Nondestructive investigation of the VHCF-endurance on cyclically loaded CFRP by X-Ray-Refractography

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Abstract. Carbon Fibre Reinforced Plastics (CFRP) are more and more used in modern civil aircrafts. These days the whole fuselage is made of this material (B787; A350). Due to strict certification standards the normal in-service loading gives a low stress level compared to the static and even the fatigue strength of the material. Hence CFRP are assumed to have an infinite life. To evaluate this assumption, fatigue tests on CFRP-specimens were performed up to 10^8 load cycles and the first inter-fibre failure was evaluated non-destructively by accompanying X-ray-refraction topography.

A tensile testing machine was integrated in a small angle X-ray scattering (SAXS) setup. X-ray refraction topography [1] was performed while the CFRP-samples were tensile loaded. This non-destructive technique enables the detection of micro-cracking and inter-fibre failure especially for CFRP. For Glass Fibre Reinforced Plastic (GFRP) X-ray refraction and in-situ loading has already been successfully used [2]. The increase of inner surfaces due to inter fibre failure was measured as a function of the stress state. Fatigue tests were performed at and below the limit of inter-fibre failure strength.

State of the art is to assume the failure of the samples under cyclic loading as the fatigue life. Accompanying non-destructive X-ray refraction measurements reflects the damage state and enables to trace its evolution even if the total failure of the specimens does not occur. This investigation technique is of high interest to give the engineer a design value of infinite life which is practically often reached due to knock down factors of certification standards. Finally the infinite life was found for cyclic fatigue loaded CFRP-samples even under high inter fibre transverse and shear loading investigated up to 10^8 load cycles.

In order to determine the influence of the matrix properties on the boarderline to infinite life, research is done on laminates while varying matrices and their fracture mechanical properties. With the evolution of early cracks and IFF monitored as well as the influence of the matrix on the crack propagation investigated an appropriate model is planned to improve lifetime prediction in CFRP.

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Content

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1. Introduction

The aim of this research project is to understand the damage evolution in FRP being subject to high cycle fatigue. This is done by combined mechanical and non-destructive testing in order to facilitate the monitoring and characterisation of damage evolution. By means of the X-ray refraction topography developed at BAM [1] it is possible to identify the internal surfaces non-destructively quantitatively and spatially resolved. Thus it is possible to identify micro-damage in the µm-scale (fibre-matrix-debonding and matrix fracture) even in complex laminates. The objective is to monitor the micro-crack-evolution in woven textiles and non-crimp fabrics to determine material constants and develop models to determine the boundary to “infinite life”. The basic idea of the investigation is represented in Fig. 1. Accompanying non-destructive testing (NDT) is mandatory to achieve information about the damage state of the material since at low load levels the total failure of the samples will not be reached.

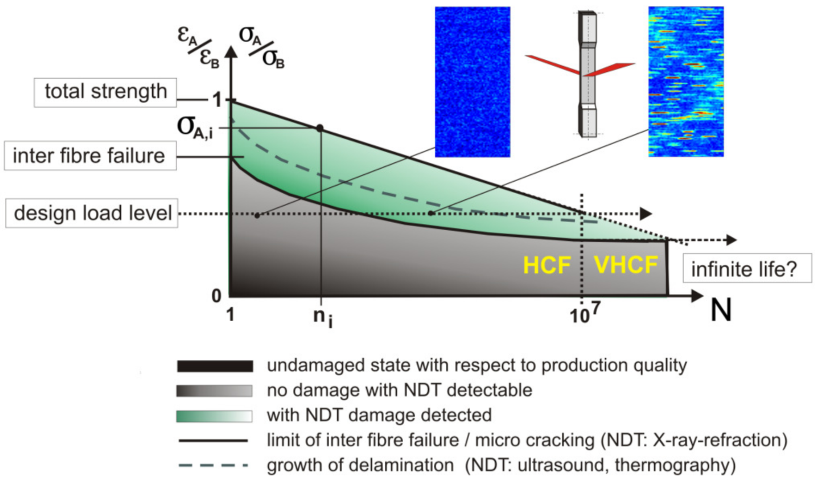

Fig. 1: Basic idea of investigation – characterisation of the damage state of CFRP due to fatigue loading before the S-N curve.

