

# NON-DESTRUCTIVE MEASUREMENT OF THE MOISTURE CONTENT IN FIBRE REINFORCED PLASTICS

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The polymers which are used as the matrix in fibre reinforced plastics composites can absorb several percent of water by weight and this can have an adverse effect on the structural integrity of the composite. It is therefore important that the moisture content of such materials can be measured, preferably by non-destructive means. This paper reviews the techniques by which sensitive, reliable and quantitative measurements may be achieved.

## 1. Introduction

Epoxy resins can absorb several percent of water by weight, dependent on the chemical composition. The equilibrium water content and the rate of water absorption are controlled by the relative humidity and temperature of the component. The rate of water absorption increases with increasing temperature. These effects can be reversed almost completely, by heating in a vacuum or dry gaseous atmosphere.

There are four routes by which water might be absorbed into a composite<sup>(1)</sup>:

- absorption by the (resin) matrix
- penetration through surface crazes and cracks
- wicking along fibre bundles through capillary action
- moisture absorption by the reinforcement (limited to aramids)

The absorbed water produces a volume increase in the resin and reduces the elastic moduli and glass transition temperature. Matrix cracking can be induced by non-uniform volume changes. The fibres constrain longitudinal expansion and hence volume changes are predominantly realised in the unreinforced directions (especially the through-plane direction). Such effects may offset the stresses produced by differential thermal contraction between the matrix and fibres during curing.

Browning *et al.*<sup>(2)</sup> reported that the resin strength was reduced by an increase in moisture content, although the failure strain may be enhanced. The presence of moisture would be expected to have the greatest effect on matrix-dominated properties. In unidirectional carbon/epoxy composites moisture does not impair the axial properties, but transverse and shear strengths and moduli are reduced.

Moisture intrusion caused by environmental exposure for extended periods is known to have an adverse effect on the structural integrity of polymer matrix composite materials<sup>(3)</sup>. Diffusion through the organic matrix leads to degradation of the polymer structure, and moisture migration along the fibre-matrix interface weakens the interfacial bond.

Moisture absorption and drying are both very slow processes<sup>(1)</sup>. Ideally, (for repair) composites should be dried to <1% weight content of moisture. For high performance composites (volume fractions of fibre around 66%) a measured moisture content of 1% will indicate a resin moisture content of 3%, when the fibres are not

expected to absorb water. Paper tissues over the area to be dried will indicate water coming from the resin. A soaked part may need three days of continuous vacuum, lamp and heat treatment at 50°C to become dry enough to repair.

The leisure marine industry has a prominent problem with excess moisture in boat hulls. Moisture may be attracted into the resin matrix by 'osmosis', which may result in localised pressure which in turn leads to blister formation. Usually these unsightly defects are found in the gel coat or at the interface between the structural laminate and the gel coat. Removal of water prior to repair is relatively easy and should only take a few weeks even when a thick laminate is encountered.

However, even extensive drying in 'desert' conditions may not prevent a recurrence of the problem. This is due to failure to remove other hygroscopic compounds almost always present in an osmotic laminate. The main culprits are the 'glycols' which may be present as residual monomer, products of resin hydrolysis, viscosity regulators or pigment dispersion media<sup>(4)</sup>. Acetic acid may also be present from degradation of the PVA emulsion binder on the reinforcement mats.

Before the advent of commercial electronic instruments, the only test for moisture in a structure was to tape a piece of polyethylene sheet over an area of the laminate. If an 'adequate' level of moisture was present then condensation would be visible. Although cheap and simple the test would only work if the laminate temperature was higher than ambient temperature, and even then was insensitive, unreliable and not quantitative.

## 2. Electrical measurement

Springer and Shen<sup>(5)</sup> examined unidirectional and ( $\pi/4$ ) T300/Fiberite 1034 composites in the temperature range 300-425 K with the material either in moist air (humidity of 0-100%) or submerged in water, during a study of moisture absorption and desorption. Attempts were made to correlate the moisture content of the material with the Rockwell hardness (0-2.5% reduction in hardness over the range 0-1.5% moisture with around 2% scatter for  $\pi/4$  specimens only: unidirectional specimens cracked) and with changes in the electrical resistance. In the pressure range 2.7-8.3 MPa the electrical resistance varied randomly between 250 and 4000  $\Omega$ , with no repeatability. At constant pressure (3.4 and 5.2 MPa) the resistance was measured at 10 minute intervals for 1 hour and again varied randomly (between 50 and 4000  $\Omega$ ). This random behaviour was observed in both unidirectional and  $\pi/4$  specimens.

Belani and Broutman<sup>(6)</sup> studied the dependence of the electrical resistivity of SP-313 prepreg unidirectional carbon fibre reinforced epoxy resins exposed to moisture or water. Measurements were carried out in three orthogonal directions (Figure 1). The laminates were found to swell transverse to the fibre direction upon absorption of moisture. Their swelling affects the contact pressure between adjacent fibres and results in a change in the effective length of the conducting path. The electrical resistance transverse to the fibre direction thus increases with increasing moisture content. The macroscopic material behaviour in the transverse direction is homogeneous, as the equipotential direction in such laminates is the fibre orientation. The electrical resistance in the transverse directions



