DAMAGE MECHANISMS OF SHORT GLASS FIBER REINFORCED POLYAMIDE 6.6 UNDER FATIGUE LOADING

E. Belmonte[a,b*], M. De Monte[b], M. Quaresimin[a], C. Hoffmann[b]

[a]*Department of Management and Engineering, University of Padova, Stradella S. Nicola 3, 36100 Vicenza (Italy)*
[b]*Robert Bosch GmbH, Corporate Sector Research and Advance Engineering – Advance Production Technology 1 – Plastics Engineering, Waiblingen (Germany)*
** Enrico.Belmonte@de.bosch.com*

Keywords: Short Fiber Reinforced Composites, Damage Mechanisms, Fatigue, Polyamide

Abstract
In this paper, the state of the fiber-matrix interface and the matrix behavior of a short glass fiber reinforced polyamide (PA66-GF35) under fatigue loading were investigated. Significant differences were observed between plain and notched specimens. Comparing the morphology of the fracture surface at crack initiation site, the notched specimens show a lower inelastic matrix deformation and a higher degree of fiber-matrix adhesion. The difference in terms of matrix behavior is attributed to the failure mode of the specimens. In the plain specimens, the damage nucleation, observed in form of a temperature spot, is followed by the unstable propagation of the crack; instead, in the notched specimens, a stable crack propagation phase was observed.

1. Introduction

Short fiber reinforced plastics (SFRPs) are widely employed in the automotive industry.
Especially in case of fatigue loading, the application of these materials for designing structural parts requires the development of failure approaches to reduce the experimental effort in predicting material behavior. Many papers about the damage mechanisms on SFRPs have been published so far [1-[11]]. In the last 30 years the experimental methods to investigate damage mechanisms have taken huge steps. In particular, the use of Field Emission Scanning Electron Microscopy (FESEM) allows to investigate the material microstructure at high magnifications enabling an improvement respect to the Scanning Electron Microscopy (SEM) in terms of resolution, magnification and image quality.
Two issues were addressed in this work: the role of the interface in the damage evolution and the matrix behavior. Firstly, it was investigated whether damage propagates at the fiber-matrix interface or in the matrix at a certain distance from the interface itself. Secondly, the fracture surface morphology was examined in order to figure out if the matrix behaves in a ductile or brittle manner.

Both the issues are fundamental to develop a damage model for the prediction of the fatigue behavior of such materials. The analysis of the degree of fiber-matrix adhesion helps to figure out whether or not a damage model has to take into account the degradation of the interface; the examination of the matrix morphology gives valuable information on the suitable constitutive law for the matrix material.

2. Experimental

The material studied in the present investigation is a short fiber reinforced polyamide containing 35wt% glass fibers (designation PA66-GF35). Fibers have a diameter of 10 μm and an average length of approximately 280 μm. Plain and notched specimens were injected along the longitudinal axis that is also the load direction. Notches are molded-in and not machined. Geometries and dimensions of the specimens are reported in Fig 1. The geometry of the notch is a slit which leads to a theoretical stress concentration factor (Kt) of 9.81 times the nominal stress. Relative humidity in the samples was kept under 0.1wt% storing them, before the tests, in a container with a drying agent (silica gel parts); they were tested thus in dry as molded conditions.

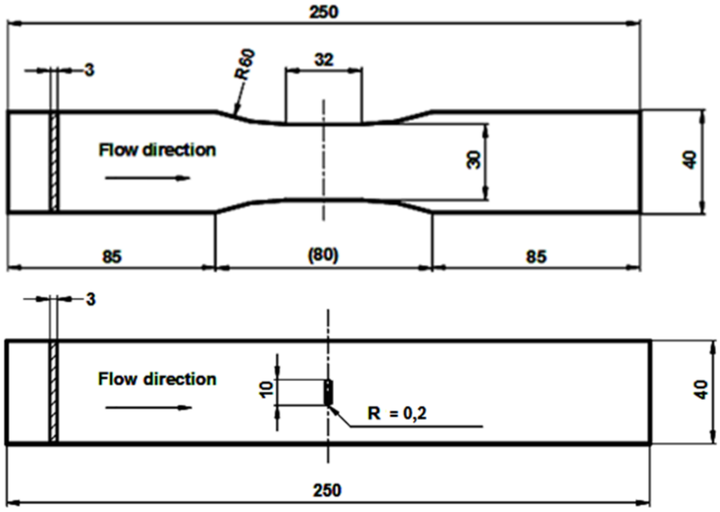

Figure 1: Specimen geometries and dimensions (in mm).