2. Experimental

2.1 Materials and test specimen

The CFRP specimens were made from a 200g/m² non-crimp fabric and 400g/m² twill style textile each made with Tenax-E HTA40 E1, 6K yarn. The matrix system was Huntsman Araldite® LY 556 / Aradur® 917 / Accelerator DY 070. Flat specimens with a length of 150mm, a width of 15mm (0°/90°-laminate) and 20mm (+/-45°-laminate) with a thickness of 1 and 2mm and tab reinforcement for clamping were used. 0°/90°- and +/-45°-laminates of each textile reinforcement were investigated.

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2.2 X-ray refraction technique

X-ray refraction [1] is caused by the effect of refraction at the interface of materials of different refractive index as known from visible light passing glass lenses. In the case of X-rays the refraction angle is below half a degree and in opposite direction due to the dispersion function of isolators. Hence Small Angle X-ray Scattering (SAXS) technique is used. In the experimental set-up (Fig. 2) a collimated X-ray beam passes the sample. At a fixed angle the refracted signal and a signal proportional to the absorption are measured. A characteristic refraction value C is determined, which is proportional to the surface per volume unit. It can be calculated from the scattering I_R and transmitted intensities I_A and the thickness d of the sample in relation to the zero values (without sample):

$$C = \left[\frac{I_R/I_{R0}}{I_A/I_{A0}} - 1\right] \cdot \frac{1}{d} \qquad (1)$$

The intensity of the refracted beam will increase if a difference of the refractive index occurs at the observed interfaces. Hence, the intensity will be higher for materials with de-bonded fibres or pores than without (Fig. 3). By calibration the absolute as well as the relative inner surfaces C are measured. In most cases the relative increase is sufficient and used in the further investigations. Scanning the whole area of the sample gives a topographic map of inner surfaces (Fig. 3 and 5). According to Lambert-Beer's law the absorption is a function of the density-proportional linear absorption coefficient µ and the thickness d of the sample:

$$I_A = I_{A0} \cdot e^{-\mu \cdot d} \qquad (2)$$

For several applications it is practical to normalise equation 1 to *ln* (I_A/I_{A0}) resulting in the relative specific surface C_m/μ, independent from variation of the number of fibre filaments due to non-perfect production of the fabrics.

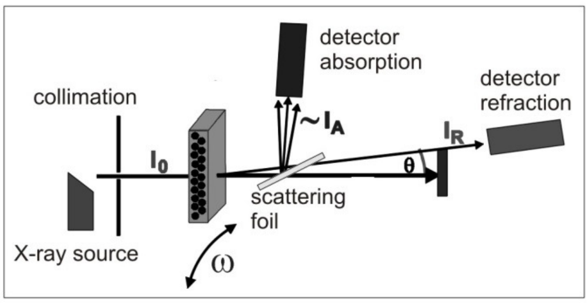

Fig. 2: Experimental setup of the X-ray-refraction technique

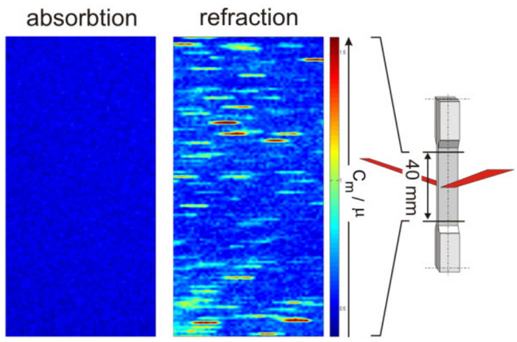

Fig. 3: The absorption and refraction signal are measured at the same position on the sample. Only in consideration of the refraction signal the cracks could be visualised.