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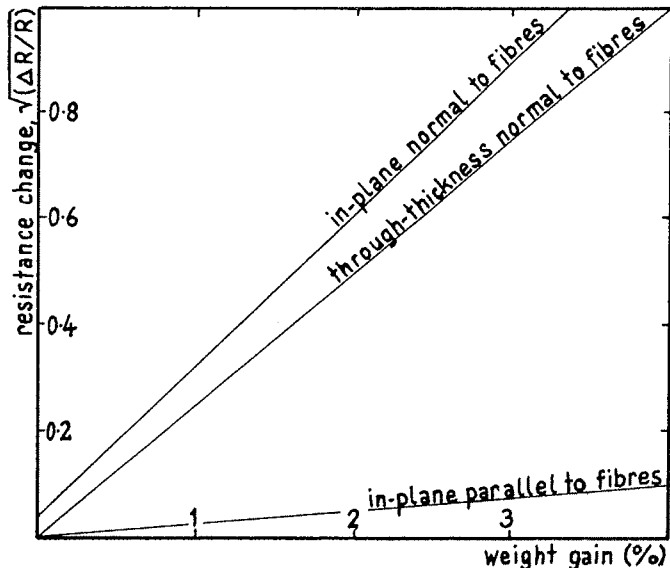


Figure 1  
Resistance change behaviour of SP-313 prepreg unidirectional carbon fibre reinforced plastics with varying moisture content (data reproduced by permission of Prof L J Broutman and the publishers, Butterworth Heinemann Ltd ©)

was shown to be proportional to the square of the moisture content.

Delmonte<sup>(7)</sup> used ASTM method C-611 to determine the volume resistivity of 60 v/o T300 carbon fibre/epoxy panels. There was little change in these values after high-humidity or high-temperature exposure, as summarised in Table 1.

Table 1: Volume resistivities of CFRP after humidity and temperature exposure<sup>(7)</sup>

680 mΩm	As Received
630 mΩm	After 48 h at 175°C
650 mΩm	Further 72 h at 23°C and 96% RH
650 mΩm	As Received, then 72 h at 23°C and 96% RH

Cotinaud *et al*<sup>(8)</sup> and Bunsell<sup>(9)</sup> studied the effects of water absorption on the electrical and dielectric properties of glass fibre reinforced epoxy resin between 0 and 100% RH, and in immersion, in the temperature range 25-90°C. The bisphenol-A epoxy resins were cured with three different hardeners; diamino diphenyl sulphone (DDS), dicyandiamide (DICY) and phthalic anhydride (PA). When dry the electrical resistivity of such glass/epoxy materials is greater than 10<sup>17</sup> Ωm. The absorption of water increased the conductivity by many orders of magnitude. The behaviour of the three materials was different, but above a threshold level of 0.6% water a continuous current was detected when a voltage of 1 kV was connected to each composite. An interconnecting network of free water molecules in the material is implied by this result.

Singh *et al*<sup>(10)</sup> have studied the temperature dependence of the DC conductivity of woven glass/epoxy laminates before and after exposure of the materials to moisture (and to various exhalates released by electronic packaging materials). The DC conductivity increases with increasing temperature. At a particular temperature the DC conductivity increases after exposure to moisture. This increase is greater at longer exposure times. The results have been explained on the basis of Fickian absorption of moisture.

### 3. Dielectric measurement

Fyall<sup>(11)</sup> discussed the poor stability of laminates in moist conditions. The dry state was defined as that at which the loss factor was constant during further drying. The materials tested are listed in Table 2. After

Table 2: Materials subjected to dielectric testing by Fyall<sup>(11)</sup>

LAMINATE	COMPOSITION
A	33 w/o rayon felt in polyester resin
B	ECC 181 glass cloth in epoxy resin
C	ECC 181 glass cloth in epoxy resin 1001 (RN48)
D	Glass fibre in Araldite epoxy resin
E	High-alkali glass cloth in polyester resin (various surface treatments)
X	Low-alkali glass laminates corresponding to laminate E

exposure to excess moisture many laminates did not regain their original values of dielectric constant or loss tangent. The moisture resistance of laminates B, C and D, after equal periods of wetting, was comparable. The moisture uptake of laminate E after 24 h at 40°C and 100% RH was so great that electrical measurements could not be made. Tests on laminates A and D ran to around 400 h and on laminates B and C over 1000 h.

Gagne and Outwater<sup>(12)</sup> reported a portable electronic moisture detector for the Naval Research Laboratory. The battery-powered instrument measured the permittivity of the epoxy resin. Abel<sup>(13)</sup> at NRL used an adaptation of a standard 'Q' meter to monitor the effects of an operational humid environment on glass/epoxy structures.

Delmonte and Sama<sup>(14)</sup> used a dielectric test to observe the extent of penetration of various acids and alkalis into glass-fibre reinforced epoxy laminates. Measurements were made not only on the immersed portion of the specimen, but also on the area outside the liquid. A continuous gradient of dielectric constant results was observed along the sample. The effects of exposed glass fibres, resin viscosity and laminating conditions of fibreglass laminates, in contact with liquid chemicals, were evident from the dielectric measurements.

Delmonte and O'Neal<sup>(15)</sup> studied the absorption of water in amine-cured epoxy laminates by changes in the weight and in the dielectric constant and dissipation factor. Changes after the immersion of laminates in acid were much more rapid than anticipated from cast resin results. Changes in the dissipation factor were most prominent during the first hour, particularly in acid and alkaline solutions. Sodium hydroxide solution gave the most pronounced changes in dielectric constant.

Kays<sup>(16)</sup> embedded parallel wire capacitive sensors in the midplane of three-ply graphite/epoxy laminates exposed to hygrothermal environments. Moisture contents were determined by weighing at discrete times during exposure. Simultaneous capacitance readings were converted to a value for moisture content. Subsequent tests were conducted with sensors at six positions in a 13-layer composite. A good correlation was found between the moisture content at the sensor location and the change in capacitance. The surface sensors in the thick laminate indicated higher moisture contents, close to saturation values.

