

This article was downloaded by:

On: 22 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



The Journal of Adhesion

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713453635>

Use of dielectric spectroscopy to assess adhesively bonded composite structures, Part III: Investigation of mechanical strength

W. M. Banks^a; P. Boinard^a; R. A. Pethrick^b

^a Department of Mechanical Engineering, University of Strathclyde, Glasgow, UK ^b Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow, UK

Online publication date: 08 September 2010

To cite this Article Banks, W. M. , Boinard, P. and Pethrick, R. A.(2002) 'Use of dielectric spectroscopy to assess adhesively bonded composite structures, Part III: Investigation of mechanical strength', *The Journal of Adhesion*, 78: 12, 1027 – 1038

To link to this Article: DOI: 10.1080/00218460215041

URL: <http://dx.doi.org/10.1080/00218460215041>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

USE OF DIELECTRIC SPECTROSCOPY TO ASSESS ADHESIVELY BONDED COMPOSITE STRUCTURES, PART III: INVESTIGATION OF MECHANICAL STRENGTH

W. M. Banks

P. Boinard

Department of Mechanical Engineering,
University of Strathclyde, Glasgow, UK

R. A. Pethrick

Department of Pure and Applied Chemistry,
University of Strathclyde, Glasgow, UK

This paper explores the way in which the data obtained from the dielectric study (reported in Part II of this 3-part series of articles), can be related to the mechanical strength of the bonded structure. Exposure of the adhesive bonded joints to a hot and wet environment leads to a loss of strength. Changes are observed in the dielectric properties on exposure to the humid environment, and an attempt is made to relate these observations to the mechanisms leading to loss in the mechanical strength of the bond. The observation of a correlation between the changes in these physical properties indicates the possibility of using dielectric spectroscopy as a powerful nondestructive evaluation (NDE) technique for bonded composite structures.

Keywords: Bonded structures; Adhesive; Moisture; Mechanical properties; Dielectric spectroscopy; Nondestructive evaluation

Received 28 September 2001; in final form 16 May 2002.

P. Boinard wishes to thank the Non Destructive Evaluation Branch, Materials Directorate of the U.S. Air Force, for the provision of a maintenance grant in support of this study (grant No F49620/97/1/0350), British Aerospace for the provision of materials, and Dr. D. Hayward at the Department of Pure & Applied Chemistry, University of Strathclyde, for his high frequency dielectric spectroscopy knowledge and advice. The help and support of Dr. Robert Crane and Dr. Charles Lee through the U.S. Air Force grant is gratefully acknowledged.

Address correspondence to W. M. Banks, Department of Mechanical Engineering, James Weir Building, University of Strathclyde, 75 Montrose Street, Glasgow G1 1XJ, Scotland. E-mail: wmb@mecheng.strath.ac.uk

INTRODUCTION

In Part I of this 3-part series of articles the effects of water ingress on the mechanical properties of a structural adhesive was reported. Part II reported the dielectric characteristics of an adhesively bonded carbon fibre epoxy composite exposed to a hot and wet environment. In this paper, the effects of the exposure to a humid environment on the mechanical strength of the bonded structures tested are reported, and an attempt is made to correlate these data with those presented in the earlier articles.

The use of adhesive bonding technology for airframes and related engineering structures offers significant advantages over the commonly used mechanical fasteners. However, these polymeric materials are influenced greatly during their service life by environmental conditions. Loss in the dynamic and mechanical properties can lead to drastic failure of the part. Degradation of bonded structures occurs when the adhesive absorbs significant amounts of water. Once the water penetrates the bond line, the adhesive properties can be altered in a reversible manner by plasticisation or in an irreversible manner by hydrolysis or crack and craze formation [1, 2]. Nevertheless, structural adhesives are selected with essentially hydrolysis-resistant chemistry and, therefore chemical attack is not generally the main driving feature for the degradation mechanism. Generally, degradation at the adhesive-adherend interface occurs by displacing the adhesive or by changing the adherend's surface chemistry. Furthermore, the diffusion of water in the adhesive tends to modify the mechanical failure mechanism of the joint from a cohesive failure in the adhesive layer to an adhesive failure at the adhesive-adherend interface [3, 4].

