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EFFECT OF HIGH ENERGY RADIATION ON TECHNICAL POLYMERS

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1. ABSTRACT

Fiber reinforced polymers (FRP) offer excellent fatigue resistance in combination with low specific weight. However, in contrast to metals, microscopic damage is usually present right from the beginning of cyclic loading and calls for insights on initiation and propagation. As with other properties, the overall performance of a composite is determined by its constituents namely fiber, matrix and fiber-matrix interface. Especially, the matrix polymer is supposedly responsible for damage initiation. To study this, different methods for altering the matrix properties can be used. The presented research makes use of Co-60 irradiation to modify the polymer on a molecular level. Six potential candidate materials for FRP are investigated, two thermoplastics and four thermosets. After irradiating the neat polymer samples with (30, 100, 200 and 500 kGy) the properties are investigated by quasi-static tensile tests and fatigue experiments. The resulting fractures are inspected visually. The results for thermoplastic materials show deteriorating tensile properties, whereas some of the thermosetting resins improve. Despite this, it could also be shown that deterioration in monotonic loading does not necessarily deteriorate the fatigue properties. The fracture surfaces indicate yielding as possible cause. Finally, it was shown that irradiation is a promising modification method for the investigation of composites and possible also a method to improve the fatigue performance.

Keywords: Fatigue, Irradiation, Fractography, Epoxy resin, Polycarbonate, Cyanate Ester

2. INTRODUCTION

Fiber reinforced materials can be found in a wide variety of applications, due to their high specific strength and stiffness. In order to achieve the best possible performance, the composite's constituents namely fiber, matrix and fiber-matrix interface need to perform their respective functions in a self-reinforcing way. Cyclic loading conditions make this interaction especially important, because every constituent has its own individual S-N curve, degradation and mechanical property, which are interacting and form the laminate's S-N curve. An important example for this interaction can be found in the damage progression of on-axis loading of composites with a unidirectional fiber reinforcement. Composites with this type of reinforcement show a shallow S-N curve with limited degradation [1]. Degradation occurs in two stages, the one predominantly controlling fatigue life is mainly controlled by matrix properties and the fiber/matrix interface [2–5], even though fibers carry most of the load. Initially damage is induced at some weaker fiber segments or flaws and result in fiber breaks at the beginning of fatigue loading or within the first load cycle [4, 5]. From there on the majority of the laminate's lifetime is defined by matrix and fiber/matrix interface properties [6], which dictates either of two modes for damage propagation, debond growth or matrix cracks transverse to the fibers with more or less fiber bridging [7–9]. As load distributes to other fibers further fiber breaks occur and the described damage cycle repeats itself [4]. This example shows that even though on-axis fatigue loading results in the best achievable fatigue performance for

a given composite [10] it is the matrix and fiber/matrix interface that dictate the performance. This is even more true for multidirectional laminates [10] or any laminate containing inhomogeneity like stitches or stabilizing yarns. As shown by Zangenberg et. al. [11] and others [12–15] even minor transverse stitching or backing fibers can deteriorate the fatigue performance and act as damage initiation sites. Therefore, nearly all practical lay-ups suffer fatigue degradation driven by matrix cracking or fiber/matrix debonding. Both failure modes are reported to depend largely on matrix fracture toughness and fiber/matrix adhesion. Only a small number of studies investigated a possible correlation between constituent properties and the composite's fatigue performance.

One investigation was conducted by Shao et. al. [16], who studied the effect of fracture toughness and fiber/matrix adhesion. They focused on on-axis fatigue loading of glass-fiber reinforced vinyl ester resin. By chemical modification of the resin, toughness and fiber/matrix adhesion were modified. Their findings show that a tougher matrix in combination with good fiber/matrix adhesion can slow down damage initiation and growth. However, both factors were always affected simultaneously by the chemical modification, which impedes observing the effects independently. Other investigations compared mainly thermosetting resins with thermoplastic matrix materials [8, 17, 18]. From these works, it was similarly found that increasing matrix fracture toughness in combination with different fiber-matrix adhesion affects initial damage growth as well as stage three of damage evolution [17]. In the final stage of degradation, typically two types of failure can be observed sudden death with no further stiffness loss or renewed degradation. Another approach pursued for the investigation of metal matrix composites is testing at different temperatures [19]. Despite the general modification concept, a transfer of the results to polymeric composites is limited. Vieille et. al. [20] also made use of temperature induced property changes at the glass transition temperature of polymers. Their results show that matrix ductility could be the main reason for differences in crack formation. Another approach mainly targeted at a changing fracture toughness are hierarchical composites. Additional particles are introduced on the sub-fiber level and can act as crack arresting inhomogeneity [21, 22].

