Damage mechanisms of short glass fiber reinforced polyamide [redacted] under fatigue loading

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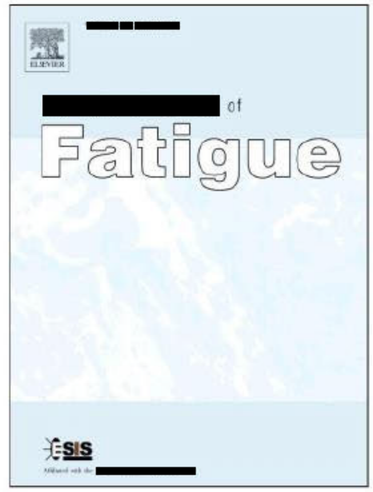

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Damage mechanisms in a short glass fiber reinforced polyamide under fatigue loading

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Abstract

This paper presents a damage investigation on a short glass fiber reinforced polyamide (PA66-GF35) under fatigue loading. Plain and notched specimens were tested at room temperature and humidity with load ratio R = 0. Electron microscopy was used to analyze the fracture surface of failed specimens and the crack path of specimens subjected to interrupted fatigue tests. Damage mechanisms were studied investigating the following fractographic features: ductile / brittle matrix fracture behavior, fiber failure / pull-out, degree of fiber-matrix interfacial adhesion. The aim of the present paper is to understand the nature of damage initiation and propagation in order to lay the foundations for the development of a multi-scale, mechanism-based lifetime prediction model for short fiber reinforced plastics.

1. Literature review

In the car engine compartment, Short Fiber Reinforced Plastics (SFRPs) are increasingly replacing metals in structural applications. High strength to weight ratio, high temperature and chemical resistance make these materials excellent candidates for under-the-hood applications. The competitiveness of SFRPs is related to the advantages offered by the injection molding process. This technology enables complex geometries to be manufactured at high production rates without the need for additional post-molding machining operations. The durability assessment of SFRPs plays a key role in the design of injection molded structural parts. Typical under-the-hood parts such as fuel rails, pump housings and sealing elements are exposed to cyclic loading due to vibrations, pulsating forces, temperature variations. Often, in the automotive industry, several prototypes are needed before beginning the series production. Lifetime prediction models aim to reduce the number of recursions before the series production by virtually optimizing the product in the early stages of the project. Lifetime predictions are accurate when the damage mechanisms are understood and properly integrated in the model. In SFRPs, damage mechanisms occur at a length scale below the macroscopic scale due to the interactions between the material constituents (fiber, matrix, fiber-matrix interface). Multi-scale models aim to bridge the scale at which the damage mechanisms occur and the macro-scale of structural applications. The first step in developing such a model is an extensive damage investigation.

The aim of this work is to investigate the fatigue damage mechanisms in plain and notched short glass fiber reinforced polyamide specimens in order to lay the basis for the development of a multi-scale lifetime prediction model. In the last 30 years, many papers were dedicated to the investigation of damage and failure mechanisms of SFRPs under fatigue loading. Considering the first works in this field in the [redacted] [1, 2], the investigation techniques have taken huge steps in terms of accuracy of observation, image quality and development of new methodologies. Fractographic examinations by means of Field Emission Scanning Electron Microscope (FESEM) on a short fiber reinforced PBT were shown recently by [redacted] [3]. Comparing the micrographs in [1, 2, 4, 5] with those in [3], the improvement of the microscopy technique in terms of resolution, magnification, image quality becomes immediately evident.

In [redacted], the authors reviewed the damage mechanisms from literature and sorted them based on specific fractographic features: ductile / brittle matrix fracture behavior, fiber failure, fiber pull-out and degree of fiber-matrix interfacial adhesion. The results from the literature were compared with the results of an extensive damage investigation carried out at Robert Bosch GmbH on plain and notched short fiber reinforced polyamide specimens (Section 5). The damage investigation was conducted by means of three methods: 1) Infrared (IR) Thermography (Section 5.1); 2) Microscopic investigation of the fracture surface (Section 5.2); 3) Microscopic investigation of the crack path (Section 5.3). Damage mechanisms were investigated at multiple scales. Macro- meso- micro- nano-scale are frequently referred in the literature. Macro-scale is the scale of the specimen. At this scale, SFRPs are treated as homogeneous materials. The objectives of the macroscopic damage analysis are: 1) Localization of the crack initiation; 2) Investigation of the failure mode of plain and notched specimens. The meso-scale is the scale of the [redacted] (RVE) [6]. At this scale, fibers and matrix are treated discretely, whereas the microstructural variables (fiber orientation, fiber volume fraction, fiber aspect ratio) are used to globally describe the RVE [7]. The analysis at the meso-scale aims to study the influence of the fiber orientation on the crack initiation / propagation. The micro-scale is determined by the fiber diameter. At this scale, SFRPs are described as [redacted] materials (fiber, matrix, fiber-matrix interface). Particular attention is given to the analysis of the fiber-matrix interface after the application of cyclic loading. Fracture in composite materials can be either cohesive or adhesive depending upon whether a layer of matrix adheres to the fibers or not. Modern electron microscopes allow the investigation of the fiber-matrix interface at such a level of detail that was impossible just a few years ago. The issue whether damage occurs at the fiber-matrix interface or in the resin, at a certain distance from the interface, is crucial for the development of a mechanism-based model. Clean fibers on the fracture surface indicate fiber-matrix debonding. Otherwise, the mechanism is matrix cracking and the analysis should be focused on the resin layer covering the fibers. During the fiber forming process, fibers are coated by a mixture of chemicals and water called sizing in order to improve the fiber-matrix adhesion. Fiber sizing can chemically and mechanically affect the resin layer at the fiber-matrix interface leading to the formation of an interphase layer with different mechanical properties from the bulk matrix. Finally, a preliminary analysis of the damage mechanisms preceding the initiation of micro-cracks is presented.

2. Literature review on the damage mechanisms investigated by means of electron microscopy

The [redacted] on the damage investigation in SFRPs under fatigue loading date back to the [redacted] and were motivated by the following reasons: 1) Comparing the fatigue behavior of different material systems; 2) Improving the mechanical properties varying the material microstructure; 3) Finding quantitative relationships between material failure modes and macroscopic material response. Historically, the damage investigation by means of electron microscopy has been based on two techniques: analysis of the fracture surface or of the crack path [1, 2, 4, 8-10]. In the former case, specimens are tested until failure. In the latter case, fatigue tests are interrupted before failure and the side surface of the specimen is polished for microscopic investigations. These two analyses offer complementary and mutually enriching points of view. The degree of fiber-matrix adhesion, the pull-out length, the amount of broken fibers can be examined through both analyses. Instead, the observation of the fracture surface is better for investigating the matrix fracture behavior (ductile / brittle) while the analysis of the side surface of the specimen enables the study of the influence of the fiber distribution on the crack path. [redacted] present some disadvantages. The analysis of the fracture surface requires the specimen separation into [redacted] limiting the access to the mechanisms responsible for the [redacted] of damage. The observation of the side surface of the specimen limits the damage analysis to [redacted] planar section of the crack without any information on the damage mechanisms along the specimen thickness. A comprehensive analysis should consider multiple sections along the specimen thickness increasing the effort in the samples preparation.

Through the traces left by the crack on the fracture surface, it is possible to study the damage mechanisms occurring due to cyclic loading in a fatigue test. The damage mechanisms reported in literature were reviewed by reference to specific fractographic features: ductile / brittle matrix fracture behavior, fiber failure / pull-out, degree of fiber-matrix interfacial adhesion. Finally, the effect of the fiber distribution on the damage mechanisms was also reviewed.

1) Matrix fracture behavior

Evidence of ductility / brittleness on the fracture surface reflects the local mode of crack advance. Ductile matrix behavior was observed on the fracture surface caused by initiation and stable Fatigue Crack Propagation (FCP) [4, 11-[redacted]. Brittle matrix behavior indicates unstable crack propagation. Ductile matrix fracture indicates matrix yielding and appears in form of polymer filaments. The degree of matrix ductility on the fracture surface is related to the length of the polymer filaments. It depends on the material system, testing conditions (temperature, humidity), load level. Analyzing [redacted] Compact Tension CT specimens under fatigue loading, Karger-Kocsis [[redacted] observed a higher ductile matrix behavior at the end than at the beginning of the stable FCP. [redacted] and [redacted] [[redacted] compared the fracture surface of conditioned [redacted] plain specimens, failed under static and fatigue loading. They observed a much larger ductile area in the latter case. [redacted] and [redacted] [[redacted] also compared the fracture surface of conditioned and dry-as-molded fatigued specimens, reporting a higher degree of the matrix ductility in the former case. [redacted] and [redacted] [[redacted] observed that the degree of matrix ductility on the fracture surface of PA66-GF30 specimens failed under fatigue loading increases with testing temperature. The same result was reported by Noda et al. [18] for a short glass fiber reinforced polyamide (PA66-GF33) tested under fatigue loading below and above the glass transition temperature of the material and by Schaaf and coworkers [19] testing a short fiber reinforced polybutylene terephthalate (PBT-GF30) under strain controlled fatigue loading at different temperatures (T = -40; 23; 120 °C).