EXPERIMENTAL PROCEDURE

Materials

The materials and the fabrication of the samples used in this study are described in Part II.

Single-Lap Mechanical Shear Test

The test consists of subjecting a simple overlap joint design to a tensile force. The specimens were loaded in a computer-controlled universal Zwick 2061 tensile testing machine. The joints were tested at a crosshead speed of 1.27 mm/min (0.05 inch/min), with the load and

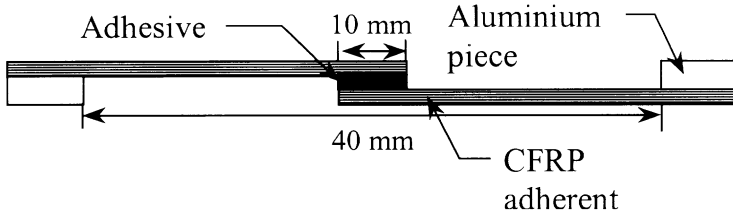


FIGURE 1 Schematic representation of the shear test joint structure.

the extension recorded by the computer interface from zero to failure load. To limit the bending due to possible eccentric load on the joints, aluminium end pieces, compensating the adherend and the adhesive thickness, were attached to the extremities of the joint. These aluminium pieces insure that, during loading, the axes of the adhesive bond line and the tensile load stay in the same plane. The specimen laminate sequence follows $[0_2/90/0_2/90/0]_s$. Figure 1 is a schematic representation of the dimensions for the shear test joint structure.

RESULTS AND DISCUSSION

Effect of Water on the Mechanical Shear Properties

A gravimetric analysis of the water uptake in the adhesive bond studied in this paper was reported in Part II. Figure 2 shows the typical evolution of the shear stress–“engineering”strain (i.e., crosshead displacement) curves for the single-lap shear test during exposure.

It can be seen that when the bonded structure is completely dry, failure occurs via a brittle characteristic. As the water penetrates the resin structure, change in the locus of the failure can be identified as being associated with a change from a brittle to a ductile mechanism. These changes in the failure mode can be observed in the fracture surfaces in Figure 3.

The failure of the unaged structure is mostly cohesive in the carbon fibre reinforced plastic (CFRP) adherends indicated by the appearance of carbon fibres in the adhesive surface. This behaviour is expected since the adhesive is a toughened epoxy resin, whereas the epoxy matrix of the CFRP adherends is more fragile [5, 6]. As the water content increases, plasticisation occurs and disrupts the local interactions in the crosslinked network of the adhesive. Moreover, swelling and possible degradation around the woven film support is thought to take place. During the ageing of the bonded structures, the failure is localised in the adhesive due to a weakening of the bulk material.