Modifying the matrix polymer is crucial for a study of its role in the fatigue damage evolution of composites. The modification methods can be classified into the following categories:

- exchanging the matrix
- exploring different thermal sensitivity of the matrix and fiber properties
- introducing another phase, which effectively forms a hierarchical composite
- altering the macromolecular structure

Each of the above strategies has its own challenges with respect to identifying the influence the matrix has on the fatigue properties. Without being exhaustive, some aspects are discussed here to show the necessity for further research. Exchanging a brittle thermosetting resin with a ductile thermoplastic matrix is at first glance the most obvious approach. Despite drastically different matrix properties, many other important aspects are also altered. This impedes a direct comparison of resulting damage initiation, because thermoplastic composites differ often in mesoscopic structure from thermosetting composites. As shown by Brunbauer et. al. [23] geometric parameters like stacking and fiber volume content can affect damage propagation, too. Even if two thermosetting resins are compared differences can be introduced by slightly different processing parameters. As shown by the comparison of Sjögren and Berglund [24] crack densities evolve differently but the pristine state in terms of manufacturing defects is also different.

The second approach relies on differences in thermal sensitivities of the constituent properties. Usually the matrix material is more sensitive to elevated temperatures, which can affect also

the cyclic response [19, 20]. However, increasing the temperature also affects residual stresses, which in effect changes the local mean stress [25].
Hierarchical composites introduce new possibilities but also a new set of processing differences. One common method is the use of particles (rubber/silica) or carbon nano-tubes [26]. Both sub-fiber constituents are interesting as toughness and stiffness can be targeted [21, 22]. However, a homogeneous dispersion is challenging especially for high particle contents, due to self-filtering and agglomeration [27]. Although improvements both on matrix and composite level are possible, porosity and additional flaws remain an issue [22].
Finally, it is possible to change the molecular structure of the resin or thermoplastic polymer used. Several studies focused on the effect fracture toughness has and used different resin formulations based on epoxy, vinyl ester or polyester for this purpose. The main benefit of this strategy is that in keeping the base resin constant the manufacture process can also remain similar, excluding geometric differences between the laminates. Furthermore, thermal expansion and therewith thermally introduced residual stresses remain comparable. All known studies pursuing this, used thermosetting resins. Another approach to modify the macromolecular structure is by high-energy radiation. The main benefit of this type of modification is the possibility to introduce the macromolecular change after impregnation or curing. A major challenge addressed with this research is that only some polymers are viable to changes without additional modifiers like crosslinking agents. The majority of polymers modified by high-energy radiation is not usable as matrix material in composites and falls within the class of commodity polymers [28]. In addition, it is hard to predict, which kind of change a polymer will undergo namely crosslinking or chain scissoring. From an extensive study on available data on technical polymers six candidate materials are chosen, which are characterized before and after treatment with different doses. The aim of the presented work is not the improvement of fatigue behavior, but identification of materials, which are applicable as matrix material and undergo significant changes in fatigue and related mechanical parameters.

3. MATERIALS AND METHODS

In total six potentially modifiable polymers have been selected, mainly based on the reported changes, which can be introduced. The different morphological structure of thermoplastic and thermosetting polymers is also considered in this selection. Two thermoplastic candidates are polycarbonate (PC) and thermoplastic polyurethane (TPU). In case of thermoplastic materials, it was also of concern if these are available as fiber reinforced tape material. From the literature it is expected that polycarbonate will suffer mainly form chain scissoring [29] but might show improved fracture strains [30]. Especially at small doses of 30 kGy crosslinking is reported to dominate [30, 31], whereas at higher doses chain scissoring is prevailing with accompanied embrittlement [32]. However, in some studies no initial crosslinking was found [29, 33]. Ether based thermoplastic polyurethane is potentially able to produce crosslinking, due to the chemistry of the soft segment. In order to characterize neat polymer samples, tensile specimens according to DIN EN ISO 527-2 1BA are injection molded according to the recommended injection parameters.
In addition, a bisphenol-a based epoxy resin (RIM135/RIMH137) is used and cured at 80°C for 4 hours. This type of epoxy typically degrades under irradiation [34–36]. Although alternatives based on bisphenol-F might improve under irradiation [37] the known embrittlement of RIM135/RIMH137 made this commercial system especially interesting, because of the significance of toughness in the fatigue of composites. Cyanate ester resins are a well-studied group of thermosetting resins, due their application in fusion/fission reactors or particle accelerators [38]. Cyanate ester resins are high temperature thermosets, which tend to be very