2) Fiber failure / fiber pull-out

The amount of broken fibers on the fracture surface depends on the loading type. In [4] it was found that the fracture surface of specimens failed under fatigue loading shows a higher amount of broken fibers than the fracture surface caused by static loading. According to [redacted] [4, [redacted] fiber failure is due crack closure effects. During the unloading part of the cycle, fibers fail by buckling and bending. [redacted] [[redacted] observed more pulled-out fibers than broken fibers on the fracture surface of [redacted] specimens failed under static loading. Testing [redacted] plain specimens under fatigue loading, Noda et al.[[redacted] observed pulled-out and broken fibers depending upon whether the temperature is above or below the glass transition temperature. [redacted] et al. [4, [redacted] noticed that the pull-out length is shorter on the fracture surface caused by stable FCP than unstable FCP. [redacted] and [redacted] [15] observed that the pull-out length is shorter for specimens failed under fatigue loading than under static loading.

3) Fiber-matrix adhesion

The quality of fiber-matrix adhesion strongly affects the fatigue strength of SFRPs. Friedrich [22] analyzed the crack propagation behavior for a short glass fiber reinforced Polyethylene terephthalate (PET) in case of good (commercial) and very poor sizing. The poorly bonded material system exhibited higher crack propagation rate. Friedrich observed mirror-clean fibers on the fracture surface of the poorly bonded material system and high interfacial fiber-matrix adhesion on the fracture surface of the commercial material. The degree of fiber-matrix adhesion strongly depends on the material system and the testing conditions. Horst [15] and Bernasconi [13] observed mirror-clean fibers analyzing the fracture surface of conditioned short glass fiber reinforced polyamide specimens failed under fatigue loading. The same result was reported by De Monte [23] testing PA66-GF35 tubular specimens under multi-axial loading in Dry As Molded (DAM) condition. Contrary to previous results, Karger-Kocsis noticed very good fiber-matrix bonding in a short fiber reinforced PA66 [24] and in a short glass fiber reinforced PBT [25] under fatigue loading.

Mirror-clean fibers on the fracture surface indicate fiber-matrix debonding. Instead, fibers covered by a resin layer suggest that damage occurs at certain distance from the interface in form of matrix cracking. Sizing can affect the resin layer surrounding the fibers leading to the formation of the interphase. Mechanical properties of the interphase differ from those of the bulk polymer ad the fibers. Hence, the transition between the fiber and the matrix should be described as a region with a specific thickness. The interphase in short glass fiber reinforced thermoplastics was investigated by Thomason in [26, 27]. Recently, Brodowsky and coworkers [28] developed an experimental technique to characterize the interphase of single fiber specimens under fatigue loading.

4) Summary of the literature review

The literature review demonstrated general agreement between different authors on the effect of failure mode (stable / unstable FCP), testing parameters (temperature, humidity) and material systems (matrix, fiber-matrix bonding) on matrix fracture behavior and fiber failure / pull-out. Instead, the degree of fiber-matrix interfacial adhesion remains a contentious issue with little agreement in the scientific community. This is due to the fact that only a [redacted] microscopes did not allow researchers to study the fiber-matrix adhesion with high accuracy. In [redacted] a comprehensive damage investigation on plain and notched short glass fiber reinforced polyamide specimens is presented. A high resolution FESEM microscope was used to study the effect of fatigue

[redacted]

loading on the mentioned fractographic features. Particular attention was devoted to the analysis of the fiber-matrix interfacial adhesion.

5) Fiber distribution at crack initiation

To complete the damage investigation, the role of the microstructure on the damage initiation / propagation should be taken into account. In fact, the worst combination of the weakest location due to local material microstructure and the local stress state creates the condition for crack initiation. The microstructure of the SFRPs is characterized by [redacted] fiber orientation, fiber aspect ratio and fiber volume fraction. The influence of the fiber orientation on the fatigue behavior of SFRPs is generally studied by machining specimens from an injection molded plate at different orientations with respect to the injection direction [4, 10, 25, 29-31]. If the fibers are aligned to the loading direction, damage was found to initiate at the fiber ends, where no sizing is expected [[redacted], 18, 19, 29, 32, [redacted].

The scenario changes if the fibers are aligned transversely with respect to the loading direction. Analyzing the fracture surface of CT specimens machined from injection molded plate crosswise to the mold flow direction (MFD), [redacted] al. [4] reported that pre-existent micro-regions of poor adhesion along the fiber-matrix interface are sites for crack initiation.

The fiber orientation also varies along the thickness showing a typical shell-core structure [[redacted], 30, [redacted] 35]. [redacted] and coworkers [[redacted] observed fiber-matrix debonding in the shell layers and matrix micro-cracks in the core layer. Analyzing tubular specimens under torsional fatigue loading, [redacted] et al. [[redacted] observed cracks nucleating in the core region where the fibers exhibit a lower alignment to the axial direction than in the skin layers. In [23] [redacted] Monte and coworkers, studied the combined effect of the local microstructure and the notch on the torsional fatigue strength of short glass fiber reinforced polyamide tubular specimens. It was found that the fatigue curves of plain and notched (molded-in V-shaped notch, $R_{notch} = 0.2$ mm) tubular specimen under pure torsional loading are almost overlapping. Instead, a significant decrease of the torsional fatigue strength was observed when drilling a hole (R = 1 mm) in the central part of the specimen. This result is due to the local microstructure which, in case of molded notch, locally increases the material strength.

The mutual position of the fibers also affects the crack initiation. [redacted] and [redacted] [2] observed that for a long fiber reinforced polyamide under fatigue loading, clusters of fibers perpendicularly oriented to the load direction are preferred sites for crack initiation. By studying a [redacted] and coworkers [[redacted] reported that the distance between [redacted] affects the damage initiation area.

3. Experimental

The material investigated in the present work is a short fiber reinforced polyamide containing [redacted] fibers (designation: [redacted]). Plain and notched specimens were injected along the longitudinal axis, which corresponds to the loading direction. Geometries and dimensions of the specimens are reported in Figure 1. The notch is a central slit of [redacted] with notch radius of [redacted] net stress concentration factor, calculated using isotropic material model, is $Kt_{net} = 9.81$.The notch was created with an insert in the mold cavity in order to reproduce the local fiber orientation at structural discontinuities in real injection molded parts. In fact, the majority of real plastic parts manufactured by injection molding are ready to use after ejection from the mold. Structural discontinuities in the mold perturb the melt flow affecting the local fiber orientation and consequently the material strength.

Relative humidity in the samples was kept [redacted] by storing them before testing, in a container with a drying agent (silica gel parts). Hence, specimens were tested in Dry As-Molded (DAM) conditions.

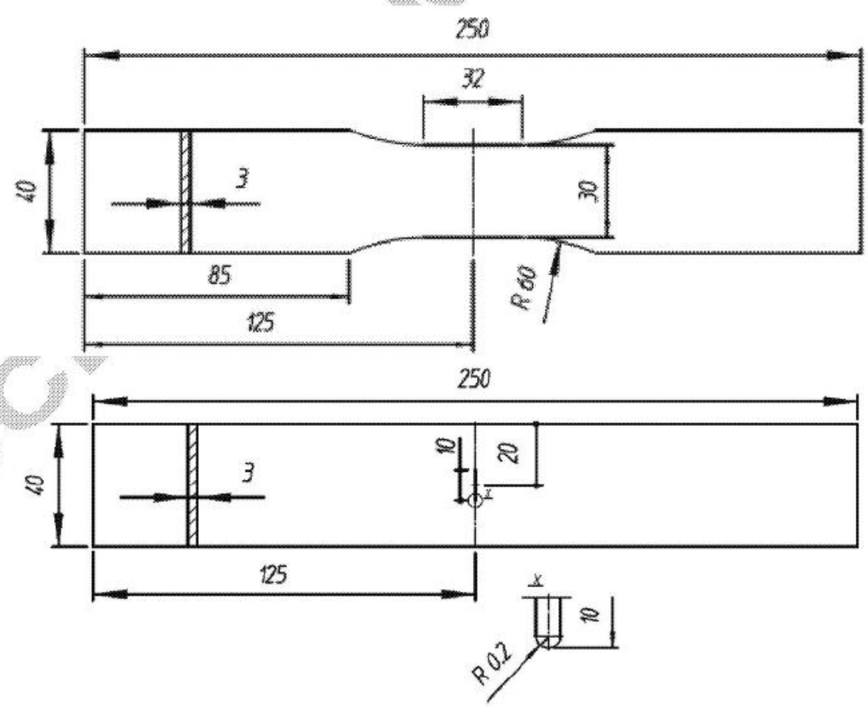


Figure 1. Specimen geometries and dimensions (in mm).

