED CLASS II DIFFUSION[282]

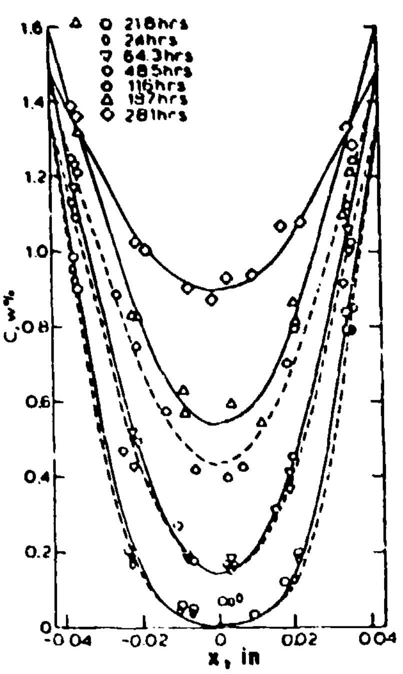

FIG. 28: MOISTURE DISTRIBUTIONS IN ANGLE-PLY TEST COUPONS FOLLOWING ABSORPTION AT 150°F/98% RH[282]

APPENDIX A — BIBLIOGRAPHY

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REPORT DOCUMENTATION PAGE / PAGE DE DOCUMENTATION DE RAPPORT

REPORT/RAPPORT	REPORT/RAPPORT
NAE-AN-4	NRC No. 20974
1a	1b

REPORT SECURITY CLASSIFICATION CLASSIFICATION DE SÉCURITÉ DE RAPPORT	DISTRIBUTION (LIMITATIONS)
2 Unclassified	3 Unlimited

TITLE/SUBTITLE/TITRE/SOUS-TITRE

Hygrothermal Effects in Continuous Fibre Reinforced Composites
4 Part I: Thermal and Moisture Diffusion in Composite Materials

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SERIES/SÉRIE

Aeronautical Note

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NOTES

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DESCRIPTORS (KEY WORDS)/MOTS-CLÉS

1. Carbon fibre reinforced plastics
2. Composite materials

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SUMMARY/SOMMAIRE

This report is the first in a series of literature reviews in which hygrothermal effects on aerospace composite materials (CM) are examined. This first report (Part I) deals primarily with fundamental aspects of the diffusion of moisture into, and from, composite materials. The effects of temperature under both steady state and transient conditions are also examined.

Subsequent reports in this series will deal with the following topics:

Part II: Physical Properties
Part III: Mechanical Properties 1
Part IV: Mechanical Properties 2
Part V: Composite Structures and Joints
Part VI: Numerical and Analytical Solutions
Part VII: Summary of Conclusions and Recommendations

A bibliography has also been prepared to serve as a source of further information. It will also serve as a reference list for the various reports in this series, and therefore it is included as an appendix.