SURFACE AND BULK STRESS/ STRAIN MEASUREMENTS IN COMPOSITE LAMINATES WITH A FIBRE-OPTIC RAMAN PROBE

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SUMMARY

Localised stress/ strain measurements in polymer based composites can now be made using a Laser Raman sensor. In this case the stress sensor is the reinforcing fibre of the composite and the detector is the Raman spectrometer. Raman stress/ strain sensor has been employed for unidirectional, as well as, multidirectional composites. The relationships between the local fibre stress or strain obtained from the Raman sensor and the far field stress or strain measured conventionally, are established.

KEYWORDS: smart structures, fibre optic, sensors, laser Raman spectroscopy

INTRODUCTION

'Smart' materials and the structures fabricated from them, are defined as materials which incorporate the functions of sensing, actuation and control. These features are integrated in all biological systems and impart to them intelligence, efficiency and superior functionality. Advanced fibrous composites already have many desirable characteristics of natural materials. By adding the biological features of sensing, actuation and control, they can taken a step further and given true life features and 'intelligence' [1]. One of the most critical functions of a 'smart' material such as a composite, is accurate sensing. Sensing capabilities can be given to structures by externally attaching sensors or by incorporating them within the structure during manufacturing. Over the last few years a new spectroscopic technique for non-destructive stress/ strain measurements in composites has been under development. This technique is based on the fact that most Raman backbone vibrational modes of crystalline fibres, shift to lower values in tension and to higher values in compression [2]. In short, bond extension or contraction changes the bond stiffness and hence the atomic vibrational frequencies. The magnitude of this Raman frequency shift can be related to external stress or strain, hence, making stress/ strain measurements in composites, possible [2]. The superiority of the Raman sensor over other existing sensors is its ability to provide independently values of fibre stress and strain from composite sample volumes as small as 1 μm [3]. In addition, this is the only technique that can directly measure stress in composites as most of the currently available non-destructive methods can only provide strain measurements [1].

The Laser Raman Technique has been already been used to monitor the composite micromechanics in single fibre geometries [3] and in full composites, to measure the residual thermal stresses and to map the stress concentration in composites containing holes [4]. All this past work was performed by translating the composite specimen in a stationary laser beam. Such an arrangement has serious drawbacks as only allows the inspection of specimens which can be physically fitted on the microscope stage of the Raman set up. More recently a novel remote laser Raman microprobe (ReRaM-I) was built and tested at QMW [5]. Flexible fibre optics have been used for the delivery of the laser light and for the collection of the inelastically (Raman) scattered light (Fig.1). Thus a complete separation of the spectroscopic and the testing stages was achieved. The use of flexible fibre optic cables permits operation of the microprobe in horizontal, vertical and multi-angle positions. Such an arrangement allows the interrogation of specimens of any size and shape and under a variety of different environments such as mechanical testing labs, factory floors, service depots, chemically hostile chambers, etc.[2,5]. In addition, the presence of a miniature CCTV imaging camera on the back of the microprobe allows the ReRaM-I to operate simultaneously both as Raman and optical microscope (Fig. 1). Therefore, the areas where Raman sampling is taken from, can be either observed on a TV monitor or videotaped for future use. Finally, the addition of integrated optics and filters at the tips of both the delivery and collection fibre optics (Fig. 1), obliterates any background interference from the fibre optic themselves and enhances confocality. For a focal spot of 1 μm in diameter, a confocal aperture of 1.8 μm has been achieved with the use of suitable optics [6].

In this work, various methods for retrieving the fibre stress or strain using ReRaM is reported. Attention is given to techniques that allow measurements not only at then near-surface plane of embedded fibres but also in fibres embedded in the bulk of a composite laminate. The stress or strain sensitivity for the various geometries is discussed. Finally, the effect that an embedded fibre optic cable has upon the mechanical properties of the composite is assessed.

