EVALUATION OF THE DEFORMATION OF POLYAMIDE MATERIALS UNDER TENSION USING LASER EXTENSOMETRY

C. Bierögel, T. Fahnert and W. Grellmann

Institute of Material Science, Martin-Luther-University Halle-Wittenberg,
D-06099 Halle/Saale, Germany

ABSTRACT

For the mechanical characterization of polymeric materials the tensile testing is the most important method to evaluate the strength and deformation behaviour. Because of the integral measurement of strain by means of mechanical extensometers exists no possibility for the description of load induced deformation mechanisms. The heterogeneity of these materials caused by the orientation produces local differences for strain and the material parameters. Additionally the local strain rates are varying to the used integral strain rate and so relaxation behaviour for the investigated volume is not comparable.

Laser extensometry as a method for measuring local strain has been used for evaluating the mechanical strain and failure behaviour of glass fiber reinforced polyamide with different fiber content. All specimens were investigated under the condition of constant cross head speed and strain controlled too. For all experiments the acoustic emission analysis was used for the determination of damage kinetics and fracture mechanisms.

The local deformation and acoustic emission parameters show different material behaviour in dependence on orientation and test conditions. For all materials the heterogeneity for constant strain rate is higher as in the case of closed loop experiments which is established by the different relaxation conditions. The results of these experiments are discussed by means of electron microscopy investigations of fracture surface. The conformity of the results of the various methods applied has proved the suitability of laser extensometry to determine the local strain and failure properties and mechanisms, too.

INTRODUCTION

The practice of polymers is strongly characterized by an increasing of product specialization and variety. At the same time an increasing of application field for the constructive used plastics like polyamide can be observed.

As a result of the wide range of polymeric materials, the different thermal and electrical properties and also many modifications in strength and toughness exist an expansive requirement to the modern material testing. This concerns specially the sensibility and precision of test methods and the qualification of the selected materials parameters. For the material development, quality assurance and failure analysis the essential relation ships between properties and structure must be well known for simulation of life time and reliability and the evaluation crack initiation and propagation too.

Under the aspect of differentiation of plastics, the evaluation of used modifiers for strength or toughness and the dimensioning of polymer components the users or developers of plastics expect qualified material parameters. These constructive parameters must be independent of methodical and technical factors of

influence. This concerns the conventional testing methods as tensile or flexural testing as well as methods of the experimental fracture mechanics used for the characterization of toughness of polymers and blends. Independent of the respective test method and the used geometry of specimen (specimens according to ISO 3167 or fracture mechanics Compact-Tension (CT-) specimens) the determined parameters of strength, deformation or toughness reflects the influence of manufacturing and loading. The geometrical influences on material parameters for instance in the fracture mechanics reach a minimum if the condition of criteria for specimen's thickness can guarantee. On the other hand the factors of production like molecular weight, degree of crystallinity, residual stress and orientation influence the deformation and fracture behaviour directly. Additional these effects will be overlapped by the realized conditions of adherence, anisotropy, geometry and content of fillers in case of composites. For blends and copolymers the required stabilization, interparticle distance and particle diameter are more important material parameters. The determined deformation, strain rate or kinetics of damage is decisive influenced by the test conditions of material testing systems. It's clearly to see that the aim of production to use invariant material parameters for materials development and optimization are not reachable with conventional test methods. So the properties of structure and morphology determine in correlation with test conditions not only the local and time dependent deformation behaviour but also the nature and extent of pre-damaging. For the interpretation or evaluation of mechanical investigations experimental results of the state of residual stress, orientation, filler content or fibre length and distribution are necessary [1-3].

As a result of these increasing requirements on applicability of mechanical or fracture mechanics parameters determined by quasi static loading more innovative methods are used in materials testing. Some of these methods are:

- hybrid test methods like ultrasonics, acoustic emission analysis or thermography simultaneously to mechanical test methods [2] and
- qualification of mechanical test methods by using digital and closed loop techniques , new physical principles of measurement [4] and optical techniques of strain measurement [5].

Independent of the chosen methods and the loading conditions the most important aim of all these researches is the event correlated interpretation or evaluation of deformation and fracture behaviour. Because of the high accuracy of load, high linearity and signal dynamic the results of mechanical testing are dominantly influenced by the used extensometer. It's well known that with the use of mechanical extensometers for plastics testing many different problems can occur. This caused because the inherent weight is too expansive and the existing high notch sensitivity of these materials the parameters may influence. As a result of these disadvantages of the mechanical strain gauges increasingly optical extensometers like video extensometers [6], field measuring systems (scanning methods, Moire-techniques, and Speckle-laserinterferometry [7]) and laser extensometers [8] are used.

