

**VERIFICATION OF COMPOSITE LAMINATES
UNDER CRYOGENIC THERMO-MECHANICAL
LOADING -
EXECUTIVE SUMMARY**

IWM Report V1209/2019

VERIFICATION OF COMPOSITE LAMINATES UNDER CRYOGENIC THERMO-MECHANICAL LOADING - EXECUTIVE SUMMARY

IWM Report V1209/2019

Dr. Jörg Hohe*

Michael Schober*

Dr. Klaus-Peter Weiss**

*) Fraunhofer-Institut für Werkstoffmechanik IWM
Wöhlerstraße 11
79108 Freiburg
Germany

**) Karlsruhe Institute of Technology, Institute for Technical Physics
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

Project No.: 425168

CLIENT: EUROPEAN SPACE AGENCY

Contents

1	Executive Summary	4
1.1	Introduction	4
1.2	Failure criteria for the cryogenic regime.....	4
1.3	Experimental validation on specimen level.....	5
1.4	Breadboard tests and evaluation	6
1.5	Verification and design guidelines	7
1.6	Conclusions	7
2	Appendix: Figures	9

1 Executive Summary

1.1 Introduction

For next generation launch vehicles and other applications in spacecraft technology, safe and reliable fuel tanks are required. For the storage of hydrogen and oxygen in the liquid state, the respective vessels have to be able to withstand temperatures in the cryogenic regime down to 20 K. The relevant loading situations are of both, static and cyclic nature. Due to the inherent brittleness of most metals in this range, and in order to save as much weight as possible, carbon fiber reinforced plastics (CFRP) is a natural material choice for the shell of such fuel tanks. In order to exploit their full lightweight potential, validated criteria for the assessment of their integrity under thermal and mechanical loads are essential.

The present research activity has been concerned with the definition and validation of composite failure criteria for application in the cryogenic temperature regime. For this purpose, a review and selection of pre-existing failure criteria has been performed. The most promising options were validated and assessed against an experimental data base. For a further validation, small breadboards including simple structural features such as holes (e.g. for bolt connections) or tapered sections were tested. The experiences made during the test campaign and assessment of the different failure criteria have been used for the formulation of guidelines regarding the application of Puck's failure criterion selected here as well as the design of laminated for application in the cryogenic regime.

1.2 Failure criteria for the cryogenic regime

Failure of composite materials in the cryogenic regime such as e.g. the temperature range of 20 K for liquid hydrogen fuel vessels may involve the development of other failure mechanisms compared to the ambient temperature regime. A major driver in this sense could be the development of microscopic residual stresses between fibers and matrix of CFRP materials induced by the mismatch in the coefficients of thermal expansion of the two constituents. The microscopic intralaminar residual stresses might be superimposed by residual stresses induced by the thermal mismatch between the effective coefficients of thermal expansion of adjacent plies with different fiber orientation. The failure of individual plies within a laminate and subsequently of the entire laminate is caused by the total stress level consisting in the contributions by the stresses due to the externally applied loads as well as the contributions due to both kinds of residual thermal stresses. The residual stress levels in general increase with decreasing temperature. On the other hand, the strength of many polymers used as matrix materials for CFRP plies strongly increase with decreasing temperature, thus inducing an opposite effect of temperature.

In order to collect the pre-existing knowledge on these effects, an extensive literature survey has been performed, considering a total of 91 publications on thermal effects in composites, concerning material stiffness, strength and toughness in the cryogenic regime, failure mechanisms, characterization procedures and experimental techniques in the cryogenic regime as well as pre-existing failure criteria. Based on the outcome of the literature study and also considering the availability in standard finite element pro-

grams, the criteria proposed by Tsai and Wu, Hashin as well as by Puck have been selected as the most promising options for failure prediction in the cryogenic regime. Based on the results of the first test campaign on specimen level (Sec. 1.3), the range of the failure criteria was down-selected to Puck's criterion.

Puck's failure criterion separates the failure assessment into the assessment against five failure modes, including tensile fiber breakage, fiber instability and subsequent failure under compressive loads, matrix tensile failure, matrix shear failure and compressive failure of the matrix occurring in a wedge-like shear mode (Fig. 1). Based on the definition of a critical failure plane, a separate criterion is provided for each failure mode, resulting in the combined failure envelope presented in Fig. 1. Puck's criterion has the advantage to be based directly on the underlying failure mechanisms rather than providing a descriptive failure envelope only. Furthermore, the mathematical definition of the failure envelope in stress space allows for an adaptation to a wide range of experimental data, also enabling limited changes in the shape of the failure envelope. The failure envelope has to be determined for every temperature under consideration separately.