EXPERIMENTAL

Materials/ Specimen Preparation

All test laminates were unidirectional Kevlar 49® / epoxy resin pre-impregnated tapes manufactured by Ciba-Geigy (type 914k-49-54.8%). 8-ply laminates were produced from the prepreg tape by curing them in an autoclave according to manufacturer's instructions. After curing the volume fraction of fibres was found to be 65% in the bulk and 70% on the surface. For measurements aimed at reinforcing fibres located near the surface of the laminate, a cleaved fibre optic is bonded to a small squared PMMA block (8mm), which is then stuck on the surface of the composite with a rubber based adhesive; so that the tip of fibre optic is just touching the surface of the composite. For the measurements in the bulk of the composite two different procedures were followed:

Fibre-optic cable embedded perpendicular to reinforcing fibres (Bulk ⊥)

The first attempt to obtain fibre strain data from a similar configuration involved (a) the attempt of a polydiacetylene (PDA) fibre on the tip of a fibre optic and (b) the incorporation of the assembly into a carbon fibre composite [7]. This configuration was only partially successful due to the effect that the size of the PDA fibre had upon the results obtained [8].

In this work, advances in the instrumentation allowed us to obtain high quality Raman response from the reinforcing fibres of the composite without the need for attaching a PDA crystal to the tip of the fibre optic. The prepreg tapes were cut to strips of 203 mm in length and 12.7 mm in width. The 8-plies were laid on top of each other with a cleaved fibre-optic

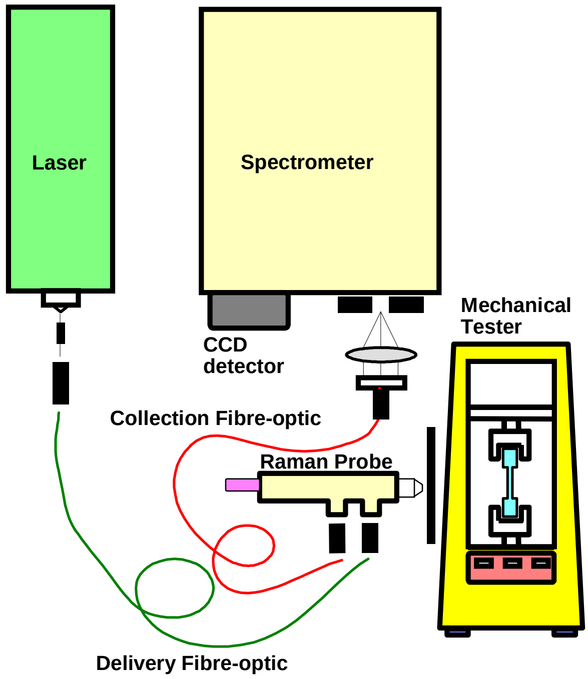


Fig. 1- Remote Laser Raman experimental set-up

embedded in the centre. Multimode fibre optic cables were made by Newport Co. (model F-MLD) of nominal core diameter of 100 μm and a numerical aperture of of 0.29, were used. The laminates were cured in an autoclave as above.

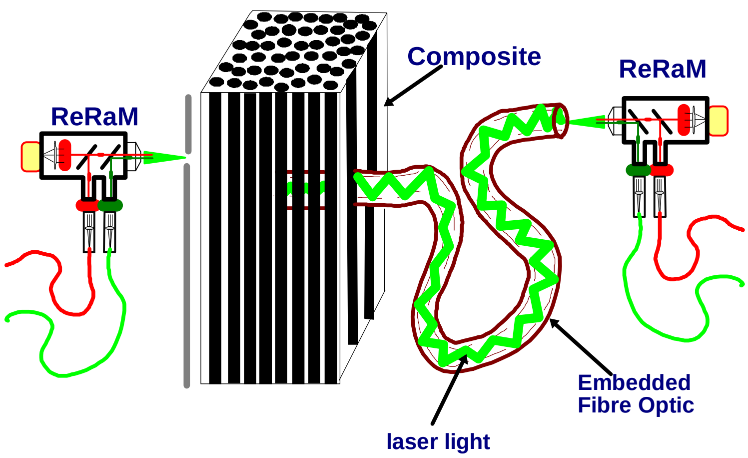


Fig. 2- Schematic of ‘surface’ (left) and ‘bulk-perpendicular’ (right) configurations. The items are not shown in scale.

Fibre-optic cable embedded parallel to reinforcing fibres (Bulk ‖)

The procedure was identical as in the case of section 2.1 but this time the cleaved fibre optic cable is wrapped in a strand of Kevlar® fibres and placed in the centre of laminates in a direction parallel to the axis of the reinforcing fibres of the laminate.

