EFFECT OF MICRO-NOTCH ON THE FATIGUE BEHAVIOUR OF SHORT FIBRE REINFORCED PEEK

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Abstract
In the present work the effect of micro-notch on the fatigue behavior of SFR PEEK was investigated and compared with unreinforced matrix. Three different materials were examined. Uniaxial fatigue tests were carried out on dog-bone specimens either plain or notched with different sizes of holes drilled on the surface of the specimen. Crack propagation from the notch was also investigated carrying out surface investigations during the test and on the fractured surfaces. For all materials the presence of a micro-notch resulted in a decrease of fatigue strength but different fatigue behavior and sensitivity to micro-notch size were observed for neat and SFR PEEK. In particular for SFR PEEK artificial micro-notches were found to have a lower influence on fatigue lifetime compared to the effects of inherent notches located at fiber ends or caused by weak adhesion between additive particles and matrix; fiber reinforcement gave rise to a negative action against crack initiation but a positive action against crack propagation.

1 Introduction
Short fiber reinforced (SFR) PEEK composites are used for safety critical components in the automotive, industrial and biomedical fields. Fatigue failure mechanisms for this class of composite materials are quite complex and not yet completely understood. Effects of processing variables on fatigue in molded PEEK and its short fiber composites were discussed in [1,2], considering fatigue crack propagation (FCP) aspects. The effects of different load ratios and levels, specimen thickness and environmental temperatures were considered in [3-5]. Fatigue behavior of SFR thermoplastics has also been studied by a classical approach based on stress-life curves (S-N). An important aspect of the fatigue behavior of short fiber reinforced PEEK which has received limited attention up to now is represented by the influence of notches. In particular short fibers reinforced composites, contrary to unfilled polymers, are full of impurities and local inherent micro-notches caused by weak adhesion between filler and matrix. As a consequence it is of interest to investigate whether local variations like those induced by scratches, notches, voids or imperfections could have a significant or small influence on fatigue lifetime compared to the effects of inherent notches present in the material. In general for short fiber thermoplastic it has been inferred that the presence of a good correlation between cyclic creep speed and fatigue lifetime could be an indication that variations in lifetimes of the specimens are mainly determined by global parameters, as creep is a phenomena of global character [6]. However direct experimental investigation for SFR-PEEK is in practice limited to rotating-bending fatigue tests on plain

and notched specimens which were carried out by Nisitani et al. [7] to compare notch sensitivity of neat and short carbon-fiber reinforced PEEK. In presence of a small hole (diameter 0.1 mm, depth 0.1mm) neat PEEK was found exhibited a high sensitivity of fatigue strength to a notch and very high fatigue crack growth rate compared to reinforced material.
In the present work the effects of a micro-notch on the fatigue behavior of SFR PEEK were therefore investigated and compared with unreinforced matrix. In particular three different materials were examined: unfilled PEEK and PEEK filled with different weight fraction of carbon short fibers or with the addition of fillers like graphite and PTFE. For each material uniaxial fatigue tests in load control were carried out up to 10^6 cycles on dog-bone specimens either plain or notched. Different sizes of micro-notch were considered by drilling a hole on the surface of the specimen with diameter of 0.4 and 1 mm respectively. Notch sensitivity of different materials was then analyzed and compared considering fatigue lifetime, fracture surfaces and failure mechanism and crack propagations aspects.

2 Materials and testing methods

2.1 Materials and specimens

Three different materials were examined in the present work: unfilled grade PEEK (NEAT), PEEK filled with 10% carbon micro-fibers, graphite and PTFE (CF10-PVX); PEEK filled with 30% carbon micro-fibers (CF30). Neat PEEK is of interest for a variety of applications and was studied as a reference for comparison purposes. CF10-PVX is mainly employed in applications in which tribological aspects are of interest due to action of PTFE and graphite as internal lubricants. CF30 is representative of a class of PEEK composites of potential interest for structural applications. For notched specimens different sizes of micro-notch were obtained by drilling a blind hole on the surface of the specimen having diameters ranging of 0.4 and 1 mm, as schematically shown in Fig. 1, and depth equal to 1.5 diameter value. Some selected specimens were also cut along longitudinal and transverse direction with respect to specimen axis. After polishing, cut surfaces were examined at optical microscope to investigate, at least qualitatively, fiber orientation.
For CF10-PVX, in which a lower amount of fiber is present and fillers were added, the material exhibited a highly heterogeneous microstructure and the distribution of fibers was not found to exhibit a distinct preferred orientation and the material. For CF30 fibers appeared instead to be preferentially aligned with specimen axis. Both CF10-PVX and CF30 fiber distribution in the gauge region of the specimen tested did not present a layered skin-core distribution.