Latishenko and Matis<sup>(17-20)</sup> used the Riga 7211 device to measure the dielectric constants at 1 MHz. Values were in the range 2.5- 3.0 for polymeric fibres (cellulose hydrate, polyester, polyvinyl alcohol and polyimide) and in the range 3.0-3.8 for the corresponding matrix materials (epoxide, polyester and polyimide). A family of lines was presented, showing a linear dependence of dielectric constant on the volume fraction of reinforcement fibre, on porosity and on moisture content at different initial values of dielectric constant. The determination of the moisture content could be used to indicate the extent of damage, as the accumulation of microcracks increases the ability to absorb water. Latishenko, Matis and Sandalov<sup>(20)</sup> described

a subsequent Type 7211 dielectric spectrometer which records the charge (polarisation) or discharge (depolarisation) current of the capacitance and has integral computer processing of the signal with harmonic Fourier analysis.

Cotinaud *et al*<sup>(21)</sup> and Bunsell<sup>(22)</sup> studied the effects of water absorption on the dielectric properties of glass-fibre reinforced epoxy resins between 0-100% RH and in immersion, in the temperature range 25-90°C (Figure 2). The bisphenol-A epoxy resins were cured with three different hardeners; diamino diphenyl sulphone (DDS), dicyandiamide (DICY) and phthalic anhydride (PA). Three distinct mechanisms of water absorption were observed in all three resin systems. The first mechanism corresponded to simple diffusion of the water molecules into the composite. Changes in properties were reversible, although leaching of the PA-cured composite was observed. The second mechanism for water absorption was observed at absorbed water levels above 0.6%. Large increases in the relative permittivity and loss factor were observed together with electrical conduction (see above). The third mechanism, seen only in immersion, was the transport of water by capillary action along microcracks in the matrix. The changes in dielectric properties were found to be directly related to the quantity of water in the composite, whereas changes produced in the mechanical properties were always irreversible.

Marsh *et al*<sup>(23)</sup> used dielectric loss techniques to study the moisture absorption-desorption kinetics and equilibria in glass/epoxy

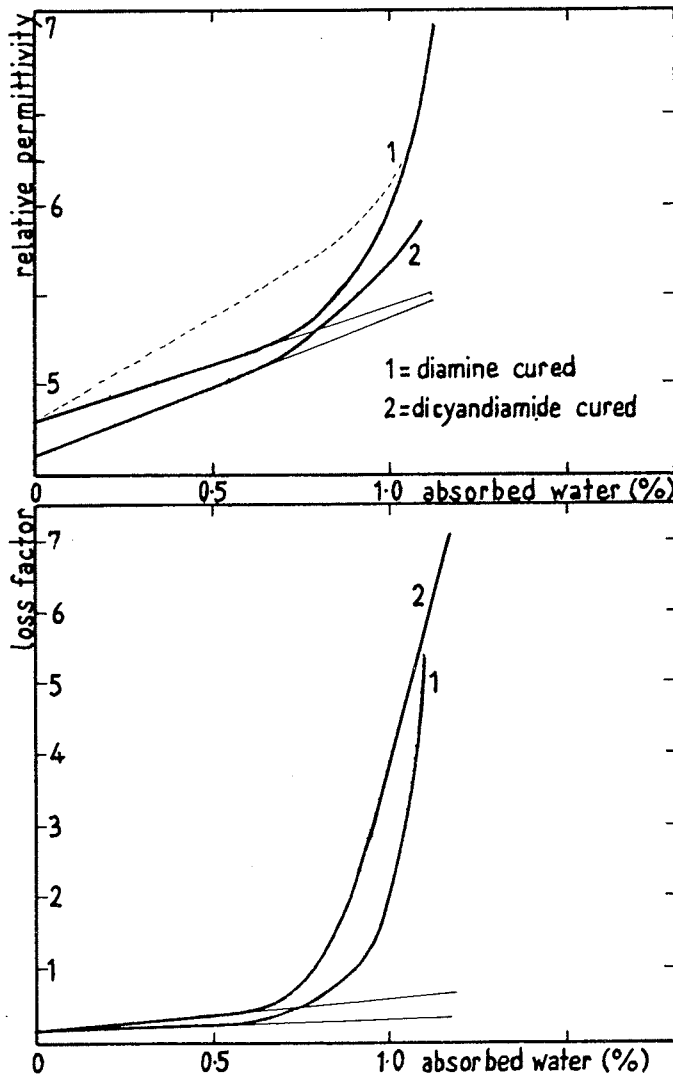


Figure 2

Variation of relative permittivity and loss factor with absorbed water content for an epoxy resin cured with different hardeners (data reproduced by permission of Prof A R Bunsell and the publishers, Chapman & Hall Ltd ©). Solid lines are for material exposed to water vapour, dashed line is for immersed material

composites and printed circuit boards. Measurements were made at a temperature of 90°C and a frequency of 200 Hz to maximise the sensitivity to field-induced polarisation processes with the relaxation losses at maximum. The measurements were a reproducible predictor of moisture content at equilibrium with the external environment. The kinetics of moisture diffusion as indicated by dielectric methods were consistent with kinetics determined by the traditional methods. The capacitance was found to be essentially independent of frequency and moisture content. The change in dielectric loss with moisture content could thus be attributed to a change in the dc resistance.

Singh *et al*<sup>(24)</sup> studied the dielectric behaviour of epoxy and polyester laminates before and after exposure to moisture (Figure 3) and various exhalates which may be released by electronic packaging materials. A strong dielectric dispersion was observed after exposure, which was reversible after moisture exposure for both types of laminate.