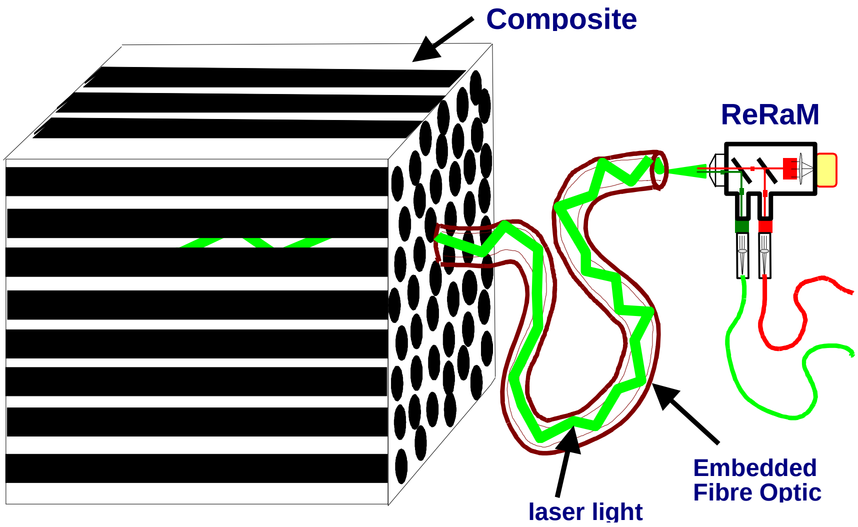


Fig. 3- Schematic of 'bulk-parallel configuration. The items are not shown in scale.

Fibre-optic cable embedded perpendicular to reinforcing fibres of angle ply laminates

The laminates were cut to the dimension and cured as in the case of section 2.1. This time the laminates were placed in $[0_2, -45, +45]_s$ order. A cleaved fibre optic cable was placed in the centre of angle ply laminates in a direction perpendicular to the axis of the reinforcing fibres of the laminate. The measurements on the 0-ply laminate is conducted by interrogating the fibres at the surface of the laminate as shown in Fig. 1.

Kevlar 49® fibres placed in the centre of unidirectional carbon / epoxy laminates

The laminates were cut to the dimension as in the case of section 2.1.1. Strand of Kevlar 49® fibres were sandwiched between in the centre of 8-ply laminates in a direction parallel to the reinforcing carbon fibres - where a cleaved fibre optic is placed perpendicular to the Kevlar 49® fibres. The laminates were cured in an autoclave as above.

Mechanical Testing

The Raman sensor was calibrated by loading single Kevlar® fibres in air and by plotting the shift of Raman frequency as a function of axial stress or strain. The composites were tested in tension following the ASTM standard procedure. Prior to testing the ends of the tensile coupons were ends are sand blasted and end-tabbed with standard, 2.4 mm thick, glass-reinforced-plastic tabs. Strain gauges of gauge resistance of 350 ± 1.0 Ω and of gauge factor of 2.03 were attached to the middle of the gauge section for each coupon. A total of 15 specimens were tested in tension; 5 of those specimens contained no embedded fibre optics, 5 incorporated 'parallel' fibre optic cables and 5 incorporated 'perpendicular fibre optic cables. Prior to testing the ends of the tensile coupons were ends are sand blasted and end-tabbed with standard, 2.4 mm thick, glass-reinforced-plastic tabs. Strain gauges of gauge resistance of 350 ± 1.0 Ω and of gauge factor of 2.03, were attached to the middle of the gauge section for each coupon. All specimens were loaded on to fracture on a 20 kN screw-driven Hounsfield mechanical tester at a strain rate of approximately 0.002 min^{-1} .