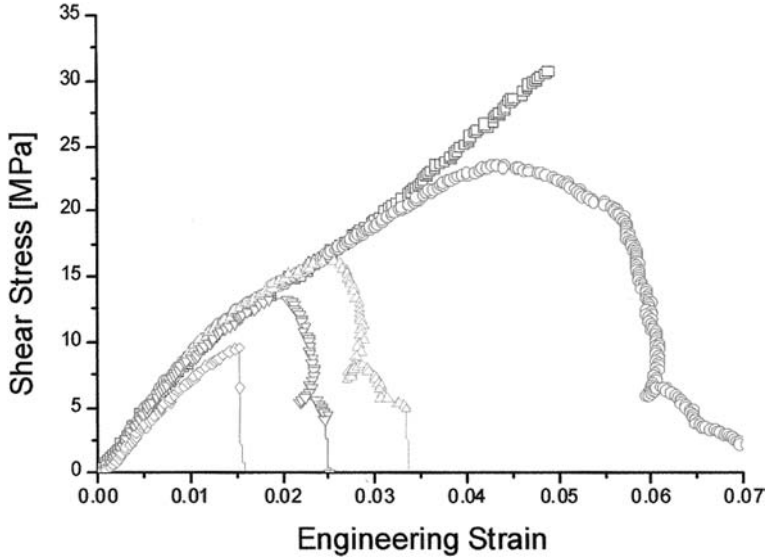


FIGURE 2 Typical stress-“engineering” strain (i.e., crosshead displacement) for the adhesively bonded composite structure during the exposure time to the hot/wet environment: (—□—) as manufactured, (—○—) after 336 h, (—△—) after 672 h, (—▽—) after 1,344 h, and (—◇—) after 2,184 h (see Color Plate IV).

As the water content increases further, the CFRP adherents start to be saturated and water appears at the interface. However, for a period of time, the bulk adhesive stays mostly dry (see picture for 672 h). Therefore, the fracture initiation changes from cohesive in the adhesive layer to adhesive at the adhesive-adherend interface. Finally, the fracture occurs almost totally at the interface after more than 2,000 h of exposure.

Six parameters were recorded during the single-lap shear test of the bonded structure: the shear modulus before, G_1 , and after, G_2 , crack initiation, the shear stress at crack initiation, τ , the shear strength, $\tau_{ultimate}$, the strain at crack initiation, γ , and the strain at rupture, $\gamma_{ultimate}$. Figure 4 shows a typical shear stress-strain curve of an unaged bonded structure and the different parameters investigated.

Figure 5 shows the variation of the mechanical shear characteristics of the adhesively bonded structure as a function of the exposure time.

All shear properties are observed to decrease with exposure to the hot and wet environment. However, there is retention of some residual

