Fatigue fracture mechanism of fibre reinforced injection moulded polyamide

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Abstract

A fatigue mechanism of bridged cracks is proposed, based on some novel experimental techniques: Measurements of creep during fatigue, through thickness strength profiles of specimens that had been subjected to a fixed number of fatigue cycles. SEM fractography was used in the normal way on broken specimens, but also on specimens that were fatigued, but not up to failure, and consequently cryogenically broken.

Damage starts with void initiation at fibre ends, the voids consequently grow along the fibre and merge into larger "cracks". For this growth process especially the tensile strength of the fibre - matrix interface is important. However not one complete crack exists, as the crack walls remain connected at a number of spots. Damage is not homogeneously distributed over the sample, but is confined to disk-like areas, similar in appearance to crazes found in amorphous plastics.

1 Introduction

Injection moulded thermoplastics reinforced with short glass or carbon fibres (SFTP’s) are being used increasingly in load bearing applications. This because of weight, cost, corrosion resistance and ease of production. This is by the injection moulding process, which makes freedom of design and integration of functions possible.

SFTP’s have a characteristically high degree of anisotropy. The anisotropy, caused by fibre orientation, and formed during injection moulding, brings about strong variations in properties: e.g. heat conductivity, elastic modulus, tensile strength and fatigue behaviour. This makes the calculation of properties of products complex.

To be able to predict the fatigue lifetime of products, fatigue behaviour
and Ultimate Tensile Strength (UTS) were investigated. A correlation between UTS and the maximum fatigue stress to attain a certain lifetime was found for GFPA.\textsuperscript{1,2} The reason for this correlation is not completely understood. Mechanisms in tensile and fatigue experiments are different, such that a similar stressing of the matrix material with different fibre orientations, in both tensile and fatigue experiments, cannot be assumed. Therefore investigation of the fatigue mechanism has started.

The mechanism in tensile experiments is well known, consisting of crack initiation and growth, accompanied by fibre pull-out. In the current investigations this mechanism could be confirmed.

1.1 Theory

The mechanism in fatigue is generally considered to consist of the following four stages:\textsuperscript{3}

1. Initiation of local damage due to cyclic deformation, generally at the locations of highest stress intensity, the fibre ends.\textsuperscript{4}
2. Initiation of microcrack.
3. Stable crack growth due to cyclic loading. Local modes of crack extension depend on local fibre orientation, matrix ductility and the degree of interfacial adhesion (Lang).\textsuperscript{5}
4. Fast (instable) crack growth in the last load cycle, which should be comparable to failure in a tensile test.

Dally\textsuperscript{6} reports for a system with the much more ductile PE matrix, and almost no fibre - matrix bonding, an entirely different mechanism. Massive debonding reduces the glass fibres from reinforcement to unbonded inclusions, giving rise to a sharp drop in modulus. The greater strains are accommodated by the matrix without failure. This process of general degradation rather than a dominant crack was also reported by Mandell\textsuperscript{7} for unnotched specimens, while Dibenedetto et al.\textsuperscript{8} also observed a similar fatigue mechanism in compact tension specimens of graphite fiber reinforced PA 6.6, conditioned to equilibrium water content.

In this paper it will be shown that the fatigue mechanism of crack growth as described above for dry as moulded GlassFibre Reinforced PolyAmide 6 (GFPA 6) cannot be applied to conditioned GFPA 6. The mechanism Dally found for GFPE does not fit to the experimental results either. A modified fatigue failure mechanism is proposed.

2 Experimental

The material used was Polyamide 6 containing 30%wt. of glassfibres; Akulon K224-G6, provided by DSM, the Netherlands. Square plates of 100x100mm\textsuperscript{2} and 2 and 5.75mm thickness were injection moulded from this. The mould has a line gate, to obtain a straight flow front. For fatigue and tensile experiments non standard dog-bone type specimens were milled from the plates, using a Roland PNC-3000 Computer Aided Modelling
Machine. Specimens were conditioned by exposing them to laboratory air for at least 1 year, giving a water content of approximately 1.5%.

The fatigue experiments were carried out on a servo-hydraulic MTS 810 bench. The load frequency used was 1 Hz, to avoid temperature increases of more than 3°K due to hysteretic heating. Earlier experiments showed the high sensitivity of the fatigue lifetime of this particular material to the test frequency. The minimum to maximum load ratio $R$ was 0.1.

During the fatigue experiments the displacement of the grips was monitored, enabling the calculation of the elastic modulus and creep. Thus we were able to get a maximum of information from one fatigue test.