brittle without modifiers and therefore usually contain modifiers [39]. Schönbacher et. al. [40] reported drastically improved properties in terms of strength. In order to repeat the results three similar cyanate ester resins are chosen for this work including one very similar to Arocy B 10 reported by Schönbacher et. al. Details given by Hamerton [39] identified this cyanate ester resin to be based on bisphenol-A, therefore the system is henceforth referred to CE-BPA. In addition, two modified versions are investigated referred to as CE-M1 and CE-M2. First trials especially with the CE-BPA system showed that it is not possible to produce tensile specimens in specimen shaped molds. One possible reason is that during cure and subsequent cooling, stresses introduced by different thermal expansion of mold and specimen lead to cracking of the dumbbell shaped specimens. In order to use the same specimen geometry throughout this research, thermosetting resins are first cured as plate and specimens are cut by CNC milling. To avoid cracking due to a too large plate size only small plates are used for cyanate esters. Table 1 summarizes all tested materials and processing parameters.

Table 1 Material overview and preparation

MATERIAL	CURE/INJECTION MOLDING	POST-CURE	SPECIMEN PREPARATION
Polycarbonate (PC)	280 °C	-	Injection molding to final shape
Thermoplastic polyurethane (TPU)	230 °C	-	Injection molding to final shape
Cyanate ester resin similar to Arocy B10 CE-BPA	1 h @ 150 °C 3 h @ 200 °C	1 h @ 260 °C	Milling from plates 200x100x2 mm³
Modified cyanate ester CE-M1	3 h @ 175 °C 2 h @ 220 °C	1 h @ 260 °C	Milling from plates 200x100x2 mm³
Modified cyanate ester CE-M2	1 h @ 150 °C 3 h @ 200 °C	1 h @ 260 °C	Milling from plates 200x100x2 mm³
Epoxy resin (EP)	4 h @ 80 °C	-	Milling from RTM plate 500x400x2 mm³

All specimens were sealed in vacuum bags, which were themselves placed in airtight casings filled with Argon, in order to avoid any contamination with surrounding air also in case of deterioration of the vacuum sealing. The samples were then irradiated with approximately 3-5 kGy/h up to exposures of 30, 100, 200 and 500kGy. Thermosetting resins were irradiated with higher doses, whereas for thermoplastic materials the focus was on lower doses (see Figure 1 for details).

The quasi-static tensile tests were done according to DIN EN ISO 527-2 with slight deviations with regard to the crosshead speed, which are described later. The tests were carried out on a Zwick RetroLine 10kN testing machine equipped with a KAP-Z force sensor of class 0.05. Strains are recorded by digital image correlation with a high-resolution single camera set-up with a frame rate of 1 fps for 600 frames (completion of yielding). For synchronization purposes both the video signal and the force recording start at a preload of 5 N. The specimens are loaded with a strain rate of 1 %/min, which corresponds to 0.6 mm/min with regard to the free clamping length of 58 mm. In order to limit the total testing time, the standard allows switching the testing speed after recording the elastic modulus. However, for this characterization campaign the testing speed was increased only after necking occurs. Preliminary tests show that neck formation is completed after approximately 8 % longitudinal strain. Therefore, the testing speed was increased to 15 mm/min after reaching 8 %. Strains are evaluated by digital image correlation until yielding occurs. For strain at break the crosshead displacement is used. Each batch for quasi-static testing consisted of at least six specimens, but only failure outside the clamps was taken as valid test result.

Based on the quasi-static testing results three materials are selected for fatigue testing. These materials are then tested in a one-stage stress controlled fatigue experiments. A testing frequency of 2 Hz and a sinusoidal waveform is chosen for this purpose. In order to exclude creep a loading ratio of R = -1 is chosen were possible. Due to the high strength in combination with a slender specimen geometry (also 1BA DIN EN ISO 527-2), this approach could not be sustained for cyanate esters. In this case R = 0.1 is used. Despite this, it can be expected that creep is not relevant for this material, due to a high glass transition temperature. All tests are performed at room temperature.
Finally, the fracture surfaces are analyzed in order to study crack initiation, propagation and final fracture for both fatigue and quasi-static specimens. In order to get a thorough understanding all fracture surfaces are investigated and the most representative ones are reported here.

4. RESULTS

4.1 Visual Changes

After irradiating the specimens, PC, TPU and EP samples show color changes. The color changed from transparent, to greenish-yellow, orange and to dark brown for PC, TPU and EP respectively (see Figure 1). In contrast, all cyanate ester resins remained unaffected. A color change is a clear indicator for changes in the molecular structure of the polymers.