EXPERIMENTAL

Measuring techniques

The strain measurements by means of laser extensometer are techniques based on principles of reflection or transmission. Specimens used for these method need reflectors for the laser beam or use the edge of specimen as handicap for the beam. In the case of tensile or compressive testing these extensometers are used in direct or diffuse measuring mode of reflection with applied gauge marks (targets) [9]. For applications of experimental fracture mechanics e.g. with compact tension (CT-) specimens the transmission mode will be preferred. Normally the measurement is based on the scanning time of a rotating mirror or prism (Fig. 1a). The laser extensometer shown in Figure 1 works with a constant laser power of 4 to 6 mW and an object distance of 200 mm. With these values and an integral initial gauge length L_0 of 160 mm the maximum resolution of extension reaches 1.5 µm. For the determination of local deformation behaviour it is necessary to apply reflectors on the specimen surface. The maximum number of reflectors is 63 whereby 62 strain-time curves are observable. In case of plastics the application of reflectors or fringes is possible by tampon- or silk-screen printing and by simple bright-dark contrasting with the help of pens or airbrush. For the measurements a minimum distance of reflectors of about 1 mm is to ensure. Starting the tensile test nearly
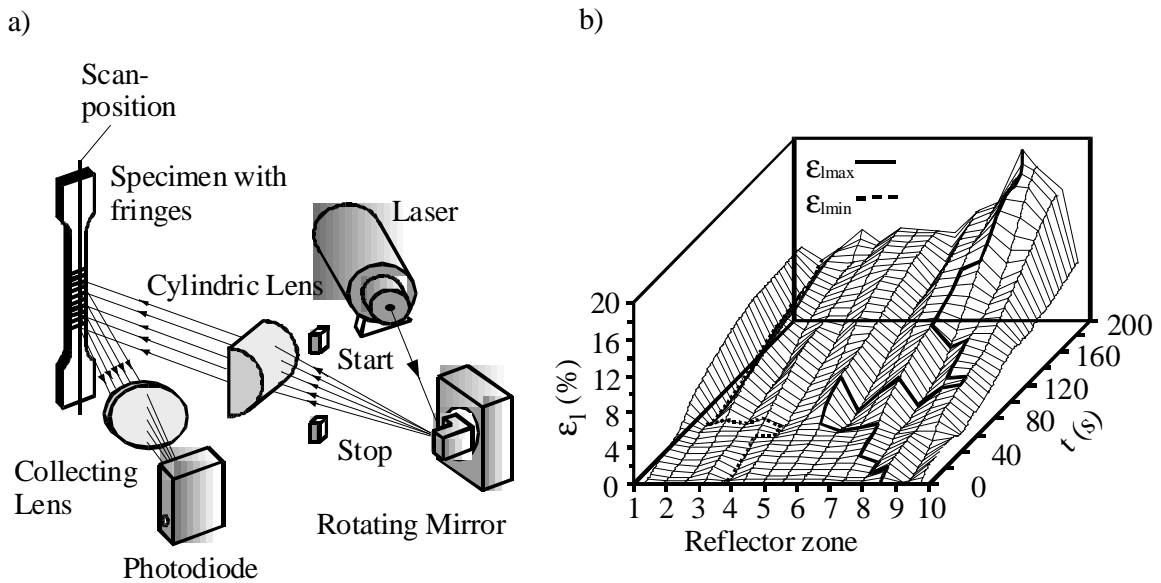
Figure 1 : Principle of local strain measurements **(a)** and local deformation behaviour of polyamide PA/GF with 30 wt.-% glass fibers **(b)**