Tensile experiments were executed on the same type of specimens, with a cross-head speed of 50 mm/min, resulting in a strain speed of 143 %/min. Tests were carried out in an environmental chamber at a temperature of 23°C and at a relative air humidity of 50%.

The assessing of fatigue damage was done by making strength profiles. Miniature test bars are milled from the specimens after subjecting these for a certain number of cycles to a fatigue load. Expected lifetime of these specimens can be accurately predicted using the creep speed - lifetime correlation. From these miniature samples, with a width of 2 mm, 85 μm thick slices are cut parallel to the surface, using a Leitz microtome. The fracture strength and fracture strain are measured for each slice, using a miniature tensile test machine. Plotting strength or fracture strain to the distance to surface gives the required profile.

SEM micrographs of fracture surfaces were made using a JEOL JSM-840A after gold coating in a Balzers SCD 040. To reveal the structure inside the specimen during the fatigue process, some specimens were first fatigued for a certain percentage of their expected lifetime, and consequently fractured after immersing them for 5 minutes in liquid nitrogen.

3 Results

3.1 Fatigue experiments

It had already been shown that especially the creep speed $V_c$ (increase in grip-displacement during secondary creep, per cycle) presents a good correlation with lifetime $N$ (number of cycles to failure) and can be used to predict the lifetime of a specimen while it is being fatigued.

Figure 1 shows a typical correlation between $\log V_c$ and $\log N$, for one load level and one specimen type. So all scatter of lifetime seen in this graph is due to specimen to specimen variations.

3.2 Strength profiles

A considerable amount of strength profiles of fatigued specimens have been made. Experimental scatter makes the profiles difficult to interpret, and no
3.2 Creep speed - lifetime correlation for one specimen type at one load level.

Maximum Fatigue stress 55% of UTS
Log Vc = -1.18LogN + 0.40

Fig. 1 Creep speed - lifetime correlation for one specimen type at one load level.

Log number of cycles to failure [ ]

For the specimens fatigued up to higher percentages of lifetime generally a very local drop in strength for a few foils exists, at the location where stress whitening lines are visible. Strength in these areas drops to a typical value of approximately 25 MPa. This is also the lowest strength value found in any of the foils, which is the most remarkable observation during these experiments: no foil was found with strength zero.

3.3 Fractography

In SEM photographs of the fracture surface two distinct areas are visible, a part where the matrix is highly deformed, the fatigue "crack" area, and a part where the matrix material shows brittle fracture, the final fast fracture area. In the ductile area high matrix drawing between the fibres is visible, and completely exposed fibres: no matrix material is seen adhering to the fibres.

Figures 3 and 4 show representative examples of cryogenically broken samples, that have been fatigued at 70% of UTS for 350 cycles, approximately 90% of the expected fatigue lifetime. In Figure 3 it is clearly seen how, although the fracture of the matrix is brittle, voids exist around the fibres.

In Figure 4 the damage has developed, a small brittle part in a
localized damage

Fig. 3 Cryogenic fracture surfaces: voids (arrows) around fibres.

Fig. 4 Brittle bridge (arrow) in ductile area. fibre diameter: 10 μm.

Microductile (cracked) area can be seen. This brittle part was broken cryogenically, and must therefore have been a bridge that was still connecting the two crack surfaces.

Figures 3 and 4 of course only give details of the damaged zone, after cryogenic fracture. We are interested in the distribution of the damage over the specimen. Therefore the entire fracture surface of some of the cryogenically broken, fatigued specimens was investigated. Close to the corner of the specimen the material is entirely microductile, except for some small zones that showed microbrittle behaviour (Figure 4). These areas are bridges between the two crack surfaces. Outside this ductile (cracked) zone a transition zone to the outer brittle zone is present. In this area voids around the (debonded) fibres are seen, as in Figure 3.

A final important observation is that not in all cryogenically broken, fatigued specimen damage could be observed. Half of the cryogenically broken specimens showed microbrittle behaviour over all of the fracture surface, no ductility could be observed in these specimens, although after fatigue, stress whitening lines were visible on the specimen.

4 Discussion/Conclusions

The correlation that was found between creep speed Vc in fatigue, and the number of cycles to failure of that particular specimen, indicates that the fatigue lifetime is determined by properties of the specimen that are of global character (for the specimen as a whole), as the creep speed is global, measured over the entire specimen length. The creep in cyclic loading is the consequence of a global fatigue damage developing in the specimen, thus making the correlation possible: A high creep speed means a rapid increase in "damage density" and therefore a low number of cycles to fracture.

