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Invited Paper

NON DESTRUCTIVE EVALUATION OF POLYMERS: SOME NEW DEVELOPMENTS

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ABSTRACT

Polymers initially intended to be a cheap substitute for metals have found their way into applications where their high specific strength (after fibre reinforcement) is important. As many of these applications are safety-relevant, one is highly interested in non destructive evaluation (NDE) of polymer materials. This paper shows which methods provide which kind of information, their advantages and drawbacks. Remote detection of delaminations, impact damage, fibre orientations, and curing processes will be highlighted for glass fibre (GFRP) and carbon fibre reinforced polymer materials (CFRP).

INTRODUCTION

Polymer composites have an increasing potential of substituting metals, because of their high specific strength, resistance against corrosion and low production costs.

Since manufacturers have a strong responsibility for their products, quality control has become an important activity that may range from the production process to maintenance and service inspection. This is the only way to make sure that low quality products are replaced early enough to avoid failure while unnecessary replacement of intact components is avoided.

In the field of metals there is a lot of experience in finding defects and interpreting the data: standard tools for non-destructive evaluation (NDE) like ultrasonics, x-rays, and eddy-currents are in use for many years. Unfortunately, these methods are sometimes less suitable for the inspection of polymer materials. Reasons are the lower atomic weight, the higher acoustic attenuation, and the low electrical conductivity. On the other hand, these properties favour other techniques which respond more sensitively to defects in polymers and their composites. It is a challenge to further develop these techniques, in order to make the new materials safer and hence more applicable to new fields.

In any case, every technique of non-destructive testing means basically that the component to be inspected is exposed to some kind of external excitation using electromagnetic, elastic, or thermal waves and that the observed response to this input characterises the component. Therefore every result shows the component and its defects under the specific aspect of the physical interaction involved. Consequently one needs more than just one method to characterise the variety of defects in polymer materials. The purpose of this paper is to demonstrate how some modern methods work and what results one can reasonably expect for applications on long and short fibre reinforced polymer materials. Of particular interest are those methods which do not require physical contact with the inspected object and which are also applicable under industrial conditions outside the laboratory.

NDE OF LAMINATES

Laminates are layered materials where each layer has a different unidirectional fibre orientation. Such materials have high strength at low weight, therefore they are very popular in aerospace applications or whenever the structure should be light. The mechanical performance depends critically on the quality of bonding between the laminate layers. That is why delaminations or impact damage caused in the production process or later on by improper use (e.g. dropped tools) are defects whose detection is important for safety reasons.

These laminates can contain carbon fibres (CFRP = carbon fibre reinforced polymer) whose thermal conductivity differs substantially from the matrix. Therefore heat transport methods respond in a sensitive way to the relevant defects. If this heat transport is modulated, one obtains phase information which is independent of how much heat was injected initially. Such measurements can be performed in a completely remote way if the heat is generated in the sample itself by absorption of intensity modulated radiation while the thermal response is analysed with respect to the modulation of emitted infrared thermal radiation. In this „photothermal" [1] technique the generated temperature modulation propagates as a „thermal wave" in the structure under test [2] and is reflected at boundaries like all other waves. The wave reflected back to the surface modifies the temperature modulation and thereby reveals the hidden near-surface defect, where „near" means a depth range which is about twice the thermal diffusion length μ given by

$$\mu = \sqrt{2\lambda / \omega \rho c}$$

(ω = angular frequency of modulation, c = specific heat, ρ = density, λ = thermal conductivity), if the signal phase is used for defect detection [3-5]. The dependence on modulation frequency provides a chance for remote depth profiling [2]. Low frequencies are required for the detection of delaminations in a depth of several mm. If such measurements are performed one after the other in a step-by-step raster image, the time required for data acquisition is too long for industrial applications. But it can be reduced by orders of magnitude if a multiplex photothermal inspection („lockin-thermography" [6-8]) is used: this modification of thermography responds only to the induced temperature modulation and displays the local phase angle as an image. This way one obtains even large scale images within typically 3 minutes which show impact damage, delaminations and even the adhesion of stringers underneath the outer surface of an airplane structure whose size can range up to several m^2. It should be mentioned that the detection of these defects is also possible for GFRP (glass fibre reinforced polymer) used for airplanes and less demanding applications.