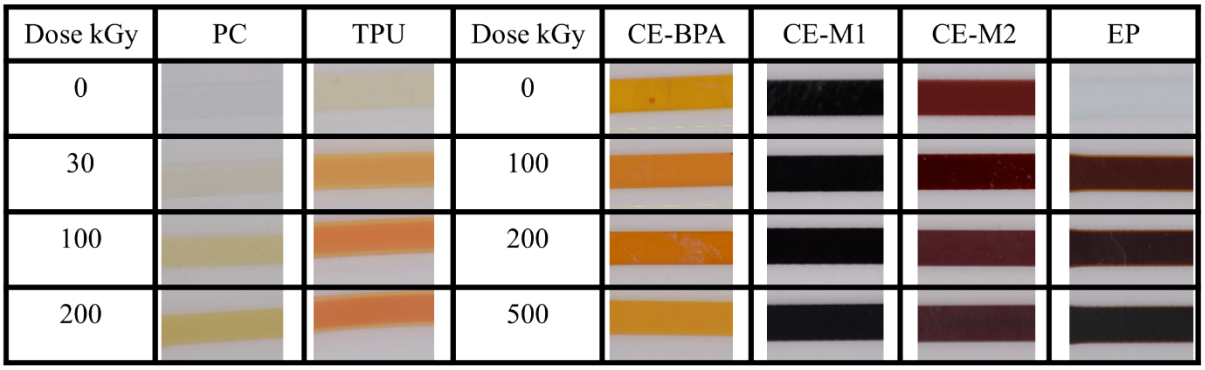

Figure 1 Induced color changes after sample irradiation and chosen irradiation doses for different materials

By visual inspection of the samples after irradiation exposure, it became clear that TPU warped under the influence of irradiation (see Figure 2). Warping occurs irregularly and sometimes lead to bend or twisted specimens. Currently it remains unclear if this is a result of the released residual stresses or uneven shrinkage, due to irradiation. Warping poses a problem for the optical evaluation of strains in a single camera set-up. Estimating the error induced by warping is hard to estimate for the irregular shaped specimens and therefore crosshead displacement is used as strain channel for TPU. Warping suggests also inhomogeneity in the TPU samples.

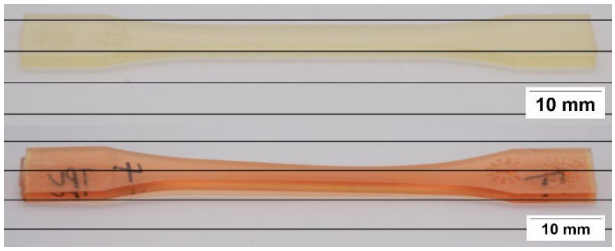

Figure 2 TPU before (top) and after (bottom) irradiation treatment

4.2 Quasi-static testing

The evaluation of the quasi-static testing results focuses on the stress-strain behavior before necking especially for the thermoplastic polymers, since excessive necking is not possible when the polymer is part of a composite. Necking was present for all thermoplastics and all doses. The corresponding yield stress increases slightly for TPU at low dose exposure of 30 kGy but declines for higher dose settings (see Figure 3). In general, TPU seems relatively stable for the applied doses. Due to excessive necking, failure strain for the thermoplastic materials is less meaningful and doesn't change much. Epoxy resin also shows necking for all doses investigated. The mean stress-strain curves shown in Figure 4 all show plastic deformation and yielding for this material. An initial decrease in yield stress is reversed for the highest dose of 500 kGy. This increase in yield stress is accompanied by a decreasing failure strain, which reduces from 6.8 % to 5.4 % from 0 and 500 kGy respectively. This inverse correlation could also be observed for 100 kGy. This dose shows the lowest yield stress with 57 MPa but the highest failure strain with 7.6 %. However, EP samples exposed to 200 kGy deviate from this trend with 56 MPa and 5.4 %.