Uniaxial fatigue tests were carried out on a Schenck Hydropulsar servo-hydraulic testing machine, equipped with a load cell of 10 kN. The fatigue tests were carried out under load control, applying a sinusoidal load function with constant amplitude. Load ratio was kept constant and equal to 0 for all the performed tests. The fatigue tests were carried out at room temperature without any control on temperature and humidity. The frequencies were chosen so as to avoid the self-heating of the specimen. The adopted criterion for choosing the test frequency was to keep the increment of temperature on the surface of the specimen less than 5°C. Fatigue results were analyzed in terms of nominal stress amplitude to the net section normalized by the quasi-static strength (UTS) at room temperature of that specific specimen, versus the number of cycles to failure N (Figure 2). Test results are reported in double logarithmic scales. The parameter k is the inverse slope of the S-N curve. Some tests on notched specimens were interrupted before failure in order to investigate the damage mechanisms on the longitudinal section of the specimen. However, these results are not reported in Figure 2. For the damage investigation analysis, two specimens failed in high cycle fatigue regime were chosen.

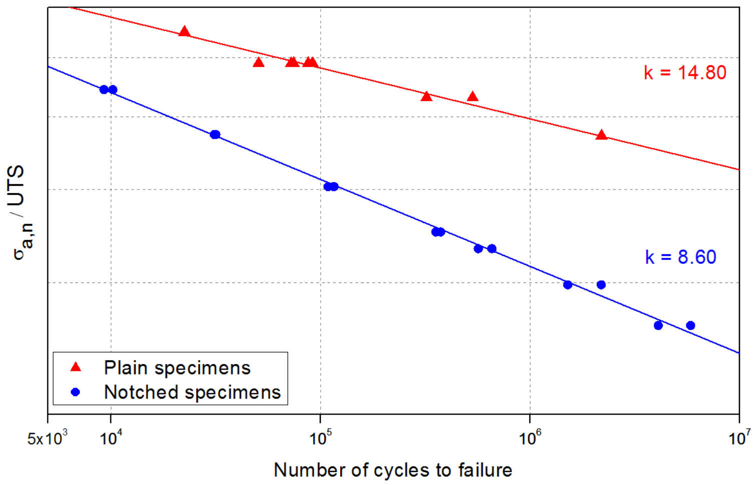

Figure 2: Normalized S-N lines for plain and notched specimens.

3. Damage investigation

3.1. Failure modes of the specimens under fatigue loading

Plain and notched specimens exhibit a different failure mode. The use of the infrared thermography shows that plain specimens fail due to the unstable propagation of a crack. Failure is preceded by a localized temperature spot occurring invariably at one of the four stress concentrations at the shoulder tips (Fig 3a). The temperature spot can be observed just few cycles before the separation of the specimen in two parts. Instead, the notched specimens show a stable crack propagation phase both at the sides of the notch (Fig 3b). The peaks of temperature, observable at a certain distance from the notch tips, indicate the front of the crack (Fig 3b). Just at the end of the fatigue test, when the crack has already steadily propagated reducing the bearing section, the separation of the specimen into two parts is preceded by the unstable propagation of a crack.

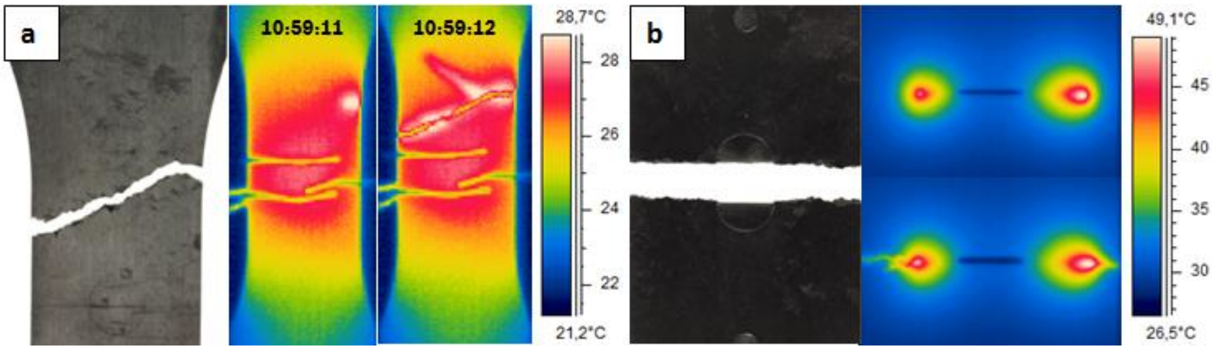

Figure 3 Crack profile and thermal analysis of (a) plain specimens, (b) notched specimens.