Another technique for remote inspection is optical interferometry where defects show up by their effect on deformation fields induced by mechanical or thermal stress. For example a delamination causing a tiny bump a few μm high can be quantified accurately by the number of interference fringes around it. However, this technique is sensitive to external vibrations and therefore in most cases less applicable under rough conditions outside the laboratory.

A very reliable technique is ultrasonic inspection where the reflection of elastic waves results in an image of defect boundaries. Both generation of the waves and the detection of their echoes are usually performed by piezoelectric transducers requiring physical contact (e.g. water coupling) with the inspected component. This may result in a time consuming raster scan where echoes are analysed subsequently to acquisition at each picture element („pixel"). Recently a method has been developed that combines ultrasonics with lockin- thermography: amplitude modulated ultrasound is radiated into the inspected object. The elastic waves propagating in the test object are more efficiently converted into heat in defective areas with locally enhanced hysteresis effects. If the amplitude of ultrasound is modulated at a low frequency each defect emits a thermal wave by which it can be detected remotely at the surface [9]. Fig. 1 shows the experimental arrangement together with an example where 7 impact damage areas are revealed [10].This dark-field technique responds selectively to defects and provides an image within several minutes.

Besides the detection of delaminations and impact damage there is another topic of interest: the orientation of carbon fibres which determines the anisotropy of strength. There are essentially three ways to determine fibre direction in a remote and nondestructive way: x-ray diffractometry [11, 12], ultrasound backscattering [13, 14], and thermal wave ellipsometry [14, 15]. All of these methods are based on the local anisotropy induced by the orientation of the cylindrical fibres. In x-ray measurements the tiny difference in the refraction indices of fibre and matrix accounts for the observed contrast. The effect of ultrasound backscattering can be visualised as specular reflection at fibres which are suitably oriented. Therefore rotation of the experimental arrangement with respect to the samples results in narrow maxima with a high angular resolution. However, this technique requires liquid coupling of the ultrasonic transducer to the inspected object. This need is eliminated in the case of locally generated thermal waves: an intensity modulated laser beam focused to a small spot on the test object generates an oscillating temperature field. Due to the thermal anisotropy induced by the carbon fibres, the lines of equal temperature and of equal temperature phase are no longer circles, but have an elliptical shape with the longer axis oriented parallel to the fibre direction. The area close to the focal spot displays the fibre orientation close to the surface while the orientation in greater depth is displayed by the outer part of the thermal wave field [14, 15]. A multi-focus array [16] shows the orientation in many points at the same time.How-

