INFLUENCE OF CONDITIONING TIME AND WET CHEMICAL SURFACE TREATMENT ON AIRCRAFT REPAIR

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Abstract

Two main aspects with high relevance for bonded composite repairs will be addressed in this paper. Firstly, the impact of the conditioning time under aircraft-relevant ambient conditions on adhesively bonded repairs is investigated. For this purpose, repair-specimens with a scarf ratio of 1:30, repaired with a soft patch repair approach, as well as unrepaired reference-specimens are used. Conditioning is carried out in a de- and anti-icing fluid (Kilfrost ABC-3) at room temperature, at 70 °C / 85 % relative humidity (hot/wet conditions) and in a hydraulic fluid (Skydrol LD-4) at 70 °C. Furthermore, dried specimens are tested as well. Quasistatic tensile tests are conducted at room temperature and at 70 °C, respectively. Secondly, an alternative method for the surface preparation is used during the repair process, besides the common sanding procedure. This alternative method is intended to alter the surface energy as well as its chemical composition by a combination of a corona with a wet chemical treatment introducing a functional silane to the respective surface. The tensile strength, the fracture and the moisture absorption behavior for specimens produced with both surface preparation methods in dependency of the ambient conditions will be discussed. In order to investigate the influence of the conditioning duration, these results will be compared to a previous study.

1. Introduction

The fact that the civil aircraft industry extensively uses carbon fiber reinforced polymers for primary as well as secondary structures and consequently needs repair methods for these, especially methods to perform bonded repairs, is well documented in literature [1–7]. This paper poses the second part of work that has been published previously at the 21st International Conference on Composite Materials (ICCM-21, 2017, Xi´an, CN) by the authors [8]. Within this initial paper the strength of repairs, the according failure modes as well as the influence of a chemical surface pre-treatment developed in previous studies [9, 10] on these, under environmental conditions encountered by aircraft [11, 12], were analyzed. Within this study, no influence of the selected environmental conditions could be observed. Consequently, the current work is intended to characterize the influence of prolonged

environmental conditioning under harsh conditions on aircraft repairs, also with the chemically modified surfaces in comparison to the common abrasive surface pre-treatment method. Therefore, the conditioning time in Kilfrost (a de- and anti-icing fluid; conditioning temperature: room temperature) was extended from 1 to 7 weeks, in Skydrol (a hydraulic fluid; conditioning temperature: 70 °C) from 6 to 12 weeks and under hot wet conditions (70 °C / 85 % r. h.) from 7 to 14 weeks.

2. Experimental

As stated in the previous section, this paper is an extension of previous work presented at the ICCM-21 [8]. Consequently, the experimental section will only give a brief overview of the materials and methods used.

2.1. Materials and specimen preparation

All tests were performed on autoclave-cured specimens (180 °C, 2 h, 6.6 bar) produced from epoxy-based, woven carbon fiber reinforced prepreg material with the following stacking sequence: $[45/0/-45/90]_S$. Two types of specimens were used, repair specimens produced according to DIN EN 6066 [13] as well as reference-specimens with similar dimensions (280 mm length, 25.4 mm width) manufactured from the virgin laminate. Repair-specimens were produced using a soft patch repair approach with a scarf ratio of 1:30 (scarf angle 1.9 °) using an epoxy-based film adhesive incorporating a polyester carrier. The surface functionalization was carried out on the tapered laminates before the repair process was initialized. Firstly the tapered surfaces were cleaned with 2-propanol provided by Carl Roth (Karlsruhe, DE) and corona-treated (Laboratory corona station PG 3001, Ahlbrandt System, Lauterbach, DE). Secondly, a functional silane (3-(2,3-Epoxypropoxy)propyl)trimethoxysilane purchased from Wacker Chemie (München, DE) was applied to the respective surfaces for 24 h followed by 3 rinsing steps with tetrahydrofuran (Carl Roth, Karlsruhe, DE) and intermitting drying with compressed carbon dioxide. Subsequently the surfaces were dried at 70 °C for 60 min. The stacking sequence of the repair plies as well as the cure cycle were the same as for the virgin laminate. Individual specimens were cut from the repaired and virgin laminate plates by a water-cooled circular saw equipped with a diamond-coated disk (Diadisc 5200, Mutronic Präzisionsgerätebau, Rieden, DE). Tapered end tabs were bonded with Loctite Powerflex Gel (Henkel, Düsseldorf, DE) to the individual specimens after conditioning, immediately before tensile testing.