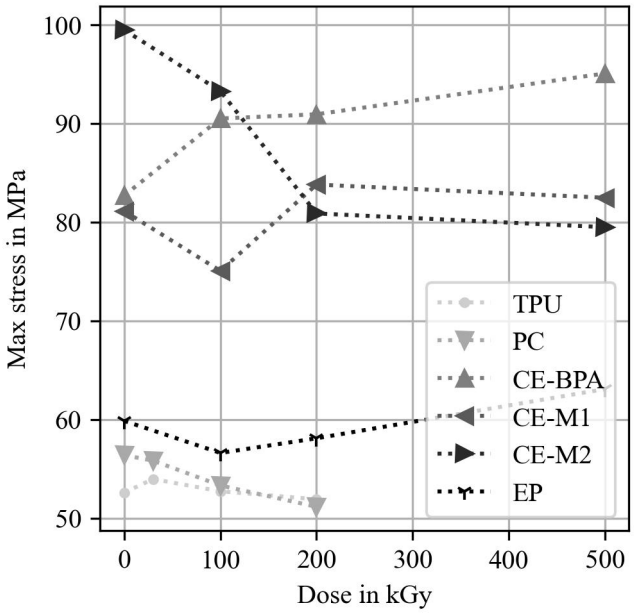

Figure 3 Failure/Yield stress

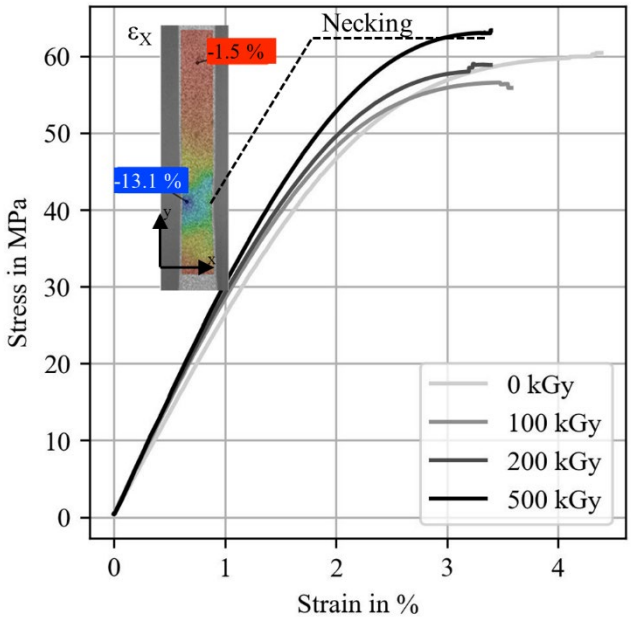

Figure 4 Effect of irradiation on the mean stress-strain curve of EP; Detail indicates neck formation by transverse strains for 500 kGy sample

In contrast to this all cyanate ester resins fail brittle at much higher loads, without yielding. The brittle nature of all cyanate ester resins leads to comparably high property scatter. Figure 3 shows the maximum stress and Figure 5 the corresponding 95 % confidence interval normalized by the mean. The low confidence in the results of all cyanate esters irradiated with 100 kGy can be attributed to a decreased number of valid results. Especially for this dosage, many specimens failed inside the grips and therefore the number of valid results is lower. This in turn decreases confidence into these results. Despite in general higher scatter for cyanate esters a tendency for improvement in terms of strength can be observed for CE-BPA, whereas CE-M1 and CE-M2 deteriorate under irradiation. The increase in strength for CE-BPA is accompanied by less brittle failure modes. This is based on the observation that non-irradiated samples fracture into multiple pieces and it is impossible without high-speed recordings to evaluate where fracture initiated. For higher doses, this changed to a single crack with no secondary damage visible. CE-M2 showed the inverse changes going from ductile to brittle because of irradiation.

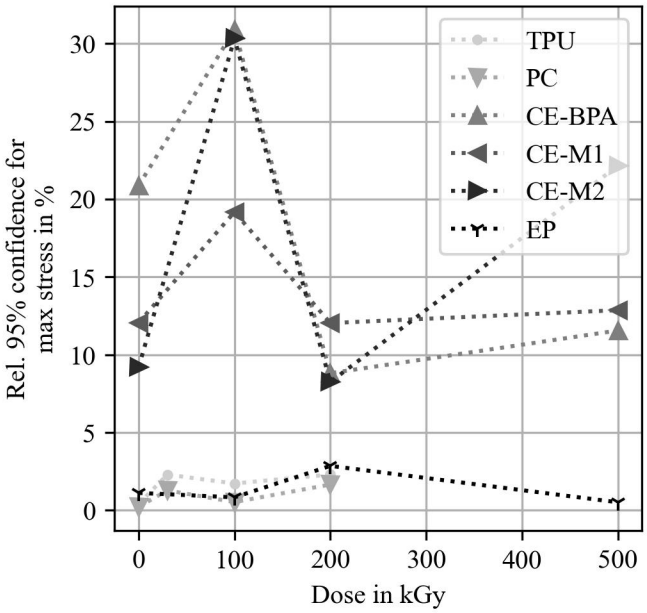

Figure 5 95% Confidence interval for maximum stress

In terms of tensile modulus, significant changes are only visible for epoxy. The tensile modulus increases mainly from 0 kGy to 100 kGy. For Polycarbonate the tensile modulus remains almost constant.