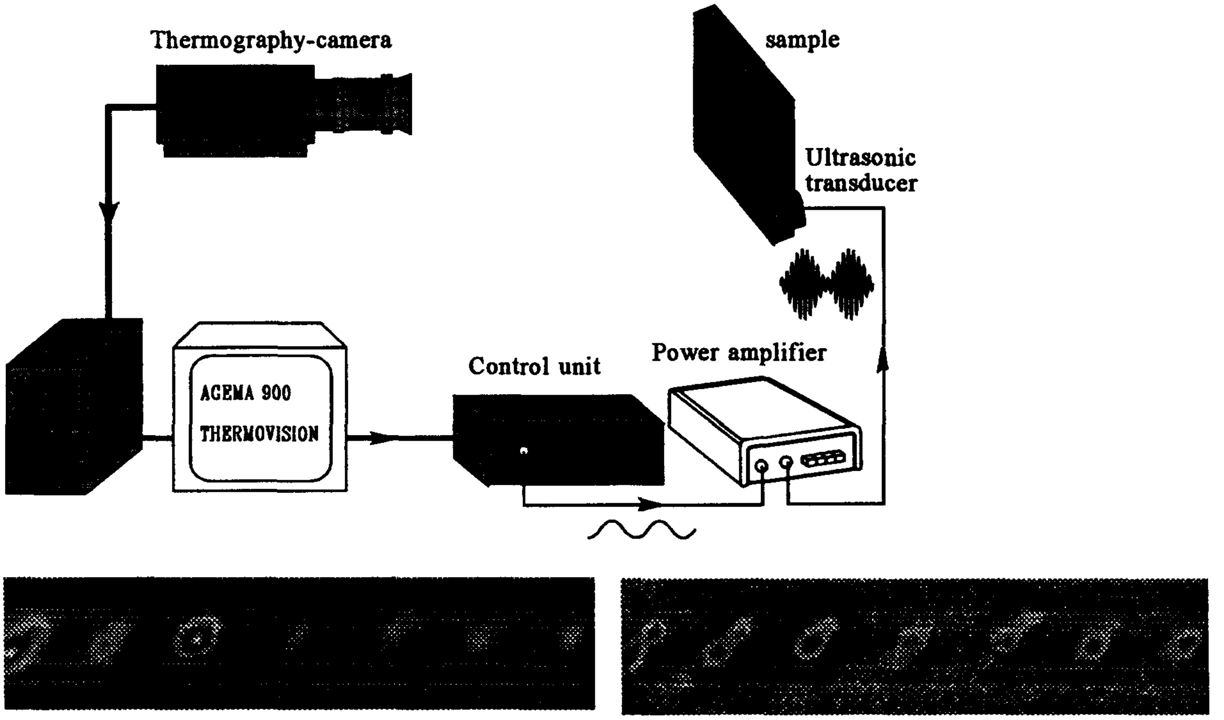

Fig. 1: *Ultrasound lockin-thermography: principle and application for the detection of impact damage in CFRP laminate. Images of magnitude (left) and phase (right).*

ever, thermal ellipsometry is applicable only to carbon fibres which have a high thermal conductivity. For fibres of other materials microwaves can be used which probe the local fibre induced anisotropy of the dielectric properties [17, 18]. Microwaves are also suited to detect delaminations in glass fibre reinforced laminates [19].

NDE OF INJECTION MOULDED POLYMER COMPONENTS

Mass production of polymer components is usually performed by injection moulding where hot and liquid thermoplastic material cools down in a mould to become a solid of the required shape. The rapid flow during the injection process causes local orientations of molecules and fibres which are frozen as the polymer cools down. The result is local anisotropy of properties. To optimise the mechanical performance one is interested to achieve proper fibre alignment or at least to avoid dangerous misalignments which might cause failure under load.

Due to the three-dimensional flow situation the fibre orientation field is significantly more complicated than in laminates. Also the fibres are much shorter (typical about 200 µm length) and the volume content of fibres is smaller (typically around 30 %). All this makes non destructive inspection more difficult. As carbon fibres are rarely used for injection moulding, thermal methods are not well suited for this application, mainly because the resulting local thermal anisotropy is too small. Microwaves, however, work so well under these conditions, that raster images clearly display fibre orientation fields and their changes caused by variation of production parameters [17]. As an example Fig. 2 shows the experimental arrangement for microwave orientation imaging together with results obtained at two different injection speeds [18]. As one image is obtained within about 20 minutes (while destructive sectioning would require many hours), one has a rather quick feedback to optimise production processes.

Such microwave raster images show the orientation as an average over the component thickness. However, there is considerable interest to know how orientation depends on depth. Recent results obtained with standing fields indicate that microwaves have the potential for remote orientation depth profiling [18].

While elastic waves were very successful in analysing fibre directions in laminates by backscattering at non-oblique incidence [13, 14], there is no indication of similar results on short fibre injection moulded components. Presumably one reason is the weaker reflection at

