Deformation Behaviour of Reinforced Polyamide Materials evaluated by Laser Extensometry and Acoustic Emission Analysis

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Introduction

As a result of the wide range of polymer materials, caused by filling and reinforcing, and the many modifications of strength and toughness, extensive requirements are placed on modern methods of material testing. This applies particularly to the sensitivity and precision of the test methods and the quality of the material parameters used. For the purposes of material development, quality assurance and failure analysis, the important relationships between properties and structure as well as damage behaviour must be well known in order that the lifetime and reliability can be simulated. In spite of the many microscopic, spectroscopic and defectoscopic tests using new or better physical principles of operation, the mechanical testing of polymers still has high importance. Suitable material parameters are required, especially for the purposes of differentiation of polymers, the evaluation of strength and toughness modifiers, and the dimensioning of polymer components by users or developers. These parameters, usually determined by conventional tensile testing, must be independent of influences of the methods used to obtain them.

It is well known that the measured parameters related to strength, deformation or toughness reflect influences of manufacturing and loading. These factors associated with the production process, such as molecular weight, degree of crystallinity, residual stress and orientation influence the deformation and damage behaviour directly. In the case of composites, these effects are overlapped by those of the adherence, anisotropy, geometry and filler content achieved.

The deformation, strain rate and kinetics of damage processes are decisively influenced by the test conditions. It is clear that material parameters suitable for material development and optimisation that are independent of production conditions are not achievable with conventional tensile testing methods. As a result of these requirements on the mechanical parameters determined with quasi-static loading increasingly combinations with non-destructive testing methods such as acoustic-emission analysis or thermography are being used in material testing. By means of this simultaneously recorded acoustic emission the registration of damage kinetics of reinforced polymers is possible. The most important aim of all such work is an event-related interpretation and evaluation of damage and fracture behaviour. For this purpose an exact knowledge of the deformation related to the damage state is necessary.

Because the conventional tensile test of polymers is overlapped by effects of creep and relaxation, strain and strain rate depend on time and localisation of the extensometer used. In fact of these effects the importance of closed-loop experiments for polymeric materials increasing. On the other hand problems are caused by the non-negligible inherent weight of the sensor and the high notch sensitivity of polymers. As a result of these disadvantages, optical extensometers such as video extensometers [1], field-measuring systems (scanning methods and laser speckle interferometry [2]) and also laser extensometers [3] are being increasingly used.

Experimental

For the experimental investigations a laser extensometer developed by Fiedler Optoelectronic GmbH Lützen was used [4] (Figure 1). In tensile testing, this extensometer is used in a diffuse reflection mode with gauge marks (fringes) applied to the specimen. The measurement is based on the scanning time of a rotating mirror or prism. The laser-prism scanner 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 a total initial gauge length L_0 of 60 mm, the maximum resolution of the extension is 0.1 µm. For the determination of the local deformation behaviour, it is necessary to apply reflecting fringes to the specimen surface. The maximum number of fringes in this case is 63, whereby 62 strain-time curves are observable. In case of polymers, the application of fringes is possible by tampon-printing or silk-screen printing or by making simple bright-dark marks with pens or an airbrush. For the measurements to be accurate, a minimum distance of the fringes of 1 mm should be ensured. To define the initial position of the applied