The density itself contains no information about inner surfaces. In the experimental setup of the refraction technique the absorption and the refraction signals are measured independently at once. In Fig. 3 the absorption and refraction mapping of a CFRP sample made of a twill fabric are compared. The absorption (Fig. 3, left) maps only slight differences of the density. Instead, the refraction-signal (Fig. 3, right) maps the micro cracks due to inter fibre failure caused by the cyclic fatigue loading. Thus, if there is no

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significant change of the density it is impossible to detect any fibre matrix de-bonding with the classical X-ray radiography which only uses the absorption proportional to mass.

2.3 Tensile strength test and in situ damage evaluation

A tensile testing machine was integrated in a small angle X-ray scattering (SAXS) setup (Fig. 4, right). X-ray refraction topography [1, 2] was performed while the CFRP-samples were tensile loaded (Fig. 4, left). This non-destructive technique enables the detection of micro-cracking and inter-fibre failure especially important for CFRP. For GFRP X-ray-refraction and in-situ loading has already been successfully used [2, 3]. The increase of inner surfaces due to inter-fibre failure was measured as a function of the stress state.
For the 0°/90°-laminate made of non-crimp fabrics the first inter-fibre failure occur at a stress level of approximately 350 to 400MPa. In steps of about 1kN (approx. 40MPa) the load was increased until the total failure of the sample.
For the 0°/90°-laminate made of twill style textile the first cracks occur above 300MPa. Since the first inter-fibre failure was detected at a certain stress level, it is assumed to reach the very high cycle (VHCF) range (Fig. 1) for the 0°/90°-laminate below this value.

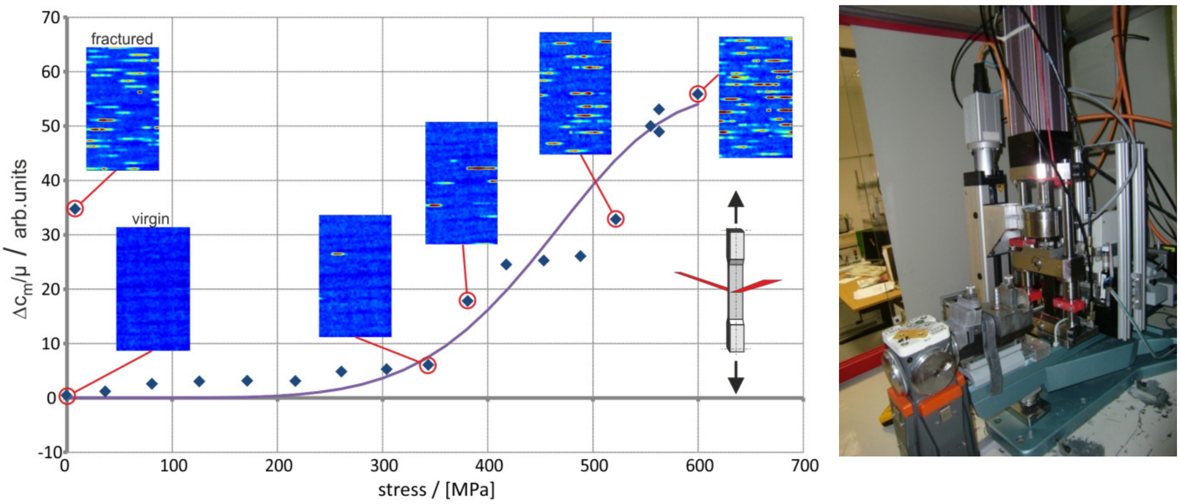

Fig. 4: left - 0°/90°-CFRP-sample of non-crimp fabric, tensile loaded and in-situ X-ray-refraction testing.
right - 15kN-elektro-mechanical tensile testing machine integrated in the X-ray scanner.

Due to the 0°/90°-fibre orientation the inter fibre failures occur perpendicular to the load direction. Beside the phenomenological degradation process of increasing inner surfaces measured and visualised with the refraction technique [2] described above (Fig. 4), the proper shape of the micro cracks is of high interest for physical interpretation. Therefore a comparative investigation was done at the Berlin synchrotron BESSY with Diffraction Enhanced Imaging (DEI) [4, 5, 6].