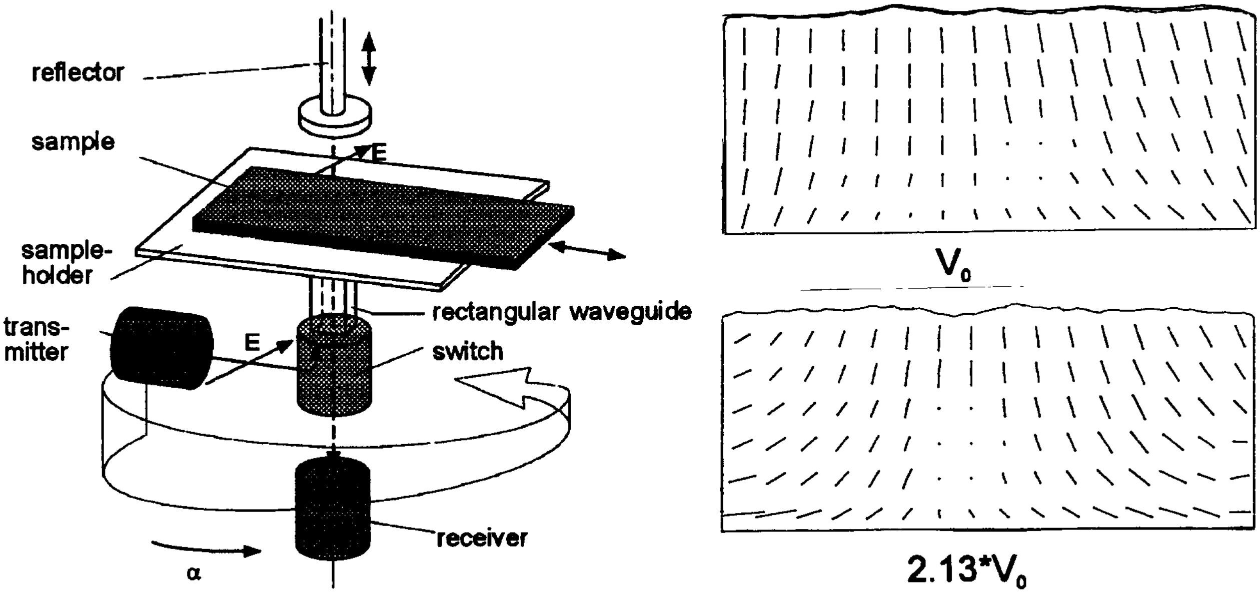

Fig. 2: *Microwaves for orientation imaging of short glass fibres in injection moulded plate: setup and examples obtained at 2 different speeds of injection.*

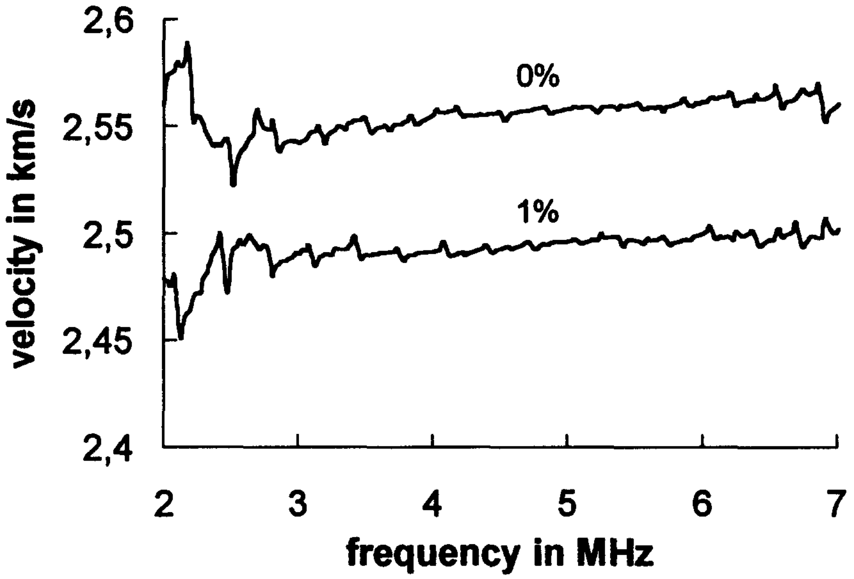

Fig. 3: *Influence of tensile loading with 1 % maximum strain on phase velocity of ultrasound.*

shorter fibres, another is that laminates have an orthotropic arrangement whereas short fibres may have orientation components vertical to the surface. Also the angular distribution is much broader: in laminates one has a small number of certain well defined directions whereas short fibres can have any direction which results in a permanent background of reflections. Therefore both thermal and mechanical waves seem to be less efficient to analyse fibre orientation fields in injection moulded components.