The stress whitening lines that are visible with fatigue, indicate local plastically deformed matrix material. The matrix material cannot deform to such a degree, without debonding from the fibres, because the fibres cannot...
Localized Damage

reach such a high strain without breaking. That the fibres are debonded from the matrix can be seen in fractography, where the fibres can be seen with no matrix adhering them. This debonding is visible in part of fracture surface, the part where the fatigue process has taken place and which can be recognised in the ductile appearance of the matrix material. The plastically deformed zones as seen in the stress whitening lines and on the fracture surface however are no real cracks. None of the foils in the strength profile experiments had strength zero, which indicates that no real (unbridged) cracks exist until the last or the last few load cycles.

To be able to observe closely the actual occurrences during fatigue, the fracture surface of cryogenically broken, fatigued specimens was studied. Because the cryogenic fracture gives pure brittle behaviour, all ductile fractured areas visible on the cryogenic fracture surface must be caused by the fatigue process. The voids that are visible mainly at fibre ends are formed during fatigue. These voids subsequently grow and merge into larger cracked areas, in which bridges exist connecting the two "crack" walls.

4.1 Fatigue mechanism

All the observations using the different methods combine to one failure mechanism in fatigue. In Figure 5 the stages of the failure process are indicated.

![Figure 5](image)

Fig.5 Visualisation of the fatigue failure mechanism for a model system, the damage develops from left to right. Numbers refer to explanation in the text.

Damage initiates at the location of highest stress intensity, the fibre ends (1). These fibre ends are badly bonded to the matrix, because at these fibre ends no coating or seizing, used to improve bonding, is present. This is because the fibres have been broken during compounding and injection-
moulding. Voids grow from this damage, mainly along the fibres (2). These voids and debonding of the fibres will largely reduce the reinforcing effect of the fibres, putting a higher stress on the matrix causing the matrix material to plastically deform in this damaged area. Increasing opening of this damaged area (3) will increase the straining of the material in front of the "craze" and will induce new voids and debonding, and growth of the "craze" in a direction perpendicular to the main stress direction. The voids continue to grow and merge into cracked areas (4). The cracked areas do not grow into one main crack though, the "crack" walls remain connected in certain locations.

The damage that is the consequence of the fatigue loading, is confined to certain areas. This can be concluded from the fact that a number of stress whitening lines are visible on the surface, after fatigue, and from the observation that on a fatigue fractured surface only part of the fracture surface is of microductile nature. The fact that only part of the cryogenically broken specimens showed ductile behaviour over part of the fracture surface also proves that damage is not homogeneously distributed over the specimen. The explanation for these phenomena is that damage exists in a craze-like manner. Damage exists in zones of limited thickness, which are oriented perpendicular to the main stress direction. These zones grow in the direction of planes perpendicular to stress during fatigue.

The creep in fatigue (Figure 1) is the result of initiation, growing and opening of the "crazes" that are formed over the length of the specimen.

Both fibre-matrix bonding and matrix ductility are crucial to the type of mechanism that will occur. In GFPA both properties depend on the fraction of absorbed water, thus causing considerable differences in test results.

Preliminary experiments have been carried out using Laser Scanning Confocal Microscopy, in cooperation with DSM the Netherlands. With this technique it is possible to focus at a level below the surface of the specimen. The technique was used to view some of the phenomena that are caused by the fatigue process. Damage in the form of debonding and void formation is visible and seen to be confined to some areas. Investigations using this method will increase knowledge about the distribution of the damaged zones.

### 4.2 Fibre - matrix interface loading

At fibre ends the local stress intensity is high, damage will occur, and debonding in a shear mode will take place at the end of the fibre, where shear stress at the interface is highest. The unloading of the fibres due to the debonding will increase the local stress on...
the matrix, which will deform. Deformation of the matrix requires lateral contraction, which is inhibited by the bonding to the surrounding fibres. This results in a tensile stress on the fibre - matrix interface. Thus for fatigue behaviour not just the pull-out (shear) strength of the interface, but also its tensile strength is of importance. This is the main difference between fatigue and tensile experiments (Figure 6): In tensile experiments the matrix cracks in a brittle manner. Loading of the fibre therefore is in pure shear, because lateral contraction of the matrix is absent. Under this shear stress the interface itself does not fail (in well bonded systems), the matrix close to the interface fails. This is shown by the matrix material that is seen adhering to the pulled out fibres.

Obviously the tensile stress on the interface that does occur in fatigue, makes the interface itself fail, which is seen in the fibres with no matrix material adhering to them.

References