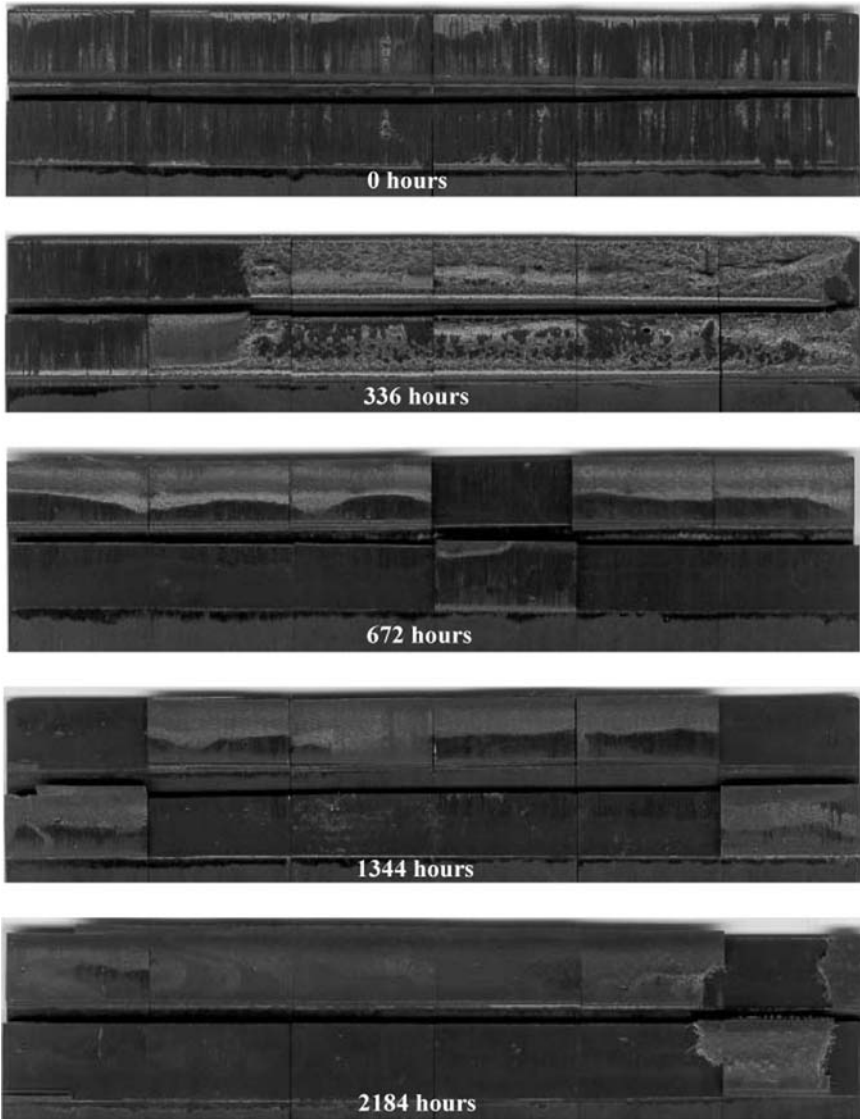


FIGURE 3 Fracture surfaces of the adhesively bonded structure after exposure to a hot/wet environment (see Color Plate V).

joint strength in all cases. All values tend towards an equilibrium, corresponding to 25% loss for the moduli, 60% loss for the shear stress at crack initiation, and 45% loss for the ultimate shear strength. This behaviour has been observed previously [3, 7, 8]. It is mainly attributed

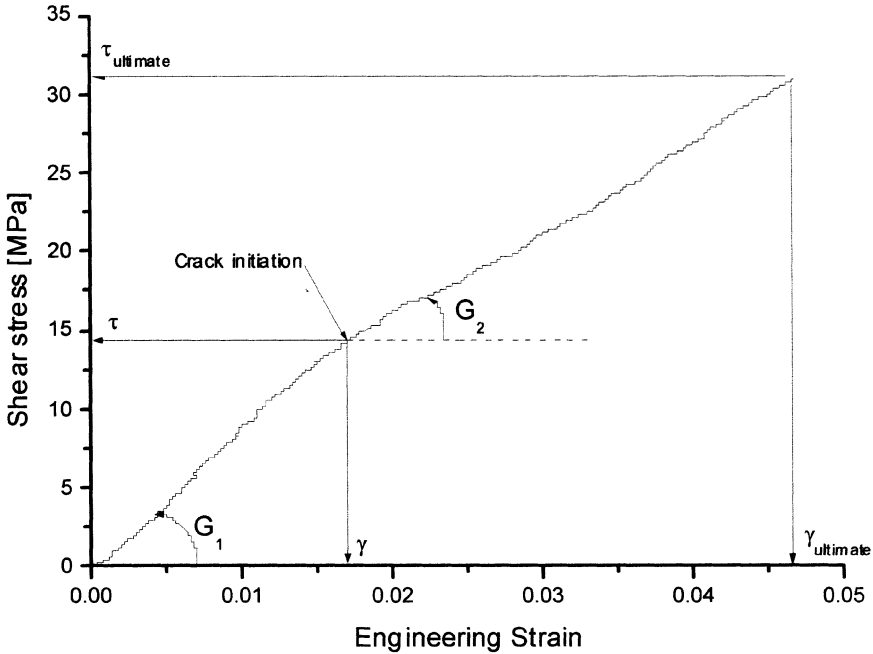


FIGURE 4 Typical shear stress–“engineering” strain (i.e., crosshead displacement) curve and the investigated parameters.

to the plasticisation of the epoxy matrix either in the CFRP composite adherends or in the adhesive. Moreover, the difference in the diffusion coefficient and the rate of sorption induced by the geometry of the structure implies that the CFRP composites will plasticise and swell earlier than the adhesive. Therefore, interfacial stresses between the two phases may occur. Such interfacial stresses have been observed by Hughes et al. [9] when they studied the dimensional stability of adhesively bonded joints under the influence of temperature and moisture.

John et al. [3] noticed that the decline in the mechanical shear properties may be accompanied by an increase in the strain at rupture. Figure 6 shows the strain at the crack initiation and the ultimate strain during the exposure time to the environment.