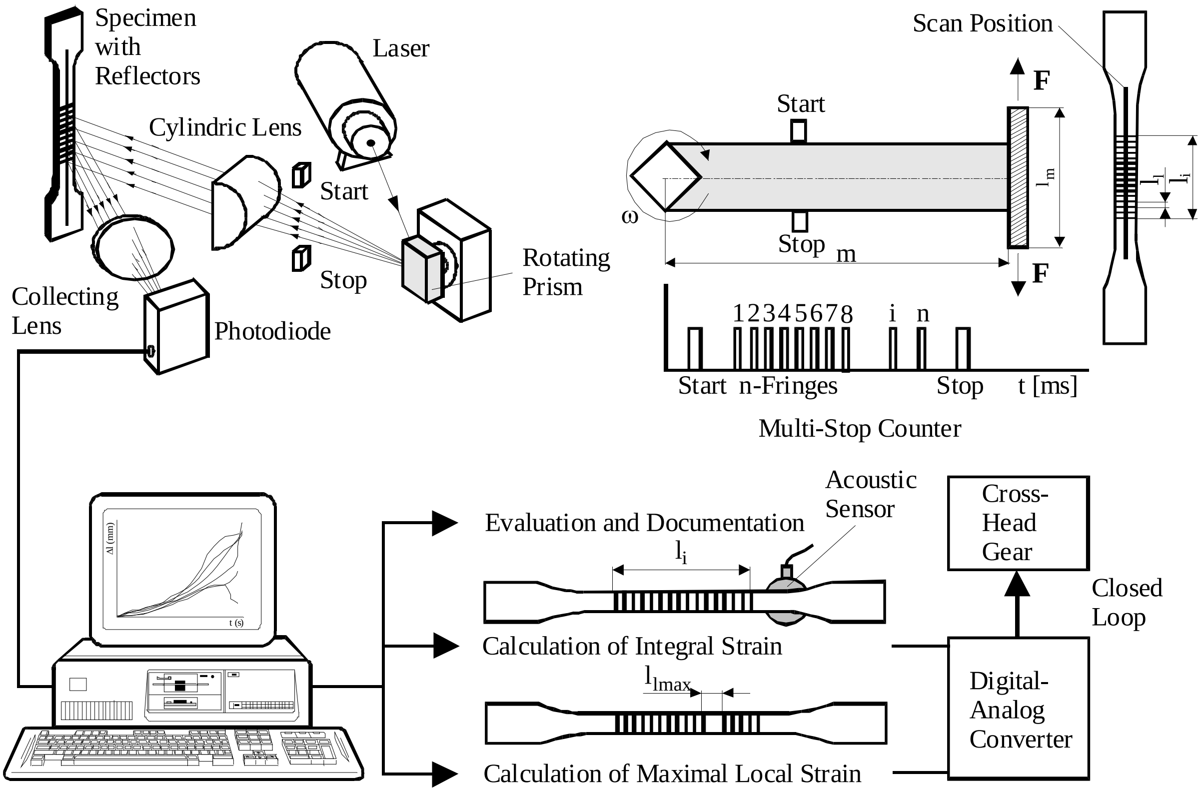

Figure 1 Principle of local strain measurement and closed-loop experiments by means of the laser extensometer

fringes, position of the applied fringes, 20 scans in the unloaded state are necessary. This means also a self-calibration of the laser extensometer, because the initial gauge length is not exactly the specified value in all cases. The start and stop diodes allow compensation of speed fluctuations and synchronisation with the system time of the universal testing machine. The recorded local extensions between the fringes are available for the calculation of the strain in relation to the measured stress, the time signal or a simultaneously recorded signal such as acoustic emission. A new feature is that the strain values between two fringes can be taken into account in the control of the testing machine, so that closed-loop experiments using the local strain can be performed. For the simultaneous acoustic-emission analysis a digital meas-

uring device ASMY4 of Vallen System GmbH Icking was used. To minimise effects of signal dumping the acoustic transducer with the resonance frequency of 220 kHz was positioned outside of the 22 applied fringes (Figure 1). The registered load of the universal testing machine serves as reference signal for the laser measurements executed simultaneously.
For the experimental investigations Polyamide materials PA6 with different contents of glass fibres (Table 1) of Leuna Miramid GmbH were used. To ensure comparable conditions and a high resolution for acoustic emission analysis the test speed was chosen to 3 %/min in case of closed-loop experiments and 3.45 mm/min (clamping distance = 115 mm) for conventional tensile tests. All specimen were conditioned to obtain an equilibrium of moisture.

Table 1 Modulus of elasticity in tension and tensile strength of materials determined in accordance to DIN EN ISO 527

Parameter	Fibre Content [wt.-%]								
	0	1	5	10	20	25	30	40	50
σ_t [MPa]	57.8	60.2	70.4	84.6	130.1	146.6	159.4	190.7	222.9
E_t [MPa]	2065	2260	2720	3570	5855	6805	7905	10830	15525

Results and Discussion

The computerised off-line evaluation of the data of the laser extensometer allows the determination of various relationships from the local and integral stress–strain diagrams [5]. For the representation of the results, an extensive graphics unit that can calculate 3D diagrams is included which offers the possibility to determine the maximum and minimum of the local strain, independent of the fringe position in the range investigated. So, it is possible to determine the strain heterogeneity H_e as the difference between the maximum and minimum of the local deformation related to the integral strain at break:

$$H_e = \frac{\varepsilon_{l\max} - \varepsilon_{l\min}}{\varepsilon_{iB}} \quad (1)$$

The results in Figure 2 show clearly that the heterogeneity of moist specimen is lower independent on glass fibre content which is caused by the reduction of residual stress. On the other hand the heterogeneity H_e also decreases (Figure 3) if the tensile test is executed under closed