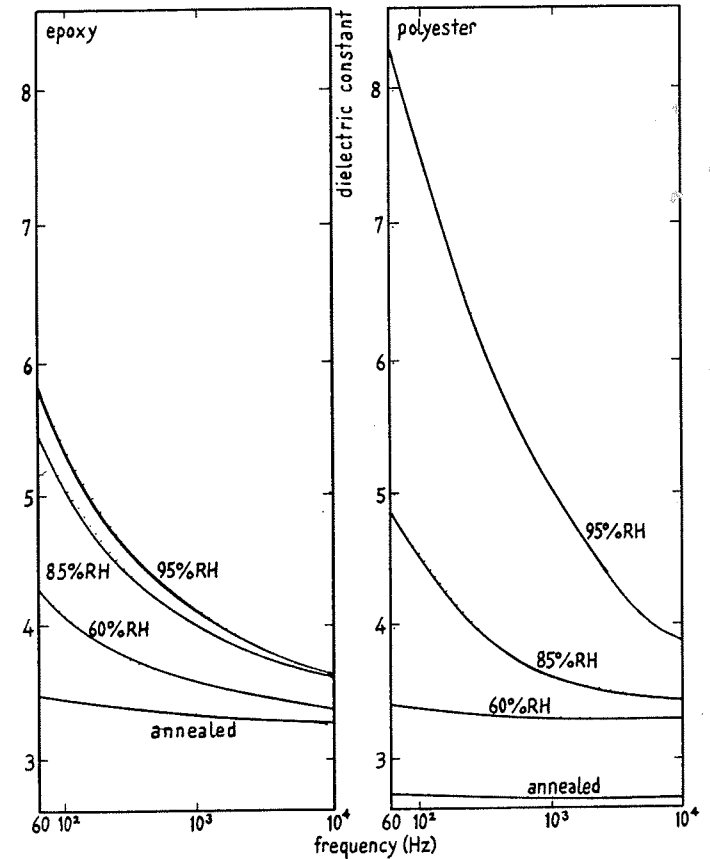


Figure 3

Frequency dependence of dielectric constant for epoxy and polyester matrix laminates as annealed or five-day moisture exposed samples (data reproduced by permission of Dr D B Singh and the publishers, Chapman & Hall Ltd ©)

Barton *et al*<sup>(25)</sup> studied the dielectric properties of a cross-linked polyether thermo-setting resin as a function of absorbed moisture, and made comparisons with a standard epoxy resin. The polyether had a much lower moisture absorption capacity than the epoxy resin and its dielectric properties were less affected by humid atmospheres. However, after taking account of the low moisture absorption capacity of the polyether, the dielectric response was greater than expected, which may be due to plasticisation of the resin by the water.

#### 4. Commercial moisture meters

The original electronic instruments were sensitive resistance meters, measuring electrical conductivity between two sharp metal probes pushed into the laminate surface. They were often set up for timber and/or masonry and did not possess the necessary accuracy and sensitivity for use on GRP. Also the sharp points on the probes could induce damage in the laminate.

Nearly all commercial moisture meters currently available monitor the electrical capacitance and/or loss of signal strength at high (radio -) frequencies<sup>(1, 26)</sup>. Typical material properties are listed in Table 3.

**Table 3: Dielectric Constants of Materials used in Composite Materials**

Material	Dielectric Constant
Water	c.81
E-Glass	6.13
Aramid	3.85
Epoxy Resin	3.62
Polyester Resin	2.7
Polyethylene	2.3

The dielectric constant, relative permittivity and loss factors can be calculated using the rule of mixtures, although contributions from fibres, resin and the interface should be included. As the water content rises, the electrical capacitance between the electrodes rises.

Moisture meters have been used for some years<sup>(1)</sup> to monitor aircraft radomes, where excess moisture reduces the radar signal strength and degrades the pilot's picture of approaching aircraft or storms. They are unsuitable for monitoring CFRP, because it conducts electricity.

Moisture meters can give an indication of bulk moisture content. Unfortunately these meters do not show moisture content directly as a percentage: they are set up to read 'green' (good), 'orange' (medium) and 'red' (wet). Surfaces must be dry before moisture checks: false readings can be obtained in areas which have been in contact with moist hands.

Moisture meters can be unduly sensitive to moisture at or close to the laminate surface, yet insensitive to moisture more than 2- 3 mm below the surface. As the instruments operate at different frequencies there can be considerable variation from one instrument to another. In some instruments the calibration facility can only compensate for varying battery voltage. An ability to adjust operating frequency to compensate for low power operation may be a critical requirement, especially for capacitance-type meters<sup>(4)</sup>.

The design of electrode spacings can be critical to instrument sensitivity. A good meter may be able to indicate readings up to 12 mm away from the electrodes. In tests on 4 mm thick GRP panels conducted for International Paints in Southampton one instrument gave near identical readings on both sides of the panel, while the other (which did not give average moisture content) indicated 25% on the wetted side and totally dry on the reverse.

One popular meter<sup>(26)</sup> uses a remote scanning head containing two concentric metal rings. The meters are very sensitive, but tend to be weighted towards surface (coatings) or near surface moisture rather than the main body of the laminate.

An alternative instrument (the Traxex Skipper) has two co-planar conductive rubber electrodes set into the body of the meter a fixed distance apart and measures the resistance between 5-25 kHz low frequency AC signals. The meters can detect moisture in laminates up to 15 mm thick and can give a good indication of the average moisture content throughout the laminate thickness. Clegg<sup>(26)</sup> suggests that the instrument is both sensitive and accurate, and 'gives by far the most meaningful indication of hull moisture content'.

Readings for all these instruments may be influenced by metal skin fittings, bulkheads, tanks, water in bilges and fillers (eg aluminium hydroxide retains moisture).

## 5. Microwaves

Microwaves are strongly absorbed and scattered by water molecules, because of a broadband rotational relaxation in the microwave

region. Many materials are transparent in the microwave band when completely dry, but became opaque to microwaves when free (unbound) moisture is absorbed. Water is a polar molecule and has dielectric properties which are a function of temperature.