As with the shear stress properties, the strain at rupture also decreases with exposure time. The ultimate strain loses up to 40% of its original value after more than 2,000 h spent in the hot/wet environment. However, the strain at which the crack is initiated stays relatively constant during the ageing period.

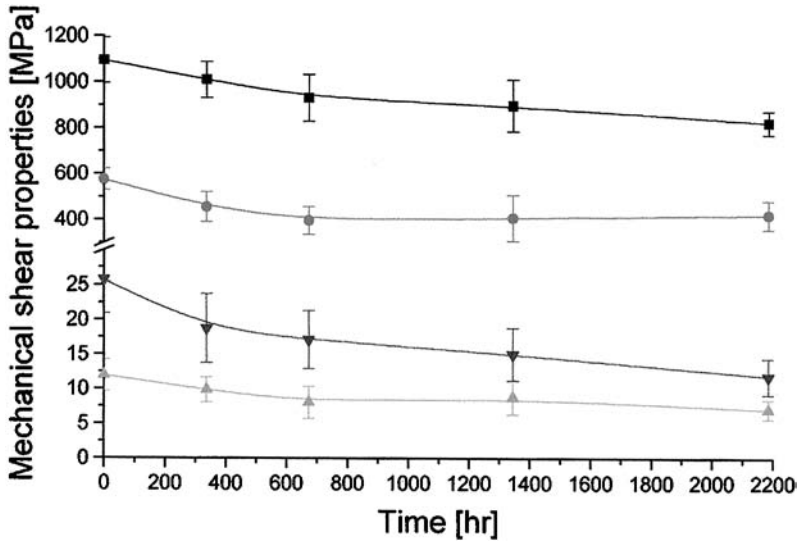


FIGURE 5 Evolution of (—■—) the shear modulus, (—●—) the shear modulus after crack initiation, (—▲—) the shear stress at crack initiation, and (—▼—) the shear strength as a function of the exposure time to the hot/wet environment (see Color Plate VI).

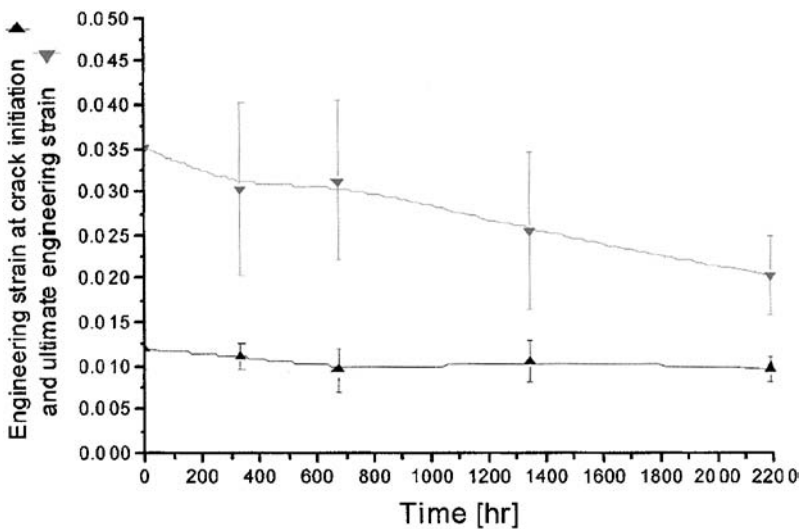


FIGURE 6 Evolution of (—▲—) the “engineering” strain (i.e., crosshead displacement) at crack initiation and (—▼—) the ultimate “engineering” strain as a function of the exposure time to the hot/wet environment (see Color Plate VII).

By integrating the shear stress over the displacement, the energy necessary to initiate cracks in the bonded structure and the total fracture energy can be calculated. The unaged structure presents a total fracture energy of 2.2 kJ/m^2 . This value falls in the range of elastomer-modified epoxy values reviewed by Bascom and Hunston [10]. The integration of the shear stress-strain curve gives access to the strain energy density, related to the volumetric energy necessary to break the single-lap shear joint. Figure 7 and Table 1 present the results of the strain energy density during the ageing period.