1.3 Experimental validation on specimen level

For assessment of the applicability of Puck's criterion under ambient and cryogenic temperatures as well as in the intermediate range, an experimental program on standard test samples has been performed. In order to account for a wide range of materials, three different reference materials have been selected, involving epoxy and thermoplastic PEEK matrices as well as high strength intermediate modulus and high modulus carbon fibers. The material has been characterized under quasi-static conditions considering ambient temperatures as well as cryogenic temperatures at 4.2 K by testing in a liquid Helium environment. In addition, a number of specimens was thermally cycled by exposure to the liquid Helium environment without mechanical load and a subsequent mechanical characterization at ambient temperature in order to separate the effect of possible micro cracking due to the cryogenic environment from the effect of mechanical loading. Part of this specimen was investigated by X-ray computed tomography prior and after the exposure to the liquid Helium environment.

The experimental characterization has been performed in two test campaigns. The first test campaign mainly consisted of standard tensile, compressive and shear experiments within and perpendicular to the fiber direction, thus providing a standard material characterization. In the second test campaign the focus was laid on more sophisticated, yet still simple experiments such as off-axis tensile and compressive experiments as well as angle-ply laminates, both with variable fiber angles. After completion of the first test campaign, an intermediate assessment and down-selection of the considered failure criteria has been performed.

In Fig. 2 an example of the non-destructive inspection of the thermally cycled specimens is presented. In this as well as in all other cases, no micro cracking due to the thermal cycle has been detected, except for one specimen which featured an initial presence of voids. Nevertheless, even in this case, no void growth due to the thermal loading has been detected. This result coincides with the observation of similar stress-strain-curves obtained in the experiments on thermo-cycled specimens and on specimens tested under similar conditions in the as-received state (Fig. 3).

Based on the results of the uniaxial material characterization under different conditions (example in Fig. 4), the chosen failure criteria were applied to the preliminary data base on all three materials (Fig. 5). It was found that - due to the pronounced differences in the tensile and compressive strengths of the reference materials perpendicular to the

fiber direction, the Tsai-Wu failure envelope either tends to over-shooting of the suspected integrity range or - when using a more conservative adaption of the parameters as in Fig. 5 - to pronounced over-conservatism in some ranges. In the same manner, the Hashin criterion was found to provide results which are not supported by the data. Thus, Puck's criterion was selected for the further investigation.

Since several ranges of the failure envelope were not provided with experimental data in the preliminary data base (Fig. 6), these ranges were provided with more data points in the second test campaign on specimen level. Subsequently, the parameters defining the Puck failure envelope were re-adapted, resulting in the more accurate failure envelopes presented in Fig. 7. In this context, Puck's criterion was found both, flexible in terms of its possibilities for adapting the shape of its failure envelope to the data and accurate in reproducing the experimental findings. The basic type of failure envelope was found to be similar in the ambient and cryogenic temperature regimes. Nevertheless, the change in temperature from ambient temperature down to 4.2 K did not only result in a self-similar change in the size of the failure envelope but also involved some changes in its shape, especially on the compressive side of the envelope in the inter fiber (σ_{22} - σ_{12} -) plane.

1.4 Breadboard tests and evaluation

In order to verify the selected criterion on a sort of structure involving more complex local stress states, experiments on small breadboards were performed. In order to allow the testing in a liquid Helium environment, designs had to be used which could be tested in a standard tensile test setup. The breadboard specimens were provided with different features inducing local stress concentrations, involving a hole (e.g. bolt hole), a tapered section with a larger radius as well as a combination of both in order to study possible interaction effects. The employed geometries are presented in Fig. 8.

The breadboards were tested under both, ambient and cryogenic conditions. For assessment of the local strains, all demonstrators were equipped with strain gauges according to Fig. 8 and Fig. 9. For the breadboard tested under ambient temperatures, a calibrated video of the tests was taken in order to identify the positions of first failure and to correlate the instant of their occurrence with the overall force-displacement response. For evaluation of the local stresses on ply level, all experiments were simulated with the finite element method (Fig. 10). The finite element results then were evaluated with respect to the local stress state triggering the observed first ply failure.

In the experiments, it was observed that failure of the demonstrators consisting of an angle ply laminate at ambient temperature in most cases occurred by an inter-fiber failure of the individual plies, triggered at positions at the hole or notch, where the local fiber direction was the tangential direction to the respective feature. Although this position was not necessarily the position featuring the highest stress level, it obviously provided a weak spot on the material side. The development of complete failure subsequently required the delamination of large ply interface areas (Fig. 11, also Fig. 13).

For the demonstrators tested in the cryogenic regime, different failure modes were observed. Although even here, inter fiber failure was observed as the dominant mode of failure, in general smaller failure regions were observed. Furthermore, the positions of final failure were predominantly found in the small cross sections of the specimens, i.e. closer to the notch roots (Fig. 12). This effect is probably caused by the higher inter fiber compressive and shear strength of the reference materials under cryogenic conditions. In some cases, the failure was triggered in an inter fiber mode, however, final failure was found to involve also fiber breakage.