Uniaxial fatigue tests were carried out on a [redacted] servo-hydraulic testing machine. The fatigue tests were carried out under load control, applying a sinusoidal load with constant amplitude. Load ratio was kept constant and equal to [redacted] for [redacted] the performed [redacted] were tested at room temperature. Relative humidity in the room was not controlled during the fatigue tests. Testing frequency was chosen so as to avoid self-heating. Fatigue tests were carried out until specimen separation. An optical method presented by the authors in [[redacted] was used to quantify the contribution of the crack initiation to the total lifetime. An Infrared (IR) camera (FLIR ThermaCAM [redacted]) was used to monitor the macroscopic damage evolution in plain and notched specimens during the fatigue tests. Optical and electron microscopy were used. The optical microscope is a Zeiss Axioplan 1. The [redacted] microscope is a [redacted] with [redacted] accelerating voltage equipped with a EDAX X-Ray Si(Li)-Detector. The specimens analyzed through the [redacted] microscope were gold sputtered in order to improve the quality of the micrographs.

Cryogenic fracture tests on specimens previously tested until crack initiation were carried out to study the damage mechanisms ahead of the crack tip. The specimens were cooled down to T = - 196 °C and broken by using two pliers.

4. Fatigue test results

S-N curves to crack initiation and to failure (specimen separation) are shown in Figure 2. Fatigue data are represented in a double logarithmic diagram, plotting the net stress amplitude (σ_a) against the number of cycles (N) to crack initiation / failure. Fatigue curves are plotted using eq. 1 for a survival probability of [redacted].

$$N \cdot \sigma_a^k = N_A \cdot \sigma_A^k \quad (1)$$

Where N_A = 1E6 cycles and $\sigma_A = \sigma_a(1E6)$. The values of σ_A, k and the scatter index $T_\sigma = \sigma_{a,10\%}/\sigma_{a,90\%}$ are reported in Table 1. Plain specimens failed suddenly by unstable crack propagation. Hence, life to failure corresponds to life to crack initiation. Instead, notched specimen exhibit stable FCP. In [37] it was found that the amount of lifetime spent in the crack initiation depends on the fiber volume fraction. For PA66-GF35 notched specimens, the crack initiation takes some [redacted] of the tot[redacted]ifetime. The rest of the lifetime is spent in stable FCP.

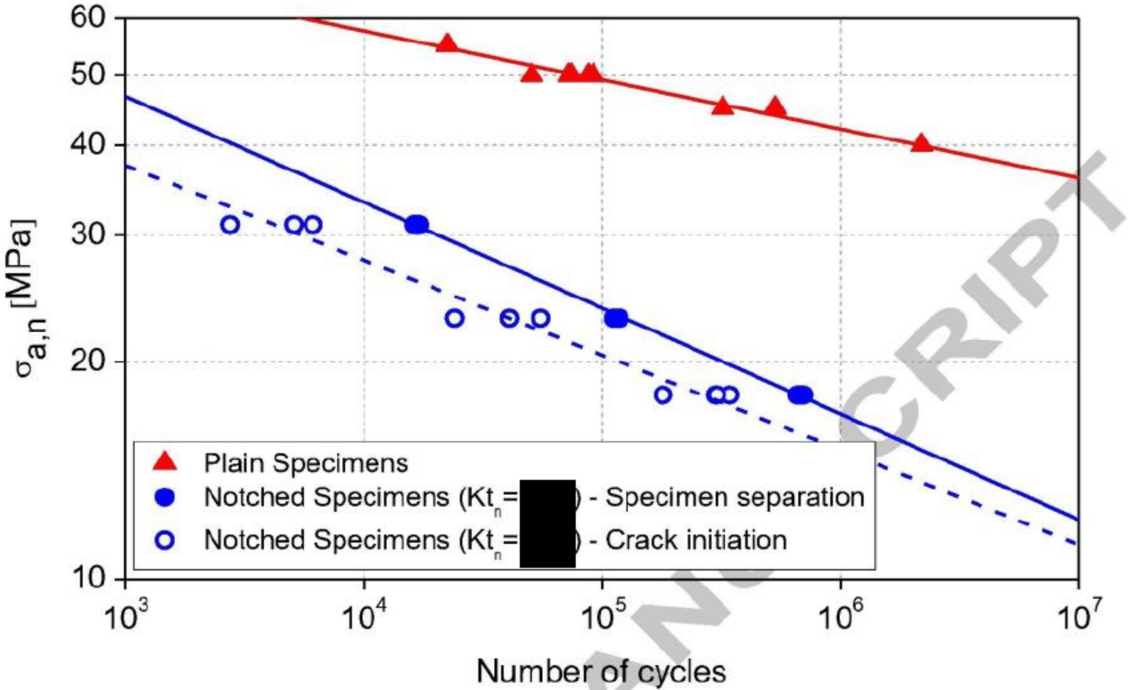


Figure 2. S-N curves up to crack initiation and up to failure of plain and notched specimens, T = RT, R = 0.

Table 1. Summary of the fatigue curve parameters for [redacted] series.

Geometry	Initiation			Failure		
	σ_A [MPa]	k -	T_σ -	σ_A [MPa]	k -	T_σ -
Plain	[redacted]					
Notched	[redacted]					

5. Damage investigation

5.1. Analysis of the failure modes of plain and notched specimens by means of IR thermography

The IR camera was used to monitor the temperature field on the specimen surface during the fatigue tests. This experimental method makes the macroscopic damage evolution before failure particularly evident (Figure 3). Plain specimens fail due to the unstable propagation of a crack. Failure is preceded by a localized temperature spot at [redacted] of the [redacted] at the shoulder tips (Figure [redacted]). The temperature spot can be observed just [redacted] cycles before the separation of the specimen in [redacted]. Similar results were reported by [redacted] et al. [[redacted]]. Instead, the notched specimens show stable FCP at [redacted] the sides of the notch (Figure 3b). The peaks of temperature, observable at a certain distance from the notch tips, indicate the crack front (Figure [redacted]).

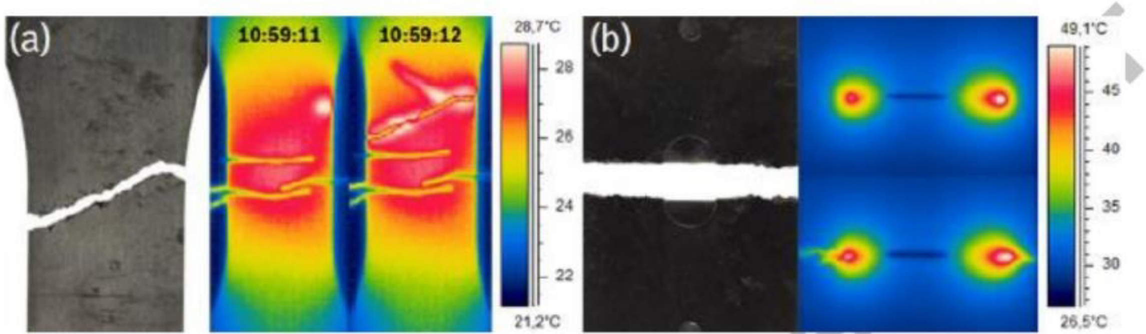


Figure 3. Crack path and temperature distribution at failure in (a) Plain specimens; (b) Notched specimens.

5.2. Analysis of the fracture surface

The microscopic analysis of the fracture surface of plain specimens reveals [redacted]. At crack initiation, where the temperature spot was observed (Figure 3a), the matrix exhibits a ductile behavior (Figure 4b). The rest of the fracture surface is comparatively brittle (Figure 4c).