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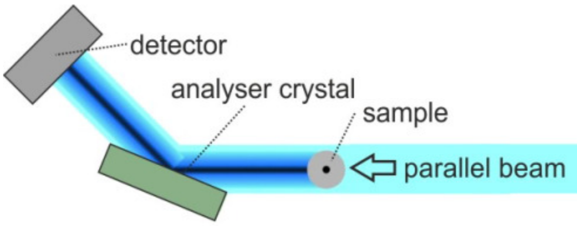

Fig. 5: Principle of DEI – refracted light is discriminated with the rocking curve of an analyser crystal.

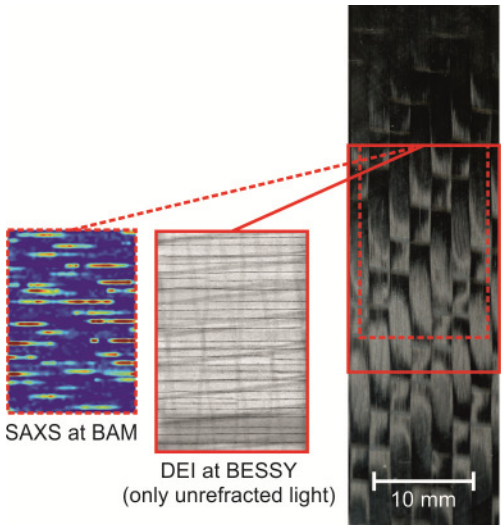

Fig. 6: Imaging transverse cracks in a CFRP-sample – from right to left: 0°/90°-CFRP, DEI at the edge and in the middle of the rocking curve, SAXS-technique.

With DEI the refracted light at cracks or pores is discriminated with an analyser crystal (Fig. 5), in contrast to the Small Angle X-ray Scanning (SAXS) technique (Fig. 2). Therefore a parallel beam of monochromatic X-ray light is mandatory which can be generated with synchrotron radiation and a double-crystal monochromator. The analyser crystal can be rotated precisely. If the Bragg-angle is adjusted to the maximum intensity only the unrefracted light complies with the Bragg condition and refracted light is missing. Thus, the cracks are imaged dark (Fig. 5 and 6).

The fractured specimen shown in Fig. 4 was investigated with DEI. The DEI images are shown comparative to the SAXS topography in Fig. 6. The DEI images are area-wide and contain 520 vs 800 pixel with a pixel-size of 28.8µm vs 28.8µm. The SAXS images are non-area-wide with a step size of 0.3mm each in x- and y- direction (horizontal and vertical) and contain 40 vs 67 pixel of 2000µm vs 50µm. The marginal values were omitted in the images due to the width of the X-ray beam of the Kratky collimation. The DEI images have a much higher resolution and visualise that the inter fibre cracks mostly pass the hole width of the specimen. Additionally the cracks are not straight but rather follow the waviness of the 90°-fibre bundles. With this finding all crack indications in the SAXS-topography can be assumed as one crack over the whole width of the specimen. The result is a crack density of approximately 1/mm. Counting the cracks of the DEI images gives the same result. For further SAXS investigations the scanning grid was optimised. Finally the specimens were scanned in 7 steps over the width and 100 steps of each 0.2mm in fly by technique over a length of 20mm.

2.4 Tensile fatigue loading and in situ damage evaluation

Fatigue tests were done in servo-hydraulic tensile testing machines at 5 up to 100Hz (0°/90°-laminate). All tests were done air conditioned at 23°C and 50% humidity. The intrinsic heating at low load levels is insignificant and hence the recorded surface temperature rise is moderate until shortly before failure. Even for +/-45°-laminate the increase of the surface temperature is only 7°C at the lowest load-level of 50MPa and a test frequency of 50Hz. The surface temperature was recorded during all fatigue tests with an infrared sensor. A maximum temperature rise of 10°C is acceptable since the maximum temperature increase inside the specimen could be assumed below 20°C. Hence the total temperature of the specimen is far below the glass transition temperature of about 120°C for this epoxy-matrix system.