3.2. Post mortem analysis of the fracture surface morphology – plain specimens

The microscopic analysis of the fracture surface of the plain specimens reveals two morphologically different areas. At crack initiation, where the temperature spot was observed (Fig 3a), matrix exhibits a ductile behavior (Figure 4a). On the rest of the fracture surface, a brittle fracture material morphology was noticed (Figure 4b). This distinction between ductile and brittle areas was reported also in [[1]-[3]].

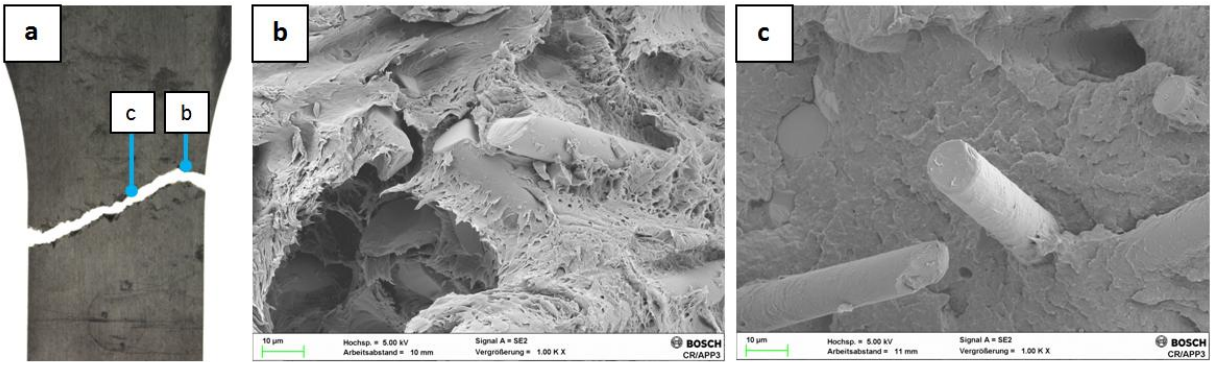

Figure 4 Fracture surface morphology of a plain specimen: (a) Investigated specimen; (b) micrograph at crack initiation; (c) micrograph far from the crack initiation position.

The examination of the fracture surface at high magnifications provides valuable insight into the state of the fiber-matrix adhesion. On the ductile, as well as on the brittle area of the fracture surface, fibers appear mostly covered by a matrix layer. However, some morphological differences can be observed. On the ductile area, fibrils of matrix bridge the fiber to the rest of the material (Figure 5a). The high stress carried by these fibrils can however cause either their failure in a ductile manner or their detachment from the fiber surface. This second mechanism may be the cause of some clean regions on the fiber surface (Figure 5a). Instead, on the brittle part of the fracture surface, fibers are covered continuously by a matrix layer. The high degree of fiber-matrix adhesion was reported also in [[1]-[3], [6]-[8]]

