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Transverse Properties of Carbon Fibres by Nano-Indentation and Micro-mechanics

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Abstract:

In this study the transverse modulus of three high modulus (M40, M46 and K63712) carbon fibres has first been measured directly by nano-indentation measurements. Transverse tensile tests on unidirectional epoxy composites were then performed, and the comparison was made between transverse fibre properties from indentation and those needed to obtain the measured transverse composite modulus using micromechanics expressions. The latter tended to underestimate values from indentation, by up to 36%, and reasons for this are discussed. Values of transverse fibre modulus determined by both methods decrease as longitudinal fibre modulus increases.

Keywords: Carbon fibre - Epoxy - Nano-indentation - Transverse tensile

1. Introduction

The performance of racing yachts is increasing continuously, thanks in part to the extensive use of carbon fibre reinforced composite materials in critical structures. There is a move towards very high stiffness fibres, but as tensile strength increases compression strength appears to decrease [1]. High performance carbon fibres can be classed in four categories, high resistance (HR), intermediate modulus (IM), high modulus (HM) and ultra high modulus (UHM). The latter are often defined as those fibres with a modulus greater than 400 GPa. All these fibres are used in sports products, but the high modulus fibres are essentially found in aerospace applications. Currently two precursers, PAN (poly(acrylonitrile)) and pitch are used. These are drawn, oxidized (150-300°C), then carbonized (1500°C). High modulus fibres are the result of a subsequent high temperature treatment, (2000-3000°C), which increases the carbon content above 99% and increases crystal size [2-4]. Over the last ten years the use of HM fibres has developed for the masts of ocean racing yachts, and particularly for 60 foot Open Class multi-hulls. These structures are generally in composite sandwich, carbon composite facings on honeycomb core, of length around 30 meters and weighing around 450 kg, manufactured from prepreg either in ovens under vacuum or autoclaves.
While the composites used in racing yachts are similar to those employed in the aerospace industry there are differences in the fabrication and quality control which make a direct transfer of knowledge difficult.

The properties of composite materials reinforced by unidirectional (UD) fibres are known to be highly anisotropic with high values of stiffness and strength in the fibre direction and poor mechanical behaviour in the transverse direction. The presence of fibres results in stress concentrations and the strength is lower than that of the unreinforced matrix. For a multi-directional composite with plies in different directions the first damage (first ply failure) often corresponds to the transverse tensile strength of the plies with unidirectional reinforcement [5].

In order to estimate ply properties using micro-mechanics expressions, the transverse modulus of the fibres is required, but this value is difficult to measure. Different techniques have been used, including direct transverse compression on single fibres [6,7], Raman micro-spectroscopy [8] and ultrasound scatter measurements [9]. An alternative approach is to measure the mechanical behaviour of unidirectional plies loaded at 90° and deduce the fibre properties [10].

The first aim of the present work is to determine the transverse modulus of different carbon fibres directly by nanoindentation measurements. Transverse tensile tests on unidirectional composites used in racing yacht construction will then be performed, and the correlation between transverse fibre properties from indentation and those needed to obtain the correct transverse composite modulus using micromechanics models will be studied.

2. Experimental procedure

2.1. Materials and preparation

Three high modulus carbon fibres and their composites have been tested, Table 1. All are or have been used in racing yacht construction. The reference fibre is M46J, which is currently the most widely used fibre for mast structures.

Unidirectional composite materials were manufactured from 14 layers of 300 g/m² areal weight *Structil* 367-2 epoxy resin prepreg. All were cured in an oven and held at 110°C for 4 hours under vacuum with the same cure cycle as that used for yacht masts. A sample of unreinforced resin was also obtained and cured under the same conditions. Three fibres, with axial modulus from 377 to 640 GPa were studied: M40J, and M46J PAN fibres from Toray, and K63712 a pitch based fibre from Mitsubishi. Mean fibre volume fraction was measured to be 52% ±1 (Table 2), and cure was checked by DSC analyses.

2.2. Test methods

Nanoindentation

The measurement of the transverse modulus of fibres with small diameters (between 5 and 10 microns) is a delicate operation. Several test procedures have been tried in this study, and the method retained involves indenting the fibre within the composite in transverse tensile specimens, Figure 1. The advantage of this approach, compared to indenting single fibres or fibre bundles, is that the fibre is restrained so that movements are blocked. The samples are the same as those used for transverse tensile testing. The sections to be indented were polished down to one-micron diamond paste.
Tests were performed on a Nanoindenter (XP, MTS Nano Instruments) and a Berkovitch indenter. The tip indentation is controlled with a frequency of 70 Hz (dynamic / Continuous stiffness measurement method). Tests were analysed using the ISO 14577 standard as described in [11].
Before each test series a calibration indentation is performed. Each test then involves contact with the surface, and loading to a depth of 100nm at a constant strain rate of $0.05s^{-1}$. The load is then maintained constant for 60s. Then the sample is unloaded at the same rate to 10% of the maximum force. This plateau enables any error due to temperature variations to be evaluated. The moduli are determined using the method proposed by Oliver and Pharr [12].
Each test involves a line of 30 indentations with a 0.5 µm spacing (Figure 1). Each line starts in the matrix and crosses several fibres. This enables the fibre and matrix moduli to be distinguished (Figure 2). Several lines are indented in different areas of the specimens. Each modulus value E_{ft} given is the mean of at least 120 indentations, based on the assumption that E_{ft} is constant across the fibre section.
The depth of indentation is an important parameter. It must not be too deep (> thickness /10), as indicated in the standard test method [16]. Also, as the fibres are made up of crystalline layers an indentation which is too deep can cause fibres to break. On the other hand, if the indentation depth is not deep enough the influence of the surface can modify the value measured (Figure 3). Figure 4 shows a typical load-unload plot. Depths used in published studies of carbon fibre composites range from 20nm to 500nm [8][17][18]. Here it was noted that above a depth of 40 nm the module was stable, a maximum depth of 100nm was therefore retained for the determination of modulus values.