1.5

Verification and design guidelines

The experiences made during the test campaign and assessment of the different failure criteria have been used for the formulation of guidelines regarding the application of Puck's failure criterion. The guideline involves three main tasks. At first, suggestions for a possible test program for parameter identification are made, considering two degrees of accuracy provided by either a minimum test program or an enhanced program. The second step is the definition of the failure envelope based on the experimental data. Finally, suggestions are made for the structural analysis of the component to be assessed, especially the required output of the finite element analysis and its subsequent processing.

The verification guideline is complemented by a guideline for the design of laminates for application in the cryogenic regime. It comprises an analysis of the load cases to be considered, a procedure for the stepwise design of the laminate involving a preliminary determination of the required fiber orientations and the stacking sequence and finally the detailed verification and possible procedure for re-design and optimization.

1.6

Conclusions

Puck's criterion allows an integrity assessment of laminate structures on single ply material level. As prerequisite, the criterion requires a finite element analysis of the stresses on ply level which are then used as an input for the integrity analysis. The integrity analysis is performed as a pure post-processing analysis using the stresses of the undamaged structure.

Following this philosophy, a numerically efficient scheme for integrity assessment is provided. The scheme provides a prediction of the instant of first ply failure, i.e. the load level or instant in loading history, where formation of the first cracks on ply level has to be expected. In the demonstrator tests, the criterion has been found to provide a slightly conservative prediction of the development of first damage. Nevertheless, at the instant of the development of the first damage events as detected by optical inspection, no pronounced loss in the overall stiffness indicating a significant structural implication of the local damage event has been observed.

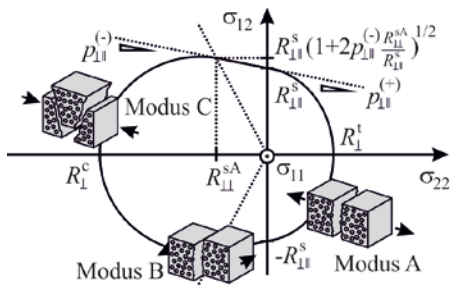
Since the criterion is based on the local stress level in the individual plies, it also accounts for thermally induced stresses, either due to anisotropic thermal mismatch between adjacent plies or due to inhomogeneous spatial and temporal temperature distributions. By this means, it is applicable throughout the entire possible temperature range, including ambient temperature and the cryogenic regime. No special treatment is necessary for thermally induced stresses since their occurrence is included on the level of structural analysis. Due to its stress-based formulation, Puck's criterion provides a criterion for assessment of brittle failure. Thus it might be considered especially useful for the cryogenic regime since failure modes tend to become more brittle with decreasing temperature. Application at any temperature level requires the determination of the material parameters for this specific temperature range.

Regarding the failure envelope at ambient temperature and in the cryogenic regime, envelopes with different sizes but similar shapes were obtained for the three reference materials considered in the present project. Nevertheless, it should be pointed out that the changes in the failure envelopes due to temperature changes might result in a change of the failure mode to be expected at the considered material point and the actual stress state. Furthermore, it should be noticed that a local first ply failure in a

specific mode may trigger total failure at this point in a different mode, i.e. inter fiber cracks with a limited size may act as triggers for fiber breakage at the respective material point due to local stress re-distribution and subsequent overloading of the fibers under continued loading although at a first glance, other positions could be suspected to be more critical.

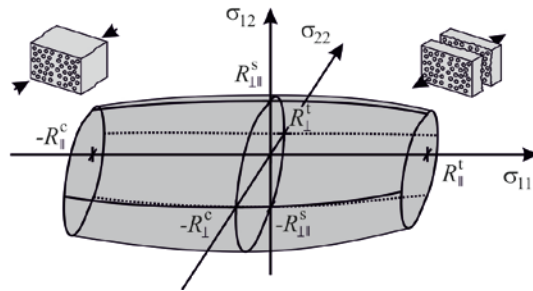
Limitations of the criterion include its restriction to first ply failure. Thus the criterion assesses only the instant of the development of first damage. It does not account for possible remaining load carrying capacity due to delaminations necessary for failure of adjacent plies with different orientations. Potential enhancements to circumvent these problems may consist in the application of damage mechanics type material models allowing for the modelling of damage effects till final material failure by means of the material model and thus during the structural (finite element) analysis of the component under consideration. By this means, a simulation of the progressive failure process and thus a direct assessment of the remaining load carrying capacity at all stages of failure would become possible.