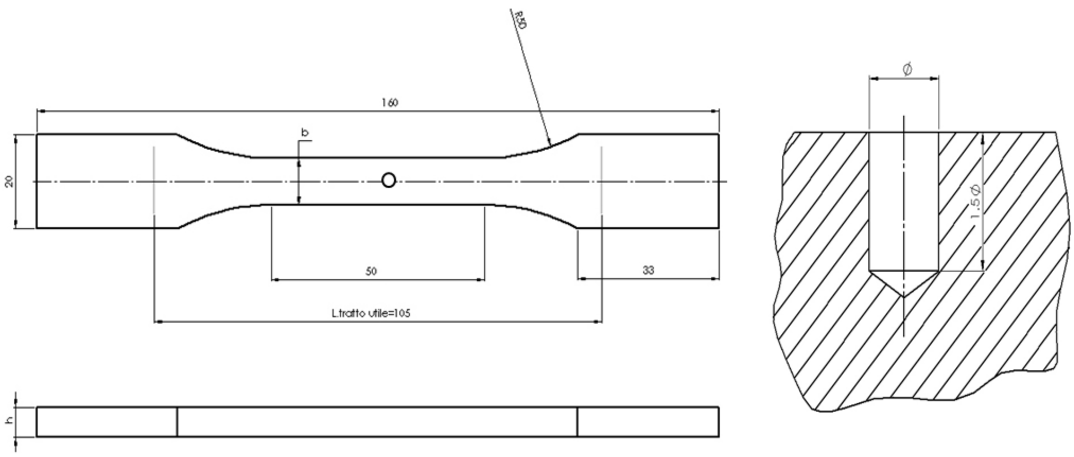


Figure 1. Specimen and micro-notch geometry

2.2 Test procedure

Load-controlled tension-tension fatigue tests were conducted on servo-hydraulic testing machine INSTRON 8501, in the range of cycles to failure from 10^3 to 10^6. Sinusoidal load cycles were applied keeping the load ratio R = (min. load)/(max. load) = 0. The cyclic frequency was set to 10 Hz. For SFR composites the response to cyclic loading is characterized by hysteresis loops resulting in self-heating. During fatigue tests, the temperature on the surface of the specimen near the notch area was monitored with an infrared pyrometer. Crack propagation was monitored using an high speed video-camera, in order to take crack pictures at different instant during the test, without interrupting it. At the end of the test, the fracture surfaces were analyzed by means of optical and electron microscopes (SEM), in order to detect and better investigate the damage mechanisms.

3 Results and discussion

3.1 Fatigue test

Fatigue life curves (S-N) for examined materials are reported in Fig. 2-4, in which maximum applied fatigue stress (σ_{max}) is plotted vs. number of cycles to failure (N_f). Test results include run-out (i.e. test stopped after 10^6 cycles). As a preliminary observation it could be noted that all notched specimen which were brought to failure failed on the section were the hole was drilled, thus indicating that for all materials the presence of a notch has some influence. On the other hand by comparing fatigue life curve and fatigue limit of plain and notched specimens some interesting differences between examined materials can be observed.

Considering neat PEEK (Fig. 2) plain specimens tested at values of maximum applied stress closer to tensile static strength did not reach thermal equilibrium and failed by excessive heating. At lower stress level specimens reached thermal equilibrium with an increase of temperature of 5°C to 20°C with respect to initial room temperature. The presence of a notch resulted in a reduction of the fatigue limit under plain condition of approximately 31% and 37% in presence of 0.4 and 1 mm diameter holes respectively. Results also indicate a change of the slope of the life-curve in the range of stress corresponding to the transition from static stress limit to fatigue limit again more significant with increasing hole diameter.