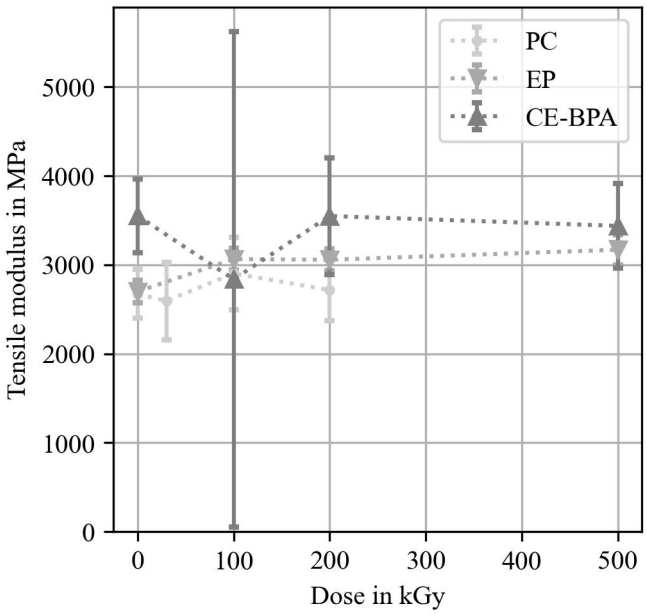

Figure 6 Tensile modulus for three materials chosen for fatigue characterization; Error bars show the 95% confidence interval

4.3 Fatigue

The tensile test results suggested PC, EP and CE-BPA to be the most promising candidate materials for later use in the modification of composites. Even though, CE-M2 changed also significantly the drastic embrittlement make it unlikely that valid fatigue tests can be performed and it is therefore not considered further. Table 1 summarizes the regression results for all three materials. CE-BPA was tested with a loading ratio of 0.1, due to the expected sensitivity for notches and especially any anti-buckling device. It can be noted that the slope of all S-N curves decreases as a result of irradiation treatment. This effect is especially prominent for

Polycarbonate, where the slope halves. The resulting S-N curve is shown in Figure 7. Similarly, EP shows a lower deterioration, due to fatigue loading. In case of CE-BPA scattering is a particular problem. The main reason for this is that most specimens fracture at the clamps. This type of failure is excluded from the PC and EP testing series, but could not for CE-BPA in order to get enough data points for an attempted regression. The bracketed values for CE-BPA in Table 1 show all samples used for this type of regression. Despite less stringent criteria for valid specimen failure, a regression is hardly possible. Notwithstanding this it can be stated that the slope is expected to be low for CE-BPA and the overall S-N curve might shift to a higher stress level if clamp breakage could be avoided. The higher ductility after exposure to 500 kGy from quasi-static tests did not lead to drastic differences for CE-BPA.

*Table 2 S-N regression results; *only estimates, due to much scattering*

Material	Dose in kGy	Slope	Stress in MPa at 5×10^5 cycles	Squared error	R	valid samples
EP	0	-0.05	25.8	0.40	-1	13
EP	500	-0.04	26.3	0.50	-1	12
PC	0	-0.28	10.4	0.68	-1	33
PC	200	-0.14	8.3	0.68	-1	12
CE-BPA	0	-0.02*	20.3*	0.18	0.1	4 (9)
CE-BPA	500	-0.01*	21.2*	0.38	0.1	4 (11)

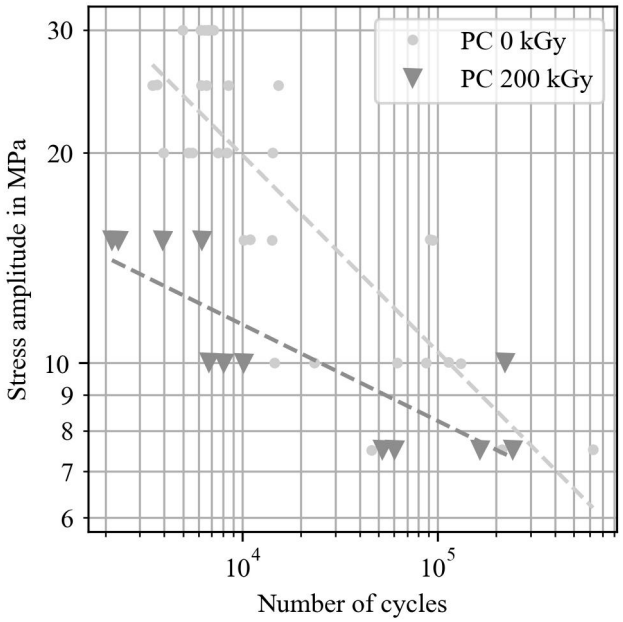

Figure 7 S-N curves of Polycarbonate for dose 200 kGy and as-molded

4.4 Fracture surfaces

Reasons for a different fatigue performance of the irradiated and the non-irradiated samples can be found in the microscopic analysis of the fracture surfaces. Due to large scattering for CE-BPA this analysis focuses on PC and EP. Figure 8 shows the fracture surfaces in irradiated (PC 200 kGy and EP 500kGy) and non-irradiated configuration. For non-irradiated samples it can be seen that yielding is present either at crack initiation or in case of PC near final fracture (see direction of crack propagation in Figure 8). In contrast to this, large scale yielding is not present