Transverse tensile tests

Transverse tensile tests were performed on at least five parallel-sided composite specimens of each material, with width 25 mm (ASTM D 3039). Elongation was measured using an extensometer. Loading rate was 1 mm/minute. In order to observe the damage mechanisms additional specimens were also tested on a small *Deben* tensile test machine inside a *Jeol 6460LV* scanning electron microscope (SEM).

Differential Scanning Calorimetry (DSC)

DSC was used to check the degree of cure of samples (absence of exotherm) and to measure the glass transition temperatures (Tg), in order to verify the assumption that the matrix was the same for all composites. *Mettler Toledo DCS 822^e* equipment was employed with a heating rate of 5°C min^{-1}.

3. Results and discussion

3.1. Analysis of fibre morphology

The morphology of these fibres varies considerably. Figure 5 shows examples of scanning electron micrographs of the three fibres. These photos clearly indicate the differences in shape and dimensions, which will affect local stress concentrations when transverse loading is applied. In order to characterise fibre geometry polished sections of each composite material were analysed, using CAD software. At least 200 fibres of each type were analysed, the results are given in Table 3 below. Equivalent diameters are shown for the two HM PAN fibres, as they are not circular.

3.2. Nanoindentation

A test was performed initially on a unidirectional glass/epoxy composite to check the approach. As glass is isotropic the transverse modulus should be close to the longitudinal value of 72 GPa [2]. The transverse value was measured to be 67.8 (±15) GPa. This is close to the expected value and suggests that at least for glass tensile and indentation moduli values are similar. The transverse moduli measured on the three carbon fibres and the glass are given in Table 4. This shows that as axial fibre stiffness increases the transverse modulus drops. This has been noted previously using other techniques [7] [19] [20] Published values vary considerably, in the range from 10 to 30 GPa for the transverse modulus of PAN fibres and from 6 to 20 GPa for pitch [7], so the present results are in the same range. This inverse relationship between axial and transverse modulus is due to the internal fibre structure. As axial stiffness increases the crystals are longer and more-closely aligned in the axial direction. The amount of amorphous material, which ensures transverse integrity, is reduced resulting in lower transverse properties [7].

3.3. Transverse tensile properties (unidirectional ply)

Table 5 shows the results from the transverse tensile tests. There is a significant influence of the fibre type on strength and a smaller dependence for stiffness. The heterogeneity of composite materials is the main cause of failure of UD plies loaded in transverse tension. Stress and strain concentrations are induced in the matrix between the fibres. De Kok has shown that a global ply strain of 1% can result in local strains greater than 5% [21]. An irregular fibre distribution accentuates this effect. As a result of these strain concentrations the transverse failure strain for UD plies is considerably lower than the longitudinal failure strain. Many previous studies have examined the parameters which influence transverse tensile strength including matrix failure strain, interface quality, fibre distribution and diameter and fabrication parameters [22-26].

Study of the propagation of cracks in the SEM reveals differences in the failure mechanisms. For the composites reinforced with PAN fibres the cracks propagate in the matrix and at the fibre/matrix interface. In the pitch fibre composites however, cracks are also noted within the fibres themselves, Figure 6. Within the composite the differences in the fibre morphology seen in Figure 5 will affect local stress concentrations when transverse loading is applied. The interface areas of unit length of PAN fibres were measured and vary from 21.8 to 25.2 μm^2, Table 3. Fibres are not perfectly circular but these areas correspond to equivalent diameters of around 5.7 μm. The pitch fibres are larger, with mean diameters around 10.5 μm. It has been noted elsewhere that the modulus of PAN carbon fibres increases as section decreases [27]. The interface areas for pitch fibres are much larger.

The transverse tensile properties are strongly influenced by the nature of the fibres. Modulus depends on fibre and matrix behaviour, while strength is also influenced by the fibre-matrix interface. The amount of the latter will increase as fibre content increases [28], due to an increase in interface area and strain concentrations between fibres. The strength prediction as dictated by crack initiation and propagation, is dependent upon local material behaviour deviations from a regular array [29].

