Comparison of the damage evolution in glass fiber-reinforced polyurethane and epoxy in the HCF and VHCF regimes investigated by intermittent in situ X-ray computed tomography

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Abstract. The aim of this work is the comparative characterization of the fatigue and damage behavior of GFR-polyurethane and GFR-epoxy with application-relevant quasi-isotropic layer setup in the high cycle and very-high cycle fatigue regimes. Therefore, a high-frequency test method based on a resonant testing system (1 kHz) has been further developed and assessed with special consideration of self-heating. In intermittent test procedures, the damage state has been explored by in situ X-ray computed tomography analysis after certain numbers of cycles. It was shown that the overall damage state in the VHCF regime is reduced by a factor of three compared to the HCF regime and accompanied by delayed initiation and propagation of delamination. The latter was proven to be the main reason for a decreased inclination of the S/N-curve in the VHCF regime by 50-60%.

1. Introduction and state of the art
Glass fiber-reinforced polymers (GFRP) are increasingly used in components that are subjected to fatigue. In major applications, e.g., wind turbines, the structures have to last more than 1E9 cycles. However, there is still insufficient knowledge about the fatigue and damage behaviors in the very-high cycle fatigue (VHCF) regime, which are essential for providing operational reliability of the structures. Furthermore, recent developments and changes in polymer matrices, e.g., polyurethane-based instead of epoxy-based GFRP used in rotor blades of wind turbines e.g. in China, lead to the need for determination of the polymer influence on the fatigue properties, i.e., damage evolution.

In the last couple of years, a lot of investigations were carried out focusing on the damage evolution in GFRP. In this context, mechanical test methods were combined with advanced techniques like X-ray computed tomography (CT), to obtain information of the internal volume structure. The potential of portraying FRP structures with high-resolution CT and its advantages is shown by various studies. In this instance, two techniques are available: conventional CT and in situ CT. Conventional CT has been used in a variety of methods to determine characteristics of FRP damage evolution. Constant amplitude tests were carried out on GFRP, in which in between test sections [1] and specimen failure [2] CT scans were used to locate proceeding damages. In situ CT is applied in an expanded way to explore the nature of FRP behavior by scanning at different levels of load [3]. Studies on FRP using in situ CT show promising potential, since they lead to significant improvements regarding result reliability by
minimizing crack closure effects, which occur because of elastic behavior. In the case of FRP, CT images carried out on an in situ loaded specimen show up to more than 200% increased delamination area and 30 to 100% more cracks than the same specimen scanned via conventional CT [4]. In this way, damage initiation and propagation can be successfully monitored. [5]

Investigations of damage evolution using in situ CT are almost exclusively limited to the low cycle fatigue (LCF) to high cycle fatigue (HCF) regime. This is mainly due to practical and economical reasons, such as the enormous test duration under standard test conditions for investigating the VHCF regime, e.g., more than 110 days for 1E8 cycles at 10 Hz. Previous approaches in the VHCF regime were therefore based on unconventional test methods with increased frequencies. However, many of these test methods are of limited use, as they are accompanied by extensive restrictions, for example with regard to maximum load. In the near past, the ultrasonic test method has been used by several researchers, which was applied on FRP in 3P bending [6] as well as axial [7]. This test method is limited to a frequency of 20 kHz. It is expected that an extensive self-heating takes place, which impairs the comparability with results from the HCF regime determined in accordance with the standard. Therefore it can be concluded that a holistic, suitable experimental method for the investigation of the VHCF regime still needs to be developed.