20 scans will be measured for definition of initial position of the applied fringes. This means also a self-calibration of laser extensometer because the initial gauge length is not exactly in all cases. The start- and stop-diodes are necessary for the compensation of speed fluctuations and for synchronisation with the system time of universal testing machine. The local stress-strain behaviour in case of 10 reflectors (Fig. 1b) shows a strain distribution which corresponds to the structure of the investigated polyamide. These 3d-diagrams give an impression about the dynamic of deformation process in dependence on time, stress or integral strain. The computerized off-line evaluation of the files allows the determination of different dependencies for the local and integral stress-strain diagrams too. For a greater elongation of specimen the rotating mirror scanner should be used (Fig. 2a). In case of small deformations measured for reinforced polymers the high resolution flat scanner is useable. This measuring system is independent on the object distance and reaches a resolution better than 0.1 µm (Fig. 2b). As a new feature for both scanners the deter-mined strain values between two reflectors are added into the evaluation of the testing machine whereby also closed-loop experiments for the local strain are available. For the control signal the reflector distance can be chosen as fixed but also as calculated maximum or minimum of strain in the whole reflector area.
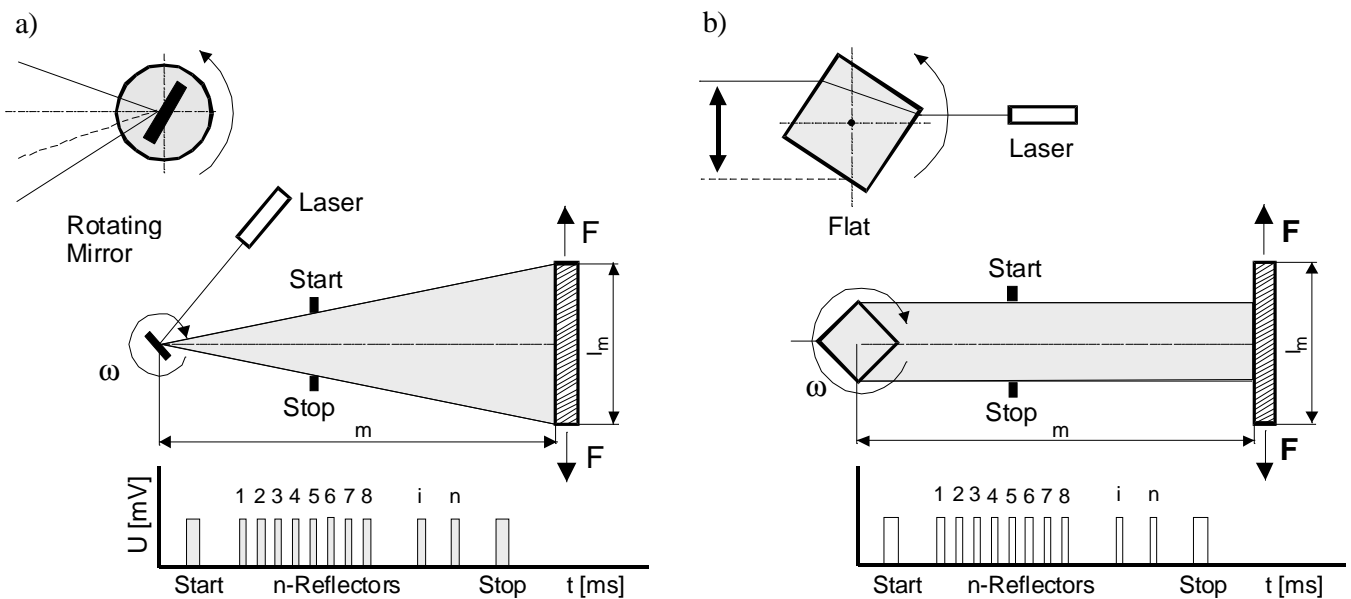

Figure 2 : Principle of rotating mirror scanner **(a)** and flat scanner **(b)**

Generally the used laser extensometers were coupled to an universal testing machine Zwick Z020. In addition to the laser extensometry the acoustic emission analysis as hybrid method was used to register the damage kinetics of the different polyamide materials. This method detects the onset of ultimate damage at composites related to the present state of stress or strain if the failure mechanisms are active. The parameter of amplitude or energy distribution calculated additionally allows to assign a change in damage processes. An exactly analysis of source mechanisms like debonding or fiber fracture is possible but very rarely [10]. For all experiments the measuring system Vallen ASMY4 with a broad band transducer at 100 up to 450 kHz was used. The sensor was mounted on specimen surface opposite to the used reflectors of laser extensometry at a non disturbing distance and the hits, amplitude and energy were recorded simultaneously.