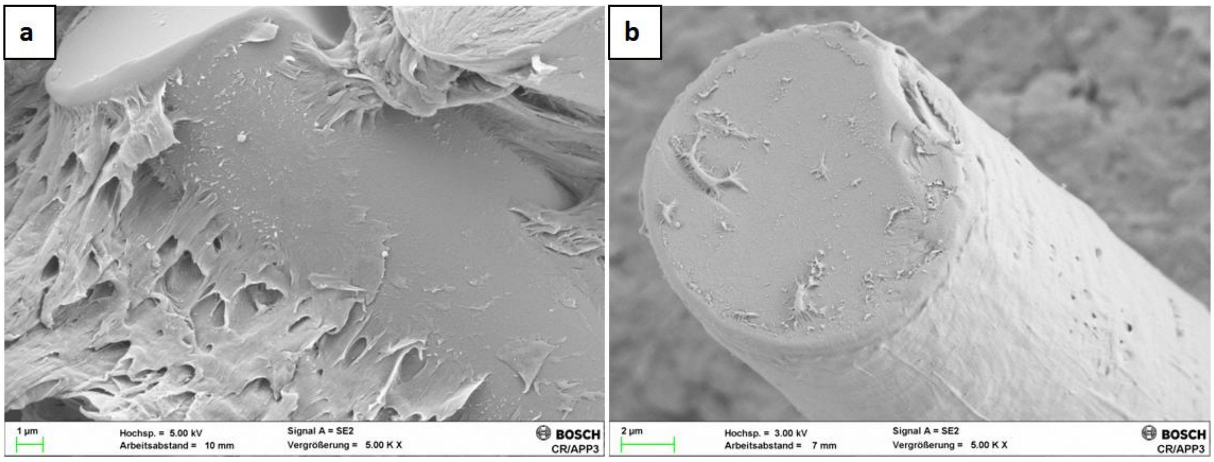

Figure 5 Analysis of the fiber-matrix interface: (a) micrograph at crack initiation; (b) micrograph far from the crack initiation position.

3.3. Post mortem analysis of the fracture surface morphology – notched specimens

As shown in Figure 3b, the lifetime of the notched specimens includes a stable crack propagation phase. The examination of the fracture surface to the naked eye reveals that the stable Fatigue Crack Propagation (FCP) is characterized by stress whitening (Figure 6a). Instead, the unstable FCP is dark (Figure 6a). This result was also reported by Lang at al.[[6]] and by Günzel and al. [[8]] for similar material systems. In Figure 6a, the stable FCP is indicated by the letter “s”, while the unstable FCP is indicated by the letter “u”. The large extension of the stable FCP indicates that a long fraction of the fatigue life was spent in the stable propagation of a crack. The analysis of the fracture surface reveals that the material morphology varies according to the crack propagation mode. On the region close to the notch root, no evidence of large inelastic matrix deformation was observed (Figure 6b). Comparing Figure 6b with Figure 5a-b, the matrix material behavior can be described neither as ductile nor brittle. Instead it seems to reflect the stepwise propagation of the crack. This fracture surface morphology is representative of the entire stable FCP. A similar description was provided in [[7]] and in [[8]], although there is no fully agreement on the terminology to be used for the classification of this kind of fracture surface morphology. In fact, in [[7]] it is described as semi-brittle while in [[8]] as micro-ductile. At the end of the FCP, extensive material ductility was observed (Figure 6c). However, as indicated in Figure 6a, the extension of this region is very small. The reason of the high matrix ductility can be explained as the material response to the high inelastic matrix deformations occurring at the end of the stable FCP. The fracture surface morphology shown in Figure 6b is similar to that observed in Figure 4b. Furthermore, some similarities in terms of fracture mode can be found. While on the stable FCP the propagation of the crack is stepwise, indicating that the crack arrests at each cycle, on the area characterized by high matrix ductility, the matrix deformation is continuous and precedes the unstable phase of the crack propagation. Moreover, the progressive reduction of the net section due to the crack growth leads to an increasing stress on the bearing section. Observing Figure 6a, it is not hard to imagine that the bearing section, at the end of the stable FCP, is reduced to half of its length leading to a higher average stress on the net section.

Right after the area showing a large matrix ductility, the matrix morphology is brittle (Figure 6c). This region corresponds to the unstable crack propagation phase and is similar to that observed in Figure 4b.