3.4. Relationship between transverse modulus of fibres and composites.

Figure 7 shows how the transverse modulus of the composite and the modulus obtained by nanoindentation vary with fibre type. The two moduli show the same trend; as transverse fibre modulus decreases so does the transverse modulus of the composite. This is in agreement with micromechanics equations which propose a linear relationship between the two values. Examples are discussed in more detail below.

3.5. Micromechanics analysis of transverse tensile properties on UD composites.

Three micromechanics models have been applied here, and these are presented below, but first it was necessary to measure the matrix properties required as input for these models.

3.5.1. Matrix elastic properties.

In order to determine the matrix properties required for the micromechanics models tests were performed on unreinforced matrix resin samples. The cure cycle is the same as that used for the composites, DSC indicated complete curing and a Tg of 116°C, slightly higher than the values for the

composites with the same resin. The Young's modulus was measured by both tensile tests and nanoindentation for comparison. The former value was retained for subsequent modelling.
Polymer modulus measured by nanoindentation are frequently found to be higher than those from tensile tests. This can be explained by the difference in test frequency (70 Hz for nano-indentation, quasi static for a tensile test) and by a hydrostatic pressure effect under the indenter [30]. Lower values at the start of the indentation curve have been attributed to scale effects, surface roughness and local oxidation [31].

3.5.2. Estimation of transverse modulus of carbon fibres using inverse micromechanics.

Once the constituent properties and the fibre content are known, there are various micromechanics expressions which allow the transverse modulus to be determined for a unidirectional composite. These are based on continuum mechanics with a unit volume (one fibre surrounded by matrix) assumed to represent the composite material. From the large number of published models three of the most popular have been retained here, those of Halpin-Tsai [32], Tsai-Hahn [33] and Hopkins-Chamis [34]. All the input values are summarised in Table 7.

Halpin-Tsai:

This model is based on Hill's self-consistent model [32]. The unit cell is a cylindrical fibre, continuous and perfectly aligned, surrounded by a resin cylinder. This is in turn surrounded by a material with the composite properties.

$$\frac{E_t}{E_m}=\frac{1+\xi\eta V_f}{1-\eta V_f} \quad \text{with} \quad \eta=\frac{\frac{E_{ft}}{E_m}-1}{\frac{E_{ft}}{E_m}+\xi}$$

E corresponds to the modulus and the subscripts m and f correspond respectively to matrix and fibre. ξ is the geometrical parameter of the reinforcement. For transverse tensile loading, ξ=2. This value gives good results for circular fibres with 55% volume fraction and was applied here. The geometrical parameter is often taken as 2 but strictly it should be recalulated for other fibre shapes or contents.

Tsai-Hahn:

An alternative model is that of Tsai and Hahn based on rule of mixtures [33]. :

$$\frac{1}{E_t}=\frac{1}{V_f+\omega(1-V_f)}*\left(\frac{V_f}{E_{f_t}}+\eta\frac{(1-V_f)}{E_m}\right)$$

For a carbon/epoxy composite Lee et al. [34] compared experimental results with those from three micromechanics models and found that the expression above gave the best prediction of transverse modulus, with a transverse tensile stress partitioning factor ω = 0.25. This value of η of 0.25 was determined empirically for T300 fibres. In another study by Gibson a value of η of 0.5 was used [5]. Both values were used here for comparison. The ω = 0.5 value was found to give a closer correlation with our experimental results and the other models.

Hopkins-Chamis :

The third model is based on a law of mixtures, which uses series and parallel representations [35]. The fibre is modelled as a square with an area equivalent to that of the circular fibre. The unit cell is divided into three parts, with a zone of width equal to that of the square fibre, containing in series a matrix zone, the fibre and matrix. This is surrounded (in parallel) by two identical resin zones.

$$E_t = E_m\left[(1-\sqrt{V_f}) + \frac{\sqrt{V_f}}{1-\sqrt{V_f}\left(1-\frac{E_m}{E_{ft}}\right)}\right]$$

Hopkins and Chamis consider the fibre to be square, but the circumference of a square and a circle are not the same. This difference changes the interface contribution, which is very important in transverse loading.

These semi-empirical models are based on idealized conditions for their unit volumes which are not necessarily realistic. The simplifications include :
A uniform fibre distribution with a regular pattern
No porosity or defects
No residual stress (whereas temperature is used to cure, the thermal expansion coefficients of fibres and matrix are different, cure shrinkage may also occur).
Perfect interfacial bonding between the resin and fibres
Absence of an interphase between fibres and matrix
Linear elastic behaviour of both fibres and matrix
Uniform curing of the composite
Perfect alignment of fibres

3.5.3. Alternative models.

The micro-mechanics models described above, while widely used are open to criticism, as they assume regular fibre arrays (square or hexagonal). An alternative but more time-consuming approach is to use finite element or finite difference modelling to study the behaviour of a representative volume element with randomly distributed fibres [23,36,37]. This representative volume, often defined based on microscopic observations, corresponds to a section containing numerous fibres. The distance between two fibres affects strain concentrations in the matrix and hence influences the transverse modulus of the ply [25]. Wongsto et al [36] showed that the transverse stiffness is higher for a random distribution than for a hexagonal array. They indicated that finite element results were close to experimental values and significantly higher than values calculated by micromechanics analyses. Finite element analyses allow more realistic material models to be applied, including effects such as matrix plasticity, interphases and scatter in fibre diameters.