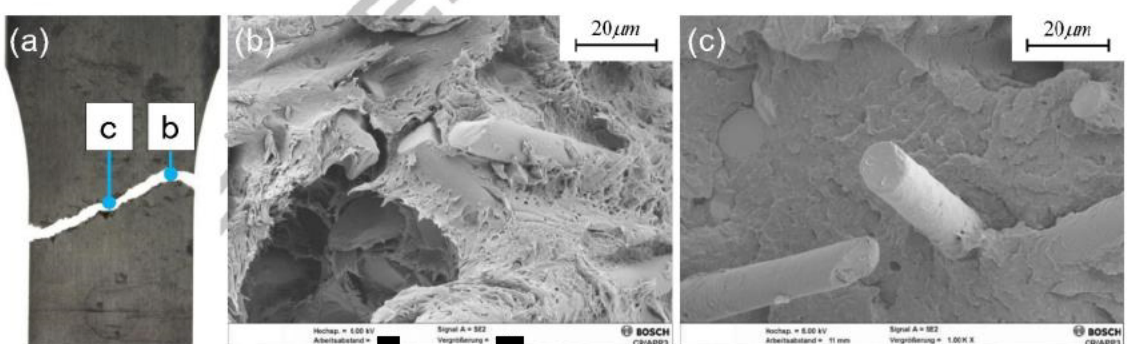
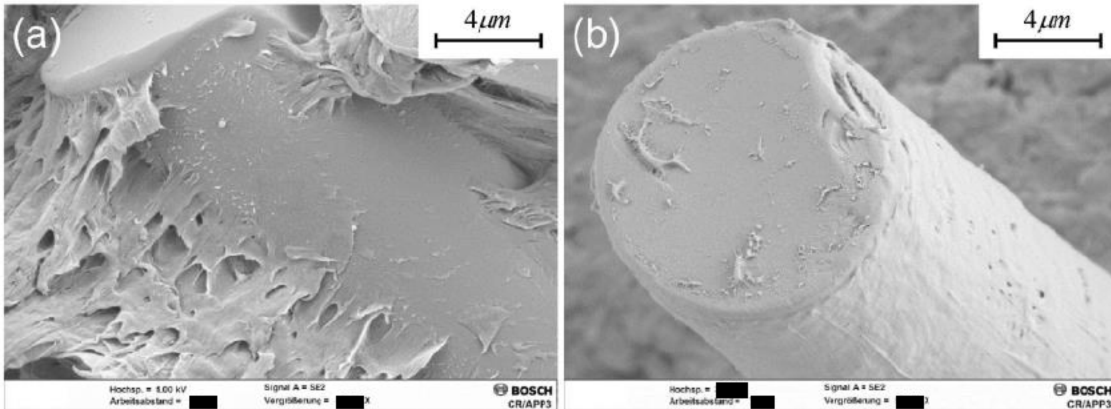


Figure 4. Fracture surface morphology of a plain specimen; (a) Investigated specimen; (b) Fracture surface at crack initiation; (c) Fracture surface caused by unstable crack propagation.

The examination of the fracture surface at high magnifications provides valuable insight into the state of the fiber-matrix interfacial adhesion (Figure 5). On [redacted] ductile and brittle areas, fibers are covered by a resin layer. Evidence of local matrix yielding in form of polymer fibrils is shown in Figure 5a. The high stress carried by the fibrils can cause either their failure or their detachment from the fiber surface. This [redacted] may be the reason of some clean regions on the fiber surface ([redacted] 5a). Conversely, on the brittle zone of the fracture surface, fibers are covered uniformly by a resin layer ([redacted] 5b).



[redacted] **5.** Degree of fiber-matrix interfacial adhesion on the fracture surface of a plain specimen; (a) Fracture surface at crack initiation; (b) Fracture surface caused by unstable crack propagation.

Failure of notched specimens occurs in [redacted] crack initiation, stable and unstable fatigue crack propagation. Fracture surface caused by stable and unstable FCP can be identified with unaided eye. The fracture surface caused by stable FCP is stress whitened and smooth ([redacted]). Instead, fracture surface caused by unstable FCP has a comparatively more irregular morphology ([redacted]). This result was also reported in [4, [redacted], [redacted]] for similar material systems. In [redacted], the stable FCP is indicated by the letter "s", while the unstable FCP is indicated by the letter "u". The fracture surface morphology varies according to the crack propagation mode. Near the notch tip, no evidence of ductile matrix deformation was observed ([redacted]). At the end of the FCP, higher degree of matrix ductility was observed ([redacted]). However, as indicated in [redacted], this region is very small. Next to this area, the fracture surface is brittle ([redacted]). This region is caused by unstable FCP and it is similar to that observed in [redacted]. Hence, unstable FCP leads to brittle fracture surface regardless of the specimen geometry. Comparing [redacted] with [redacted]-d, the matrix material behavior can be described neither as ductile nor as brittle. A similar description was provided in [[redacted], [redacted], [redacted], 40], although there is no full agreement on the terminology to be used. Hereinafter, the term "microductility" is associated with the evidence of ductile deformation at micro-scale only. The degree of the matrix ductility depends in fact on the scale of observation. An example is provided in [redacted] 7. [redacted] shows a low magnifications FESEM micrograph of the fracture surface caused by stable FCP. The micrograph shown in [redacted] 7b is an enlargement of [redacted]. In this case, the ductile matrix response is evident. Moreover, matrix cavitation around particles is observed.

The pull-out length on the fracture surface caused by unstable FCP is longer when compared with stable FCP. This result confirms the observations in [4, 15, 41]. During the stable FCP, crack propagates in a fiber avoidance mode. This mechanism was also observed in [5, 14, 19, 22, 41, 42]. Therefore, either the fibers are slightly below the free surface of the crack, or just a small portion of them is visible from the observation of the fracture surface. On the fracture surface caused by unstable FCP, the high amount of released energy leads to a more irregular crack path. Fibers are extracted from the fracture surface. [redacted] are observed on [redacted] the fracture surfaces caused by stable and unstable FCPs. This indicates that fiber failure is not the dominant mechanism.

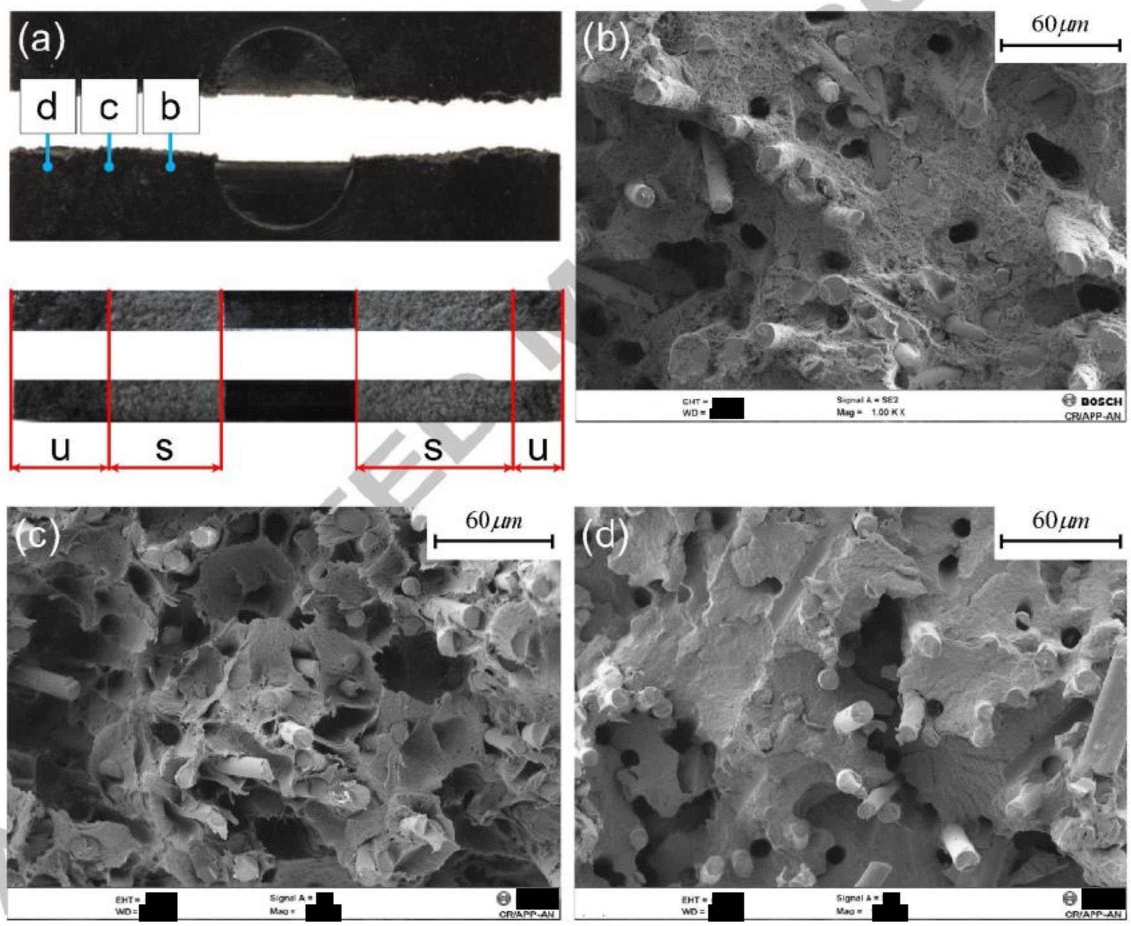


Figure 6. Fracture surface morphology of a notched specimen; (a) Failed specimen; [redacted] and “u” indicate the fracture surface caused by stable and unstable FCP respectively; (b) Fracture surface at crack initiation; (c) Fracture surface at the end of the stable FCP; (d) Fracture surface caused by unstable FCP.