2 Appendix: Figures



© J. Hohe, Lecture notes Composites I, II, Siegen University

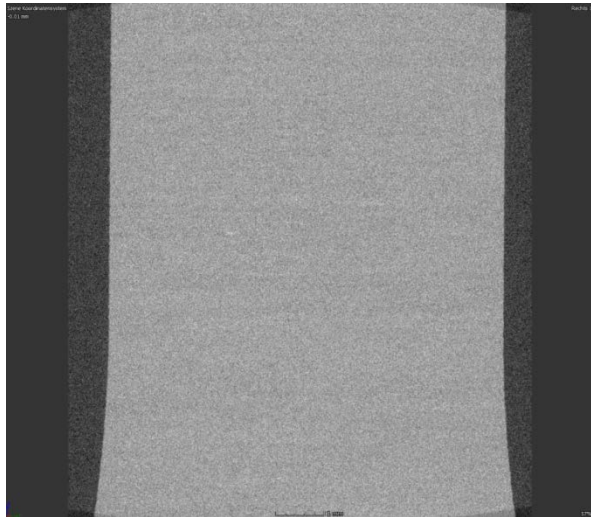
(a) inter-fiber failure envelope



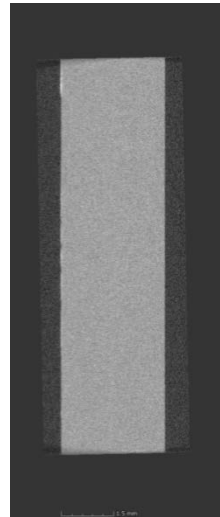
© J. Hohe, Lecture notes Composites I, II, Siegen University

(b) complete plane stress failure envelope

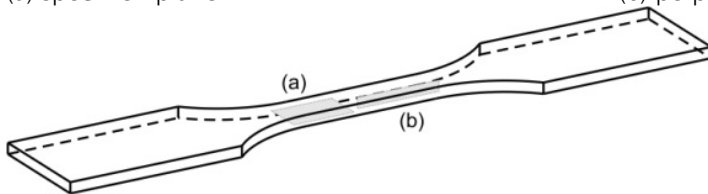
Fig. 1: Puck criterion, failure envelope and basic failure modes for plane stress states.



(a) specimen plane

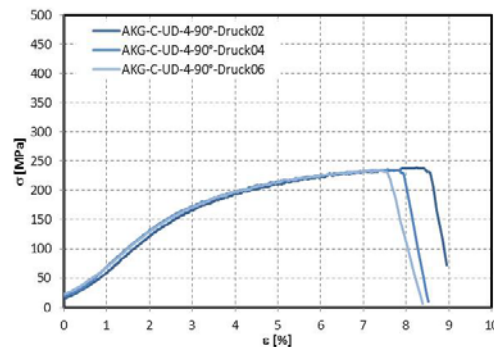


(b) perpendicular to specimen plane

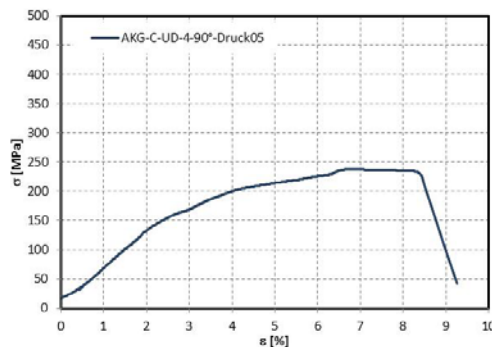


(c) section orientation

Fig. 2: Non-destructive inspection using X-ray computed tomography (Material C).



(a) as received



(b) thermocycled

Fig. 3: Effect of thermocycling (Material C).