In principle, it has to be discussed whether a characterization of the fatigue properties in the VHCF regime is necessary at all. According to Talreja [8], FRP exhibit an endurance limit represented by region III in the fatigue-life diagram. In this case, the strain amplitude is too small to initiate matrix cracks and/or to be able to trigger a crack growth rate sufficient for failure. In investigations of Wu et al. [9], Hosoi et al. [10], and Adam and Horst [11] the theory of Talreja is supported. For example, Hosoi et al. did not detect any damage in quasi-isotropic GFRP up to 3E8 cycles at a relative maximum stress of 0.2, which is why a limit value with regard to damage initiation and thus endurance limit was assumed. Adam and Horst investigated the damage evolution in GFRP up to 1.5E8 cycles. At an initial strain of 46-51% of the static strain to inter-fiber breakage, no or marginal delamination occurred. Based on this, the authors assumed a limit value of the initial strain representing an endurance limit.

In recent test series, findings contrary to those of the research groups listed above can be found. In tests on CFRP [12], GFRP [7] and flax fiber-reinforced EP [13], no endurance limit or trend in this respect can be identified. Weibel et al. [12] detected a significant increase in crack density under 3P bending up to 1E9 cycles and failure occurred at approx. 2E9 cycles on additional specimens. Investigations on unidirectional GFRP under axial tensile loading show similar trends [7]. Although the S/N curve shows a flattening from the HCF to the VHCF regime, failure still occurs in tests up to approx. 1.6E9 cycles, accompanied by an initial strain amplitude of only approx. 0.11%. With reference to the theory of Talreja, this proves that even at low strain amplitudes failure of FRP structures can still occur. Although it cannot be ruled out that an endurance limit may be present at even lower strain amplitudes and/or other structural properties, recent research results show the need to investigate the VHCF regime up to at least 2E9 cycles.

This work focusses on the comparative characterization of the fatigue and damage behavior of GFR-polyurethane and GFR-epoxy with application-relevant quasi-isotropic layer setup in the HCF and VHCF regimes. Therefore, an intermittent test procedure has been applied to determine the damage evolution and interaction stepwise after certain numbers of cycles. For the investigations in the VHCF regime, an unconventional high-frequency test method based on a resonant testing system (1 kHz) has been further developed and assessed with special consideration of self-heating.

2. Material

In this study, GFR-polyurethane (GFR-PU) and GFR-epoxy (GFR-EP) are comparatively investigated. Both GFR-PU and GFR-EP are based on an application-relevant quasi-isotropic layer setup of [45/-45/0/90]s with a fiber volume fraction of 46%. The fiber material is Hex-Force 07781 1270 (continuous E-glass, 9 µm fiber diameter) in 8h satin fabric. The fibers are showing a Silan TF970 sizing, which is suitable for both polymers.
In Table 1 the properties of the neat resins are listed. The EP is the Hex Ply 914 matrix system certified for use in the aviation industry. The GFR-EP was manufactured by FACC (Austria) in an autoclave process at 7 bar and 175 °C for 1 hour. The newly PU thermosetting resin was developed by Rühl (Germany) and is based on a multi-stage modification of existing resin systems suitable for a vacuum RTM manufacturing process. The final configuration used in this work is a combination of the slowly reacting polyol purpreg 185-2L IT with internal release agent and the low-viscosity isocyanate puronate 905. In the production process of GFR-PU at the Institute for Material Technologies and Plastic Processing (Switzerland), both components are mixed in a high pressure device and injected with a rate of 7 g·s⁻¹ for 41 s into the high pressure tool, in which a preform is fixed. The curing process takes place at 35 bar and 85 °C for 300 s. Further details on the manufacturing process can be found in [14].

|                        | Young’s modulus $E$ (GPa) | Tensile strength $\sigma_M$ (MPa) | Strain to failure $A$ ($10^{-2}$) | Glass trans. temperature $T_g$ (°C) |
|------------------------|---------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| Polyurethane           | 3.1                       | 63                                | 9.1                               | 130                               |
| Epoxy                  | 3.9                       | 48                                | 1.5                               | 190                               |

Table 1: Properties of the neat resins of polyurethane and epoxy [14].