Since the mechanical characteristics of the joint structure at crack initiation remain the same during the exposure, little degradation of the adhesive is believed to have occurred. The tough epoxy matrix retains its cohesive strength, and the only decrease (at the start of the exposure) is related to the possible degradation of the support films inside the adhesive bond lines. The dielectric data obtained previously during ageing of the bonded structures confirm these assumptions (see Part II). Therefore, the loss of the strength of the adhesive is related to changes occurring at the interface. It has been shown that the presence of water at the adhesive-adherend interface modifies the

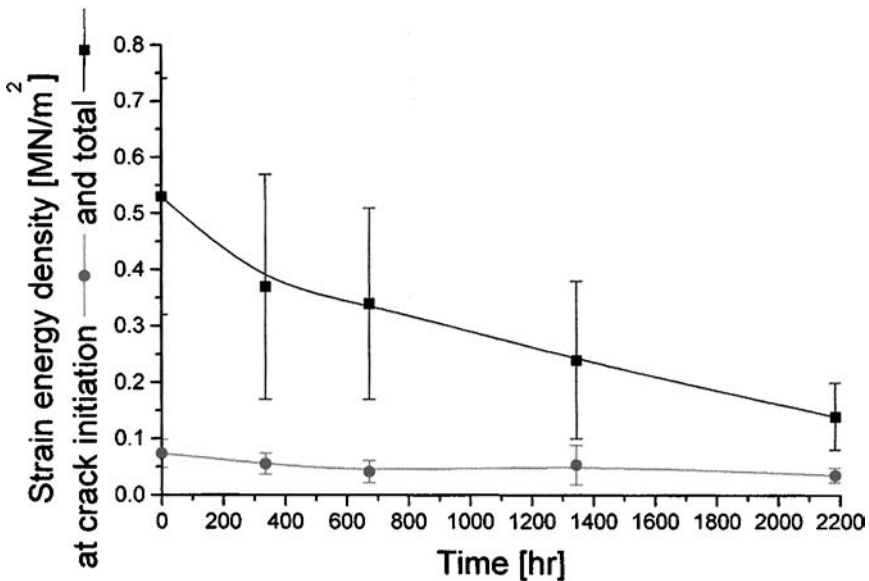


FIGURE 7 Evolution of the (—●—) initiation crack and (—■—) total strain energy density as a function of the exposure time to the hot/wet environment (see Color Plate VIII).

TABLE 1 Variation of the Strain Energy Density as a Function of the Exposure Time

Ageing time (h)	Strain energy density (MJ/m ³)	Loss in the strain energy density (%)
0	0.53 ± 0.21	N/A
336	0.37 ± 0.20	-30
672	0.34 ± 0.17	-36
1344	0.24 ± 0.14	-55
2184	0.14 ± 0.06	-74

work of adhesion [4, 11]. The work of adhesion, W_S , of the structure can be expressed as

$$W_S = \gamma_s + \gamma_a - \gamma_i, \quad (1)$$

where γ_s , γ_a , and γ_i are the surface tensions of the adherend, adhesive and interface, respectively, and W_S is a positive value implying that the interface is thermodynamically stable. As water penetrates the structure, W_S becomes

$$W_S = \gamma_{sl} + \gamma_{al} - \gamma_i, \quad (2)$$

where γ_{sl} and γ_{al} are the surface tensions of the adherend and adhesive, respectively, altered by the presence of water molecules. In this equation W_S becomes negative. The interface is thermodynamically unstable. Therefore, the presence of water molecules provides a driving force for the displacement of the adhesive from the substrate. This is shown in Figure 3, where the failure for the last measurement is dominantly adhesive failure.