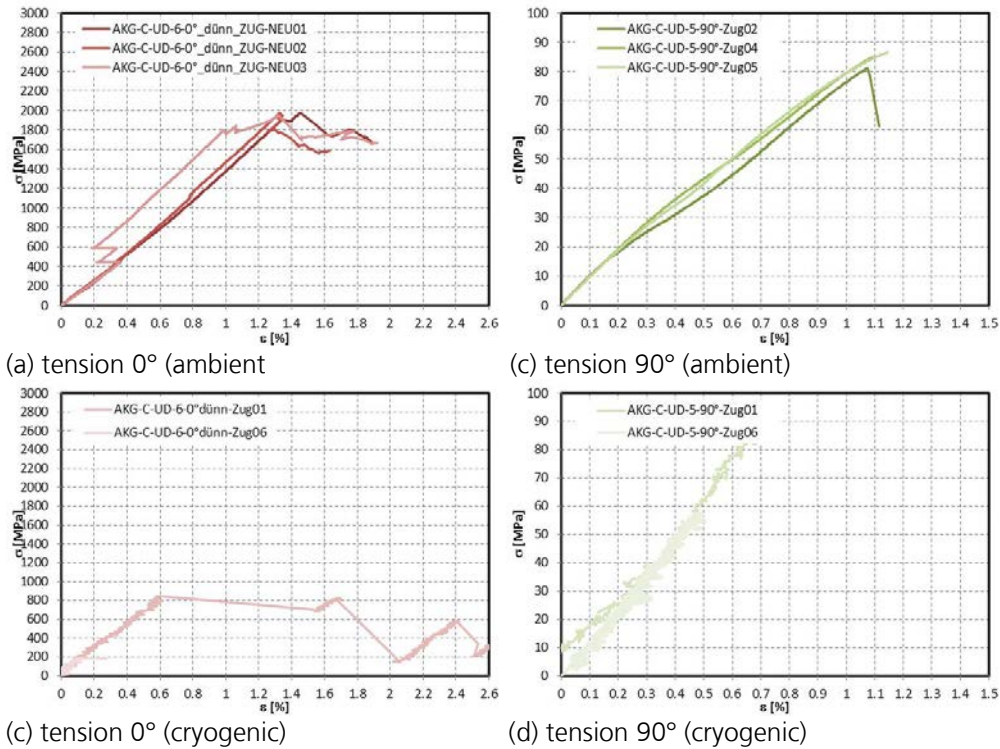


Fig. 4:
Uniaxial characterization
(Material C).

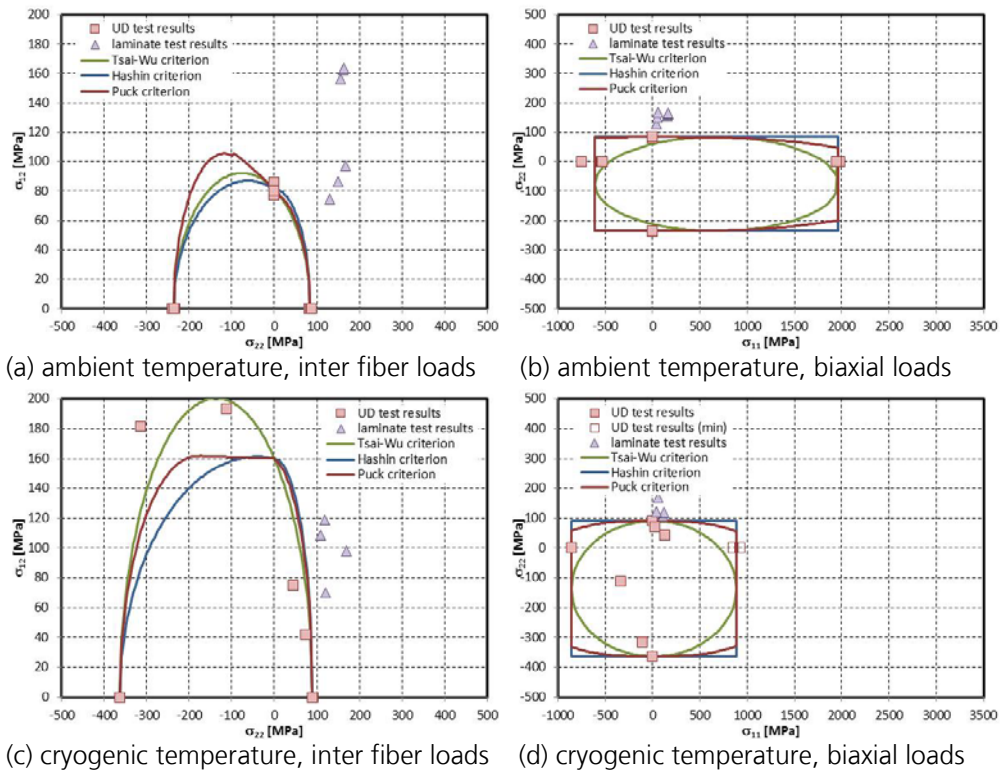


Fig. 5:
Application of selected
failure criteria
(Material C).