3. Experimental methods

For the investigations of the damage evolution, an intermittent test procedure was applied, in which mechanical tests were stopped after certain numbers of cycles to determine the damage state via in situ CT. Two different kinds of mechanical testing systems for the LCF to HCF regime and the VHCF regime were used and will be presented in the following subsections. A special CT specimen geometry (Fig. 1) is necessary due to restrictions of the in situ CT-stage. The dimensions allow a CT-scan of the whole measurement length at a time and the shape of the specimen results in an almost completely homogeneous stress distribution in the measurement length, shown for an elongation of 10 µm in Fig. 1.

![Fig. 1: Specimen geometry and simulation of the stress distribution for an elongation of 10 µm.](image)

3.1. Fatigue tests in the LCF to HCF regime

The investigations in the LCF to HCF regime were carried out stress-controlled with the use of a servo-hydraulic testing system (Shimadzu EHF-UV100, $F_{max} = \pm 100$ kN) with a sinusoidal load time function under tension-tension loading ($R = 0.1$) in a climatic chamber at 23°C. Due to the CT specimen geometry, a special adapter was used (Fig. 2). With the adapter, the specimen is mechanically clamped by combining friction and pinning.

The frequency was varied stress-related between 1.5 Hz (200 MPa) and 30.0 Hz (100 MPa) to realize a constant induced energy rate and thereby a similar self-heating. More information regarding the
principle of induced energy rate can be found in [15]. The fatigue tests for investigating the damage evolution were carried out at a relative maximum stress of \( \sigma_{\text{max},r} = 0.55 \) related to the tensile strength.

3.2. Fatigue tests in the VHCF regime

In view of economical and practical reasons, investigations in the VHCF regime need to be carried out at higher frequencies. Therefore, in this study, a resonant testing system (Rumul Gigaforte, \( F_{\text{max}} = \pm 25 \text{kN} \)) was used, which works at a frequency of about 1 kHz. The further test parameters and the specimen adapter were similar to the ones applied in the HCF regime.

The use of this testing system leads to an improved comparability between the results in the HCF and VHCF regimes compared to the use of an ultrasonic testing system, which was applied in multiple previous studies, e.g., [7, 12], due to the lower frequency (1 kHz vs. 20 kHz). However, there is still a big difference between the frequencies in the HCF and VHCF regime. That is why special attention was paid to the self-heating. Therefore, specimens made of GFR-EP were provided with thermocouples on the surface as well as embedded in the core. The results have been used to determine a suitable stress range, in which a sufficient comparability of the test results is assumed.

Figure 3a) shows the stress-dependent development of the surface and core temperature at simultaneous air cooling. The surface temperature remains almost constant with increasing maximum stress. The core temperature increases significantly by about 40 K from an initial temperature of 30 °C between 60 and 140 MPa, showing a linear behavior. Thus, the difference between surface and core temperature increases with increasing stress. In order to evaluate the influence of air cooling, additional tests were carried out without air cooling (Fig. 3b). In this case, the core temperature increases exponentially, so that a temperature of well over 100 °C (> 50% Tg) is already reached at \( \sigma_{\text{max}} = 90 \) MPa. In contrast to the tests with air cooling, the surface temperature shows a qualitatively comparable course with only a slight increase in the temperature difference from the core temperature.

From the investigations it can be concluded that the concept of evaluating the specimen temperature on the basis of surface temperature measurements with simultaneous air cooling must be considered improper. On the basis of the above-mentioned findings, the low self-heating detected by Flore et al. [7], among others, is to be questioned with regard to their validity. It can be assumed that the core temperature is significantly higher. Lower fatigue strengths compared to investigations with conventional test methods, such as those determined by Weibel et al. [12], can therefore probably be justified by the temperature increase in the core.

