NON DESTRUCTIVE ANALYSIS OF COMPOSITE MATERIALS USING DIELECTRIC ANALYSIS

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Abstract

Dielectric measurements are widely used in the laboratory to probe the dynamics of molecules, particularly the dynamics of polymer molecules. The dielectric technique exploits the fact that many molecules, although electrically neutral posses an asymmetric distribution of charges which can be approximated to an electric dipole. The (usually thermal) motion of the molecule can be detected by the interaction of this dipole with a time varying electric field.
The great advantage of the technique is that no transducers or sensors are required; the direct application of an electric field produces a directly measurable electric response over a frequency range of MHz to GHz.
This paper discusses the practical application of dielectric measurements to composite structures and the information that can be obtained on the state of the polymer in polymer composite matrix materials.

1 Introduction

Over the last ten years, the application of the dielectric test method to the study of adhesively bonded structures has been explored at the University of Strathclyde[1-14]. The initial investigations looked at the use dielectric measurements to study the ageing of adhesively bonded aluminium – epoxy - aluminium joints in which the joint formed a waveguide for the propagation of the electrical signal. Study of the electrical impedance as a function of frequency combined with investigation of the reflection coefficient in the time domain allowed exploration of the variation of the dielectric characteristics of the resin in the bond and also the homogeneity of the bond line. Fortunately carbon fibres are sufficiently electrically conducting for them to provide an equivalent structure to allow a similar experiment to be performed to that using the metal bonded structures and whilst the peaks are somewhat broader the technique has been shown to apply equally to carbon fibre –epoxy –carbon fibre bonded structures.

The general conclusions which emerged from the study indicated that there was a correlation between the changes in the dielectric characteristics of the resin and the ingress of moisture into the joint. Water has a high dielectric permittivity and it is relatively easy to see fractions of a percent uptake within the bond line. Water exhibits two relaxation features; the first at approximately 20 GHz and associated with ‘free’ water located in micro fissures and cracks and ‘bound’ water which is molecularly dispersed throughout the resin. The latter is able to plasticize the resin and lowers the glass to rubber transition temperature [T_g] with a consequential decrease in the mechanical strength of the joint. This process is to a large extent reversible and drying the adhesive can lead to a regain of its original strength. Study of the mechanical properties of a range of joint structures has shown that there is a correlation between the changes in the amount of ‘bound’ water and the loss in mechanical strength of the joint.

Time domain reflectometry (TDR) measurements indicate that physical changes occur in the bond line and are associated with the creation of voids as a consequence of internal stresses in the resin. The studies reported have established the utility of the method for both metal and carbon fibre bonded structures.

A challenge for composite scientists is the study of glass reinforced plastic structures which do not contain a natural conducting path for the electrical signal. Studies at Strathclyde and also at Case Western Reserve University[15] and the University of Missouri[16] have explored the direct application of high frequency microwave techniques to the study of defects in non conducting materials. The correlation between gravimetric and high frequency dielectric spectroscopy data has previously been demonstrated using end of coaxial line measurements establishing the necessary link with the previous studies on bonded structures[13].

2 Experimental work

A Teradyne capacitance bridge C357 operating at 1 kHz was used to make low frequency dielectric measurements. Measurements at microwave frequencies were carried out using the network Hewlett Packard 8720C operating over a frequency range 7-12 GHz. The signal was applied to the sample via a tuned resonant cavity with a small aperture at one end. The aperture allows the observation of the evanescent wave interaction with the material as is detected in terms of a change in amplitude and frequency of the resonance. The method used is similar to that used previously[15,16] and allows detection of changes in the permittivity of the material.

3 Examples of dielectric studies of non conducting composites; Low frequency measurements

As indicated above it is possible to observe the ingress of moisture into a composite structure by measurement of the change in the permittivity of material as a function of location. The principal problem with such measurements is that electrodes have to be applied to both sides of the sample under test and this not universally practicable. It is possible to use a single sided inter-digitated electrode structure of the form shown in figure (1) to achieve a penetration of the electric field into the material.

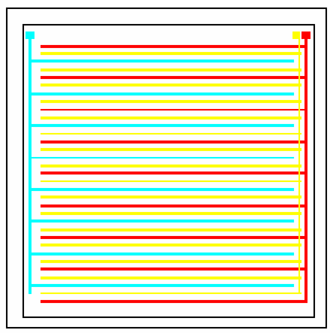

Fig. 1. Inter-digitated electrode structure allowing single sided low frequency measurements of a GRP structure.