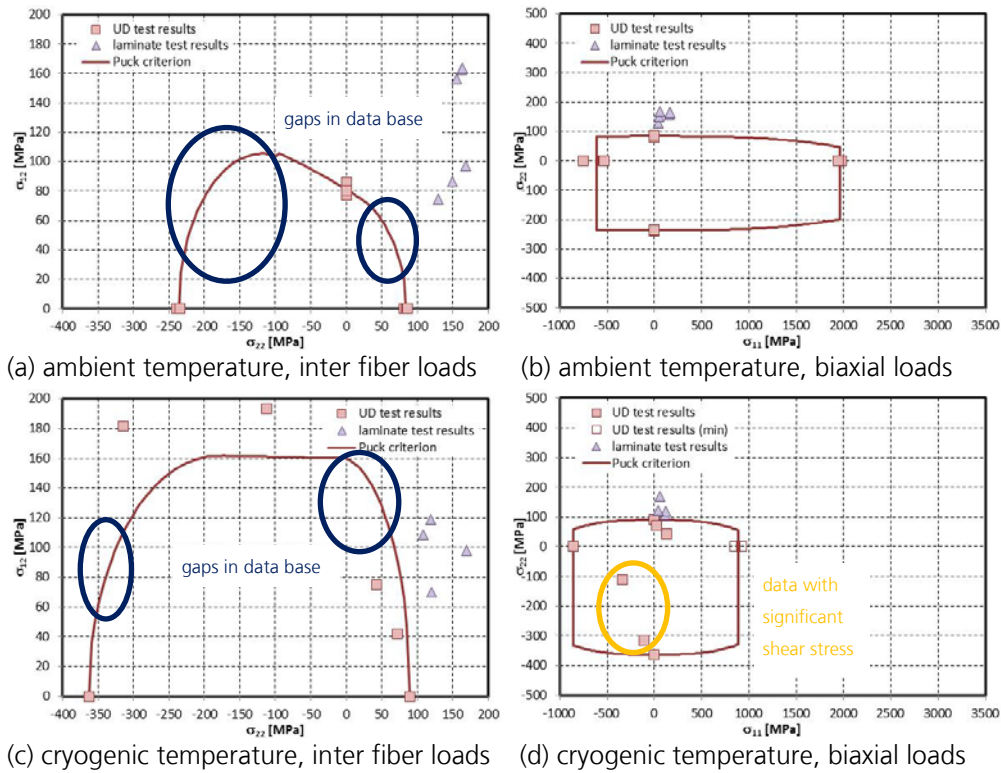


Fig. 6: Shortcomings of data base for Puck's criterion (Material C).

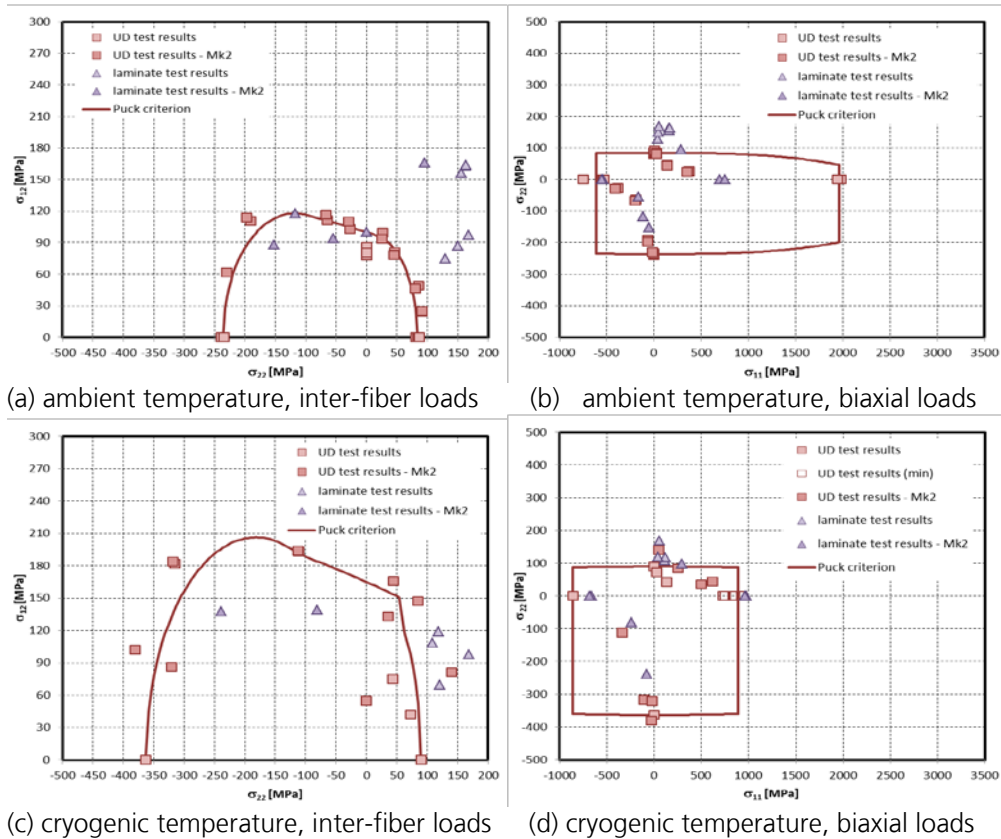


Fig. 7: Application of Puck's failure criterion based on enhanced data base (Material C).