While defects in laminates are very often planar and parallel to the surface, they may have almost any kind of geometry in injection moulded components (e.g. bubbles and cracks). We found that microwaves and interferometry are suited for remote NDE. Contacting ultrasonic pulse-echo techniques are applicable as well if the effect of damping can be reduced by working at lower frequencies. We found that the propagation of elastic waves is modified by damage, as is shown in Fig. 3 by comparing data obtained before and after subjecting GFRP samples to 1 % strain [21] in tension test. In some cases ultrasonic lockin-thermography mentioned above may be successful, too, at least in those situations where the hidden defect generates an enhanced hysteresis effect or stress concentration. As compared to conventional ultrasonics, this technique provides an image of the imaginary part of local stiffness, and the ultrasound is injected just by clamping a transmitter to the component which is inspected within about 3 minutes.

NDE FOR PROCESS MONITORING

Polymer materials may undergo changes during the production process or due to deterioration. In order to understand these developments one must investigate e.g. their speed and their asymptotic behaviour. We found that dielectric and mechanical spectroscopy are able to monitor these changes, as the following examples will demonstrate.

In dielectric spectroscopy the investigated sample acts as a medium in a capacitor to which an oscillating voltage of variable frequency is applied. The current as a response to this input is analysed. This way one obtains the frequency dependent complex dielectric constant ε. Since many polymers contain dipoles it is obvious that their tendency to align along the electric field results in a frequency behaviour that can be described by a Debye-relaxation model which contains mainly the relaxation time τ as a parameter. The inverse value of this time is the angular frequency where the real part of the dielectric function ε(ω) has its steepest (step-like) decrease. During polymerisation processes this time changes. Therefore a frequency scan (which takes only several seconds if analyser is used) reveals the state of curing a network which is of practical interest e.g. if one wants to monitor adhesives [22].

While dielectric spectroscopy requires the applicability of electrodes to the inspected sample, the situation may be less demanding if mechanical spectroscopy is performed. Here one investigates how the sample reacts to mechanical input, and the observed response (consisting in a spectrum of eigenfrequencies) characterises the stiffness under various kinds of oscillating strains. There are basically two kinds of mechanical excitation: a short pulse has a broad frequency spectrum and hence excites a wide range of eigenfrequencies which are determined by the Fourier-transform of the vibration observed after the input pulse. Alternatively a frequency sweep of a sinusoidal excitation can be used to investigate the frequency dependent mechanism of response. This method is slower, but it may be advantageous if one wants to monitor only frequency changes within a small spectral range, e.g. of an eigenfrequency. There is one more advantage: while mechanical spectroscopy („vibrometry“) is usually performed in a contacting way with an attached piezoelectric transducer, continous wave excitation may be achieved by absorption of intensity modulated light [23]. If interferometric detection is used to analyse the oscillation, then one can perform laser vibrometry in a remote way where the results are no longer affected by attached sensors or transmitters [24].This way samples can be investigated which are very small, sticky, or hot. Applications are e.g. polymerisation, drying processes, and water diffusion [25, 26]. As an example for remote vibrometry on a small polymer sample, Fig. 4 shows the anisotropy of vibration frequency induced by molecular orientation caused by previous strain [27]. As the frequency resolution may range up to 10^{-5} or 10^{-6}, one has a very sensitive indicator for in-situ monitoring of processes, as shown previously by an experiment on the sintering process of ceramics [23].

Since there are no problems related to the reproducibility of sensor attachment, remote laser vibrometry is also suited to detect statistical fluctuations of component geometries and density distributions in the production line (as a feedback for process control). If the initial eigenfrequencies are known which characterise the component like a fingerprint, then changes observed later on during inspection (after certain intervals of service) may be used as criteria to decide upon further safe use.