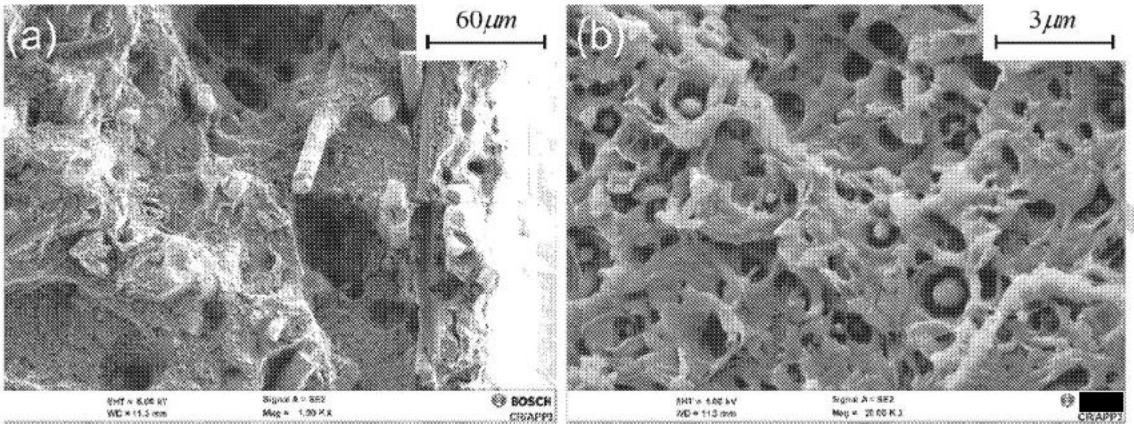


Figure 7. Fracture surface caused by stable FCP (notched specimen); (a) Low magnification; (b) High magnification.

So far, it is not clear if cavities observed on the fracture surface precede or follow the crack onset. In order to study this mechanism, a fatigue test of a notched specimen was interrupted at crack initiation (Figure [redacted]). Then the specimen was cryo-fractured at T = - 196 °C (Figure 5.8b). This test has a twofold aim: 1) Investigate whether the cavities can be observed also in the region ahead of the crack tip. 2) Investigate if there are pre-existing foreign particles in the material. If so, evidence of particles should be provided also on the cryo-fractured surface. Figure 5.8c-d shows the fracture surface caused by stable FCP and the cryo-fractured surface. Cryo-fracture leads to brittle fracture surface.

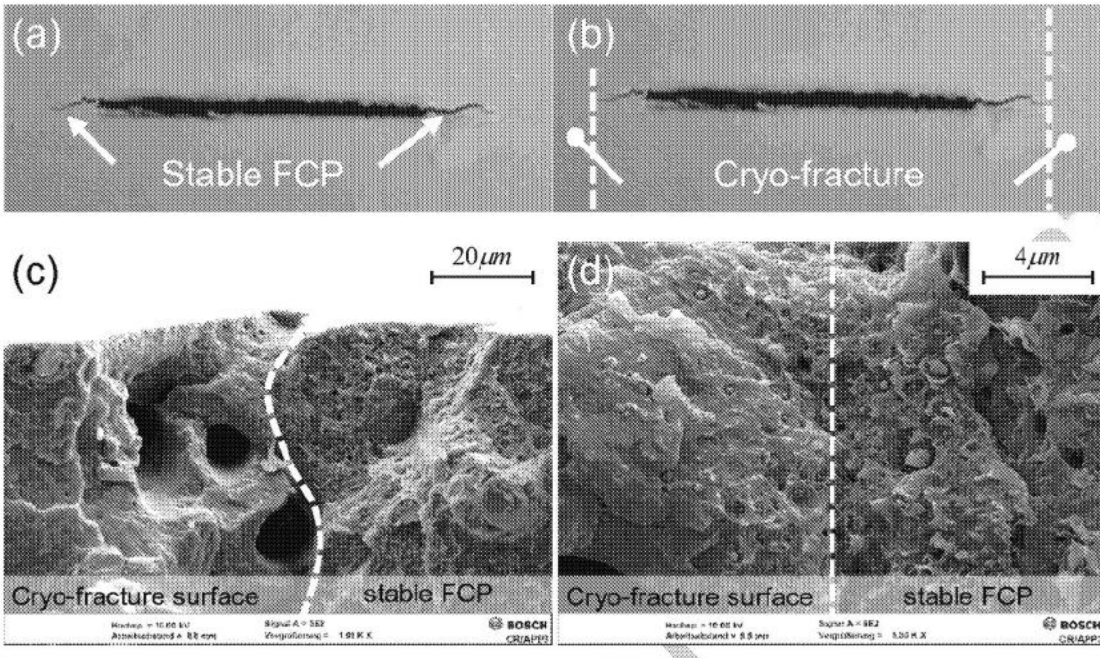
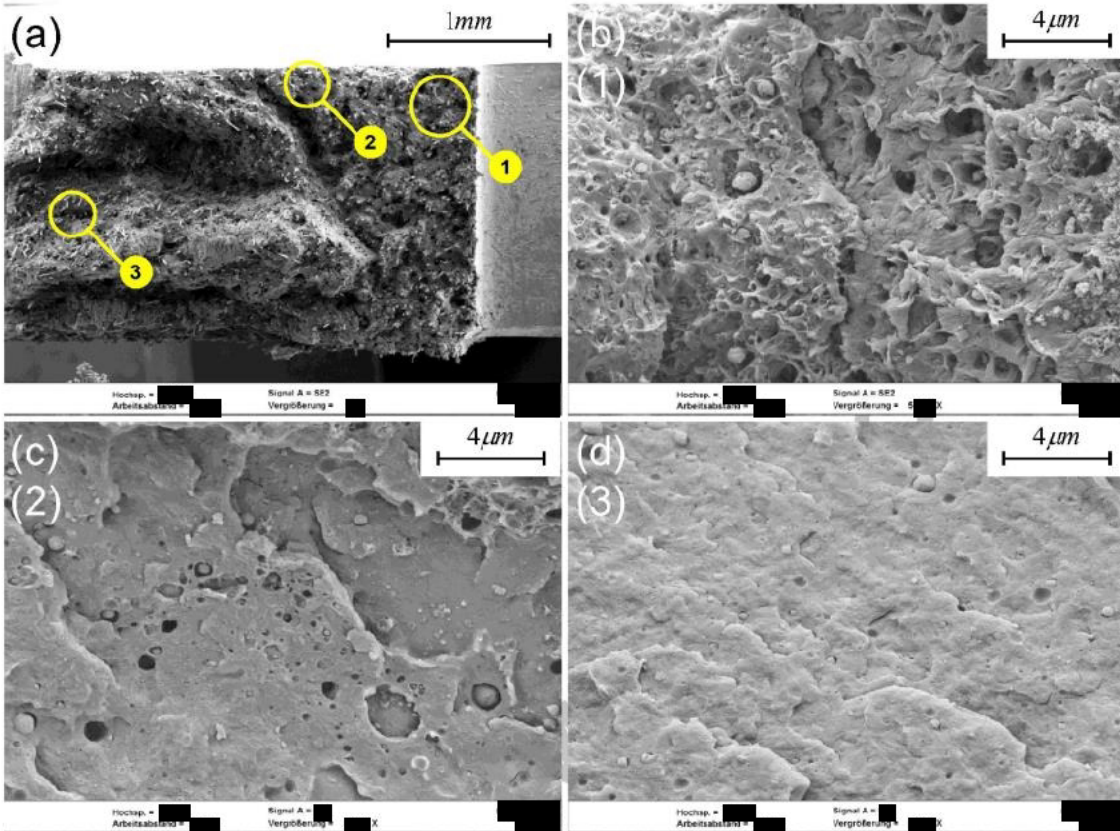


Figure 5.8. Stable FCP and the cryo-fractured surface: (a) Fatigue test was interrupted at crack initiation; (b) Cryo-fracture; (c) Fracture surface, low magnification; (d) Fracture surface, high magnification.

Figure 9 shows FESEM micrographs of different regions on the fracture surface of the cryo-fractured specimen shown in Figure 5.8. Cavities and particles are observed on the fracture surface caused by stable FCP (Figure 9b). On the region immediately ahead of the crack tip, a certain amount of cavities is observed (Figure 9c). Cavities can also be observed on the cryo-fractured surface (Figure 9d), far away from the crack tip. However they are few in number and also smaller. This preliminary investigation suggests that coalescence of voids precedes the formation of a micro-crack. The result would confirm some experimental evidence of voids in a short fiber reinforced polyamide under static loading, recently provided in [11, [redacted]].



[redacted] **9.** Fracture surface of notched specimen. Fatigue test was interrupted at crack initiation and then cryo-fractured. (a) Overview of the fracture surface; (b) Fracture surface caused by stable FCP region; (c) Cryo-fractured surface ahead the crack tip; (d) Cryo-fractured surface far from the crack tip.

[redacted] [redacted] shows examples of particles on the cryo-fracture surface, immediately ahead of the crack tip. The smooth fracture surface is interrupted by cavities of different sizes. Some cavities are filled by particles, some others are instead empty. A difference in terms of surface appearance between the particle and the matrix around it can be also appreciated in [redacted] [redacted]. This experimental evidence seems to suggest that the particles are not made by the same material of the matrix.