Materials

All investigations were executed on different reinforced polyamide types with contents of 10, 20 and 30 wt.-% of glass fibers. For the comparison of structural state injection-moulded specimen and plates with a thickness of 4 mm were produced. Afterwards from the plates dumb-bell specimens were cawed out and conditions of materials all conventional and closed loop tensile tests were performed with an integral test speed of 5 %/min. The required reflectors were printed by silk-screen at a distance of 1 mm and by using of 21 fringes an initial integral gauge length of $L_0 = 42$ mm creates. The evaluation of local deformation behaviour and the calculated heterogeneity of specimen volume permits conclusions from changing of material behaviour caused by the deformation if additional microstructure methods of investigation are used [11]. For this purpose the maximum and minimum of local strain independent from the measuring zone was calculated (Fig 3a) and the integral strain related heterogeneity was plotted versus integral strain (Fig. 3b). The determined maximum of local strain rate can also be used as actual state for the comparison with the integral strain rate as planned status in closed loop tensile tests.
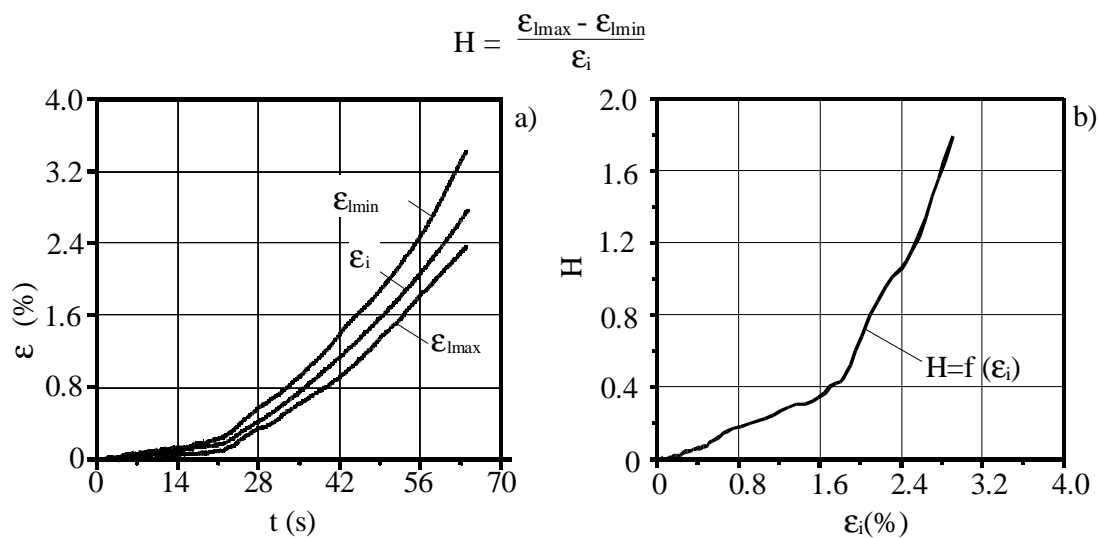
$$H = \frac{\varepsilon_{lmax} - \varepsilon_{lmin}}{\varepsilon_i}$$

Figure 3 : Determination of strain heterogeneity H for a polyamide with 10 wt.-% glass fibers, local and integral strain-time curves **(a)** and function of heterogeneity in dependence on integral strain **(b)**