Kaelble and Dynes<sup>(27)</sup> used NMR spectroscopy, dynamic mechanical spectroscopy (1-100 Hz), ultrasonic wave velocity and attenuation (2.25 MHz), microwave dielectric constant (2.64 GHz) and microhardness measurements for the study of moisture degradation in graphite/epoxy composites. The microwave dielectric properties were evaluated from perturbation measurements of cylindrical rod samples in a right-cylindrical microwave resonant cavity. The measured values are extremely high ( $\epsilon' = 850$ ) and exhibited a 2% frequency shift between dry and moisture aged (maximum of 2.7% by weight of water) composites.

Umeda *et al*<sup>(28)</sup> used a microwave system to study the extent of absorbed water in GFRP. The test piece (40 x 30 x 15 mm) was boiled in water and measured after removal of water from the surface. The measuring point moves in a counter-clockwise direction on the polar diagram as the volume of absorbed water increases, since both  $\epsilon$  and  $\tan \delta$  increase with the water content. The shift of phase angle,  $\Delta\Phi$ , increases continuously during the boiling period for bulk resin; however in the glass-fibre composite  $\Delta\Phi$  initially increases, but decreases after 4 h. The flexural strength also decreases after boiling for 4 h, and this is attributed to the generation of interfacial cracks.

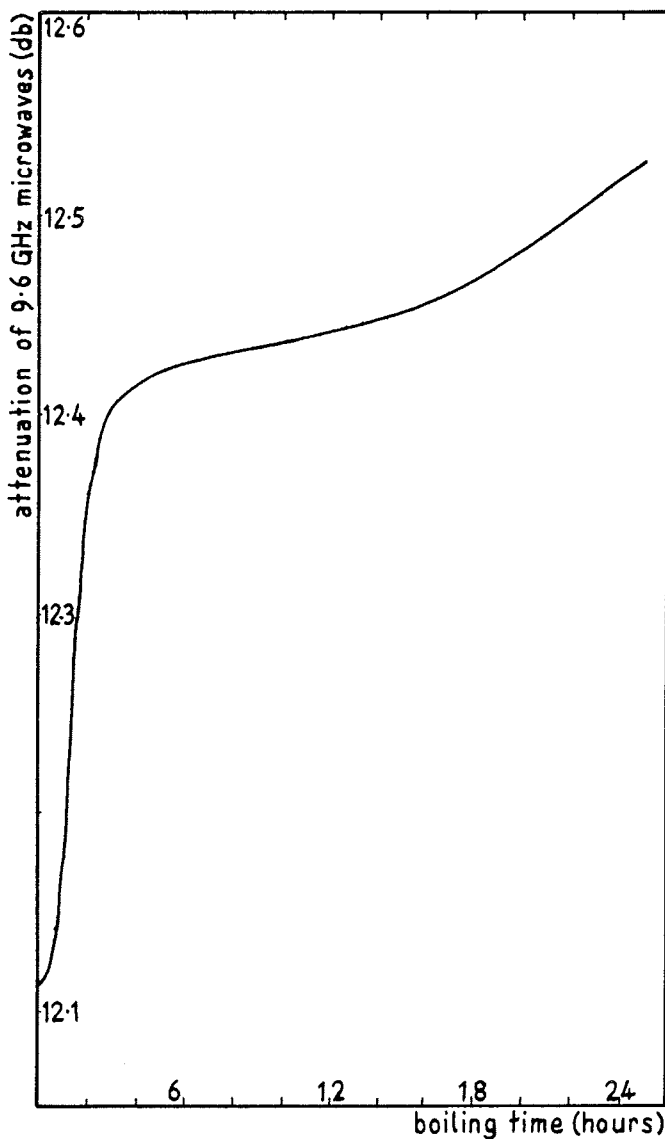
King and Stiles<sup>(29, 30)</sup> used an open-ended coaxial 6 GHz microwave resonator to determine the feasibility of monitoring moisture, which may in turn be a diagnostic indicator of the structural integrity and ageing of composites. The initial experiments were conducted on granulated dielectric materials and dry organic pith. Latorre and King<sup>(31, 32)</sup> calibrated the system using triethylamine in which moisture was injected in precisely known quantities. The homodyne detection system could measure changes in water content of the model materials to less than 100 parts per million.

Carr<sup>(33)</sup> investigated two microwave methods to detect absorbed water in carbon-fibre reinforced epoxy resin. Detection was feasible by the measurement of attenuation at 9.6 GHz. An insertion loss of around 60 dB  $\text{cm}^{-1}$  was obtained for radiation polarised perpendicular to the fibre direction. A change of 0.4 dB after boiling for 25 h was found in a 2 mm thick sample (Figure 4). Measurements of the permittivity of the sheet were impossible because of the high effective conductivity, estimated at 10 ( $\Omega\text{m}$ )<sup>-1</sup>.

## 6. Infrared

Boulay *et al*<sup>(34)</sup> measured the far-infrared (570  $\mu\text{m}$  wavelength) transmissivity of glass and polyester fibre reinforced epoxy sheets with differing percentages of absorbed moisture. The beam from a methanol laser was transmitted through such sheets with only small scattering losses, while the water absorption coefficient was more than an order of magnitude greater than that for dry material. An empirical approach based on a single-line transmissivity measurement was proposed as a fast and simple alternative to the spectroscopic approach for the bulk moisture analysis of polymeric materials.

Trebor Industries (USA)<sup>(35)</sup> have recently introduced a battery-powered hand-held moisture meter which uses 'advanced near-infrared technology'. The near infrared spectrum is normally defined as between the wavelengths 750-2600 nanometers. The instrument uses a rotating optical filter to select wavelengths with low reflectance (1200, 1450, 1940 nm) or high reflectance (1300 nm) in the presence of moisture<sup>(36)</sup>. The reflected signal is monitored by a lead sulphide detector. The moisture content is proportional to the ratio of absorbed to reference light levels. The meter is a non-contact device with an indicator to allow the maintenance of a constant 150 mm (6") distance from the sample being measured. Examples of moisture content values in synthetic polymers include nylon pellet (0.5-5%), polyethylene film (0.2-9%) and polycarbonate board (0.1-4%).