after irradiation treatment. Progression marks can be observed especially for PC in both states. However, the spacing between striations is bigger for non-irradiated samples. The non-irradiated EP samples on the other hand show a yield surface at the beginning of the crack, which is then followed by a small region with progression marks (PM in Figure 8). This small area of presumably stable crack growth transitions quickly to unstable cracking with branching. In contrast to this, the region with progression marks is much bigger for the irradiated samples (see Figure 8) and takes up a large fraction of the total surface. Where there are no progression marks visible for the irradiated sample crack blunts can be identified just before and inside the area marked by striations. Crack blunting is associated with slower crack growth, because the crack is arrested and renewed initiation of a sharp crack tip is necessary for crack propagation [41].

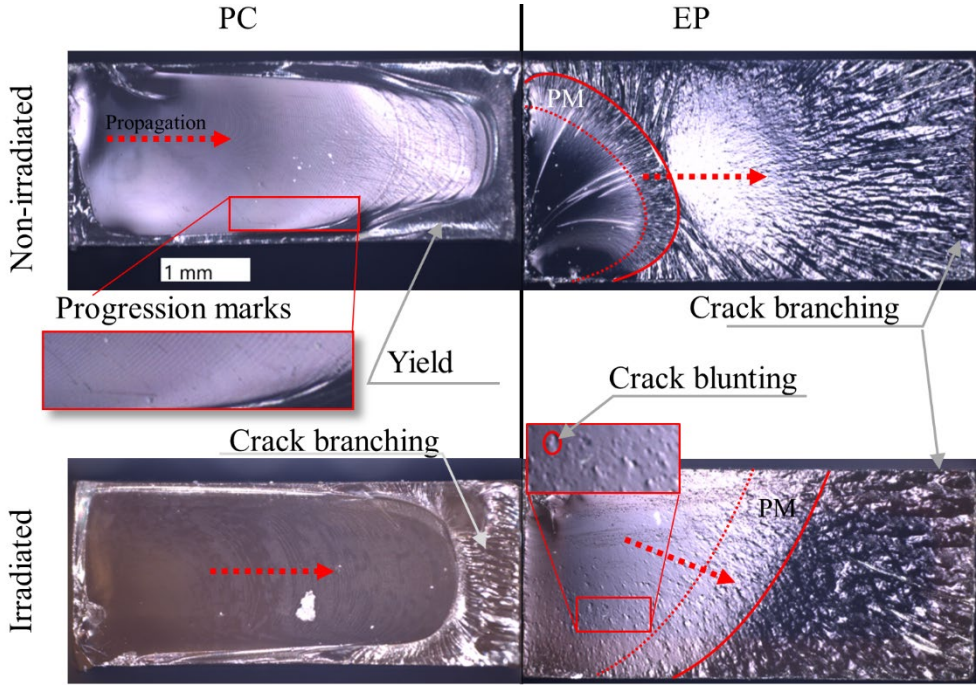

Figure 8 Fracture surfaces of the irradiated (PC 200 kGy/ EP 500 kGy) and non-irradiated samples after fatigue loading; Red lines for EP mark an area with identifiable crack arresting lines

Due to limited necking for the epoxy specimens, it is also possible to investigate the role of plasticity and yielding in quasi-static fracture. Figure 9 shows two representative fracture surfaces of epoxy samples. The shown fracture surfaces are mainly oriented normal to the loading direction. Parallel lines visible in both fracture surfaces are therefore likely shear bands angled to this plane. A small area limited by a round transition zone is present in both fracture surfaces and could mark a reorientation of the shear bands for the 0 kGy configuration. In case of the 500 kGy samples a similarly shaped transition zone marks a zone with rapid crack growth and crack branching [42]. As shown by Cantwell et. al. [42] a similar transition takes place for tensile testing of epoxy at different temperatures. The 0 kGy fracture surface has similarities to the high temperature fracture surface (105°C), whereas the 500 kGy corresponds to an intermediate temperature (85°C). This surface features both a smooth and a rough area.