RESULTS

The 3d-distributions of different specimen states are shown in Figure 4. At the beginning of the tensile test local deformation behaviour is nearly homogeneous independent on preparation of the specimen. It is clearly to see that the local deformation of transversal to injection-moulding direction milled specimen (Fig. 4a) show a lower heterogeneity as the specimen longitudinal milled (Fig. 4b). Because the state of orientation concerning to the gauge length for longitudinal milled specimens is nearly symmetric the maximum of local strain occurs in the middle of the investigated volume. Specimen which are produced by in-
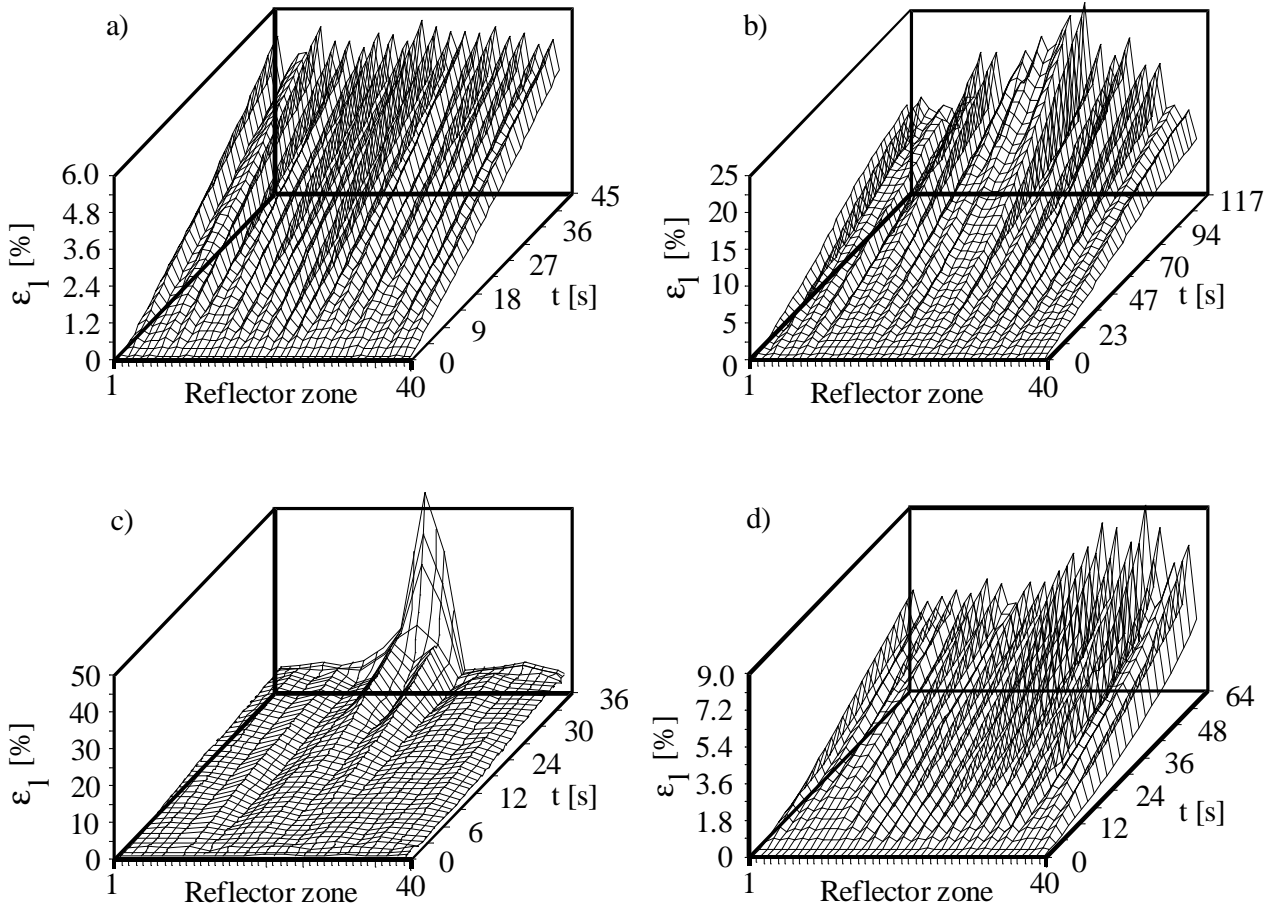
Figure 4 : Local deformation behaviour for polyamide with 10 wt.-% glass fibers of specimen milled longitudinal to injection-moulding direction **(a)**, milled transversal to injection-moulding direction **(b)**, notched dumb-bell specimen with 20 wt.-% fibers **(c)** and unnotched dumb-bell specimen too **(d)**