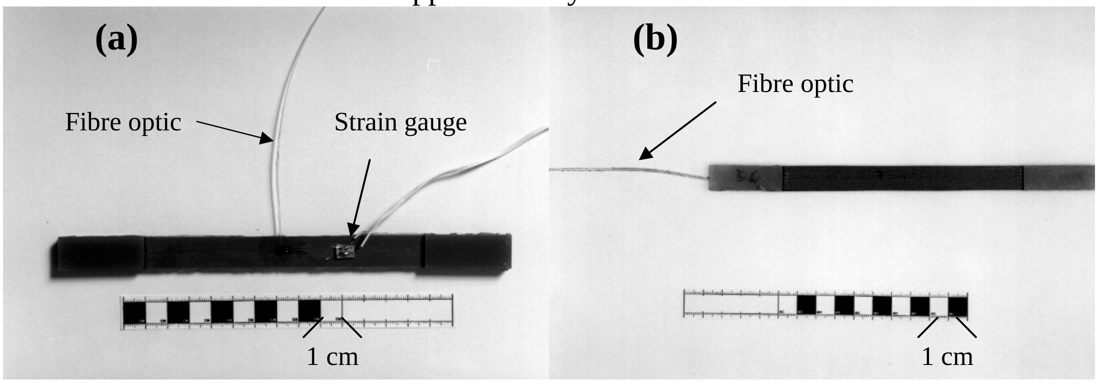
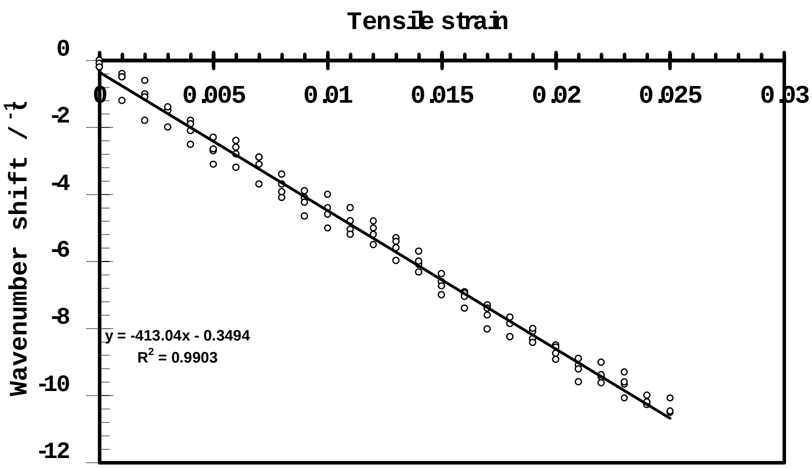


Fig. 4- Photographs of ‘bulk-perpendicular’ and ‘bulk-parallel’ standard composite tensile coupons.

Laser Raman Experiments

The input laser light of an argon-ion laser excited at 514.5 nm, was directed to the objective of the ReRaM and then was focused onto the free tip of the embedded fibre optic. For moderate levels of laser power the 514.5 nm wavelength was not found to cause any damage to the fibre. The light emitted by the fibre optic was non-polarised and, therefore, the calibration of the stress sensor in air was also carried through using non-polarised laser light. The 180^0 scattered light from the bulk or the surface of the composite was collected via the same embedded fibre optic and, by means of the ReRaM, was directed to the single monochromator for analysis and Raman light detection. The laser power was maintained constant throughout each test. In the case of the *Bulk* ⊥ experiments, 3 mW of incident laser radiation was directed to the embedded fibre optic and measurements were taken at 10 sec exposure intervals during the mechanical loading of the composite. For the *Bulk* ∥ experiments, a laser power of 7 mW was used and measurements were taken every 20 sec. At each Raman measurement, both the strain in the laminate- measured by means of the attached strain gauge- and the corresponding applied load, were independently recorded.



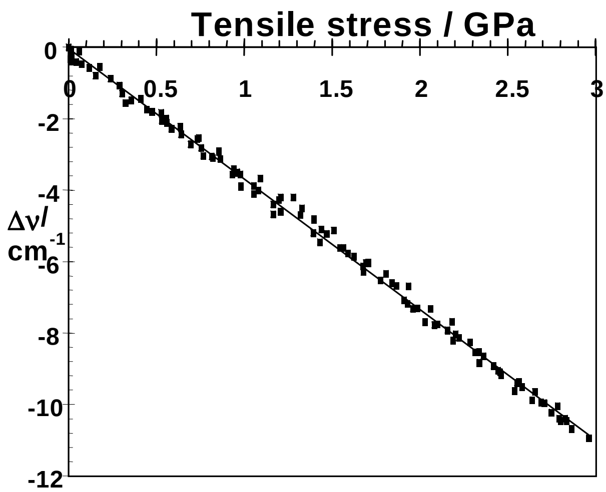


Fig. 5- Raman frequency shift as a function of tensile strain (top) and tensile stress (bottom) for the Kevlar 49® fibres (Raman sensor) in air. These curves are used as calibration curves for the conversion of the Raman frequency values into values of fibre stress or strain in composites.