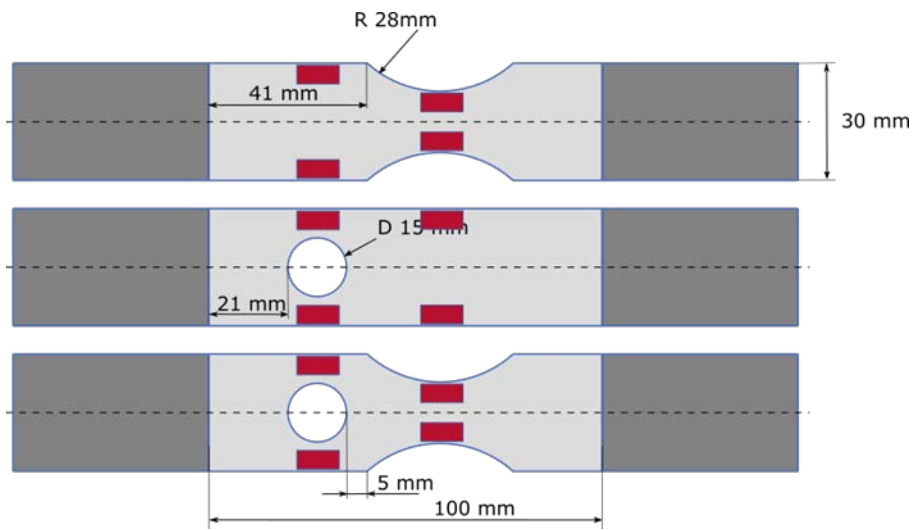


Fig. 8:
Breadboard design and instrumentation.

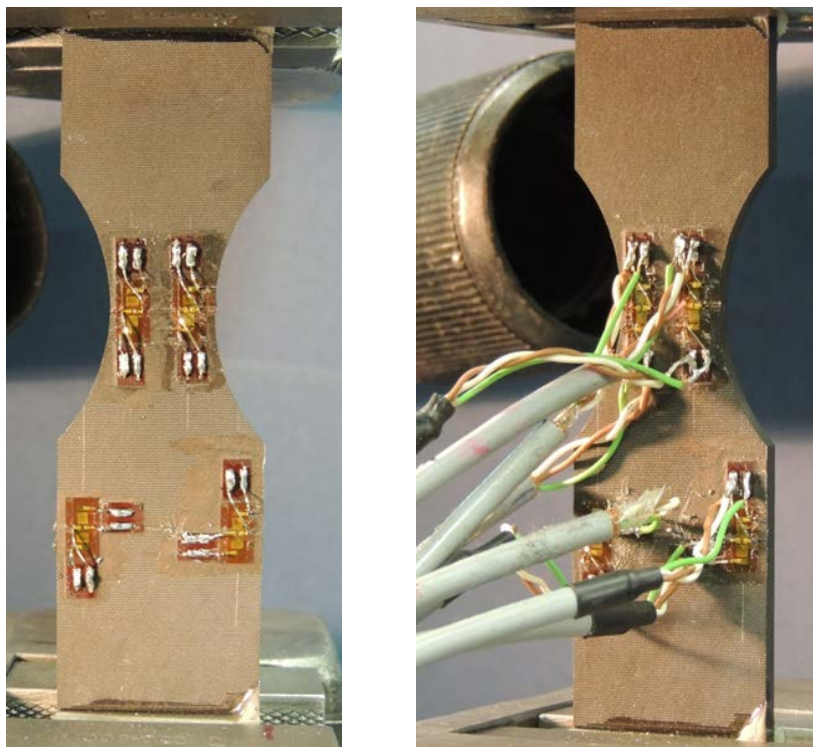


Fig. 9:
Breadboard instrumentation.

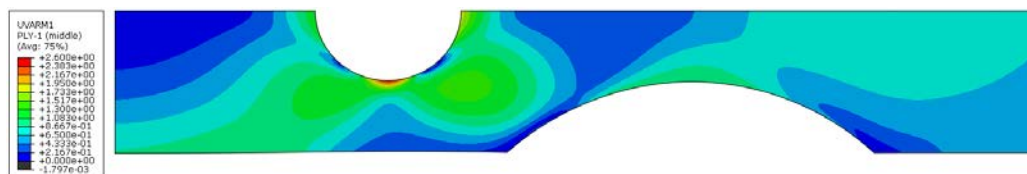


Fig. 10:
Evaluation of local stress states using the finite element method.