jection-moulding directly have a very low orientation near the shoulder opposite to the injection point. In fact of this local deformation distribution show the maximum in this range and detects the area of ultimate failure early (Fig. 4d). If there exists a notch the difference between the maximum of local strain and the out side ranges increases rapidly. Because of the very high deformation speed and the stress state near the notch after crack initiation the deformation is only localized in this damage zone. The comparison between
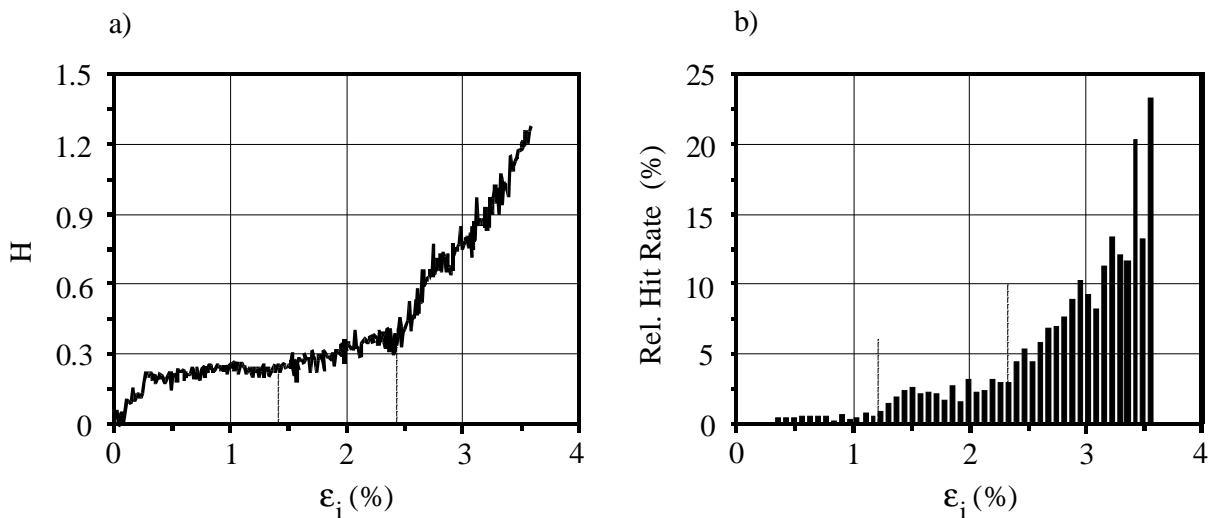

Figure 5 : Strain heterogeneity **(a)** and relative hit rate of acoustic emission **(b)** for dumb-bell polyamide specimen with 30 wt.-% glass fibers (unnotched)

the acoustic activity and the strain heterogeneity is shown in figure 5 for polyamide 6 with 30 wt.-% glass fibers. After starting the conventional tensile test the heterogeneity calculated from the difference between the maximum and minimum of local strain increases and stays on a constant level up to 1.4 % of integral strain. At this point which is characterized by the onset of acoustic emission the heterogeneity increases and reaches strongly slope at 2.4 % of strain. It can be seen that acoustic emission at the same point increases rapidly. The reason of increasing homogeneity and acoustic emission is the forced crack propagation caused by internal processes of changing and damaging. The inspection of these dependencies in the 2d-diagrams shows a correspondence of the acoustic onset and time of splitting in local deformations. Indepen-
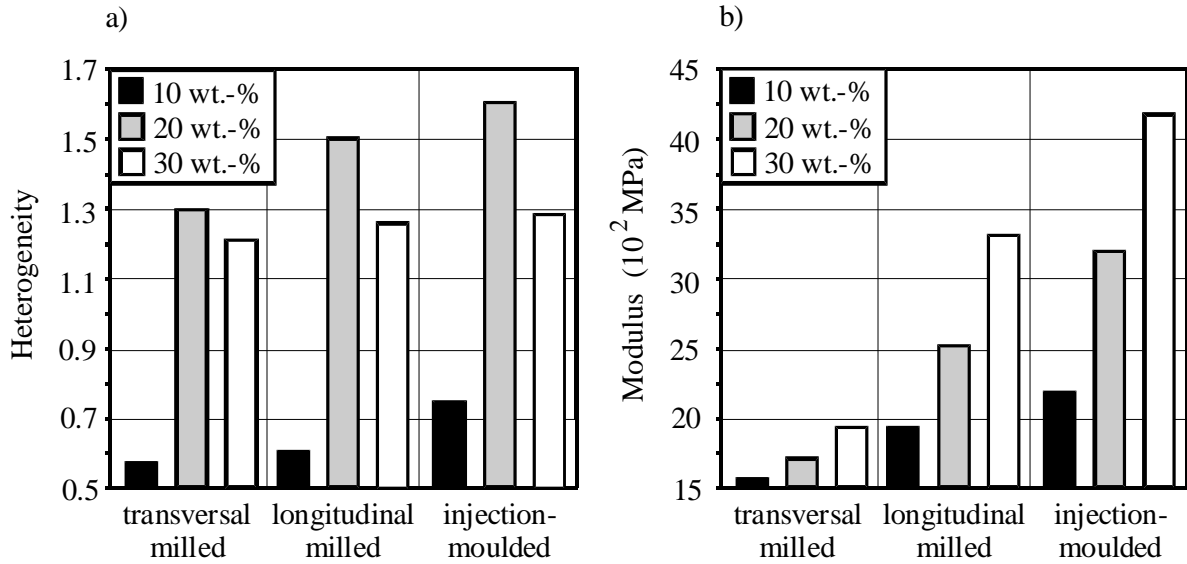