RESULTS AND DISCUSSION

As mentioned earlier, the basis of the laser Raman sensing technique is the stress or strain dependence of the Raman vibrational frequencies of almost all reinforcing fibres. Raman frequency shift can be mapped as a function of tensile stress and of tensile strain for Kevlar 49® fibres free standing in air. Both relationships can be considered linear within experimental error although, in general, the Raman frequency vs. strain relationships can be best fitted with cubic spline polynomial curves (3). The slope of the least-squares-fitted straight lines represent the sensitivity of the stress or strain sensors and can be used to convert Raman frequency into values of fibre stress or strain in composites. The slope of the least-squares-fitted straight lines for stress is -3.8 cm^{-1} GPa^{-1}, and for strain it is -4.38 cm^{-1} $\%^{-1}$.

In Table 1 the effect of the presence of an embedded fibre optic upon the integrity of the composite is investigated; as can be seen the presence of a fibre optic cable perpendicular to the reinforcing fibres reduces the tensile strength by approximately 10%, whereas the presence of a fibre parallel to the reinforcing fibres have no practical effect upon the tensile strength of the composite.

Table 1- Ultimate tensile strength data of all composites tested in this work

No of Specimen	**Ultimate Tensile Strength of Bulk ⊥ coupons / GPa**	**Ultimate Tensile Strength of Bulk ∥ coupons / GPa**	**Ultimate Tensile Strength of As-received Coupons**
1	1.18	1.33	1.43
2	1.25	1.38	1.39
3	1.32	1.41	1.35
4	1.23	1.35	1.41
5	1.19	1.42	1.27
Mean/ GPa	**1.24**	**1.38**	**1.37**
STD /GPa	**0.06**	**0.04**	**0.06**

The stress sensor measurements shown in Figs. 6, 8 and 10 indicate a 1:1 relationship between applied stress on individual fibres of the composite and the fibres stress measured by ReRaM . The strain sensor measurements shown in Figs. 7, 9 and 11 also indicate that a 1:1 relationship between composite strain, as measured by the attached strain gauge, and fibre strain, as measured by the ReRaM is attained.

Fig. 12 shows the measured stress by ReRaM on fibres of +45 ply in relation to applied stress over the whole composite coupon $[0_2, -45, +45]_s$. As expected, the axial fibre stress is only a fraction of the applied composite stress. However, a closer inspection of the data points reveals that the relationship is not linear and therefore one may be able to extract useful information upon the deformation mechanisms in these geometries based on the relationship of the fibre stress at angle θ to that of the applied stress in the axial direction.

Finally, the possibility of using Kevlar 49® fibres as stress sensors in composite which incorporate reinforcing fibres that exhibit a weak Raman signal has been investigated. Fig. 13 shows the relationship between the Kevlar 49® fibre strain to that of the applied composite strain. The data points lie on a least-square-fitted straight line the slope of which is unity. Thus, a small small amount of Kevlar 49® fibres can be used to assess the fibre strain in carbon composites and indeed in any composite, such as glass fibre reinforced polymers, for which the Raman signal can not be obtained directly from the reinforcing fibres. The fibre stress can also be obtained but, in this case, the ratio of the tensile moduli of the Kevlar 49® fibre to that of the reinforcing fibre of the compsite must be known.

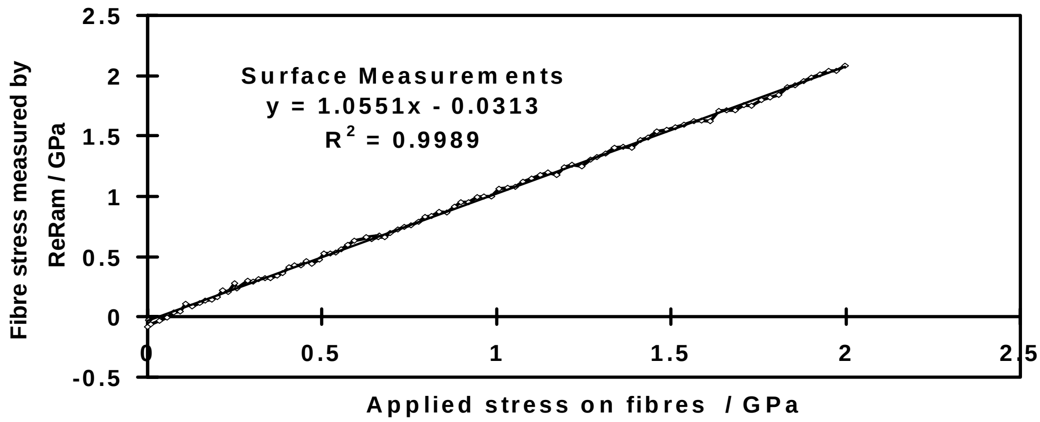


Fig. 6- Fibre stress measured by the remote Raman microprobe near the surface of the laminate as a function of the applied stress on individual fibres of the composite.