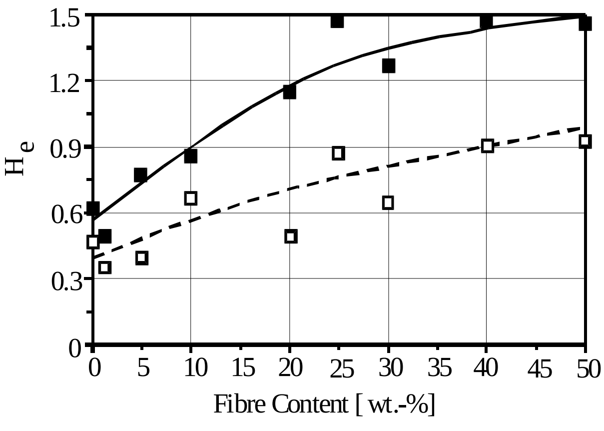

Figure 2 Comparison of heterogeneity for dry -■- and moist specimen -□-

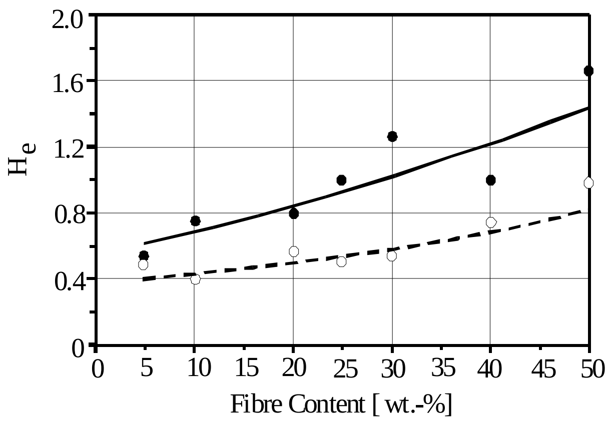

Figure 3 Comparison of conventional -●- -○- and integral closed loop tensile test

loop conditions. Simultaneous experiments with laser extensometry and acoustic-emission analysis under different load conditions show that a connection exists between the acoustically measured damage kinetics and the kind of closed loop experiment, for example for Polyamide with 30 wt.-% of glass fibres. In contrast to the integral closed loop experiment (Figure 4) the test with local maximum closed loop induces a near constant acoustic behaviour up to 2 per cent of integral strain (Figure 5). For all reinforced Polyamides the amplitudes and energy of acoustic emission are lower as in conventional test and integral strain closed loop experiment. The decreasing strength and increasing strain at break are caused by the improved relaxation conditions especially in the case of closed loop by means of local maximum strain.

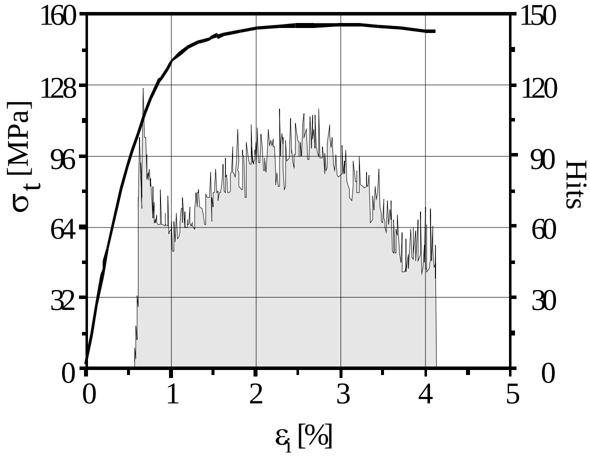

Figure 4 Stress-strain behaviour and acoustic hits for integral closed loop tensile test

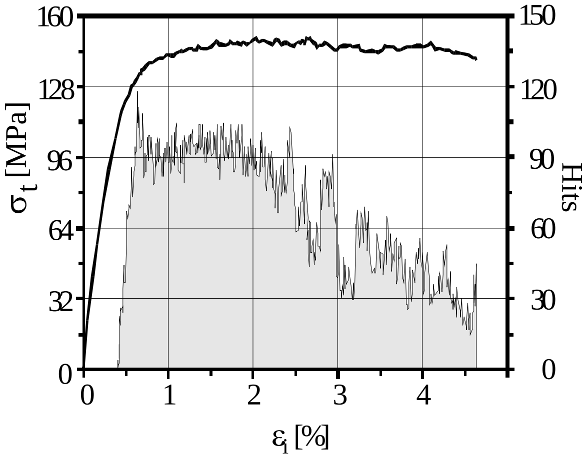

Figure 5 Stress-strain behaviour and acoustic hits for local maximum closed loop tensile test

Conclusion

The experimental possibilities of measurement and evaluation provided by the laser extensometers described above demonstrate the versatility of these modern optical extensometers. One of the main advantages, especially for plastics, is that the method is contactless and can be applied to specimens of different dimensions. The information that it can provide, about local and integral strain as well as strain rate, and calculated parameters such as the heterogeneity, is very important for material development and polymer testing. The observed distribution of local deformation at the specimen surface correlates with the results of non-destructive methods such as acoustic-emission analysis. From this point of view, these optical extensometers are an innovative technique for the testing and development of polymeric materials.

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