3.6. Transverse fibre modulus, comparison between micromechanics and indentation results

Table 7 shows the input data for the micromechanics models and Table 8 and Figure 8 show the results. The calculated modulus values are quite similar for each model. Miyagawa [8] compared predictions of the models of Mori–Tanaka, Uemura, and Halpin-Tsai with two finite element models (2D and 3D). He showed that for T300 (HR) carbon fibres, the Halpin-Tsai analysis provided results closest to those measured using nano-indentation and micro-Raman spectroscopy. The model predictions presented here are globally similar to the nanoindentation values, but tend to give lower modulus values. This is coherent with previous results by the authors on glass/polyester (inverse modulus from Halpin-Tsai equations of 63.6 GPa for E-glass fibres) and with the FE models discussed above. In addition to the various simplifying assumptions of the micromechanics models it should also be noted that the transverse modulus values measured by indentation are obtained in a compression mode rather than in tension. Various previous studies have examined the differences between tensile and compressive moduli in the fibre axis direction and generally concluded that compression properties, both modulus and strength, are lower than those measured under tensile loading [38].
Much less work has been performed in the transverse direction but a previous study on pitch fibres measured transverse compression response directly [7] and reported modulus values in the range from 6 to 20 GPa. The value obtained in the present work (10.7 GPa) is in that range. Those authors suggest that the variation is related to differences in microstructure but showed that the orientation (radial or disordered) has less influence than inter-crystal bonding. It should be noted that in order for the differences noted in Table 8 to be only caused by the difference between tension and compression modulus would require this difference to be unexpectedly large.

4. Conclusion

The properties of unidirectionally reinforced carbon/epoxy composites are very anisotropic, with high longitudinal properties and poor transverse behaviour. The latter is of interest as their transverse strength often corresponds to the damage threshold of composite structures. Transverse modulus depends on the transverse fibre modulus but the latter is hard to measure and few values have been published for high moduulus carbon fibres.

In the present study nanoindentation has been used to measure transverse fibre modulus of different high modulus carbon fibres. The technique was first checked on glass fibres, then three high modulus carbon fibres were indented. Their transverse moduli ranged from 9 to 15 GPa, lower transverse moduli corresponding to higher axial modulus.

Transverse tensile behaviour of unidirectional composites reinforced with these three fibres indicated a strong influence of fibre on stiffness, strength and failure strain. This information is important for the design of racing yachts. Analysis of the transverse modulus indicated a strong correlation with fibre properties. From micromechanics analyses of these composites it was possible to calculate transverse fibre modulus values and compare these with the nanoindentation results. The trend of decreasing transverse stiffness with increasing fibre axial modulus was again observed. The micromechanics expressions were similar to but tended to underestimate the directly-measured nanoindentation values. This may result from the many simplifying assumptions in these expressions, though the difference in loading may also influence the results. Further studies are looking at fibre-matrix interfacial properties of these high modulus composites and the transfer to global composite properties.

References

[1] Hamada H., Oya N., Yamashita K., Maekawa Z.I.: Tensile strength and its scatter of unidirectional carbon fibre reinforced composites. J. Reinf. Plast. Compos. 16(2) 119-130 (1997)
[2] Guigon M., Microstructures des fibres de carbone, Ph.D.Thesis Université de Technologie de Compiègne, (1985).
[3] Chung D. D. L.: Carbon Fiber Composites, edition Butterworth-Heinemann (1994)
[4] Luyckx J. : fibres de carbone, technique de l'ingénieur article A 2 110 (in french)
[5] Gibson R.F: Principles of composite material mechanics. Engineering mechanics series. McGraw-Hill International Editions, New York, (1994).
[6] Kawabata S.: Measurement of anisotropic mechanical property and thermal conductivity of single fiber for several high performance fibers, Proc. 4th Japan-US. Conference on Composite Materials 253-262 Technomic Publishing Co., Lancaster PA (1989)
[7]. Fujita K, Sawada Y., Nakanishi Y.: Effect of cross-sectional textures on transverse compressive properties of pitch-based carbon fibers, Mater. Sci. Res. Int. 7 116-121 (2001)
[8] Miyagawa H, Mase T., Sato C., Drown E., Drzal L., Ikegami K.: Comparison of experimental and theoretical transverse elastic modulus of carbon fibers, Carbon 44 2002-2008 (2006)
[9] Ueda M., Takiguchi T.: Measurement of transverse Young's modulus of fibers by angular characteristics of ultrasonic scattering, Adv. Compos. Mater, 1 (4) 309-320. (1991)
[10] Meurs P., Scheurs P., Peijs T., Meijer E. : Characterization of interphase conditions in composite materials, Composites Part A, 27, 781-786 (1996)
[11] Fischer-Cripps C.: Nanoindentation, Springer (2002)
[12] Oliver WC, Pharr GM, An improved technique for determining hardness and elastic-modulus using load and displacement sensing indentation experiments, J. Mater. Res. 7 1564-1583 (1992)
[13] Bunsell A.R., Fibre reinforcements for composite materials, Elsevier Composite Materials Series, 482-484 (1988)
[14] Torayca standard TY-030B-01, method for determination of tensile strength, tensile modulus of elasticity, and elongation at break
[15] Morita M.: A study of relationship between composite strength and mono-filament strength, Progress in science and engineering of composites, Paper presented at the 4th Proceedings ICCM , Tokyo, p107-111 (1982)