Correlation between Dielectric Spectroscopy and Mechanical Behaviour

One of the main purposes of this work was to demonstrate the suitability of dielectric spectroscopy to assess the durability of adhesively bonded composite structures under the influence of water ingress. The durability was defined in relation to residual mechanical strength of the joint. The aim was to correlate the residual mechanical strength of the structure as a function of its water content. In Part II it was established that the change in the dielectric characteristics of the adhesively bonded structure due to water sorption can be monitored and a useful dielectric tool has emerged, termed the Ng factor (see Part II). Figure 8 relates the total strain energy density involved to

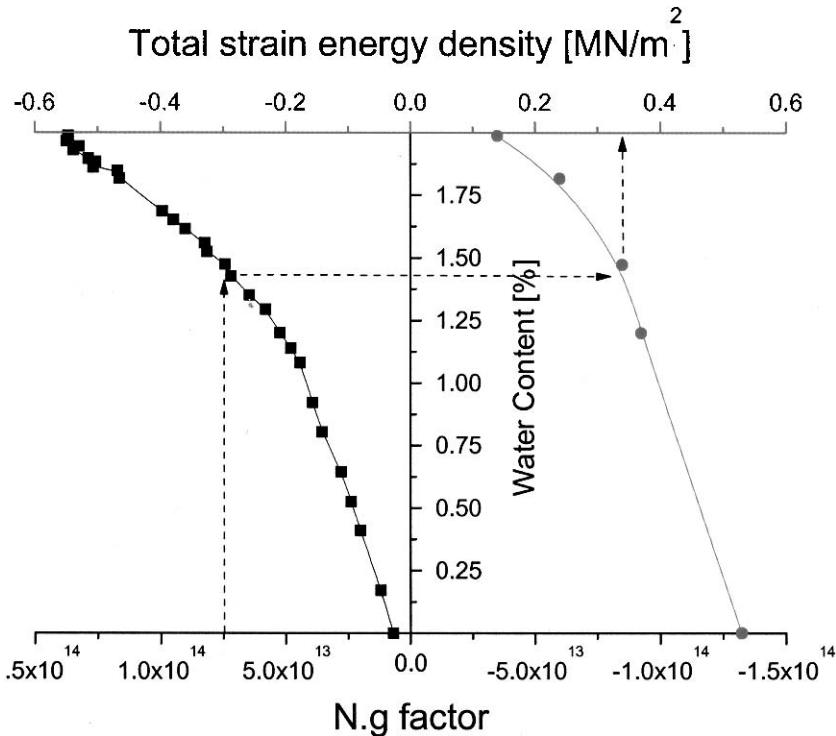


FIGURE 8 Correlation between the dielectric and mechanical characteristics via water content of the adhesively bonded composite structure immersed in the hot/wet environment (see Color Plate IX).

separate the two adherends and the $N.g$ factor. Figure 8 shows the master curve of the relationship between dielectric measurements and mechanical shear strength for the specific 914C/T300 adherends-AF163-2K adhesively bonded structure investigated in this study in a hot/wet environment.

This master curve can be used to estimate the strength of the bonded system at any given time, knowing that it is only true for systems and environmental conditions identical to those studied in this work. It is hoped that this will be the first step in the development of a series of master curves for alternative systems. The determination of the dielectric properties of the adhesively bonded structure allows the assessment of the water content in the structure by simply superimposing the dielectric value onto the master curve and crossing toward the water content axis. By crossing from the water content