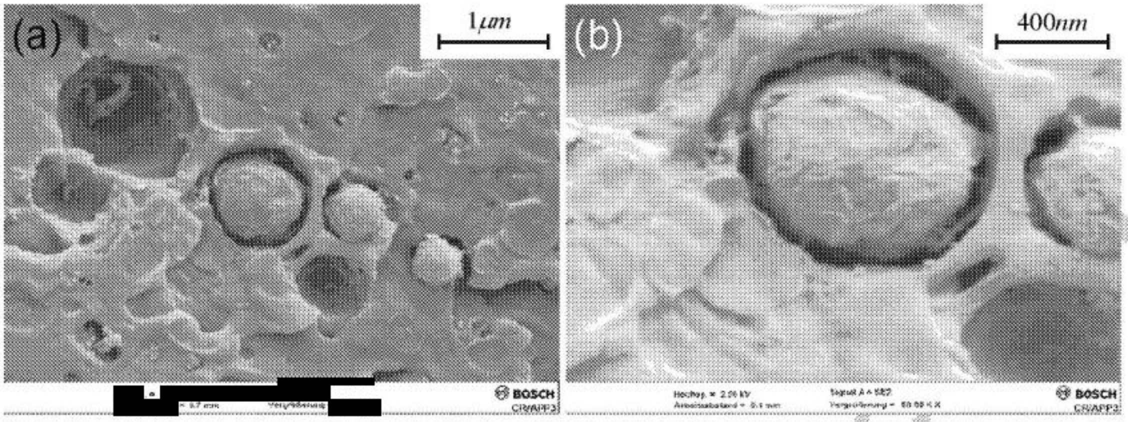
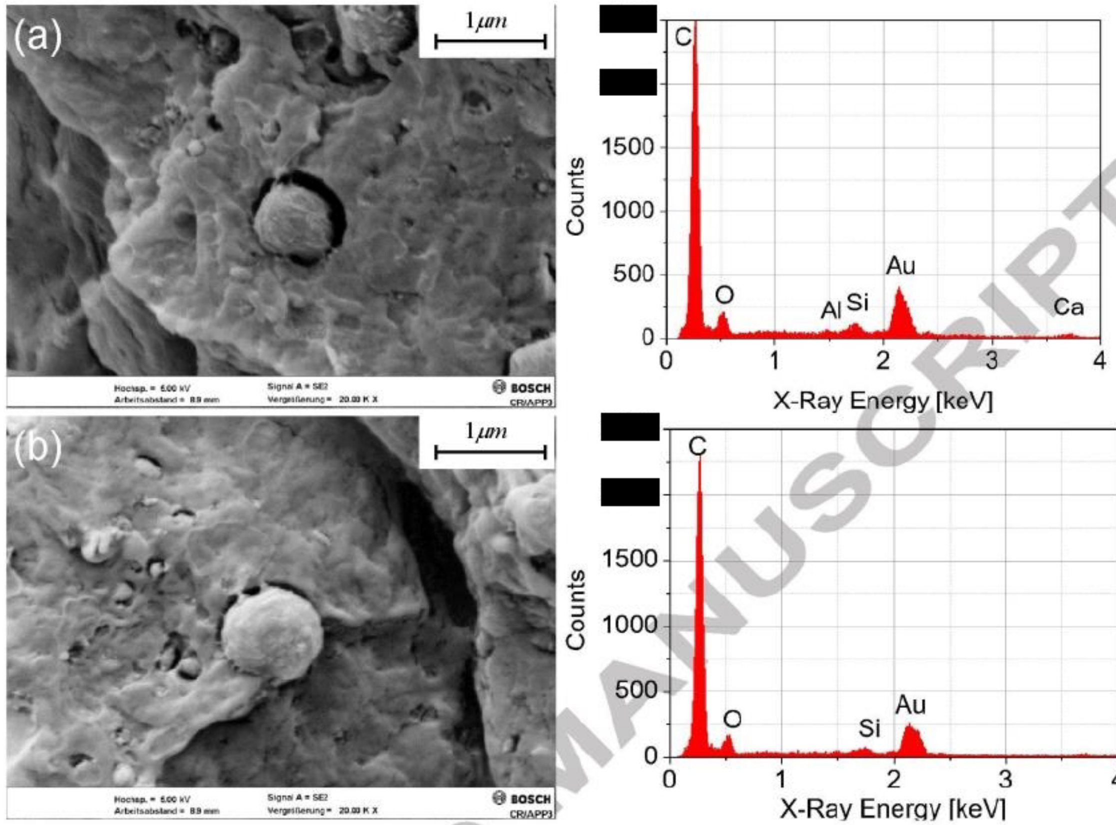


Figure 10. Particles on the cryo-fractured surface ahead the crack tip.

Energy-dispersive X[redacted] ([redacted]) analyses of the particles were also performed to study their elemental composition (Figure [redacted]). The [redacted] analysis was performed with an excitation voltage of [redacted]. The peaks of carbon, oxygen and nitrogen (between the [redacted]) are attributed to the polymer. A peak of gold is due to gold-sputtering. Smaller peaks of Silicon and Calcium were also noticed and may be related to the occurrence of a fiber fragment in the analyzed volume. Hence, the [redacted] analysis did not provide clear indication on the nature of the observed particles. It is worthwhile noting that the spatial resolution of the [redacted] detector is on the order of 3 μm and it is probably too coarse for the elemental analysis of particle of diameter 0.1 - 0.3 μm. The aim of this preliminary analysis was to highlight a new damage mechanism occurring below the micro-scale and preceding the formation of a micro-crack. For better understanding of this mechanism, further investigations are needed using more accurate experimental techniques.



[redacted] **11.** EDX analysis of particles found on the cryo-fractured surface ahead the crack tip.

The fracture surface shown in [redacted] is caused by stable FCP. Therefore, between the [redacted] described in [redacted], the area shown in [redacted] was examined at high magnifications in order to observe the state of the fiber-matrix interfacial adhesion. [redacted] shows [redacted] perpendicular to fracture surface, ([redacted]) and the other lying on the fracture surface, ([redacted]) near the free surface of the notch. In [redacted], fibers are covered by a resin layer. The microscopic examination suggests that damage occurs at a certain distance from the fiber-matrix interface. A thorough examination reveals that the resin layer is thicker for the fiber perpendicularly oriented to the fracture surface than for the fiber lying on the fracture surface.

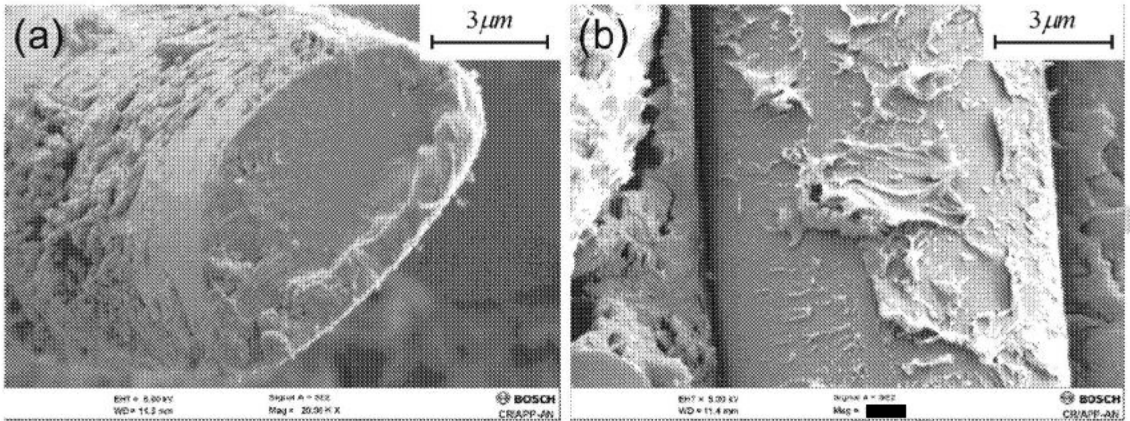


Figure 12. Fiber-matrix adhesion on the fracture surface of a notched specimen caused by stable FCP; (a) Fiber perpendicular to the fracture surface; (b) Fiber lying on the fracture surface.

5.3. Analysis of the crack path

In this [redacted]on, damage mechanisms were investigated on the side surface of the specimens. Fatigue tests were interrupted before failure using the IR camera ([redacted]on 5.1). Samples at crack initiation were machined from the specimens and analyzed by means of optical and field emission scanning electron microscope. The goals of this section are: 1) To analyze the damage mechanisms at crack initiation in plain and notched specimens; 2) To study the fiber orientation distribution at crack initiation.