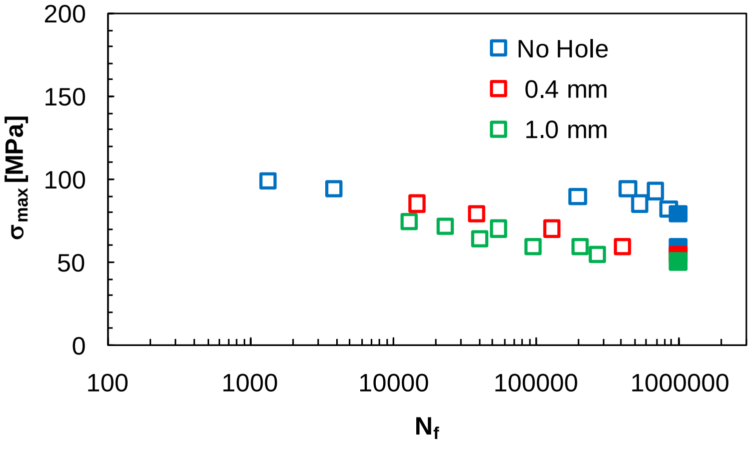


Figure 2. Fatigue life curve for plain and notched neat PEEK

Considering CF30 (Fig. 3) fatigue data for plain specimens confirmed good fatigue properties of SFR PEEK, with CF30 plain specimens exhibiting a higher fatigue strength compared to neat PEEK due to the reinforcing effect of the fibers and being able to withstand 10^6 loading cycles at stress level up to about 60% of static strength. It should be noted that stress-life curve for plain specimens in this case is more scattered. This is possibly due to some slight difference at a microstructural level (and consequent inherent micro-notch distribution) that could be also related to the different sampling positions on the original semi-finished plate from which the specimens were obtained. Data dispersion is lower for notched specimens, suggesting that in this case this effect is somewhat masked by the presence of the hole.

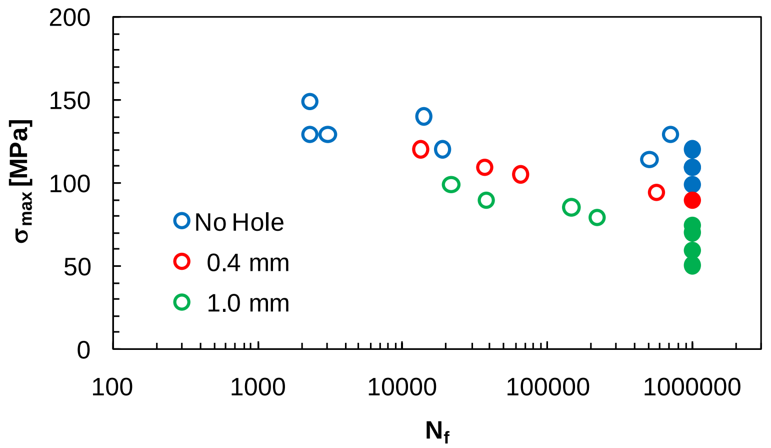


Figure 3. Fatigue life curve for plain and notched CF30

The presence of a notch resulted in a reduction of the fatigue limit under plain condition of approximately 15% and 25% in presence of 0.4 and 1 mm diameter holes respectively. Also in this case a change of the slope of the life-curve in the range of stress corresponding to the transition from static stress limit to fatigue limit can be observed, more significant with increasing hole diameter. Finally, considering CF10-PVX, in which PTFE and graphite were added and a lower amount of fiber is present, a different behavior can be observed, as shown in Fig. 4. Compared to neat PEEK the fatigue behavior of both plain and notched specimen was very different. In particular considering maximum applied stress was about 40% lower than neat PEEK for plain CF10-PVX specimens but they were able to withstand 10^6 loading cycles at stress level up to about 90% of static strength. For this material the addition of particles for lubrication purposes is likely to exert a detrimental effect on static and fatigue strength.