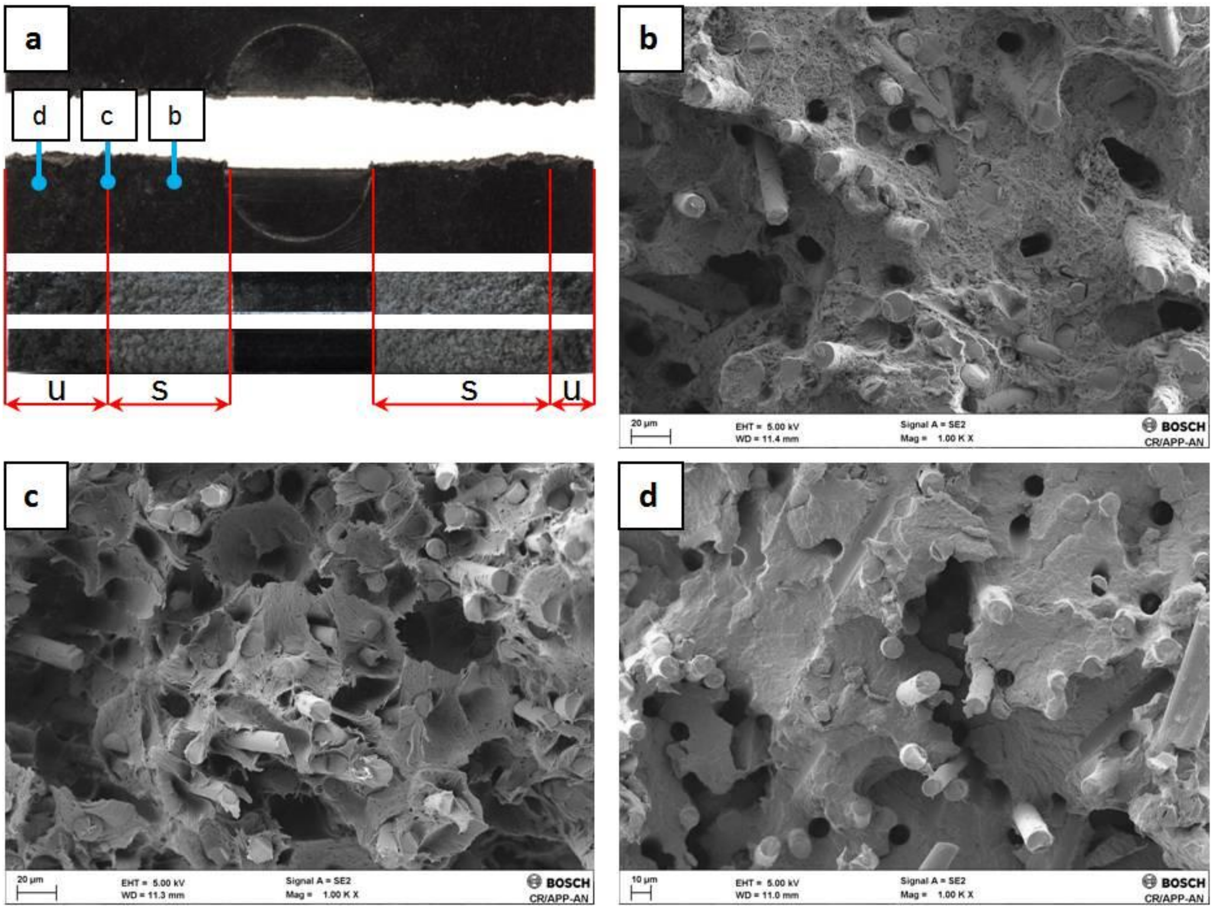

Figure 6 Fracture surface morphology of a notched specimen (a) Investigated specimen, “s” and “u” indicate respectively the stable and the unstable phase of the crack propagation (b) micrograph at crack initiation on the stable FCP (c) micrograph at the end of the stable FCP; (d) micrograph on the unstable FCP.

Since failure of the mechanical components occurs generally at geometric discontinuities, the damage scenario caused by the progressive stable crack propagation is more representative in this sense than that based on the unstable crack propagation. Therefore, between the three areas described in Figure 6, the area shown in Figure 6b was examined at high magnifications in order to observe the state of the fiber matrix adhesion. Figure 7 shows two fibers (one perpendicular to fracture surface, Figure 7a and the other lying on the fracture surface, Figure 7b) localized near the free surface of the notch. In both cases, fibers are covered by a matrix layer. The microscopic examination suggests that damage involves the matrix rather than the fiber-matrix interface. A thorough examination reveals that the matrix layer is thicker for the fiber perpendicular oriented to the fracture surface than for the fiber lying on the fracture surface. That is due to the fact that the stress field at the fiber-matrix interface is influenced by the local orientation of the fiber respect to the loading direction.
This result is in contrast with [[1], [5]] and in accordance with [[11]]. However, the micrographs shown by Horst [[1]] and Bernasconi [[5]] refer to material in conditioned state. As explained by Horst [[1]], interface can absorb water leading to a loss of the material properties. Instead, Friedrich [[11]], for a short fiber reinforced PET, shows fibers covered by a matrix layer on the fracture surface of compact tension (CT) specimens tested in dry-as-molded conditions. The failure mode of the CT specimens is characterized by the stable crack propagation and it is thus similar to that exhibited by the notched specimens tested in this work.