[redacted] [redacted] shows the damage analysis on the side surface of a plain specimen. Evidence of damage was found using the reflected light microscope ([redacted] [redacted]). This region was thoroughly investigated by means of FESEM. Figure 13 shows typical mechanisms occurring just before the specimen failure. Several micro-[redacted] were observed indicating that a macro-crack forms due to the coalescence of micro-[redacted]. An example of fiber failure is shown in [redacted] [redacted]. [redacted] shows a small crack occurring between the matrix and a fiber fragment. Figure [redacted] shows a crack at the end of a longitudinally oriented fiber. Here the crack propagates at a distance of [redacted] from the fiber surface. [redacted] [redacted]f represent [redacted] pictures of the region 4. Voids can be observed close the fiber ([redacted] [redacted]). Coalescence of voids seems to represent the damage stage preceding the formation of a micro-crack in the matrix. If one compares [redacted] [redacted]with [redacted]9b-c, it can be deduced that the size of the voids is comparable.

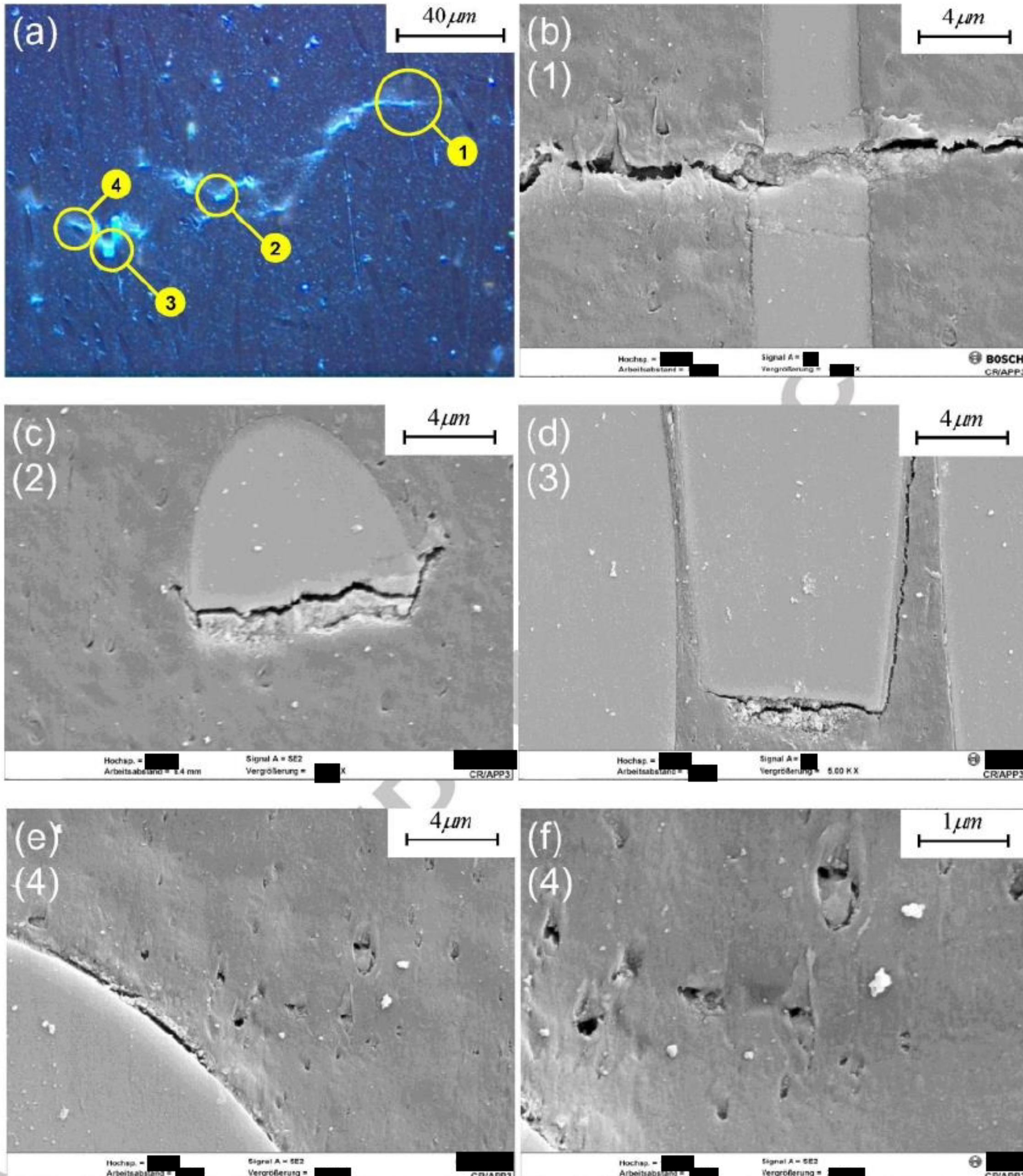


Figure 13. Damage investigation on the side surface of a plain specimen, interrupted fatigue test; (a) Damage zone observed through optical microscopy; (b) Fiber failure; (c) Crack at fiber fragment; (d) Crack at the fiber end; (e),(f) Voids in the matrix.

[redacted] shows the side surface of a notched specimen subjected to interrupted fatigue test. The crack does not initiate at the notch tip as it would be expected if the material was homogeneous. At higher magnifications ([redacted]-c), it is possible to examine the fiber orientation distribution around the notch. At the close vicinity of the free surface of the notch, fibers are oriented through the thickness. This is deduced observing the circular footprints left by the fibers on the polished section. [redacted] from the free surface of the notch, the fibers are mostly in-plane oriented. Around the notch, fibers are unevenly distributed. They tend to agglomerate and form clusters. It is worthwhile noting that the location of crack initiation corresponds with a cluster of through-the-thickness oriented fibers (Figure [redacted]d). The analysis of the side surface of other specimens confirmed the occurrence of fiber clusters around the notch. As well as being a geometric discontinuity, the insert also represents an obstacle for the melt stream during injection molding affecting the local fiber orientation. The analysis of the crack propagation shows that the crack exhibits a zig-zag path (Figure [redacted]. As soon as the effect of the insert vanishes, the crack follows the direction imposed by the in-plane fibers growing in a fiber avoidance mode (Figure [redacted]).

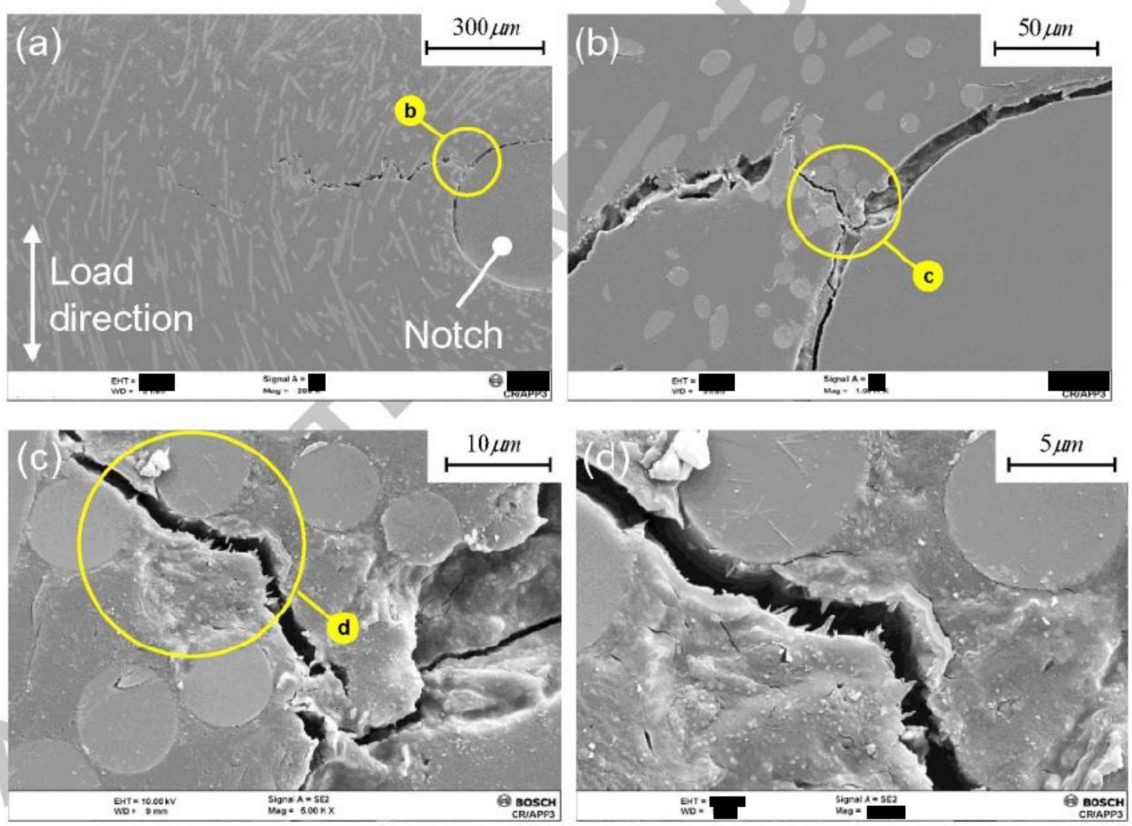
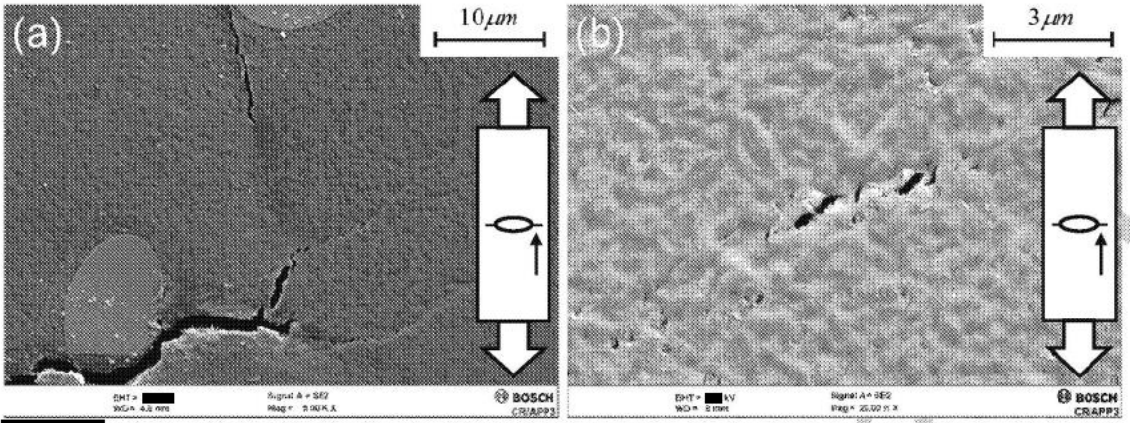