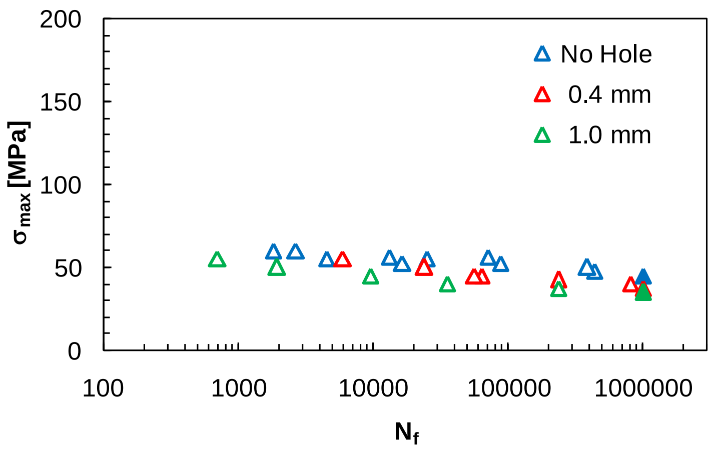


Figure 4. Fatigue life curve for plain and notched PVX

Their effect is preminent with respect to that of reinforcing fibers (which are anyway in lower weight fraction) as they result in inherent defects due to their weak adhesion with matrix. Comparing diagrams in Figs. 3-4 it can be deduced that lower fatigue strength and scatter of PVX is due to the stronger effect and more uniform distribution of such particles-induced defects with respect to the inherent micro-notches present in CF30.

For CF10-PVX the presence of a notch results in a further reduction of the fatigue limit under plain condition of approximately 15% and 20% in presence of 0.4 and 1 mm diameter holes respectively. Overall the observed behavior indicated a high sensitivity of neat PEEK fatigue characteristics to the presence of notches. For CF30 the percentage reduction of fatigue limit from plain to notched condition is lower than for neat PEEK, indicating a somewhat lower influence of the notch on the fatigue behavior. Even lower percentage reduction could be observed in presence of fillers and short fibers for CF10-PVX. These results are in line with literature finding [7].

3.2 Surface investigations

In order to investigate failure mechanisms of the different materials examined, the fracture surfaces of notched specimens were observed both macroscopically both at optical and electronic microscope, as shown in Fig. 5a-c.