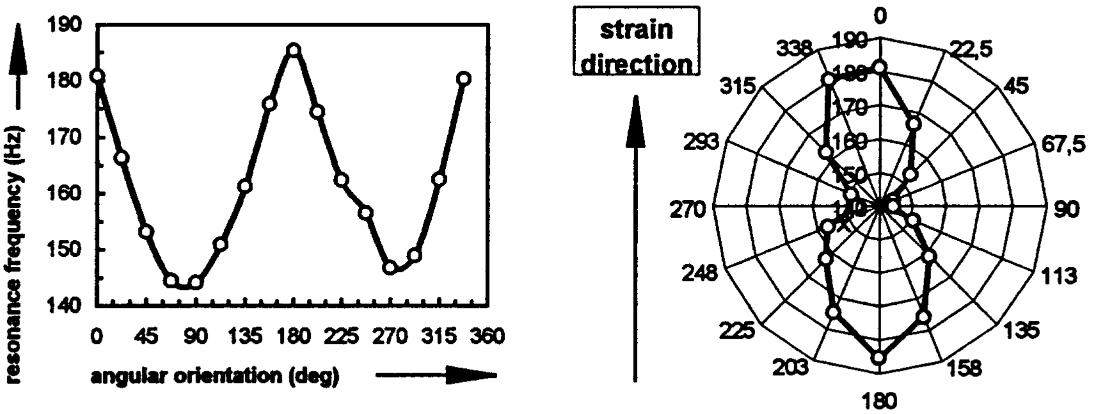

Fig. 4: *Anisotropy of vibration due to strain induced molecular orientation. Cicular PVC sheet (40 mm diameter, 0.4 mm thickness) clamped at various angles along its diameter. The polar plot shows that the higher frequency is obtained for bending along the molecular chains.*

CONCLUSION

Though polymer materials have physical properties different from metals for which conventional NDE methods have been originally developed, they can be tested using new and powerful methods. Depending on the kind of information that is required and on the specific needs of the component to be inspected, one has to select the most promising method.

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REFERENCES

[1] NORDAL, P.-E.; KANSTAD, S.O.: *Photothermal Radiometry.* Physica Scripta 20 (1979), p. 659-662

[2] ROSENCWAIG, A.: *Photoacoustic Spectroscopy.* American Lab. 11 (1979), p. 39-49

[3] BUSSE, G.: *OptoacousticPhase Angle Measurement for Probing a Metal.* Appl. Phys. Lett 35 (1979), p. 759-760

[4] BENNETT, C.A.; PATTY, R.R.: *Thermal Wave Interferometry: a Potential Application of the Photoacoustic Effect.* Appl. Opt. 21 (1982), p. 49-54

[5] LEHTO, A.; JAARINEN, J.; TIUSANEN, T.; JOKINEN, M.; LUUKKALA, M.: *Magnitude and Phase in Thermal Wave Imaging.* Electr. Lett. 17 (1981), p. 364-365

[6] BEAUDOIN, J.L.; MARIENNE, E.; DANJOUX, R.; EGEE, M.: *Numerical System for Infrared Scanners and Application to the Subsurface Control of Materials by Photothermal Radiometry.* Infrared Technology and Applications. SPIE vol. 590 (1985), p. 287

[7] KUO, P.K.; FENG, Z.J.; AHMED, T.; FAVRO, L.D.; THOMAS, R.L.; HARTIKAINEN, J.: *Parallel Thermal Wave Imaging Using a Vector Lockin Video Technique.* Photoacoustic and Photothermal Phenomena (P. Hess and J. Pelzl Eds.), Springer-Verlag, Heidelberg (1988) p. 415-418

[8] BUSSE, G.; WU, D.; KARPEN; W.: *Thermal Wave Imaging with Phase Modulated Thermography.* J. Appl. Phys. 71 8 (1992), p. 3962-3965

[9] RANTALA, J.; WU, D.; BUSSE, G.: *NDT of polymer materials using lock-in thermography with water-coupled ultrasonic excitation.* NDT & E International (1995), in print

[10] WU, D.; KARPEN, W.; STEEGMÜLLER; R.; DIENER, L.; RITTER; S.; BUSSE; G.; RANTALA, J.: Nondestructive evaluation of lyered and coated materials (Van Hemelrijck & Anastassopoulos (eds.). lst Joint Belgian-Hellenic Conference on Nondestructive Testing, Balkema, Rotterdam. (1996), p. 213-2169

[11] HENTSCHEL, M.P.; LANGE, A.; VOSSHENRICH, B.; PIOTTER, V.: *Orientierungsverteilung mit der Röntgen-Raster-Diffraktographie bestimmen.* Kunststoffe 80 (1990) 12, p. 1367-1370