[16] ISO 14577 Metallic materials – Instrumented indentation test for hardness and materials parameters, (2002)
[17] Godara A, Raabe D., Green S.: The influence of sterilization processes on the micromechanical properties of carbon fiber-reinforced PEEK composites for bone implant applications, Acta Biomaterialia 3, 209-220, (2007)
[18] Marx D. T., Riester L.: Mechanical properties of carbon-carbon composite components determined using nanoindentation, Carbon, 37 1679-1684 (1999)
[19] Yamashita Y, Kawabata S, Okaka S, Tanaka A: Mechanical Characteristics of PBO single fiber, Paper presented at the 7th Asian textile conference, New Delhi (2003)
[20] Ueda M., Takiguchi T, Measurement of Transverse Young's Modulus of Carbon Fibres by Angular Characteristics of Ultrasonic Scattering, Paper presented at the ultrasonic symposium, 1081-1085 (1990)
[21] de Kok J.M.M., Deformation, Yield and Fracture of Unidirectional Composites in Transverse Loading, Ph.D. Thesis, Eindhoven University of Technology, The Netherlands (1995).
[22] Benzarti K., Cangemi L., Dal Maso F., Transverse properties of unidirectional glass/epoxy composites: influence of fibre surface treatments, Composites Part A 32, 197-206. (2001)
[23] de Kok J.M.M., Meijer H.E.H.: Deformation, yield and fracture of unidirectional composites in transverse loading: 1. Influence of fibre volume fraction and test-temperature, Composites Part A, 30, 917-932 (1999)
[24] Keusch S., Haessler R.: Influence of surface treatment of glass fibres on the dynamic mechanical properties of epoxy resin composites, Composites Part A, 30, 997-1002 (1999)
[25] Gusev A.A., Hine P.J., Ward I.M.: Fiber packing and elastic properties of a transversely random unidirectional glass/epoxy composite, Composite Science and Technology, 60, 535-541 (2000)
[26] Baley C., Davies P., Grohens Y., Dolto G., : Application of Interlaminar Tests to Marine Composites. A Literature Review Appl. Compos. Mater. 11, 96-126, (2004)
[27] Kobets L., Deev I.: Carbon fibres: structure and mechanical properties, Compos. Sci. Technol., 57, 1571-1580, (1997)
[28] Dengn S., Ye L.: Influence of fiber-matrix adhesion on mechanical properties of graphite/epoxy composites : I. Tensile, flexure, and fatigue properties. J. Reinf. Plast. Compos., 18, 1021-1040, (1999)
[29] Adams D.F., Inelastic analysis of a unidirectional composite subjected to transverse normal loading. Journal of Composite Materials 4 (1970) pp 310-328.
[30] Briscoe B. J., Sebastian K. S., The elastoplastic response of poly(methyl methacrylate) to indentation. Proceedings of the Royal Society of London 1996;452.
[31] Hu Y., Shen L., Yang H., Wang M., Liu T., Liang T., Zhang J., Nanoindentation studies on Nylon 11/clay nanocomposites. Polymer Testing 2006;25(4):492.
[32] Halpin J.C, Kardos J.L.: The Halpin-Tsai equations: a review, Polym. Eng. Sci., 16, 344-352, (1976)
[33] Hahn HT, Tsai SW, Introduction to Composite Materials CRC Press (1980) 378-405
[34] Lee S-Y.,. Springer G.S: Effects of cure on the mechanical properties of composites. J. Compos. Mater., 22, 15-29, (1988)
[35] Hopkins D.A. and Chamis C.C., Unique set of micromechanics equations for high-temperature metal matrix composites, ASTM STP 964 (1988), pp. 159–176
[36] Wongsto A., Li S. Micromechanical FE analysis of UD fibre-reinforced composites with fibres distributed at random over the transverse cross-section. Composites Part A 36, 1246-1266. (2005)
[37] Zhang L., Ernst L.J., Brouwer H.R, Transverse behaviour of a unidirectional composite (glass fibre reinforced unsaturated polyester). Part I. Influence of packing geometry. Mechanics of Materials 27, 13-36(1998)
[38] Oya N.,. Johnson D. J, Longitudinal compressive behaviour and microstructure of PAN based carbon fibres, Carbon 39, 635–645(2001)