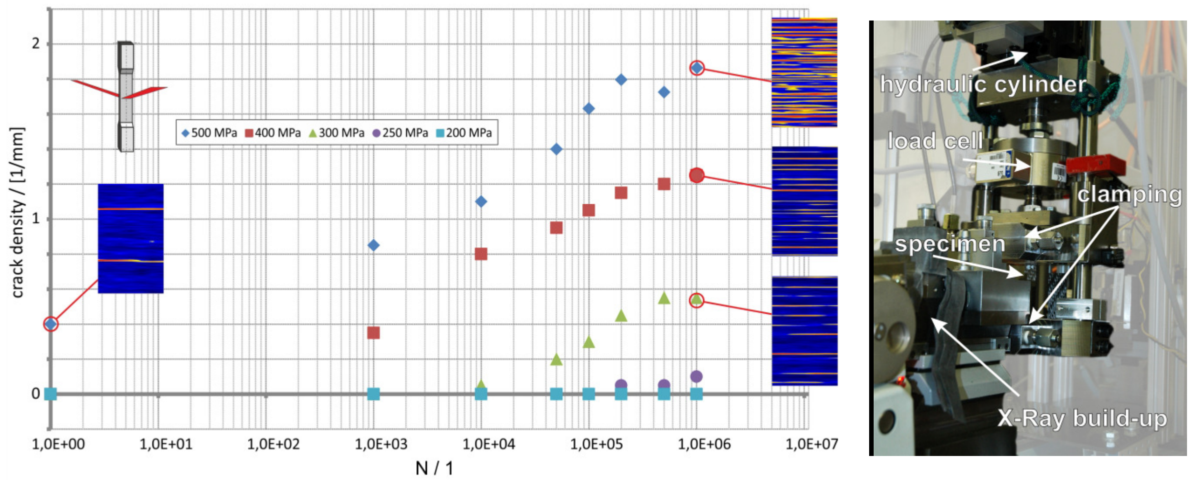

Fig. 7: left - 0°/90°-CFRP-sample of non-crimp fabric, tensile cyclic (fatigue) loaded and in-situ X-ray-refraction testing. right - 10kN servo-hydraulic tensile testing machine integrated in the X-ray scanner.

Two test rigs of servo-hydraulic tensile testing machines were built-on with a load capacity of max. 10kN. One load frame was integrated in the SAXS scanner (Fig. 7, right) instead of the electro-mechanical tensile testing machine (Fig. 4, left). The specimens were scanned after a certain number of load cycles (Fig. 7, left) und the crack density was measured while a preload was applied to open the cracks. These in-situ measurements were done up to 10^6 load-cycles at different load levels (Fig. 7, 8 and 10). In a second load frame the very high cycle fatigue tests were performed up to 10^8 load-cycles. After a certain number of load cycles the fatigue experiments were stopped and the specimens were scanned in a second SAXS scanner unloaded. All results are summarised in the Figures 9 and 11.

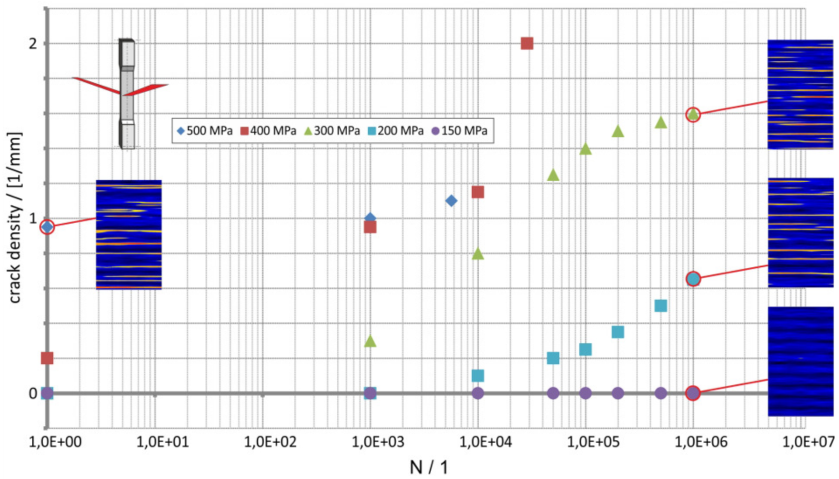

Fig. 8: 0°/90°-CFRP-sample of twill style textile, tensile cyclic (fatigue) loaded and in situ X-ray-refraction evaluation. Load ratio R=0.1