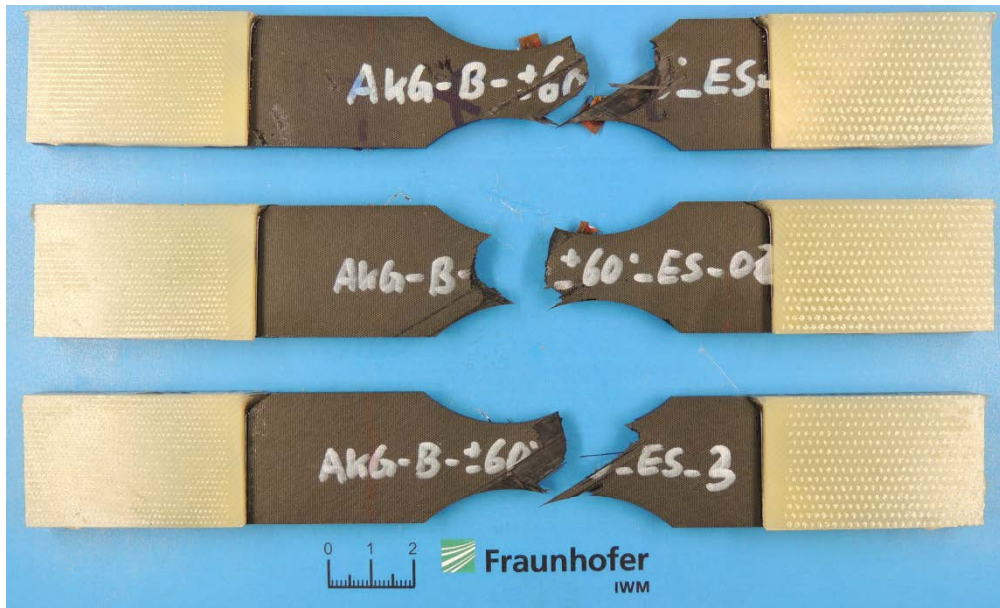


Fig. 11: Failure mode of demonstrators at ambient temperature.

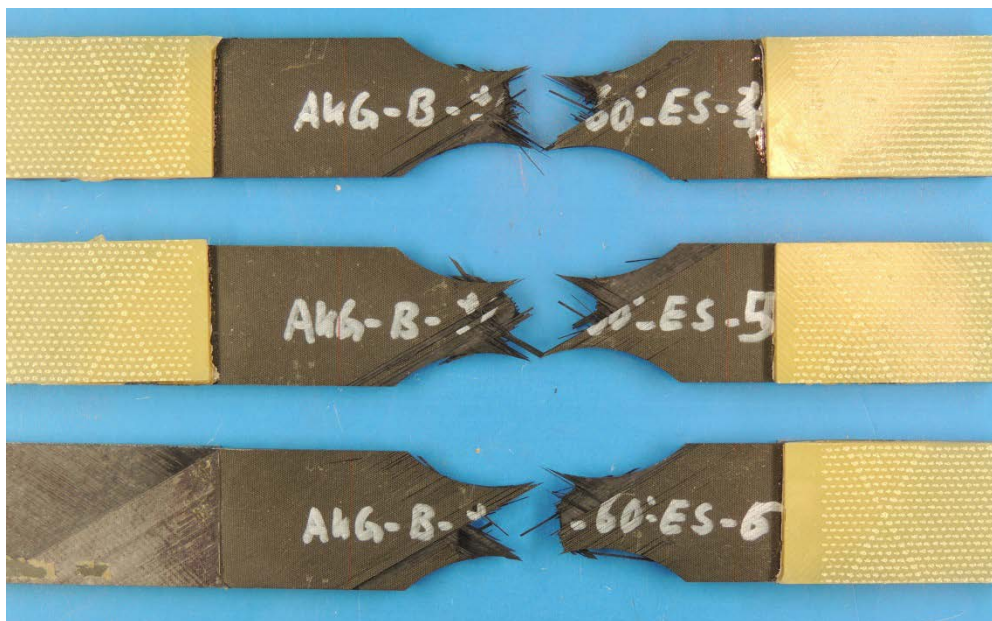


Fig. 12: Failure modes of demonstrators in the cryogenic regime.

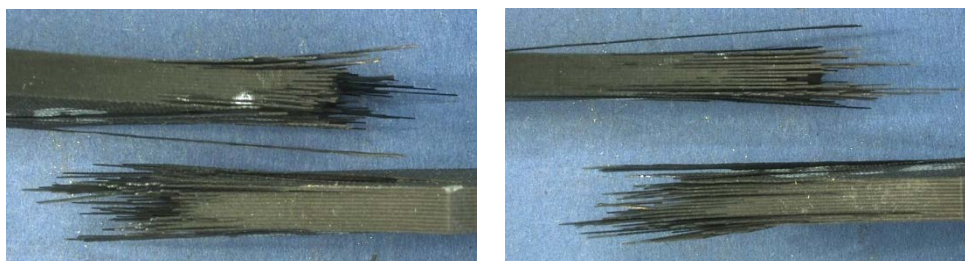


Fig. 13: Delamination of demonstrators.