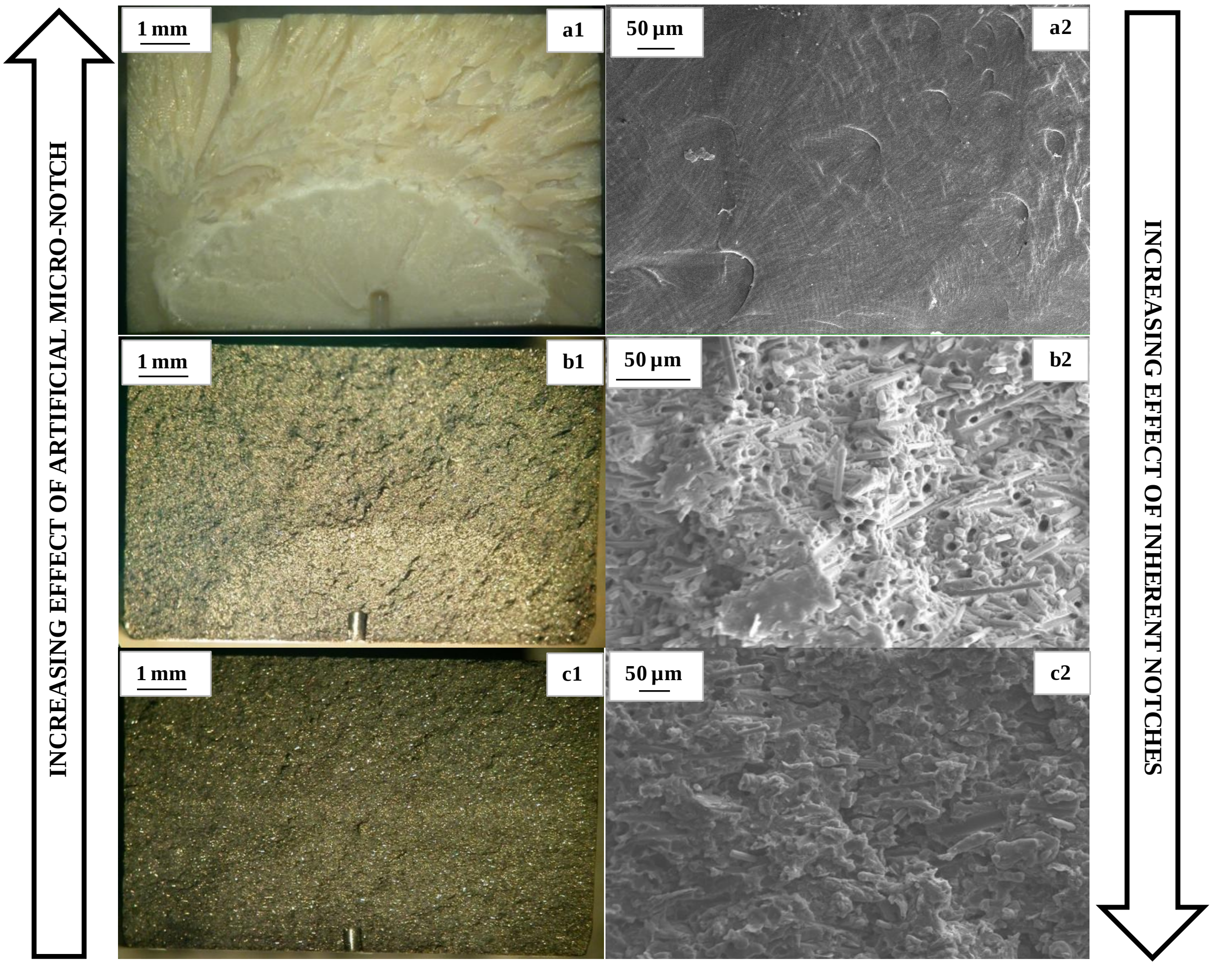


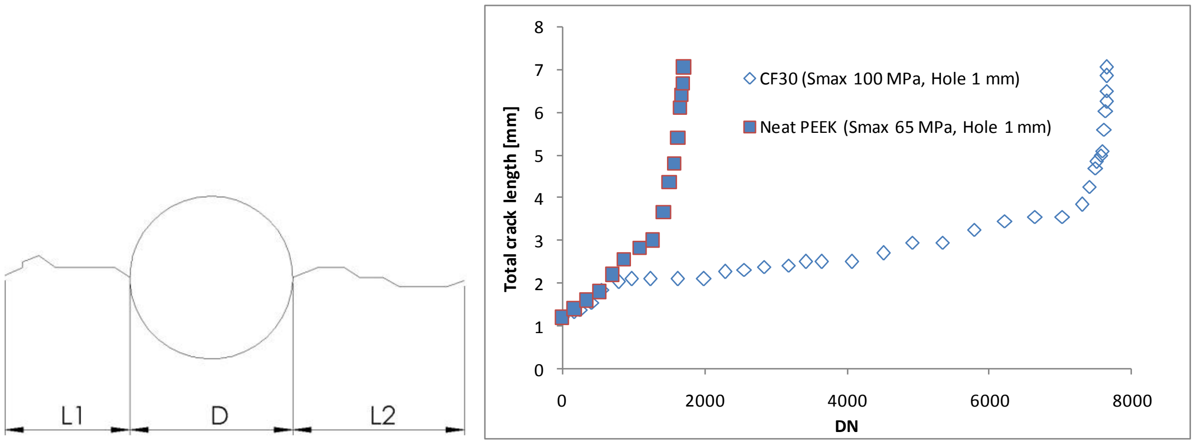
Figure 5. Fatigue fracture surfaces of notched specimens: 1: optical microscope, 2: SEM microscope; (a) Neat PEEK, (b) CF30, (c) CF10-PVX PEEK