**Figure 4**  
Variation in the attenuation of 9.6 GHz microwaves with boiling time for a unidirectional EXAS carbon fibre reinforced epoxy resin with 50% by volume of fibres and 2.5% void content. The initial high absorption rate was attributed to a resin-rich surface layer (data reproduced by permission of Mr A D Carr and publishers, Technomic Publishing Co Inc ©)

## 7. Nuclear magnetic resonance

Nuclear magnetic resonance in a complex molecule is generally achieved by placing the material in a strong constant magnetic field, and then applying a perpendicular radio-frequency (RF) alternating magnetic field. The nuclei absorb or emit energy at characteristic frequencies of the RF field, and the amount and frequency of this energy can indicate the chemical structure of the material under test<sup>(37)</sup>.

The Bohr concept of the atom comprised negatively charged particles in orbits around a heavier positively charged nucleus. On this classical model, a particle of charge  $e$  and mass  $m$ , describing a circular orbit of radius  $r$  with a velocity of  $v$ , will possess an angular momentum  $p$  and a magnetic moment  $\mu$  which are related<sup>(38)</sup> by the equations:

$$p = mvr \quad (1)$$

$$\mu = \frac{1}{2} \cdot evr \quad (2)$$

The gyromagnetic ratio of these two parameters,  $\gamma$ , is independent of the orbital details; thus:

$$\gamma = \frac{\mu}{p} = \frac{e}{2m} \quad (3)$$

and in the presence of a magnetic field  $B$  the orbital particle is subjected to a torque proportional to  $\mu$  and  $B$ . This results in a precession of the axis of rotation about the direction of the magnetic field with a frequency inversely proportional to  $p$ , such that:

$$\omega_0 = \frac{\mu B}{p} = \gamma B \quad (4)$$

where  $\omega_0$  is the characteristic frequency, known as the Larmor frequency.

According to classical theory, the precessing particle should continuously lose energy, so that the orbit spirals inwards until  $\mu$  and  $B$  become parallel. However, quantum theory indicates that only certain orbits are permitted and that transfer between these orbits is accompanied by a quantum change of energy. Any particle with spin should behave in a similar manner to the orbiting particle and have a characteristic Larmor frequency. If a large number of nuclei are forced to change simultaneously from one spin energy state to another, then a measurable induced magnetic field may be detected. By steadily varying either the magnitude of the magnetic field  $B$  or the frequency of the radio frequency field  $\omega$ , the condition at which the nuclei change state may be detected as a weak RF signal or as a minute loss of power in the driver circuit. These two methods of detection are known respectively as nuclear induction or nuclear resonance absorption. The general term, nuclear magnetic resonance, abbreviated to NMR, is applied as a name for both manifestations. In physical systems changes of state do not occur simultaneously, and decay or relaxation times are influenced by the environment of the nucleus. In NMR, two major groups of decay times are distinguishable:

$T_1$ : the spin-lattice interaction, or longitudinal relaxation time; and  
 $T_2$ : the spin-spin interaction, or transverse relaxation time.

The frequency of precession is an atomic constant characteristic of a particular nucleus, but the relaxation times are determined by the experimental conditions. Factors affecting the relaxation times include the uniformity of the applied field, shielding due to the bulk of the material, the time to achieve thermal equilibrium in the specimen and the boundary conditions arising from the sample container.

If the nuclear spins in a sample are aligned with a uniform magnetic field  $B_0$  and a stronger magnetic field normal to  $B_0$  is applied ( $B_{90}$ ), then the nuclear moment will deflect; this is identified by the spin-lattice relaxation time  $T_1$ . The removal of the transverse magnetic field allows the nuclei to realign with the original uniform field  $B_0$ , identified with the spin-spin relaxation time  $T_2$ . In the realignment with the original field, the nucleus will perform the characteristic Larmor precession.

Recent advances in NMR spectroscopic techniques have led to the ability to measure chemical shifts in solids<sup>(39)</sup>. Combined with FT (Fourier Transform) electronics, cross polarisation (CP) and magic angle spinning (MAS), this has led to a massive expansion of the capabilities of NMR.

Kaelble and Dynes<sup>(27)</sup> have reported that Witt has shown that the presence of moisture affected both the free induction decay (FID) and the  $T_1$  and  $T_2$  relaxation times in graphite/epoxy composites. The NMR technique indicated that the absorbed water was tightly bound to the epoxy resin, which is in agreement with the high temperature (>120 °C) required for internal moisture to affect the molecular motion and low frequency damping of the composite.

King *et al*<sup>(40)</sup> used a 30 MHz transient hydrogen-NMR system to study a single graphite/epoxy specimen. The dried specimen had a single free induction decay time of 8  $\mu$ s. After exposure to 8% RH

at 52°C, the 8 μs part of the FID did not change, but a component with a longer relaxation time occurred which increased with the duration of the exposure (10 μs after 24 h, 24 μs after 72 h and division into two relaxation times after 336 h). A weight loss method indicated 0.8% moisture content in the composite after 336 h.