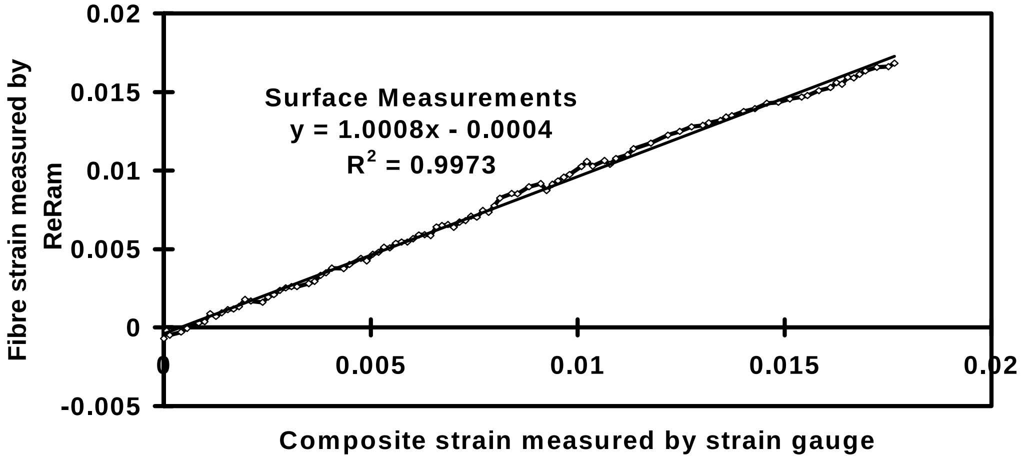


Fig. 7- Fibre strain measured by the remote Raman microprobe near the surface of the laminate as a function of the strain in the composite measured by means of an attached strain gauge.

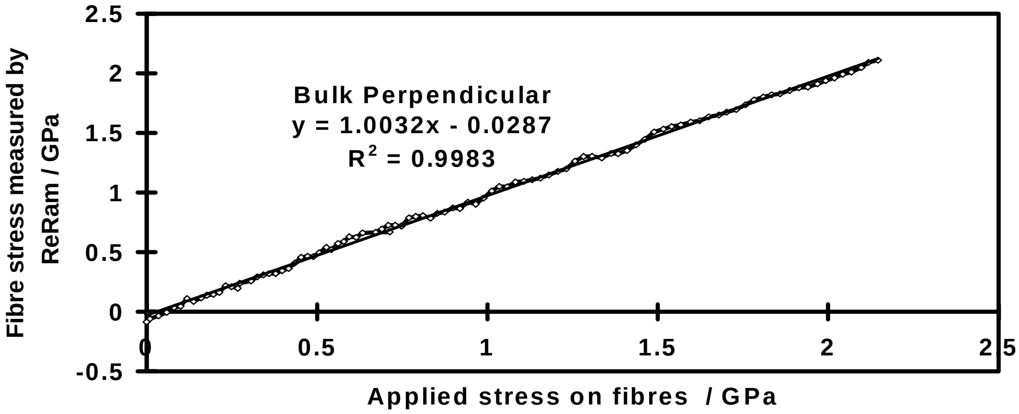


Fig. 8- Fibre stress measured by the remote Raman microprobe using an embedded fibre optic in a direction perpendicular to the reinforcing fibres of the composite, as a function of the applied stress on individual fibres of the composite.