Tables

fibre	manufacturer	type	Modulus (GPa)	R (MPa)	density
M40J	Toray	PAN	377	4410	1.77
M46J	Toray	PAN	436	4210	1.84
K63712	Mitsubishi	Pitch	640	2600	2.12

Table 1: Selected fibres and their tensile properties [data Toray, Mitsubishi]

fibre	resin	density	V_f (%)	Tg (°C)	ILSS (MPa)
M40J	Structil R367/2	1.49	52	111	62 (2)
M46J	Structil R367/2	1.53	53	106	65 (1.3)
K63712	Structil R367/2	1.67	52	108	46 (2.6)

Table 2 : Selected composites and their properties

	Surface area (μm²)	Circumference (μm)	Equivalent diameter (μm)
M40J	25.2 (1.78)	18.2 (0.6)	5.8
M46J	23.5 (1.31)	17.7 (0.5)	5.6
K63712	86.6 (3.21)	33.0 (1.1)	10.5

Table 3: Mean fibre measurements (standard deviation), from measurements by CAD software on over 200 fibres in each composite.

fibre	Transverse Modulus (GPa)	Longitudinal modulus (supplier values) (GPa)
M40	15 (±4,9)	294
M46	14 (±5.7)	377
Pitch K637	10.7 (±3.1)	640
E-glass	67.8 (±15)	70

Table 4: Transverse modulus of fibres measured by nanoindentation and suppliers' values of longitudinal modulus.

		Transverse tensile properties		
	V_F (%)	E_T (GPa)	σ (MPa)	Failure strain (%)
M40J	52	6.64 (±0.32)	30.2(±0.8)	0.45 (±0.02)
M46J	53	6.15 (±0.40)	24.3 (±1.0)	0.40(±0.01)
Pitch K637	52	4.98 (±0.15)	21.1 (±1.2)	0.42 (±0.02)

Table 5: Results from transverse tensile tests, mean values and standard deviations.

	Tension test	nanoindentation	DSC
Matrix resin	E (GPa)	E (GPa)	Tg (°C)
367-2	3.1 (±0.2)	4.5 (±0.1)	116

Table 6 : Measured matrix properties

	Properties	V_f	Model parameters
M40J	E_f =377 GPa	52%	
M46J	E_f =436 GPa	53%	
Pitch K637	E_f =640 GPa	52%	
Matrix	E_m = 3.1 GPa		
Halpin-Tsai			ξ=2
Tsai-Hahn			ω = 0.25 and 0.5
Hopkins - Chamis			

Table 7. Input values for micromechanics models

fibre	Halpin-Tsai	Tsai-Hahn		Hopkins - Chamis	Nanoindentation
		ω=0.25	ω=0.5		
M40	11.82	9.01	11.32	15.11	15 (±4.9)
M46	10.16	7.95	11.23	9.5	14 (±5.7)
Pitch 637	6.76	5.79	6.18	7.78	10.7 (±3.1

Table 8: Results for transverse modulus of carbon fibres estimated using micromechanics models and comparison with measured nanoindentation values.

Figures

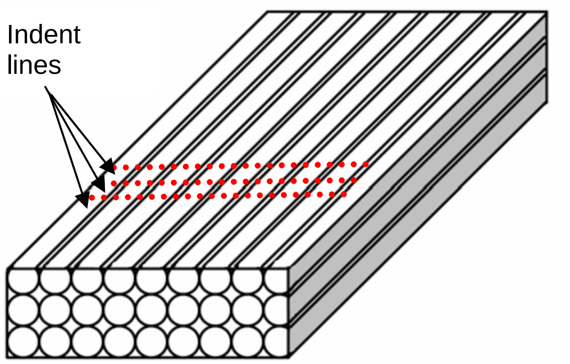

Figure 1: Schematic diagram showing indentations.

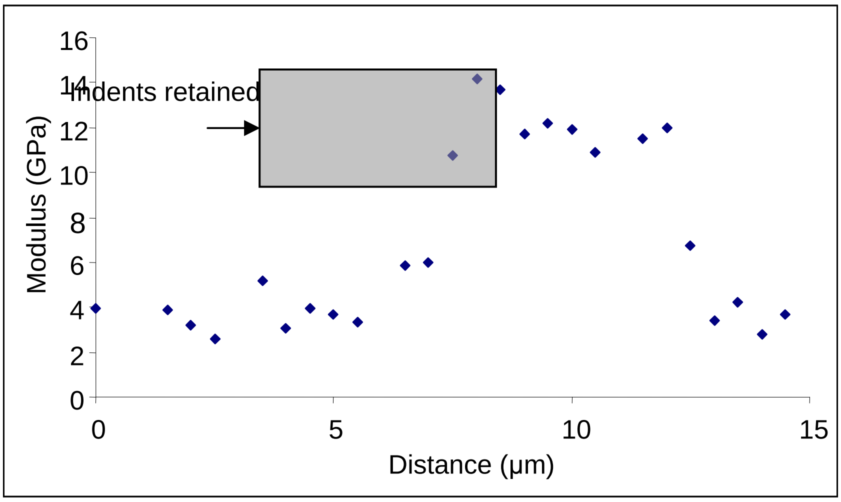

Figure 2 : Example of values measured along one indent line across matrix and fibre.