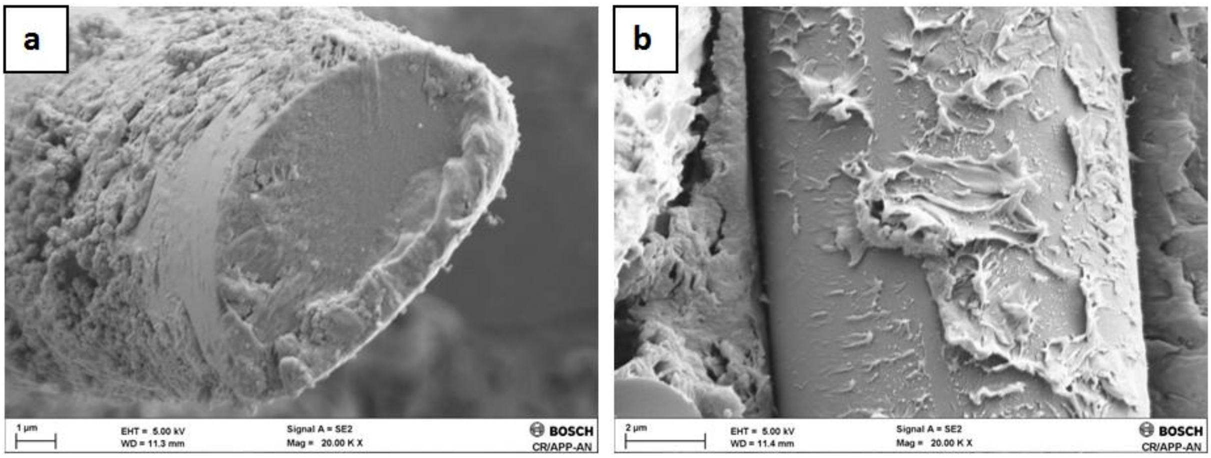

Figure 7 Analysis of the fiber-matrix interface, notched specimens: (a) fiber perpendicular to the fracture surface; (b) fiber lying on the fracture surface.

The analysis of the longitudinal section of the specimens subjected to interrupted fatigue tests (Figure 8) confirms the results of the previous analysis. Figure 8a shows a fiber avoided by the crack but covered by a matrix layer. Figure 8b provides an example of the mechanism “fiber pull out” in which the fiber extracted from the fracture surface is completely covered by a matrix layer indicating that damage occurs in the matrix rather than at the fiber-matrix interface. These results are opposite respect to those shown by Lang et alii [[6]]. In fact, they observe clean fibers clearly debonded from the matrix, concluding that damage propagates directly at the fiber-matrix interface.