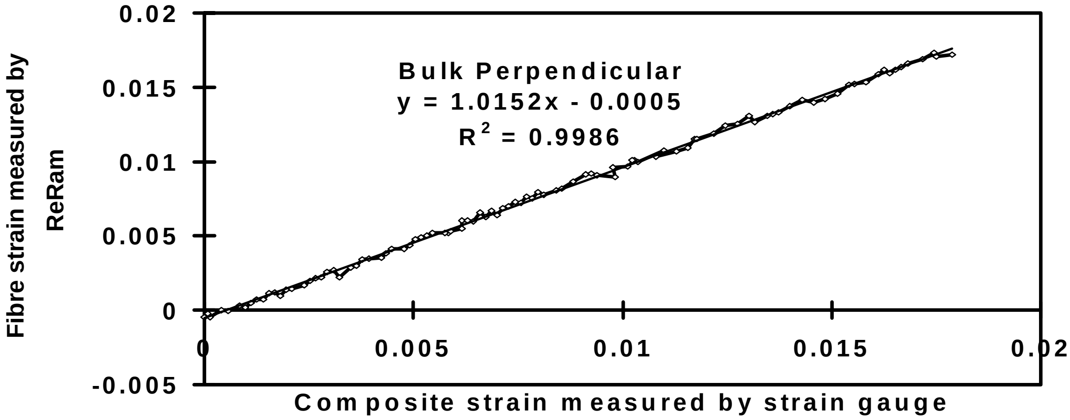


Fig. 9- Fibre strain measured by the remote Raman microprobe near the surface of the laminate as a function of the strain in the composite measured by means of an attached strain gauge.

CONCLUSIONS

A new stress/ strain sensor has been built and tested. The technique is based on the stress or strain sensitivity of the Raman frequencies of the reinforcing fibres which are the load supporting elements of all the high performance composites. Measurements can be conducted on the near-surface of the laminate and also in the bulk. The latter are performed by employing fibre optic cables to transport the exciting laser radiation to the embedded reinforcing fibres in the composite. It has also been demonstrated that the Raman stress/ strain sensor can be employed for unidirectional, as well as, multidirectional composites. In all cases the axial stress/ strain in the fibre at a given ply can be measured. The Kevlar 49® sensor can also be incorporated in composites that contain fibres whose Raman signal is considered too weak for accurate measurements.

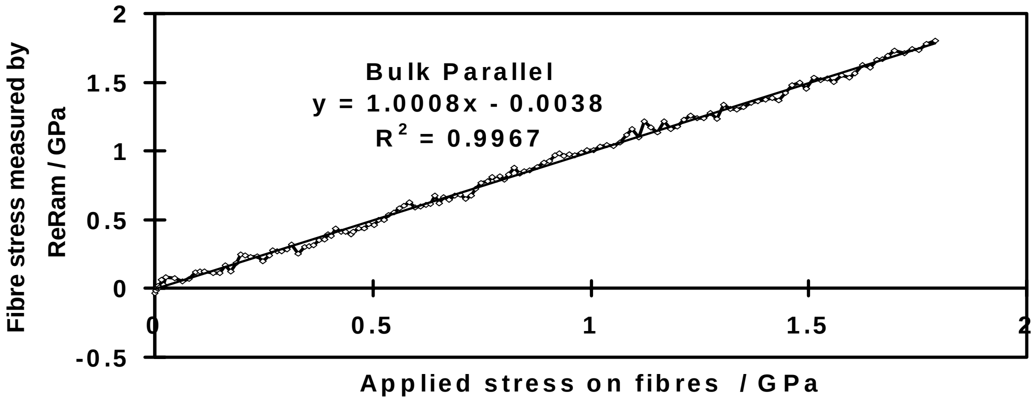


Fig. 10- Fibre stress measured by the remote Raman microprobe using an embedded fibre optic in a direction parallel to the reinforcing fibres of the composite, as a function of the applied stress on individual fibres of the composite.

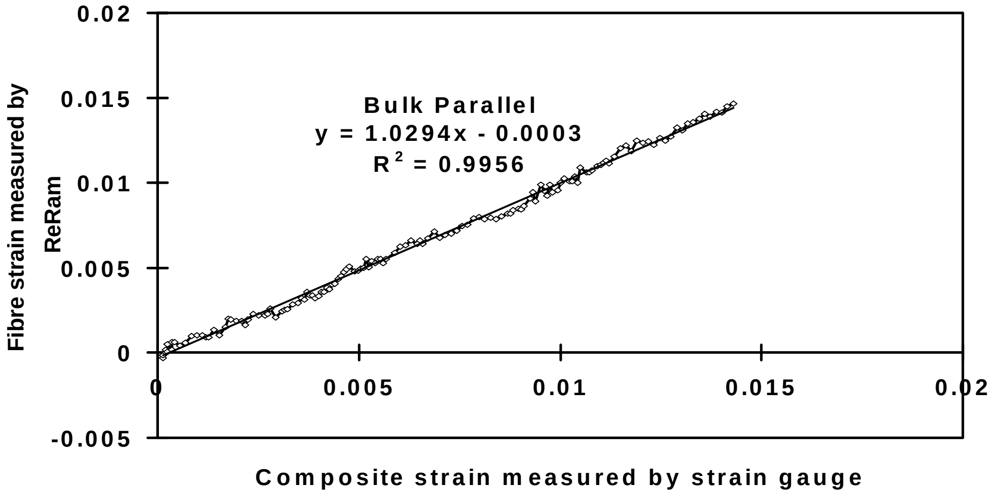


Fig. 11- Fibre strain measured by the remote Raman microprobe using an embedded fibre optic in a direction parallel to the reinforcing fibres of the composite, as a function of the strain in the composite measured by means of an attached strain gauge.