For the resonant testing system, the temperature development in the core confirms that this test method can only be used validly on GFRP in a low stress range (VHCF). In this range, the temperature development in the core can be limited to, e.g., approx. 20 K for \( \sigma_{\text{max}} = 90 \) MPa by using air cooling.
The testing system was therefore used exclusively in a stress range of \( \sigma_{\text{max,r}} = 0.25 \) to 0.35, taking into account the temperature development and following VHCF-specific threshold values of Hosoi et al. [16] and Jeannin et al. [13]. For the investigations of the damage evolution in the VHCF regime, a relative maximum stress of \( \sigma_{\text{max,r}} = 0.275 \) was applied, resulting in a calculated temperature increase in the core of approx. 23 K, which is considered permissible for FRP in Vieille and Taleb [17]. Furthermore, the maximum temperature stays below 35\% Tg for GFR-PU and 25\% Tg for GFR-EP, respectively, and is thereby significantly below the threshold value of 50\% Tg defined by Backe et al. [6].

![Stress-dependent self-heating of GFR-EP specimens at a frequency of 1 kHz. a) Comparison of surface and core temperatures and b) influence of air cooling.](image1)

**Fig. 3:** Stress-dependent self-heating of GFR-EP specimens at a frequency of 1 kHz. a) Comparison of surface and core temperatures and b) influence of air cooling.

### 3.3. In situ CT investigations of the damage evolution

CT investigations were carried out with Nikon XT H 160. The system contains an X-ray tube with a maximum voltage of 160 kV and a maximum power of 60 W and allows, with the 10082-pixel detector, voxel sizes down to 3 \( \mu \text{m} \). In situ load application was performed using an in situ CT stage (Deben Uk CT5000, \( F_{\text{max}} = \pm 5 \text{kN} \)). A glass-like carbon tube with a wall thickness of 3 mm transmits the force between the lower and upper clamping device. X-rays can pass through this homogeneously constructed tube under a small, uniform damping. [5]

![3D CT Volume, Defect analysis, Front view, Top view with layer orientations.](image2)

**Fig. 4:** Procedure of in situ CT defect analysis [5].

The defect analysis is done using in situ acquired 3D CT volumes containing the damage states of the specimens. For an illustration of the 3D damage volume, the specimen material is hidden, and only
the contrast agent is visible (Fig. 4). Besides, the contrast agent residues on the surfaces of the front and back are hidden to provide an insight into the specimen. The front and top view of the 3D damage volume are used to analyze the damage state and distribution with the usage of single layer defect analysis for all layer orientations, to separate the occurring damages and their locations. [5]

4. Results

4.1. Fatigue behavior

Figure 5 presents the material-specific S/N curves. The S/N curve of GFR-PU (Fig. 5a) shows a similar shape to GFR-EP (Fig. 5b), although the tensile strength is reduced by approx. 15%. In recent publications various approaches for modelling the S/N curve are used. In this study, the S/N curve is separated into three regions, which are representing the (I) LCF, (II) HCF, and (III) VHCF regime. As a demonstration of the insufficient description by simpler approaches, a power function is shown in a grey dotted line across all regions as well as across regions II and III. The entire power function leads to an underestimation of fatigue strength in the LCF regime and an overestimation in the VHCF regime, which is critical with respect to a conservative design. With a power function for the joint description of regions II and III, fatigue strength in the VHCF regime is consistently underestimated, which does not allow an ideal utilization of the material potential. The separation of the S/N curve into three regions, on the other hand, leads to a very good agreement of region-specific power functions with the test results and is therefore recommended for a comprehensive description of the fatigue strength.

\[ \sigma_{\text{max}} = a \times N_f^n; n = k^{-1} \]  

(1)

For GFR-PU, the ratio of the inclination index k (Eq. 1) for regions II and III \((k_{\text{II}}/k_{\text{III}} \approx 10.5/15.9 \approx 1/1.5)\) is almost identical to GFR-EP \((10.9/17.5 \approx 1/1.6)\). The inclination index k presents the slope of the S/N curve described by Basquin’s law. It can be summarized that for quasi-isotropic GFRP there is a transition region from the HCF to VHCF regime, which is accompanied by a change in the slope of the S/N curve of 50 to 60%. The course of the S/N curve is thus comparable to that of face-centered cubic metals whereby their change in the slope is more pronounced and entails a change in the failure mechanism from primarily surface damage to internal damage. A deviating or delayed development of damage in the VHCF regime was also suspected for FRP as a reason for the changing inclination from region II to III \([6, 11]\) and will be demonstrated in the following subsection. The results do not show any trend regarding an endurance limit, which confirms recent findings in other studies \([7, 12]\).