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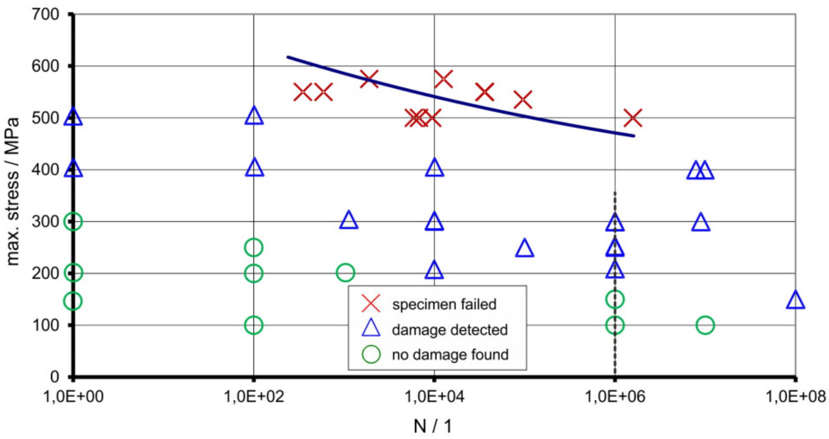

Fig. 9: S-N-curve of 0°/90°-CFRP of twill style textile – failed, damaged and no detectable damage. Load ratio R=0.1

2.4.1 Testing of 0°/90°-laminate

The 0°/90°-CFRP specimen of non-crimp fabric do not fail at and below a stress level of 500MPa (R=0.1) up to 10^8 load cycles, even though the first inter fibre failure occur at the first load cycle (Fig. 4 and 7). The maximum crack density is doubled to approx. 2/mm compared to the static strength test with ca. 1/mm at fracture. In the fatigue tests the maximum crack density decreases with stress level. Below 200MPa there is no micro cracking detected up to 10^6 load cycles. Thus technically infinite life could be assumed at this load level in respect to the laminate, the load ratio and the taken material concerning fibre, fabric and matrix.

An overview of the fatigue tests on 0°/90°-CFRP specimen of twill style textile is summarised in Fig. 8 and 9. The total failure of the samples emerged in the fatigue tests at and above 400MPa upper limit, load ratio R=0.1. Above 300MPa inter-fibre failure occurs at the first load cycle however the specimens does not fail up to 10^7 and hence reach the VHCF region.

Below a stress level of 150MPa no increase of micro cracking could be observed up to 10^7 load cycles (Fig. 9). This is approximately 50% of the inter-fibre failure stress in static loading. Again technically infinite life could be assumed at this load level in respect to the laminate, the load ratio and the taken material concerning fibre, fabric and matrix. There is a transition region from undamaged to damaged state between 150 and 300 MPa upper limit stress. X-ray refraction enables to show an increasing crack density with the stress amplitude (Fig. 8).

2.4.2 Testing of +/-45°-laminate

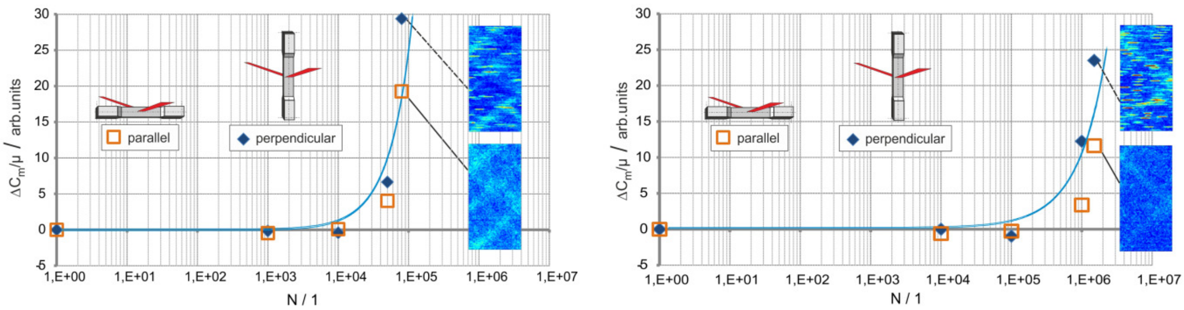

Fig. 10: +/-45°-CFRP-samples of twill style textile, tensile cyclic (fatigue) loaded and in-situ X-ray-refraction testing parallel and perpendicular to the samples length direction. Load ratio R=0.1.
left: maximum stress 75MPa, right: maximum stress 60MPa