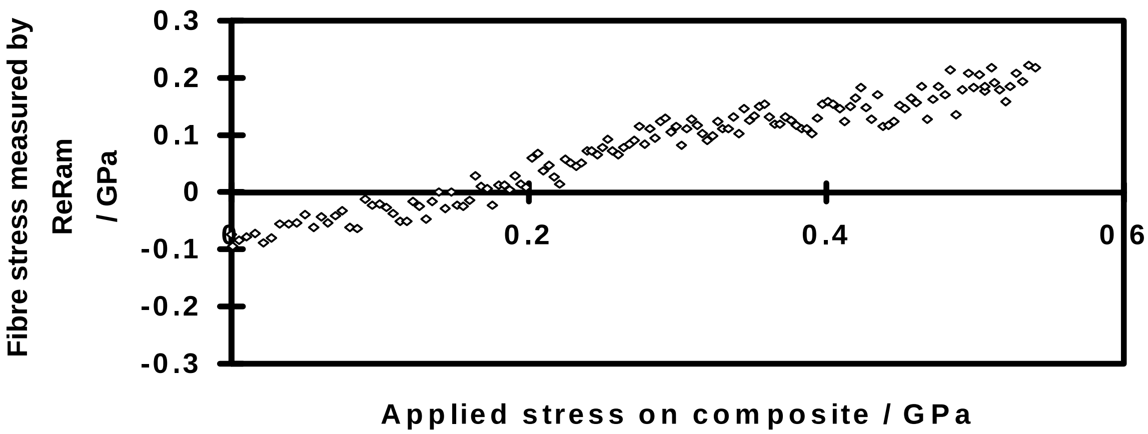


Fig. 12- Fibre stress measured by the remote Raman microprobe using an embedded fibre optic in a direction perpendicular to the +45-ply, as a function of the applied stress over the whole composite coupon. Geometry of the coupon is $[0_2, -45, +45]_s$.

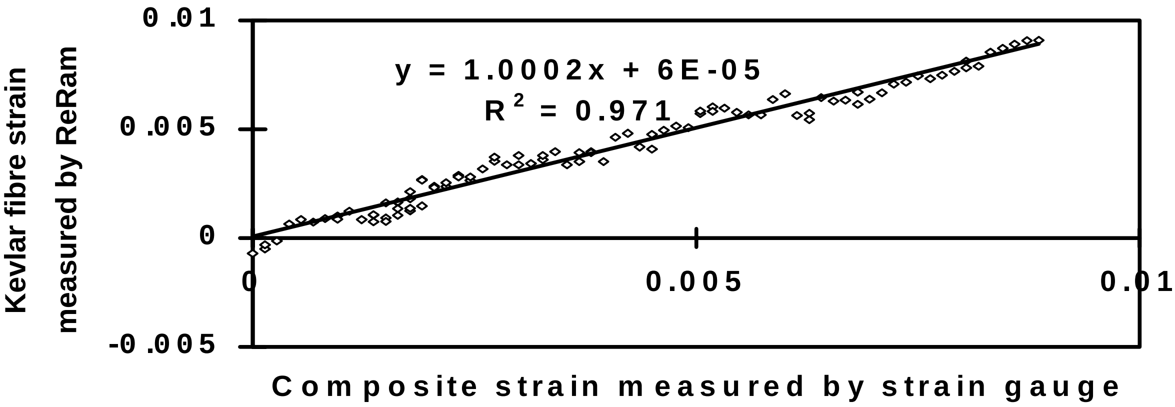


Fig. 13- fibre strain measured by the remote Raman microprobe using an embedded fibre optic in a direction perpendicular to Kevlar 49® fibres which are incorporated between 8-ply carbon / epoxy composite, as a function of the strain in the composite measured by means of an attached strain gauge. The solid line is a least-squares-fit to the experimental data.

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