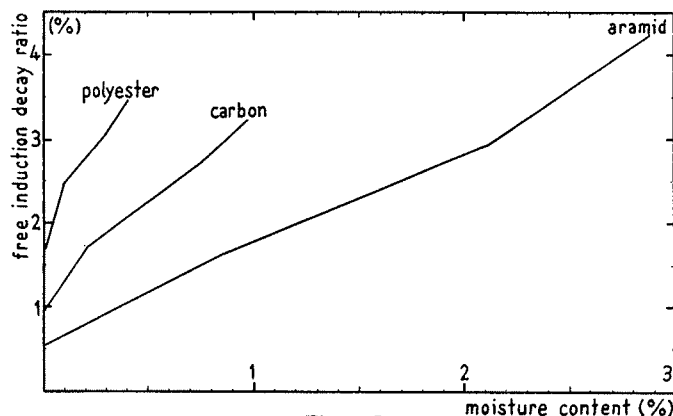
Matzkanin<sup>(41, 42)</sup> conducted an investigation of the feasibility of using 30 MHz pulsed hydrogen NMR to non-destructively determine the amount of moisture in organic matrix composites and the extent of moisture-induced mechanical degradation. Two composite material systems were studied: 8 ply ± 45° S2-glass fibre in SP-250 resin and Kevlar 49 Fibre in Reliabond 9350 resin. Long-term environmental conditioning for four months at 95% RH and 52°C was undertaken. Reductions in tensile strength were 4.3% in a fibreglass with 1.3% moisture content and 14% in a Kevlar composite with 4.6% moisture content. Absorbed moisture levels as low as 0.2% produced measurable NMR signals, consisting of distinct multiple components attributed to moisture in various states of molecular binding. Good correlation was obtained between the NMR signal output and the dry-weight moisture percentage. Experiments on fractured specimens, subjected to additional environmental exposure, showed that NMR is capable of distinguishing the free moisture entering a composite through cracks and fissures from that moisture absorbed into the composite structure. Analysis of the NMR signals indicated that plasticisation of the resin matrix, rather than microcracking, was the most likely cause of mechanical degradation. Experiments on the fibres showed that no proton-NMR signal was observed from the glass fibre, although moisture absorbed by the Kevlar fibre could be detected.

The important feature of the Matzkanin NMR results is that not only can the amount of moisture be determined, but the method can also distinguish between water in different binding states. Pulsed NMR free induction decay (FID) signals were analysed following a 90° RF pulse. This measurement allows a determination of the nuclear spin-spin relaxation time ( $T_2$ ) which provides information on molecular coupling and on nuclei in different chemical states. Signal average techniques were used to reduce the signal-to-noise ratio. The NMR FID signals were generally characterised by a large-amplitude fast-decaying component ('solid component':  $T_{21} = 6\mu\text{s}$ ) identified with chemically bound structural hydrogen in the composite, and a slower-decay lower-amplitude component associated with the absorbed moisture. The Kevlar composite (1.36% moisture) exhibited three slower relaxation times ( $T_{22} = 80 \mu\text{s}$ ,  $T_{23} = 157 \mu\text{s}$  and  $T_{24} = 253 \mu\text{s}$ ) whilst fibreglass (0.37% moisture) showed only two relaxation times ( $T_{22} = 28 \mu\text{s}$  and  $T_{23} = 71 \mu\text{s}$ ). The absence of hydrogen from the glass fibre structure is associated with the reduced number of relaxation times. As the moisture levels increase, the time constants also increase, indicating that motion of the moisture is increasingly constrained, as shown in Table 4.

**Table 4: Variation of relaxation times with moisture content**

Kevlar Composite					Parameter	Fibreglass Composite				
0.49	1.36	2	2.83	4.6	% moisture	0.19	0.37	0.62	0.78	1.27
70	75	82	113	178	$T_{22}$ (μs)	13	33	35	45	54
175	157	175	200	230	$T_{23}$ (μs)	62	70	76	99	125
-	210	228	235	245	$T_{24}$ (μs)					

Matzkanin<sup>(43)</sup> has reported results on glass fibre and Kevlar fibre-reinforced plastics, and lists the problems which NMR is capable of addressing as including the measurement of moisture, environmental degradation, modulus variations, degree of cure, impact damage and hydrogen concentration variations. Matzkanin<sup>(44)</sup> has demonstrated the use of one sided NMR using U-shaped magnets and flat detection coils to allow measurements to be



**Figure 5**  
Variation of the free induction decay ratio (%) of the voltage amplitudes at 125 μs/20 μs (after the start of the transmitted radio frequency pulse for the hydrogen nuclei within the NMR sensor) for polyester, carbon and aramid fibre reinforced epoxy laminates, plotted against moisture content (data reproduced by permission of Prof G A Matzkanin and the publishers, Butterworth Heinemann Ltd ©)

made from a single surface. The sensitivity of this approach is illustrated by Figure 5.

Lawing *et al.*<sup>(45)</sup> used a broadline NMR spectrometer to investigate the temperature dependence of water-soaked graphite/epoxy (TGDDM/DDS) cylindrical unidirectional composites. The narrow line consistently observed in the broadline NMR spectra in the temperature range (25°, -42°C) studied arose from an intermediate state between free liquid water and bound solid-state water, probably due to hydrogen bonding with the polymer. As the temperature was lowered, some of the intermediate-state signal became indistinguishable from the broad background line, due to the protons in the resin. The intermediate signal reappeared on raising the temperature, and the water line was still present below 0°C. Ratios of the areas beneath the narrow and broadline NMR absorption were a function of temperature:

Temperature (°C)	25	-5	-20	-42
Area Ratio (%)	8.5	3.3	2.5	2.3

The room-temperature ratio of 8.5% corresponds to a ratio of mobile water in the composite of 1.2% by weight. The spectrum was isotropic upon rotation of the fibre axis with respect to the direction of the externally applied magnetic field, which is in marked contrast to studies of certain other fibre systems reported elsewhere.