At a macroscopical level for neat PEEK the presence of a crack propagation region expanding radially from the notch is clearly noticeable. A more localized SEM analysis revealed the presence of different striation patterns on the fracture surface and of parabolic or dimpled regions which are considered to be formed by the intersection of major crack front with secondary cracks nucleated ahead in the plastic zone. For CF30 at a macroscopical level on the fracture surfaces the presence of two distinct regions can again be noticed, suggesting the presence of a crack propagation area in which more localized SEM investigation showed the presence of different failure mechanism involving fiber pull-out, matrix failure and debonding at the interface between matrix and fibers lying in planes transversal to the loading direction. Finally for CF10-PVX a significantly different fracture surface was observed since at a macroscopical level a crack propagation region could not be clearly identified and plain and notched specimens presented similar aspect. SEM investigation showed the presence of a rather heterogeneous and irregular aspect, with areas in which some of the above mentioned failure mechanisms could be noticed, such as fiber/matrix separations along the interfaces of fibers oriented parallel to the fracture surface, and in some cases fiber breakage and pull-out, as well as cavities and regions with different degrees of matrix deformation. Overall fracture surface investigations combined with results of fatigue tests indicated that the combinations of fillers and short fibers results in a material which is full of impurities and in which fibers and particles can act as preferential crack nucleation points. As a result crack nucleation could be less directly related to the presence of the artificial notch.

3.3 Crack propagation investigations

Fatigue crack propagations aspects for neat and SFR PEEK in presence of notches were first studied by Nisitani et al. [7]. They found that for neat PEEK plain specimens fatigue crack initiation was of the point-initiation type, with a crack propagation life extremely short compared with the total life due to very high crack propagation rate. Fatigue life was controlled by the behavior of a crack initiation and the material exhibited a high sensitivity to a notch. On the contrary for CFR PEEK they found that fatigue crack initiated from near the fiber ends due to stress concentration effects but crack growth rate was much less (up to two orders of magnitude in the case of CF30) than that in the neat matrix [2,5,7,8]. They concluded that fiber reinforcement gave rise to a negative action against crack initiation, since micro-fibers and particles can act as preferential crack nucleation points, and a positive action against crack propagation, since micro-fibers oppose crack propagation by dissipating energy in different ways (pull-out, fracture and debonding).

In order to investigate these aspects on the examined materials, the crack propagation phase was monitored, as explained in section 2.2, considering the total length (hole + lateral cracks) as observed on the surface of the notched specimen (Fig. 6a).



(a) (b)

Figure 6. (a) Scheme adopted for total crack length evaluation, (b) Example of fatigue crack propagation of notched neat PEEK and CF30 PEEK specimens

As an example of the behavior observed experimentally the total crack length during crack propagation phase before final failure is reported in Fig. 6b, where ΔN is the number of cycles starting from the moment the crack was detected on the surface of the specimens (i.e about

38000 cycles for neat PEEK and 14.000 cycles for CF30). Despite the fact that tests refer to different loading conditions, with higher applied maximum stress for CF30 PEEK, by estimating crack growth rate on can observe that the short fiber reinforced PEEK exhibited a significantly lower crack growth rate ($\Delta a/\Delta n$ = 0,3 μm/cycle) compared to neat PEEK ($\Delta a/\Delta n$ = 1,5 μm/cycle). For PVX crack growth rate could be evaluated only for a test at 50 MPa, with a 0.4 mm hole diameter. In that case a crack growth rate of about 0.06 μm/cycle was found. Although these results seem in line with the interpretation previously discussed the experimental data available are unfortunately too limited. Overall these results substantially confirmed that for neat PEEK most of the fatigue lifetime is spent for crack nucleation but after crack initiation propagation is very fast. On the contrary, for the reinforced material, filling fibers or particles could be in general considered as "weak points" regarding nucleation (see for example Fig.7) but they also act as obstacles to crack propagation, so that after crack initiation failure is more progressive.