By careful design of the electrode spacing it is possible for the field to penetrate to a distance of several millimetres and in principle a depth penetration of greater than 20mm is feasible. A computer simulation of the potential profile is shown in figure (2)

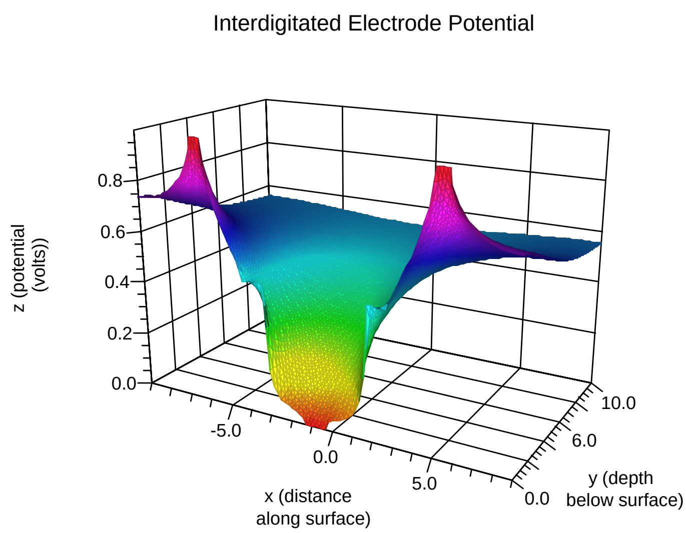

Fig. 2. Inter-digitated electrode potential profile showing field penetration to a depth of several millimetres below the surface.

The advantage of this type of probe is that it allows the characterisation of the dielectric properties of a structure in which access is only possible from one side. This approach has been trialled successfully on the hull of a GRP vessel, figure (3).

Fig. 3. Mine Counter Measures vessel of the type in which measurements were successfully performed.

The technique demonstrated that there were detectable variations in the permittivity as the probe was scanned from above to below the water line and indicated that water had permeated into the GRP. A separate study of the effect of water exposure on the GRP[10] has shown that although the GRP appears to be completely cured, in thick structures such as the hull of a vessel it is possible for several percent of un-reacted monomer to be retained and on exposure to water over a long time this residual monomer can be leached out. The un-reacted monomer acts as a plasticizer for the matrix and its removal leads to an increase in rigidity of the matrix and a greater propensity for environmental stress cracking to occur. The loss of monomer results in the creation of micro voids and these are rapidly filled with water when the hull is immersed. The high sensitivity of the dielectric technique to the ingress of water makes it easy to follow the creation of these void structures and can also allow detection of delamination and other damage deep within the hull structure.

4 Microwave studies of composite structures

As has been demonstrated by Tabib-Azar[15] and Zoughi[16] the study of evanescent wave propagation into insulating materials can be used to provide a sensitive method of monitoring changes in the permittivity of a material being scanned. As an illustration of this method data are reported on a GRP pipe which has been exposed to hydrochloric acid gas. In certain areas there is internal blistering as shown in figure (4). This internal blistering is indicative of chemical attack on the pipe structure and is an early indication of prospective failure of the pipe structure. The pipes contain an internal carbon fibre coating which aids detection of changes in the internal structure. A scan of the outer surface, figure (5) indicates the region of the blister shown in figure (4). The dielectric measurement is made by only accessing the surface of the pipe and demonstrates the potential of the method for the NDT of pipe structures.

Fig. 4a

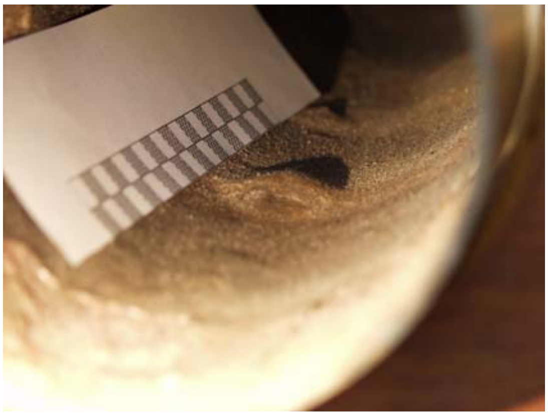

Fig. 4b

Figs. 4a & 4b. Blistering inside a GRP pipe which carries hydrogen chloride gas.

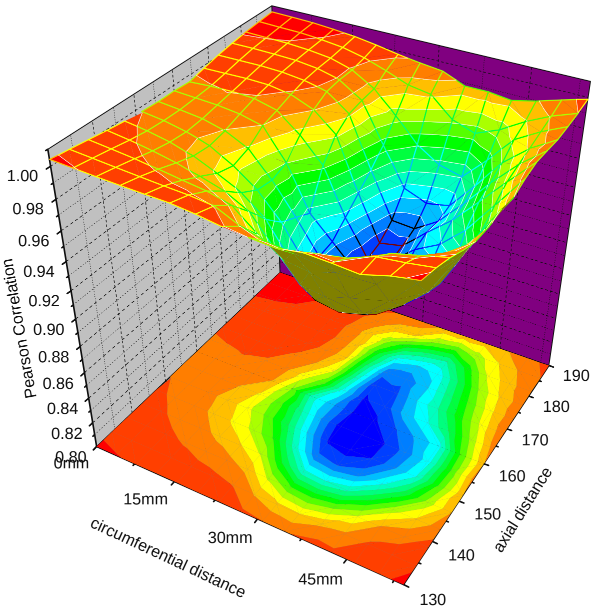

Fig. 5. Map of the change in the reflection coefficient over the area of a blister.

5 Conclusions

The dielectric NDT method developed for the study of adhesive joints can be readily extended to the investigation of heterogeneities in insulating pipe structures. The technique has an advantage over other NDT methods for the sensing of the distribution of water in a structure and this signature can provide useful information as to the potential for failure in certain situations.

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7 References

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