Fuller *et al.*<sup>(46-48)</sup> continued the above study and examined four environmental cases: soaked in H<sub>2</sub>O, soaked in D<sub>2</sub>O, dried, and not treated. Proton spectra were obtained at 56.4 MHz fixed radio frequency, with signal/noise enhancement by time averaging. The first group of samples (cured resin only) at room temperature were examined dry and soaked. The dry samples had a single broad peak due to protons in the solid state. The peak width was 10 G. The soaked samples exhibited a broad peak and a narrow line due to the water absorbed by the sample. The narrow line was 1 G wide, which indicated that the water was in an intermediate solid/liquid state. At elevated temperature (68°C) a narrow line became visible for the dry samples, and the water line of the soaked samples was sharpened by the increased mobility of the water.

In Fuller's second group of samples, graphite-epoxy composites, the spectra were measured as the angle between the fibre and the magnetic field was changed. There was no change in the spectra of either dry or soaked samples with angle. It was concluded that either water was only associated with the bulk resin, or water associated with the fibres did not have a preferred orientation. In a third group of experiments, a sample was dried and soaked in D<sub>2</sub>O, whereupon a narrow line appeared in the proton spectrum. It was concluded from this that hydrogen exchange was taking place between the resin and the absorbed water, probably at the hydroxyl group of the opened epoxy ring. The frequency of the band in the infrared spectrum (wave

number 3300) is consistent with a hydroxyl stretching frequency. It may of course be that the exchange occurs in unreacted DDS curing agent.

Batra and Graham<sup>(49)</sup> have shown that continuous wave (CW) NMR can be used to measure small traces (a few mg) of moisture in composites of both graphite-epoxy and glass-epoxy. A radio-frequency magnetic field ( $H_{rf}$ ) of 15.9932 MHz, simultaneous with a static field ( $H_0$ ) of 3.7558 kG, was applied with a parallel sweep field of  $\pm 50$  G.  $H_{rf}$  was orthogonal to  $H_0$ . In composites containing moisture, the protons are loosely bound in the water when compared to the protons in the epoxy matrix. Tightly bound protons have strong coupling of their magnetic moments. Consequently their interactions are strong and the line width is fairly large. For protons in water, the line widths are very small due to the rapid tumbling of the nuclei which largely averages out the dipolar fields generally responsible for broadening the line. The NMR spectra of composites containing moisture consists of a very sharp proton line superimposed upon a very broad background signal. Since the number of nuclei is proportional to the area under the line in the NMR absorption curve, the moisture content in a given sample can be estimated quantitatively from the NMR spectra. A plot of resonance intensity against milligrams of water showed a linear relationship in the range 0-40 mg.

## 8. Positron annihilation

It has long been realised that moisture may affect the mechanical properties of fibre-reinforced plastics. Furthermore, the moisture profile through the laminate thickness can cause large stress gradients due to swelling of the composite. Consequently, it is of much interest to determine the moisture content in all composite structures. For small test specimens it is simple to determine moisture content and diffusion constants by drying the specimen, weighing it and then monitoring weight as a function of moisture absorption. The moisture profile may then be calculated for a larger panel using Fick's diffusion laws. For a real structure, however, there is a need to be able to measure, non-destructively, the moisture content and its depth distribution.

A technique which seems suitable for such measurements is based on the dependence of positron lifetime on the moisture content of the composite specimen<sup>(50-52)</sup>. The positron is the antiparticle of an electron. When a free positron, usually obtained from one of the artificially radio-active isotopes  $^{22}\text{Na}$ ,  $^{58}\text{Co}$ ,  $^{64}\text{Cu}$  or  $^{68}\text{Ge}$ , encounters an electron, the two annihilate each other with a lifetime of 125 picoseconds, emitting a pair of gamma rays. When a positron enters a molecular medium such as a polymer, there are at least two annihilation routes possible<sup>(50, 51)</sup>. There is one short-lifetime component related to direct annihilations with molecular electrons and to the self-annihilation of singlet positronium (parapositronium). The second longer lifetime, of the order of a few nanoseconds, is believed to relate to the annihilation of triplet positronium (orthopositronium) by the so-called pick-off process. This latter delayed positron annihilation includes the effects of interaction of the positronium with the surrounding media and responds sensitively to the changes in the properties of the medium produced by diffusing water.

Singh *et al*<sup>(50, 51)</sup> used a conventional fast-slow coincidence system to measure positron lifetimes for different moisture contents in the studied materials. Positron lifetime is measured with respect to a reference time determined by the detection of a nuclear gamma ray emitted simultaneously with the positrons which were emitted from a  $10 \mu\text{Ci}$   $^{22}\text{Na}$  source. The positron lifetime decreased linearly with the moisture content of the Narmco 5208/T300 carbon/epoxy specimen. For industrial applications, the experimentally determined curve might be used as a calibration curve for determining the moisture content of objects fabricated from the same material.

The positron technique used by Singh *et al*<sup>(50, 51)</sup> appears to have sufficient sensitivity to monitor moisture content in conventional

carbon/epoxy systems, even though the saturation weight percent of water is of the order of only 1%. Thus, for uniformly distributed moisture in these materials continuous-energy positron-emitter radioactive sources, as discussed above, are quite adequate. However, for specimens having nonuniform moisture distribution, the positron annihilation characteristics will vary depending on where the positrons are thermalized. Those positrons which stop in the drier regions of the composite will have different lifetimes from those which stop in moist regions. The resulting spectrum will be a complex mixture of spectra characterised by different lifetimes. Thus, for investigating nonuniform moisture distributions in fibre-reinforced plastics, positrons of appropriate, well-defined energies are needed. The only practical means for producing monoenergetic positron beams of various energies is to analyse, magnetically, positrons emitted from appropriate radioactive sources. Such a technique was used by Singh *et al*<sup>(52)</sup> for a preliminary investigation of moisture distribution in polymers.

## 9. Summary

Electric, dielectric, microwave, infrared and NMR techniques have each been used for the non-destructive measurement of the moisture content in fibre-reinforced polymer matrix composites.

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## 12. Moisture meters

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