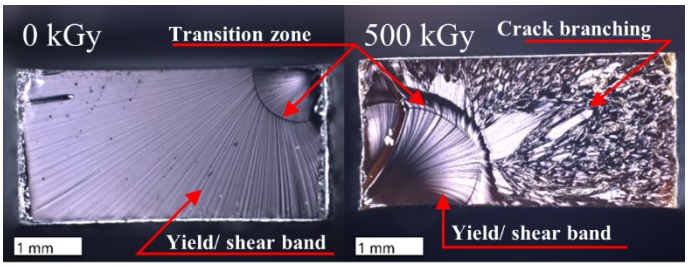

Figure 9 Quasi-static fracture surfaces of epoxy samples for 0 and 500 kGy

5. DISCUSSION

The results show that irradiation treatment mostly deteriorates quasi-static properties like yield or maximum stress. In the case of TPU inhomogeneity can arise and become an issue in a composite. It would be problematic if warping is the result of non-uniform volume changes instead of freed residual stresses. This along with other induced changes are hard to predict and seem to be very specific for a given polymeric system, as many of the reported changes were not or only partially present after irradiation treatment. For example, an increase in fracture strain for PC could not be observed, mainly because neck formation took place until it was restrained by the clamps. Instead of deteriorating properties of bisphenol-A based epoxy an increase in fatigue resistance and tensile strength was observed. Only for the bisphenol-A based cyanate (CE-BPA) ester resin the reported increase in strength was confirmed. Modifiers on the other hand can prohibit those changes.

Especially polycarbonate featured a contradiction, when quasi-static results and fatigue performance are compared. This is because tensile properties suggest a deterioration whereas the S-N curve shows an improved fatigue performance. However, taking the fracture surfaces of both EP and PC into account crack branching, crack blunting and shear band formation are affecting the fatigue performance. For PC specimens, the formation of crazes and shear bands interact as could be seen from the differently spaced crack progression marks for both configurations. This effect is termed shear-craze competition [43]. According to Takemori [44] each progression mark corresponds to a shear band arresting a crack travelling through a craze. The steady crack propagation starts with a craze degrading during cyclic loading until crack propagation becomes possible. The crack then travels through the crazed polymer until it is arrested by shear bands forming near the former craze tip. This reduces the local overstress and stops the crack until a newly formed craze is deteriorated enough to permit further propagation. With this explanation in mind, the molecular morphology after irradiation treatment permits only shorter distances for crack propagation in PC and hence slows fatigue deterioration. A decreasing yield stress is therefore not necessarily contradicting a better fatigue performance, because shear band arresting of the crack could be more likely.

On the other hand, this explanation cannot be assumed for epoxy resin, because yield stress and fatigue performance increased simultaneously as a result of irradiation. In any case this is not surprising because craze formation is unlikely for epoxy resins [45]. The increased yield stress for this material could be made responsible for a later onset of fatigue crack growth. If crazes are not present in this material localized yielding near an inhomogeneity might be the crack initiator. In addition to a retarded crack onset, it also travels slower, due to the formation of several blunts until unstable cracking sets in.

6. CONCLUSION

The results show that by irradiation treatment a significant modification of technical polymers without any modifiers is possible. Furthermore, the comparison between quasi-static loading

and fatigue loading show that deteriorating properties like for example yield stress not necessarily correlate with the fatigue performance of a polymer. This is a new aspect with respect to technical applications of irradiation treatment of polymers and possibly composites, because these effects are seldom studied. Many earlier investigations used mainly tensile tests to assess the polymer's applicability for irradiation treatment. Only in very rare cases are these tests followed up with fatigue tests. From the performed tests the following conclusions can be drawn:

1. Irradiation induced changes depend largely on the detailed polymer chemistry and state. Chemical modifications can alter the resulting changes drastically (compare CE-BPA and CE-M1 and CE-M2).
2. Property changes depend on the applied dose and can reverse between doses (see tensile results of EP). Interpolation is therefore only possible between close dose settings.
3. Most technical polymers degrade in terms of quasi-static tensile properties like yield stress, tensile modulus and strain at break.
4. Fatigue degradation expressed in terms of slope of the S-N curve improves for epoxy resin and polycarbonate after irradiation treatment.
5. To correlate the macroscopic properties of quasi-static tensile tests with fatigue properties a detailed analyses of the fracture morphology is necessary.
6. Color changes of the polymer after irradiation can only roughly indicate induced changes, as can be seen from the significant colorization of TPU without accompanying changes in the tensile test.
7. Irradiation can lead to warping for some materials.

7. ACKNOWLEDGEMENTS

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