[12] EKENHORST, D.; LANGE, A.; HENTSCHEL, M.P.: *Röntgen-Refraktion.* Materialprüfung 39 (1997), p. 175-179

[13] PFEIFER, T.; VON WACHTER, F.K.; SCHUSTER, J.: *Backscattering - Neues Ultraschallprüfverfahren für Faserverbundbauteile.* Ingenieur Werkstoffe 2 No. 9 (1990), p. 46-49

[14] DIENER, L.; WU, D.; STEEGMÜLLER, R.; BUSSE, G.: *Nondestructive Evaluation of Fibre Orientation:* Non Destructive Testing. (Van Hemelrijck & Anastassopoulos (eds.). lst Joint Belgian-Hellenic Conference on Nondestructive Testing, Balkema Rotterdam. (1996), p. 245-249

[15] KRAPEZ, J.C.; CIELO, P.; MALDAGUE, X.; UTRACKI, L.A.: *Optothermal Analysis of Polymer C omposites.* Polymer Composites 8 (1987), p. 396-407

[16] KARPEN, W.; WU, D.; STEEGMÜLLER, R.; BUSSE, G.: *Depth Profiling of Orientation in Laminates with Local Lockin Thermography.* Proc. QIRT '94, Paris: Editions Européennes Thermique et Industrie (1995), p. 281-286

[17] HOLDEN, A.; ALLAN, P.S.; BEVIS, M.J.; DIENER, L.; BUSSE, G.: *Monitoring the Multiple Live-Feed Moulding Process with Microwave Orientation Imaging.* German Plastics, 82 (1992), p. 28-29

[18] DIENER, L.: *Zerstörungsfreie Kunststoffprüfung mit Mikrowellen im Nahfeld offen endender Hohlleiter.* Thesis, Fak. Verfahrenstechnik, Universität Stuttgart (1997).

[19] DIENER, L.: *Microwaves for Raster Imaging of Local Anisotropies in Polymer Materials.*Review of Progress in Quantitative NDE 14 (1995) p. 615-622

[20] DIENER, L.: *Microwave Near-Field Imaging with Open-Ended Waveguide - Comparison with Other Techniques of Nondestructive Testing.* Res. Nondestr. Eval. (1995) 7, p. 137-152

[21] STANULLO, J.; BOJINSKI, S.; GOLD, N.; SHAPRIO, S.; BUSSE, G.: *Ultrasonic Signal Analysis to Monitor Damage Development in Short Fibre Reinforced Polymers.* Ultrasonics International '97, Delft, The Netherlands, July 2-4, 1997 (submitted).

[22] BUSSE, G.; BINDER, B.; BOHNACKER, A.; DIENER, L.; ELSNER, P.: KARPEN, W.; OTA, M.; RIPPEL; W.; RITTER, S.; WU, D.: *Zerstörungsfreie Prüfung von Polymerwerkstoffen - Neue Entwicklungen und Anwendungen.* 13. Stuttgarter Kunststoffkolloquium, Hüthig-Verlag, Heidelberg (1993), p. 25-40

[23] WHITE, R.M.: *Generation of Elastic Waves by Transient Surface Heating.* J. Appl. Phys. 34 (1963), p. 3559-3567

[24] LYAMSHEV, M.L.; STANULLO, J.; BUSSE, G.: *Thermo Acoustic Vibrometry. Remote „in situ" Monitoring of Ceramic Sintering.* Materialprüfung 37 1-2 (1995), Carl Hanser Verlag, München, p. 22-24

[25] GEISSE, D.; NIXDORF, K.; BUSSE, G.: *Cure Monitoring of Thin Adhesive Layers.* ICSV'5, University of Adelaide, South Australia Dec. 15-18, 1997 (submitted)

[26] DÖTTINGER; CH.; STANULLO, J.; LYAMSHEV, M.L.; BUSSE; G.: *Remote Vibrometry for Characterisation of Materials and Processes.* ICSV'5, University of Adelaide, South Australia Dec. 15-18, 1997 (submitted).

[27] DÖTTINGER, CH.: *Laservibrometrie: Aufbau und Erprobung einer Apparatur.* Thesis for a diploma, Fak. Verfahrenstechnik, Universität Stuttgart (1996).