Figure 6 : Heterogeneity **(a)** and tensile strength **(b)** for different fiber contents and producing methods

dent of the amount of integral strain there also seem to be an effect of localization in the distribution of local deformation. As expected the mechanical properties like modulus of elasticity increase with higher orientation from transversal milled specimen to injection-moulded one (Fig. 6b). On the other hand it can be seen that the anisotropy shown by the heterogeneity increases also (Fig. 6a) to a higher orientation state. But it is clearly to see that with increasing fiber content the modulus of elasticity increases heavier as the determined heterogeneity. The comparison of acoustic energy (Fig. 7) shows different time dependencies for the investigated specimen types. The first energy band reaches very high energy values near 3000 and is observable for all specimens. As a result of inspection of the fracture surfaces this energy range as well as the accompanying amplitudes are caused by debonding mechanisms. In fact of the mainly transversal direction of fibers to the load line in case of transversal milled specimens only this damage mechanism can be detected by acoustic emission analysis. On the fracture surface of these specimen types no broken fibers
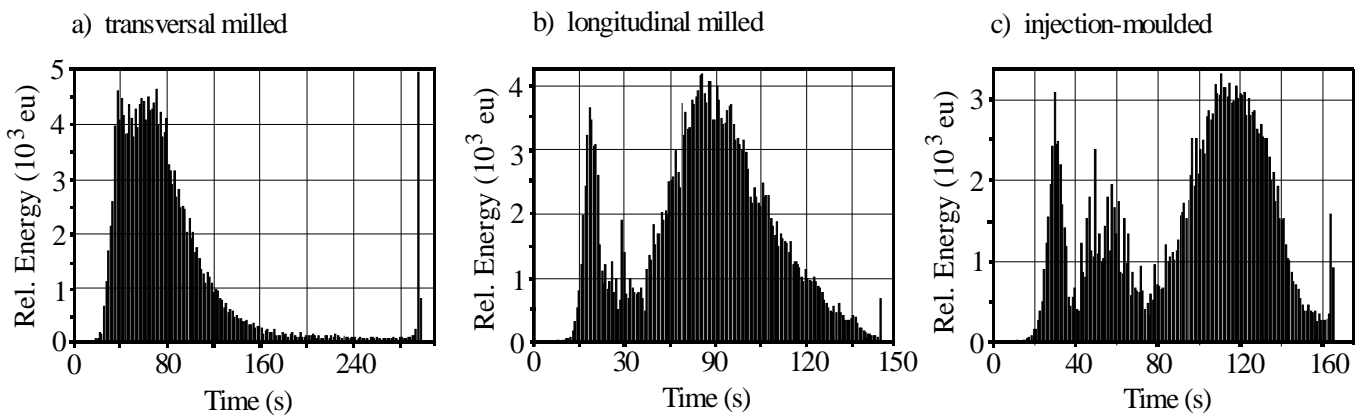

Figure 7 : Relative acoustic energy versus test time for polyamide with 10 wt.-% glass fibers of specimen milled transversal to injection-moulding direction **(a)**, milled longitudinal to injection-moulding direction **(b)** and injection-moulded dumb-bell specimen **(c)**

could be found. At higher testing times a second energy band is detectable for longitudinal milled specimens which shows the same distribution as the third band for injection-moulded dumb-bell specimens. For both specimen types fiber pull out and fiber fracture can be seen on the SEM-images (Fig. 8b and c). The energy of these damage mechanisms are nearly the same as for debonding but event or hit rate is very high.
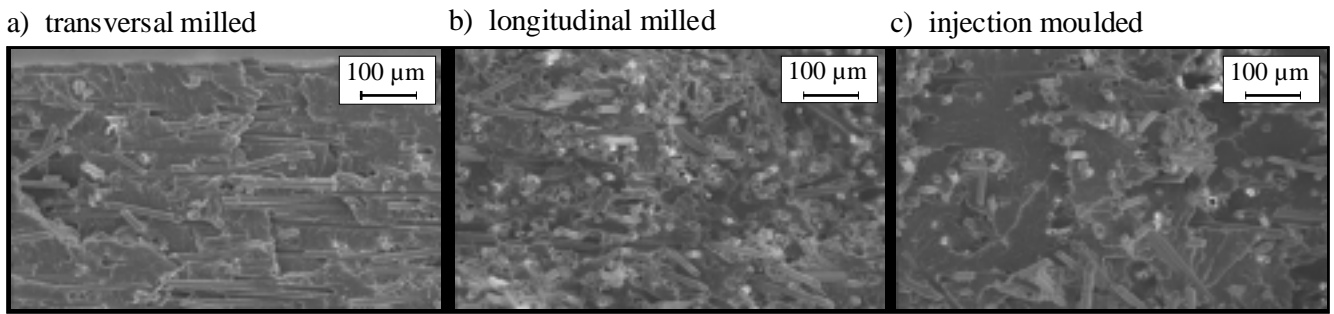
a) transversal milled   b) longitudinal milled   c) injection moulded