![Fig. 5: S/N curves for a) GFR-PU and b) GFR-EP from the LCF to VHCF regime.](image-url)
4.2. Damage evolution

The damage evolution is exemplarily illustrated in Fig. 6. Generally, the damage evolution is characterized by a gradual development. In the HCF regime, directly after the test start, inter-fiber cracks form in the 90° layers and subsequently in the ±45° layers. In the further course of the test, these damage mechanisms proceed, accompanied by intra-layer delamination in the 0°/90° layer, which originate from meta delamination. In the following, existing cracks and intra-layer delamination grow. Additionally, the amount of meta delamination increases significantly in the 0°/90° layers and first inter-layer delamination occur between the 0°/90° and ±45° layer. The delamination proceed over the entire measuring length and spread towards the center of the specimen, indicating the subsequent failure.

GFR-EP consistently shows a more extensive damage state. In particular, the number of inter-fiber cracks is significantly increased, which can be attributed to the lower ductility of EP compared to PU. However, the fatigue strength is higher than that of GFR-PU. This is due to the fact that the formation of delamination is reduced. GFR-PU has a significantly higher proportion of delamination, which are more critical in terms of fatigue properties than inter-fiber cracks. The reduced fatigue strength can therefore be attributed to a decreased interlayer strength, which results, amongst others, out of a pore content of approx. 1.2 vol.-% (GFR-PU) compared to 0.0 vol.-% (GFR-EP).

In the VHCF regime, the damage mechanisms and the general damage evolution are similar to those in the HCF regime, although a reduced crack density is formed. This results in a reduction of the damage volume by a factor of three compared to the HCF regime. However, the main difference in the damage evolution in the VHCF regime is represented by a delayed initiation and propagation of delamination. In the HCF regime, first delamination occur after approx. 10% of the fatigue life. In the VHCF regime, on the other hand, no delamination can be detected in GFR-PU up to 79% and in GFR-EP, after 89% of the fatigue life first delamination formation is visible. It can therefore be assumed that delamination will probably also be initiated in GFR-PU in the further course, which will initiate the failure. Based on the findings it is reasonable to assume that the delayed initiation of delamination, as a critical damage mechanism, leads to the decrease of inclination of the S/N curve in the VHCF regime by 50-60%.

![HCF and VHCF Damage Evolution](image)

Fig. 6: Comparison of the damage evolution in GFR-PU and GFR-EP in the HCF and VHCF regimes.

5. Conclusions

In this study the necessity for improved alternative test procedures in the VHCF regime was proven. In particular, the influence of increased frequencies has to be assessed by paying special attention to the self-heating. For the 1 kHz resonant testing system, a suitable stress range was identified by measuring
the stress-dependent increase in temperature with the use of embedded thermocouples. In intermittent test procedures, the damage state has been determined by in situ 3D X-ray computed tomography. It was shown that GFR-PU exhibits decreased fatigue strength compared to GFR-EP, although the overall damage state of GFR-EP is significantly higher. This contradiction has been explained by the increased proportion of delamination in GFR-PU, which are much more critical than the higher amount of interfiber cracks in GFR-EP. In the VHCF regime the general damage evolution is similar to the one in the HCF regime, although much less extensive and delayed. The damage volume is reduced by a factor of three compared to the HCF regime. However, the most important difference was proven to be the delayed initiation and propagation of delamination, which was considered the main reason for the change in the slope of the S/N curve of 50 to 60% from the HCF to VHCF regime. Although the slope of the S/N curve changes, a trend regarding the existence of an endurance limit could not be identified.

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