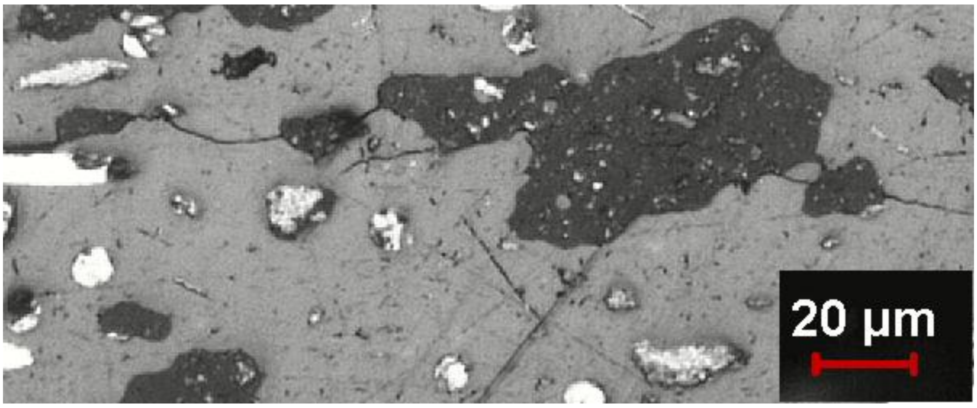


Figure 7. Micro-cracking at the interface between filler particles and matrix in CF10-PVX PEEK

As a final consideration, following previous reasoning, the fatigue limit of SFR PEEK could be possibly regarded as the non-propagation threshold of inherent or early formed micro-cracks generated by the mismatch between filler and matrix, having approximately the same size of that typical of the filler from which they originate. Under this assumption, as an interesting future development, a sort of transposition of the Murakami's approach [9] proposed for metallic material could be attempted also for these materials and in particular experimental testing in presence of even smaller hole diameters could help evaluation of ΔK threshold and the presence of short crack effects for these materials.

4 Conclusions

Different fatigue behavior and sensitivity to micro-notch size were observed for neat and SFR PEEK and depending on the kind of reinforcing phase. In particular for short fiber reinforced composites the fatigue behavior observed experimentally was significantly influenced by the presence of notches but notch sensitivity decreased as the size of the notch decreased. Overall results indicate that since SFR composites, contrary to unfilled polymers, are full of impurities and local notches due to the presence of fibers, local variations (scratches, notches, voids, imperfections) could have a small influence on fatigue lifetime compared to the effects of collective notches (fiber ends or additive particles) present in the material and fiber reinforcement gave rise to a negative action against crack initiation but a positive action against crack propagation.

References

[1] Saib K.S., Isaac D.H., Evans W.J., Effects of Processing Variables on Fatigue in Molded PEEK and Its Short Fiber Composites, *Materials and Manufacturing Processes*, **9(5)**, pp. 829–850 (1994)
[2] Saib K.S., Isaac D.H., Evans W.J., The effect of short carbon fiber reinforcement on fatigue crack growth in PEEK, *Composites Part A*, **27A**, pp.547–554 (1996)
[3] Brillhart M., Fatigue fracture behavior of PEEK, *Journal of Reinforced Plastics and Composites*, **12**, pp. 943-950 (1993)
[4] Brillhart, M., Gregory B. L., Botsis J., Fatigue Fracture Behaviour of PEEK. I. Effects of Load Level, *Polymer*, **32(9)**, pp.1605-1611, (1991)
[5] Brillhart M., Botsis J., Fatigue Fracture Behaviour of PEEK. II. Effects of Thickness and Temperature, Polymer, 33(24):5225-5232 (1992)
[6] Horst J.J., Spoormaker J.L., Fatigue fracture mechanisms and fractography of short-glassfiber-reinforced polyamide 6, *Journal of Materials Science*, **32**, pp.3641-3651 (1997)
[7] Nisitani H., Noguchi H., Kim Y.H., Evaluation of fatigue strength of plain and notched specimens of short carbon-fiber reinforced polyetheretherketone in comparison with polyetheretherketone, Engineering Fracture Mechanics,
[8] Friedrich K., Wlater R., Voss H., Karger-Kocsis J., Effect of short fiber reinforcement on the fatigue crack propagation and fracture of PEEK-matrix composites, *Composites*, **17**, pp. 205-216 (1986)
[9] Murakami Y. Metal fatigue: Effects of small defects and nonmetallic inclusions, Elsevier, Oxford (2003)