Figure 8 : SEM-images of fracture surfaces for polyamide with 30 wt.-% glass fibers of specimen milled transversal to injection-moulding direction **(a)**, milled longitudinal to injection-moulding direction **(b)** and injection-moulded dumb-bell specimen **(c)**

In dependence on decreasing fiber content this energy becomes broadband and on the fracture surface are more broken fibers are detectable. For the injection-moulded dumb-bell specimen exists a second narrow band energy range with values up to 2000 relative units. Because of the sandwich structure of these specimens and the different states of orientation in the investigated volume matrix deformation processes can also be dominantly. Here defect mechanisms like crazing or shear yielding are of importance as well as friction of fibers at the crack flanks. The time separation of these different mechanisms is caused by the load state and the deformation speed obviously. Because that at every point of conventional tensile test the conditions for relaxation are different the material shows not comparable deformation and damage processes which can not corrected by means of integral closed loop tensile testing exactly. In figure 9 the results
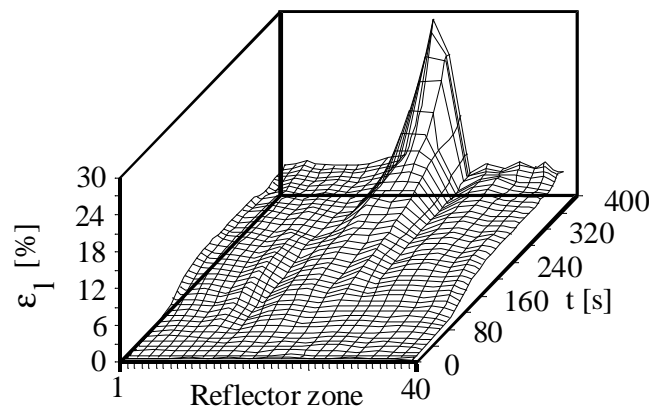

Figure 9 : Local deformation behaviour for polyamide with 20 wt.-% glass fibers of dumb-bell specimen in case of closed loop tensile testing

of closed loop experiments controlled by the local deformation are shown for example for polyamide with 20 wt.-% of glass fibers. In this case the maximum of local strain independent of localization on specimen surface is searched and token as control value for the control process. In comparison to the conventional tensile test (Fig. 4d) it can be seen that the strain distribution is very homogeneous up to the point of unstable crack propagation (Fig. 9) and the dependencies on the realized state of orientation are smaller. The acoustic emission analysis visible by the energies and amplitudes shows dominantly debonding over the whole range and only near the macroscopic failure damage processes like pull out and fiber fracture.

SUMMARY

The experimental possibilities of measurement and evaluation of the described laser extensometer demonstrate the versatility of these modern optical extensometers. One of the main advantages especially for plastics is the contactless application for different geometry's of specimen. This information about local and integral strain as well as strain rate or calculated parameters like heterogeneity are very important for material development and plastics testing. Under the aspect of loading conditions the laser extensometer shows every failure by clamping or practices of tensile testing. The distribution of local deformation at the observed specimen surface correlates with the results of non-destructive methods like acoustic emission analysis or thermography. By means of an expansive evaluation the localization of active damage processes regarding to the time or area is also possible. Because the results of in situ-experiments always related to the integral strain there exists now the chance for the correction of the strain to the local damage zone or compliance. Among the multiple capability of such laser extensometers for determination and representation of local deformation behaviour the controlling of engineering and true strain is possible if the requirements of mathematical algorithms are satisfied. The independence of existing specifications of universal testing machines allows the application and coupling of laser extensometers for all testing systems.

From this point of view these optical extensometers are an innovative technique for the testing and development of materials. For the enhanced evaluation of materials, simulation of reliability and life time the laser extensometry is a valuable instrument for material testing of polymers.

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