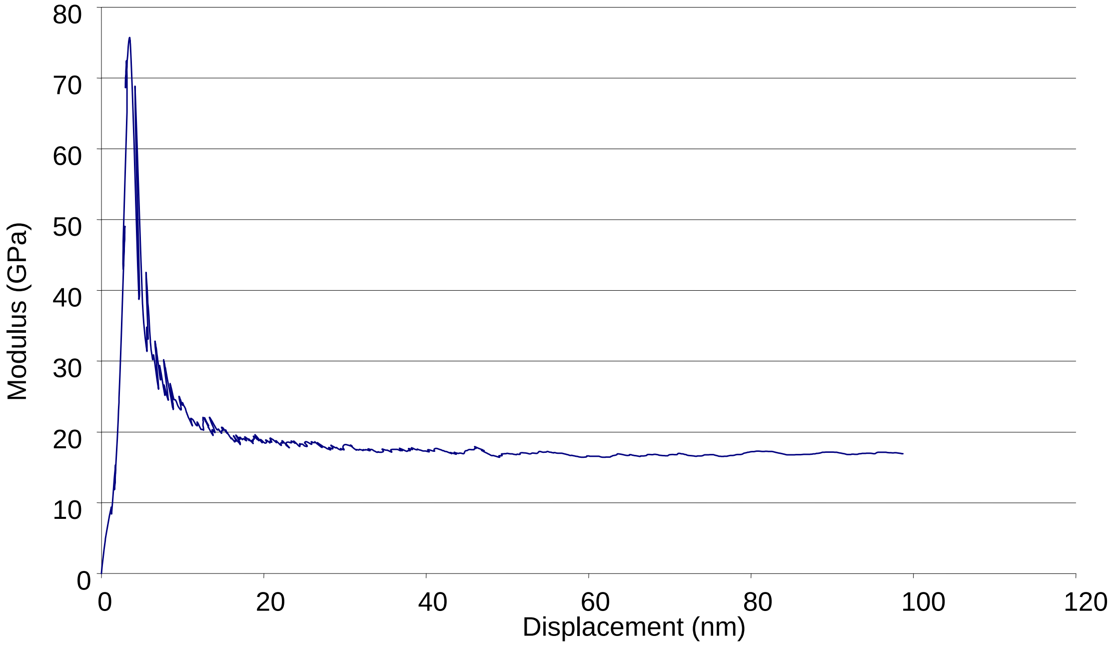

Figure 3 : Variation of modulus with penetration depth

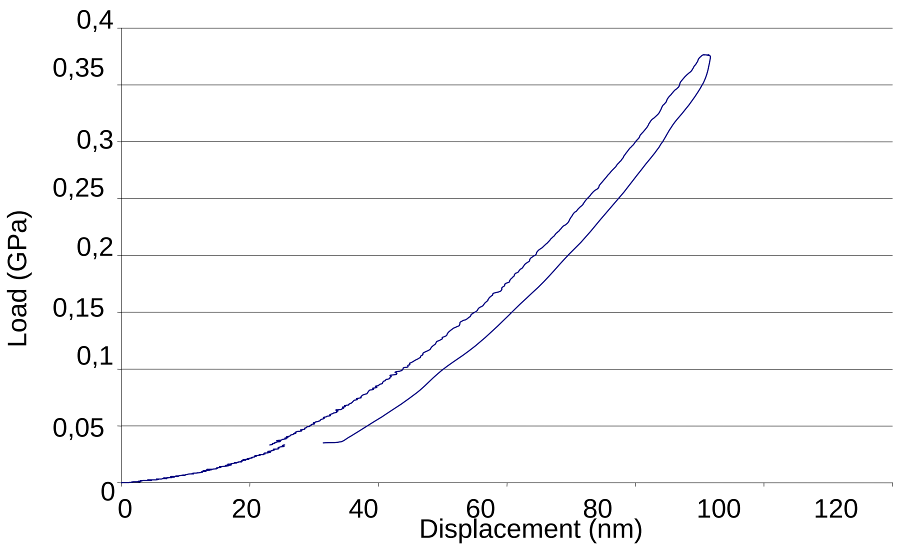

Figure 4 : Load-displacement plot for nanoindentation

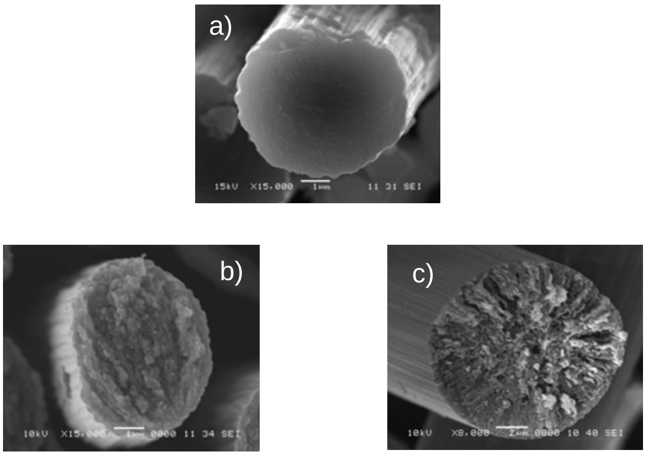

Figure 5 : a) M40J,b) M46J, c) K63712 Fibre cross-sections.