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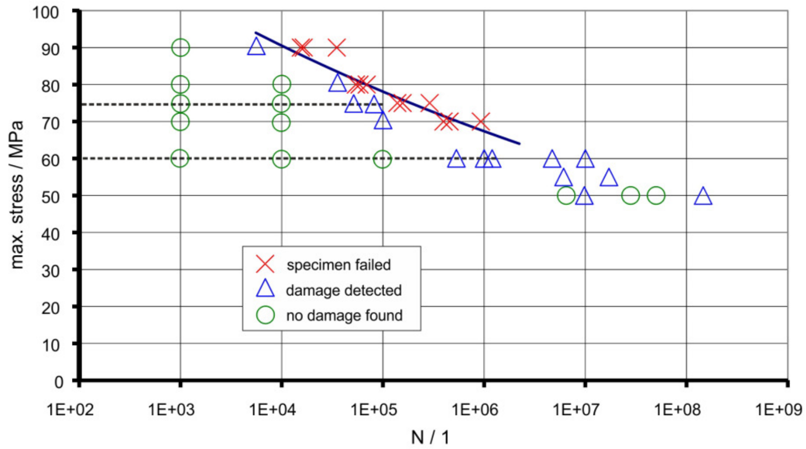

Fig. 11: S-N-curve of +/-45°-CFRP of twill style textile – failed, damaged and no detectable damage. Load ratio R=0.1

The overview of the fatigue tests on +/-45°-laminate is summarised in Fig. 10 and 11. The failure in fatigue loading is found at and above a maximum stress of 70MPa up to 10^6 load cycles. The region of no damage is below 50MPa. That's also approximately 50% of inter-fibre shear strength. The VHCF region could be reached at an upper stress limit of 60MPa. Fortunately the scatter of number of load cycles to failure of the S-N-curve is much lower compared to the 0°/90°-laminate. Figure 10 depicts the transition from undamaged to damaged state by fatigue tests on constant stress levels at 75 and 60MPa. Due to the +/-45°-fibre orientation two perpendicular oriented X-ray refraction measurements have to be done [2, 3]. Two different failure mechanisms occur. First perpendicular to the specimen length direction matrix cracks appear in the crossings of the fibre bundles and subsequent intralaminar fibre matrix debonding occurs due to the inter-fibre shear loading. Both effects can be distinguished by the perpendicular measurements. In parallel configuration only the fibre matrix de-bonding is visualised. In perpendicular configuration both – the fibre matrix debonding and the matrix cracks become visible. The scattering orientation function is proportional to $\cos^2$□ [2, 3]. Hence both the +/-45°-fibres and/or the fibre-matrix de-bonding contribute to the parallel and perpendicular scans. In contrast the matrix cracks contribute only to the perpendicular scanning configuration. Neither the matrix cracks nor the fibre-matrix-debonding travel over the whole width of the sample rather distribute statistically over the specimen. Due to this micro crack behaviour no crack density could be defined as for the 0°/90°-laminates. Therefore the normalised refraction value was plotted vs. the number of load cycles to characterise the damage state.

2.5 Influence of fracture mechanical properties

In earlier investigations at BAM [9] it was shown, that the matrix has a strong influence on the micro-crack formation and finally on the total lifetime. For that purpose an epoxy resin had been cured to a different degree of curing. This material was then used to cut modified SENT-specimens for a fatigue crack propagation test. In consequence of about 2 % difference in the degree of curing, the crack propagation rate differed by factor 10 (Fig. 12). Furthermore these differently cured epoxy systems were used as matrix material

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for a fatigue tests on ±45°-GFRP-specimens. The S-N-curve of the less cured specimens which lies one magnitude under the curve of the higher cured specimens, is shown in figure 13.