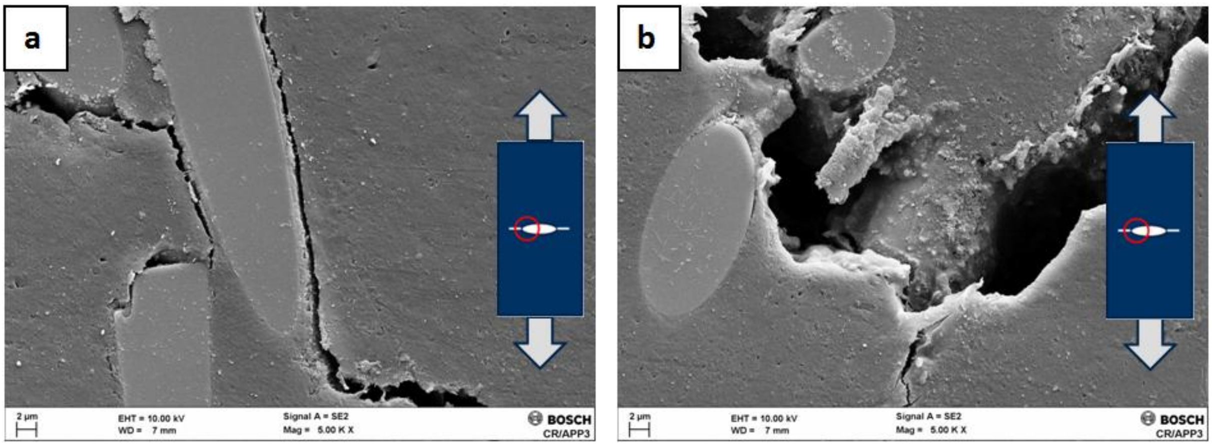

Figure 8 Analysis of the fiber-matrix adhesion on the longitudinal section of notched specimens subjected to interrupted fatigue tests (a) crack in the matrix along a fiber (b) Fiber pulled-out covered by a matrix layer.

4. Conclusions

The use of FESEM enabled an analysis at high magnification of the fracture material morphology of a short glass fiber reinforced polyamide undergoing fatigue loading. The following conclusions can be drawn. 1) The morphology of the fracture surface reflects crack nucleation and propagation mode. Hence, the damage scenario is not the same for plain as well as for notched specimens. 2) The fracture surface morphology of notched specimens at crack initiation does not provide any evidences of large inelastic matrix deformation suggesting no need to develop damage model which takes into account plasticity 3) Both the analysis of the longitudinal section of specimens subjected to interrupted fatigue tests and the analysis of the fracture surfaces reveal that the fibers are mostly covered by a matrix layer indicating that damage is not propagating at the fiber-matrix interface but in the matrix, some micrometers from the interface itself. An extensive comparison of the results shown in this work with those reported in the literature will be presented in a second paper in order to figure out if the damage mechanisms are in some ways dependent on the significant improvements of the fiber-matrix interface properties.

References

[1] J. J. Horst J. L. Spoormaker. Fatigue fracture mechanisms and fractography of short-glass fiber-reinforced polyamide 6. *Journal of Materials Science*, volume(32):3641-3651, 1997.

[2] B. Klimkeit, S. Castagnet, Y. Nadot, A. El Habib, G. Benoit, S. Bergamo, C. Dumas, S. Achard. Fatigue damage mechanisms in short fiber reinforced PBT+PET GF30. *Materials Science and Engineering: A*, volume(528):1577-1588, 2011.

[3] M. De Monte, E. Moosbrugger, K. Jaschek, M. Quaresimin. Multiaxial fatigue of a short glass fibre reinforced polyamide 6.6 - fatigue and fracture behavior. *International Journal of Fatigue*, volume(32):17–28, 2010.

[4] M. De Monte, E. Moosbrugger, M. Quaresimin. Influence of temperature and thickness on the off-axis behaviour of short glass fibre reinforced polyamide 6.6 – cyclic loading. *Composites: Part A*, volume(41):1368-1379, 2010.

[5] A. Bernasconi, P. Davoli, C. Armanni. Fatigue strength of a clutch pedal made of reprocessed short glass fibre reinforced polyamide, *International Journal of Fatigue,* volume(32):100-107, 2010.

[6] R. W. Lang, J. A. Manson, R. W. Hertzberg. Mechanisms of fatigue fracture in short glass fiber-reinforced polymers, *Journal of Material Science,* volume(22):4015-4030, 1987.

[7] J. Karger-Kocsis, K. Friedrich. Fatigue crack propagation in short and long fiber-reinforced injection-molded PA 6.6 composites. *Composites*, volume(19):105-114, 1988.

[8] S. Günzel, S. Hickmann, C. Wittemeyer, V. Trappe. Effects of Fiber Orientation and Moisture on the Crack Growth in Short Glass Fiber Reinforced Polyamide. *Advance Engineering Materials*, Volume(14):867-872, 2012.

[9] J.F. Mandell, D.D. Huang, F.J. McGarry. Crack Propagation Modes in Injection Molded Fiber Reinforced Thermoplastics. In: *Short Fiber Reinforced Composite Materials*, ed. B.A. Sanders ASTM STP 772, Philadelphia, 1982.

[10] J.F. Mandell, F.J. McGarry, D.D. Huang. Fatigue of glass and carbon fiber reinforced engineering thermoplastics, *Polymer Composites*, volume(2):137-144, 1981.

[11] K. Friedrich. Microstructure and fracture mechanical properties of short fiber reinforced thermoplastic P.E.T.. *Colloid and Polymer Science*, volume(259):808-811, 1981.