Figure 14. Fiber distribution at crack initiation for a notched specimen, interrupted fatigue test; (b), (c), (d) are magnified views of the crack.

[redacted] shows examples of matrix-cracking at the tip of a crack. [redacted] micrographs suggest that the crack forms by coalescence of voids.



[redacted]. Matrix cracking (notched specimen)

[redacted] [redacted] shows damage mechanisms along the crack path in different specimens. Crack propagates in a fiber avoidance mode ([redacted] [redacted]b). Fibers are covered by a resin layer. Figure 16c provides an example of fiber pull-out in which the fiber extracted from the fracture surface is completely covered by resin layer. This layer is around half a micrometer thick ([redacted] [redacted].The FESEM micrographs shown in [redacted] [redacted] indicate that damage occurs at a certain distance from the fiber matrix interface in form of matrix cracking and not in form of fiber-matrix debonding. Hence, the lifetime prediction model should be able to reproduce the damage initiation and propagation at the resin layer covering the fibers. These results confirm the observations of the fracture surface in [redacted]. They are valid for the material system and for the testing conditions reported in this paper.

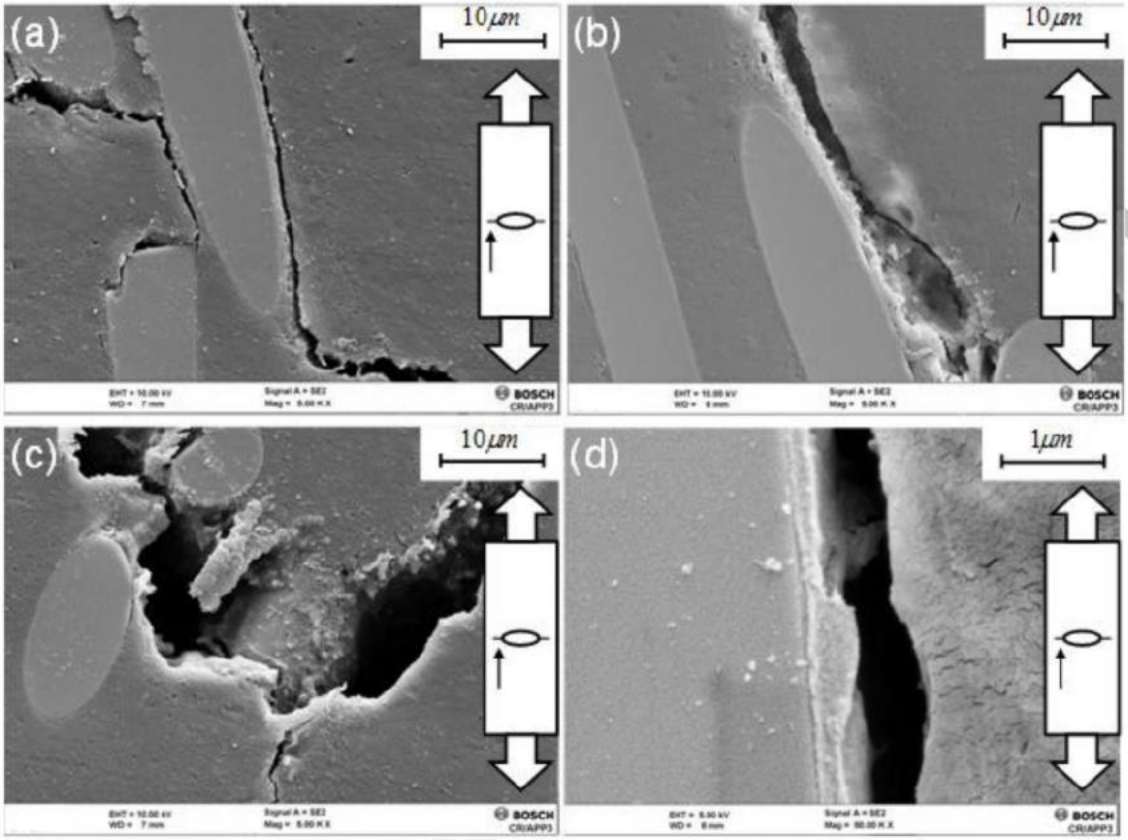


Figure 16. Damage mechanisms along the crack path (notched specimen).

6. Conclusions

In this work a damage investigation on a short glass fiber reinforced polyamide under fatigue loading is presented. The use of IR thermography enabled the localization of the damage initiation site. The damage mechanisms were studied by means of optical and electron microscopy investigating the following fractographic evidences: ductile / brittle matrix fracture behavior, fiber failure / pull-out, degree of fiber-matrix interfacial adhesion. The results can be summarized as follows.

1. *Failure modes*. Plain and notched specimens exhibit different failure modes. Plain specimens fail due to unstable FCP preceded by a local temperature spot at shoulder tips. Failure scenario of notched specimens follows [redacted]: 1) Crack initiation; 2) Stable crack propagation; 3) Unstable crack propagation.
2. *Damage mechanisms*. The analysis of the fracture surface reveals that the material response varies according to the crack propagation mode (stable / unstable FCP). Matrix fracture behavior is micro-ductile on the fracture surface caused by stable FCP and brittle on the fracture surface caused by unstable FCP.

[redacted] fractographic analysis showed that fibers are mostly covered by a resin layer. This result, valid for the material system and the testing conditions presented in this work, indicates that damage does not occur at the fiber-matrix interface in form of fiber-matrix debonding but in the resin, at a certain distance from the interface in form of matrix cracking. A better understanding of the effect of sizing on the matrix at the fiber-matrix interface would be required to determine to what extent sizing affects the resin layer around the fibers leading to the formation of the interphase.

Cavities were found on the fracture surface caused by stable FCP. With the aim to investigate this damage mechanism, fatigue tests of a notched specimens were interrupted at crack initiation and then cryo-fractured. Cavities were also observed on the cryo-fracture surface immediately ahead of crack tip. Far from the crack tip, cavities are fewer and smaller. This preliminary analysis suggests that coalescence of voids in the matrix precedes the formation of a micro-crack. Evidence of voids was proved analyzing the side surface of plain and notched specimens after interrupted fatigue tests.

3. *Fiber distribution at crack initiation.* The insert used in the mold for creating the notch affects the fiber orientation at crack initiation. Clusters of through-th[redacted]thickness oriented fibers characterize the fiber orientation distribution around the notch. The crack was found to initiate in the matrix between through-the-thickness oriented fibers.

   In plain specimens, micro-cracks were found at the end of fibers aligned to the loading direction. Some evidence of fractured fibers (longitudinally oriented with respect to the loading direction) was also provided.

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Highlights

- Damage mechanisms on plain and notched [redacted] specimens undergoing fatigue loading have been investigated by means of electron microscopy.
- Clusters of through-the-thickness oriented fibers located around the notch affect the crack initiation position.
- Damage initiates in the matrix in form of nano-voids and cavitation around nano-particles.
- Damage evolves in form of matrix cracking at a certain distance from the fiber-matrix interface and not in form of fiber-matrix debonding at the interface.