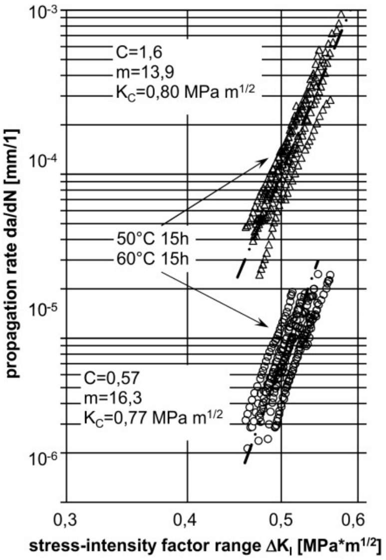

Fig. 12: Crack propagation rate of differently cured epoxy resins [9].

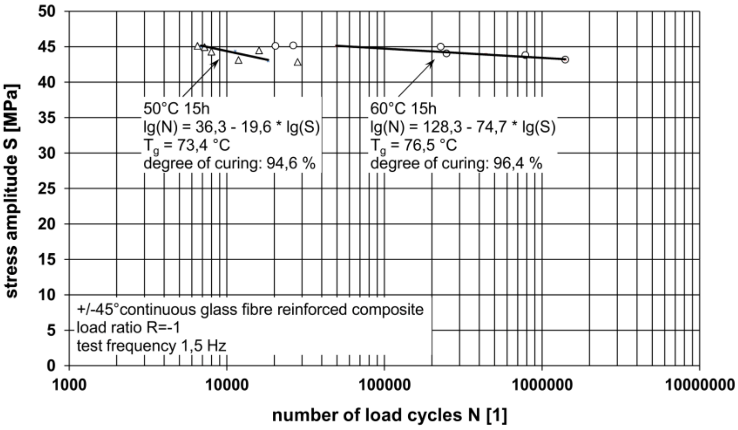

Fig. 13: S-N-curves of differently cured ±45°-GFRP-specimens using the same epoxy and curing cycle [9].

In order to determine the influence of the matrix properties on the borderline to infinite life, research on laminates while varying matrices and their fracture mechanical properties is going on. With the evolution of early cracks and IFF monitored as well as the influence of the matrix on the crack propagation investigated an appropriate model is planned to improve lifetime prediction in CFRP.

3. Discussion

It is well known [8] to visualise inter-fibre failure with the classical radiography at low X-ray energies, however contrast agent has to be used to mark them and only cracks connected to the sample surface will be detectable. With the X-ray refraction technique thin micro cracks and inter-fibre failure could be visualised without contrast agent averaged over the volume [1] and hence an in-situ investigation of increasing micro damages in parallel to mechanical static [2, 3] and fatigue loading [7] is possible.

A stress level of 50% of static inter-fibre failure seems to be the limit to the infinite life of CFRP either for intralaminar transverse and shear loading. This is in the region where the airliners and wind-turbine blades are designed nowadays due to certification standards. Thus, the fatigue of composite materials plays an underpart regarding the service life of such applications. Finally it has to be stated that these results are only valid for the investigated materials.

In an ongoing project the influence of the sizing of the fibres and the matrix are investigated to improve the limit of infinite life regarding inter-fibre failure.

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4. Conclusion

An infinite life was found for cyclic fatigue loaded CFRP-samples under high inter-fiber transverse (0°/90°laminate) and shear (+/-45-laminate) loading investigated up to 10^8 load cycles. State of the art is to take failure of the samples as the fatigue life whereas accompanying non-destructive X-ray-refraction measurements reflects a level of damage even if samples failure not occurs. These results and investigation technique are of high interest to give engineers a design value of infinite life which is practically often reached due to knock down factors of certification standards. Further investigations at different load ratios (especially *R=-1*) are running. A high influence of the load ratio on the stress level of infinite life is assumed and well known for the fatigue life of FRPs.

5. Acknowledgement

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