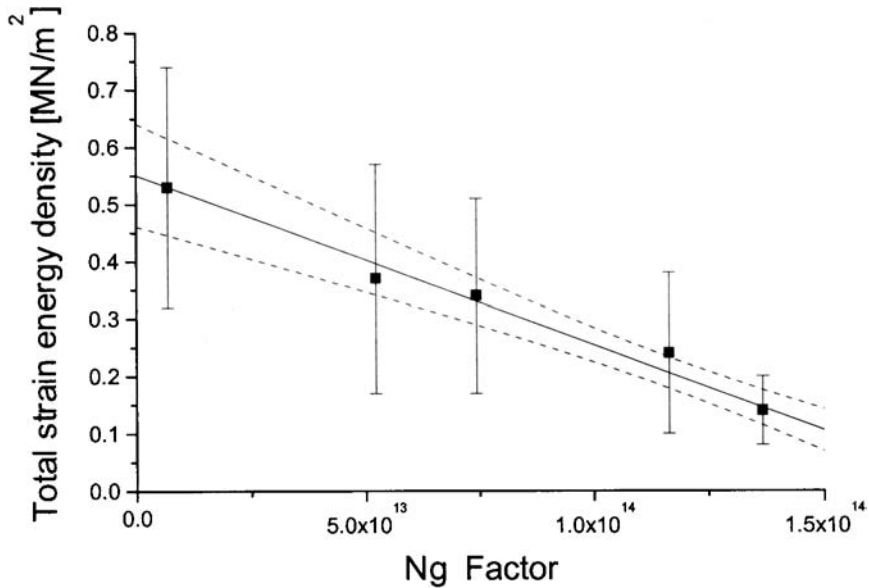


FIGURE 9 Total strain energy density as a function of the exposure time: (■) experimental data, (—) best linear fit, and (- - -) confidence band.

value towards the strain energy density curve, the strength remaining in the joint can be determined in terms of the total strain energy density. An interesting aspect of this master curve is the identification of the adhesive plasticisation around 1.25% water content by the dielectric feature (see Part II) related to the start of high strength loss in the mechanical properties. This simple interpretation of the dielectric data allows identification of plasticisation and mechanical degradation of the adhesive layer prior to drastic failure of the bonded structure. It may help in the decision to repair or replace the bonded part.

Figure 9 presents the direct relationship between dielectric and mechanical properties. It relates the total strain energy density required to separate the two adherends and the Ng factor.

CONCLUSION

The epoxy adhesive studied in this work showed two interesting characteristics. First, the total strain energy density decreases with exposure time. However, around 26% strength is retained even after more than 2000 h in the harsh environment. Secondly, the strain

energy density at crack initiation does not vary significantly over the ageing period. It is therefore believed that the loss in strength is due to interfacial phenomena.

The mechanical strength of the bonded structure has been related to its dielectric characteristics. Plasticisation and strength loss have been identified on a master curve demonstrating the vast potential of dielectric spectroscopy as a nondestructive evaluation method for bonded composite structures.

REFERENCES

- [1] J. Comyn, *Developments in Adhesives—Part 2* (Applied Science Publishers, Barking, UK, 1981) pp. 279–313.
- [2] S. J. Shaw, *Chemistry and Technology of Epoxy Resins* (Blackie Academic & Professional, Glasgow, 1993) pp. 206–255.
- [3] S. J. John, A. J. Kinloch, and F. L. Matthews, *Composites*, **22**(2), 121–126 (1991).
- [4] A. J. Kinloch, *Adhesion and Adhesives: Science and Technology* (Chapman and Hall, London, 1987) pp. 339–404.
- [5] Technical data, Prepregs for advanced composites: Fibredux 914, Ciba-Geigy.
- [6] D. H. Kaelble, J. Moacanin, and A. Gupta, *Epoxy Resins—Chemistry and Technology* (Marcel Dekker Inc., New York, 1988) pp. 603–651.
- [7] M. R. Bowditch, *Inter. J. Adh. and Adh.*, **16**(2), 73–79 (1996).
- [8] K. B. Armstrong, *Inter. J. Adh. and Adh.*, **16**(1), 21–28 (1996).
- [9] E. J. Hughes, J. Boutilier, and J. L. Rutherford, *Adhesive Joints* (Plenum Press, New York, 1984) pp. 137–150.
- [10] W. D. Bascom and D. L. Hunston, in *Treatise on Adhesion and Adhesives*, R. L. Patrick, Ed. (Marcel Dekker, New York, 1989) vol. 6, chap. 4, pp. 123–185.
- [11] A. J. Kinloch, M. S. G. Little, and J. F. Watts, *Acta Materialia*, **48**, 4543–4553 (2000).