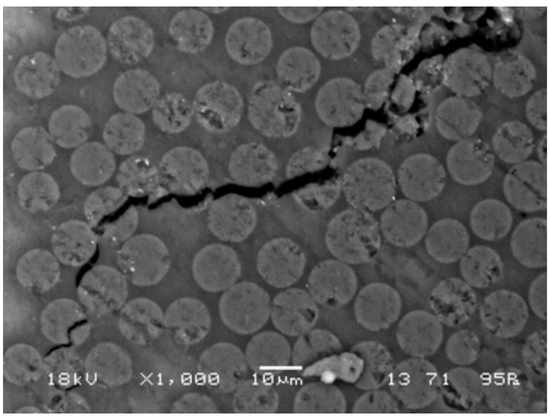

Figure 6: Transverse tensile failure in SEM of unidirectional Pitch fibre reinforced composite, loaded vertically.

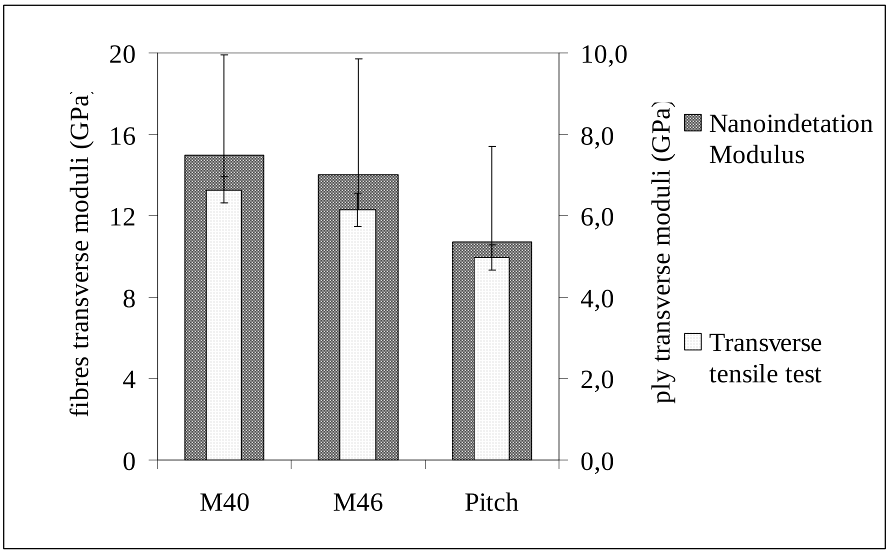

Figure 7 : Transverse moduli values for different fibres and composites.

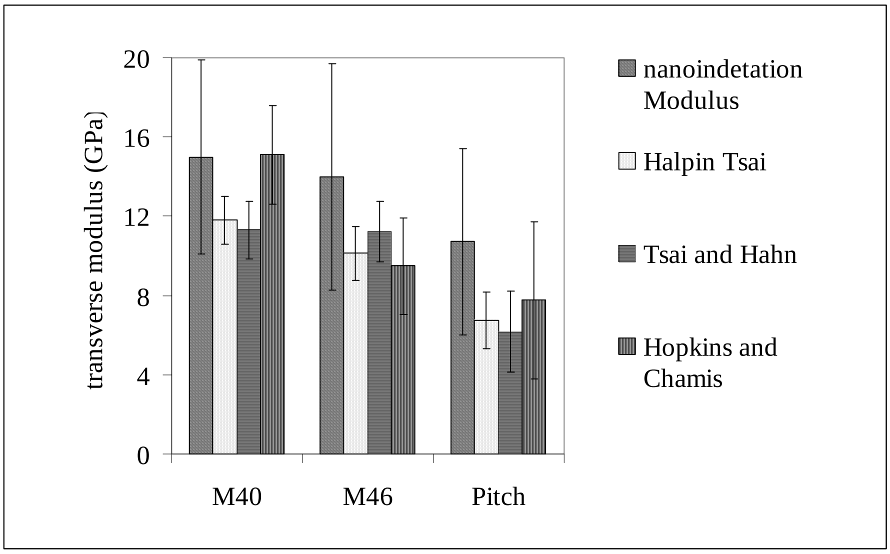

Figure 8 : Transverse modulus of carbon fibres. Comparison between nanoindentation and values calculated from inverse micromechanics modelling