2.2. Specimen conditioning and tensile testing

Repair- as well as reference-specimens were conditioned in accordance with ASTM D5229 [14] in Kilfrost ABC-3 (Kilfrost Limited, Haltwhistle, GB), a de- and anti-icing fluid at room temperature (RT) for up to 7 weeks, in Skydrol LD-4 (Eastman Chemical B.V., Capelle aan den Ijssel, NL), a hydraulic fluid, at 70 °C for up to 12 weeks and under hot/wet conditions (70 °C / 85 % r. h.) for up to 14 weeks (Climate chamber CTC256, Memmert, Schwabach, DE). Specimen drying was performed in a heating chamber on all specimens for at least 4 days at 70 °C prior to conditioning. Specimens in dried state tested at RT were dried for 4 days; the ones tested at 70 °C were dried for the 4 days as well as for 7 weeks, respectively.
Iterative mass measurements were conducted in order to assess the specimens´ mass gain and consequently calculate the relative moisture content ΔM following equation [14]:

$$\Delta M, \% = \frac{(W_i - W_b)}{W_b} \times 100 \quad (1)$$

With W_i, the current specimen mass, g and W_b, the baseline specimen mass, g after 4 days of drying at 70 °C.

The specimens were removed from conditioning, wiped off with lint-free wipes and stored in a sealed glass container to prohibit influences from the lab´s ambient conditions. Immediately after the weighing procedure, the specimens were put back for further conditioning. Regarding the conditioning in Skydrol at 70 °C it has to be noted that the moisture absorption could not be evaluated due to the low moisture uptake [8].
Quasistatic tensile tests were performed according to DIN EN 6066 [13] at either RT or 70 °C on a universal tensile/compression testing machine (Z250, Zwick, Ulm, DE) with a test speed of 2 mm/min. A load cell with a load bearing capacity of 250 kN was used. The load introduction was achieved with wedge-screw grips designed for 250 kN maximum load. The initial distance between the wedge-screw grips was set to 160 mm. The tensile tests were performed at RT and 70 °C, respectively. For 70 °C testing, specimens were held at this temperature for a settling period of 10 min per specimen in the heating chamber installed at the test machine.
For the calculation of the tensile strength, the maximum load was divided by the thickness of the parent laminate and the width of the specimens (mean of three individual measurements per specimen). A digital single-lens reflex camera equipped with a standard lens (EOS 600D and EF-S 18-135mm f/3.5-5.6 IS, Canon Inc., Tokyo, JP) was used for the pictures of the fracture patterns.

3. Results

Figure 1 shows the relative moisture content as a function of the time of exposure in Kilfrost for repair- and unrepaired reference-specimens. After 7 weeks of exposure, both specimen types absorbed approximately twice the amount of moisture when compared to exposure for one week. After one week the repair-specimens´ moisture content reached 0.25 % [8], after 7 weeks 0.58 %. The reference specimens absorbed 0.22 % of moisture [8], after 7 weeks 0.53 %. The repaired specimens absorbed a higher amount of moisture; the type of surface modification did not influence the moisture absorption behavior.
Immersion in Kilfrost for 1 week did not lead to a decrease in tensile strength compared to the dried specimens (see Figure 2). Despite the fact that the specimens´ moisture content was increased substantially by the extension of the exposure time in Kilfrost, the tensile strength remained constant within the standard deviation ranges at 89 ± 4.8 % (functionalized) and 89 ± 6.1 % (sanded) compared to the 1 week exposure. After the extended immersion time, all the specimens failed by fiber failure in the taper-region (Figure 3), which is a similar result to the previous study [8].

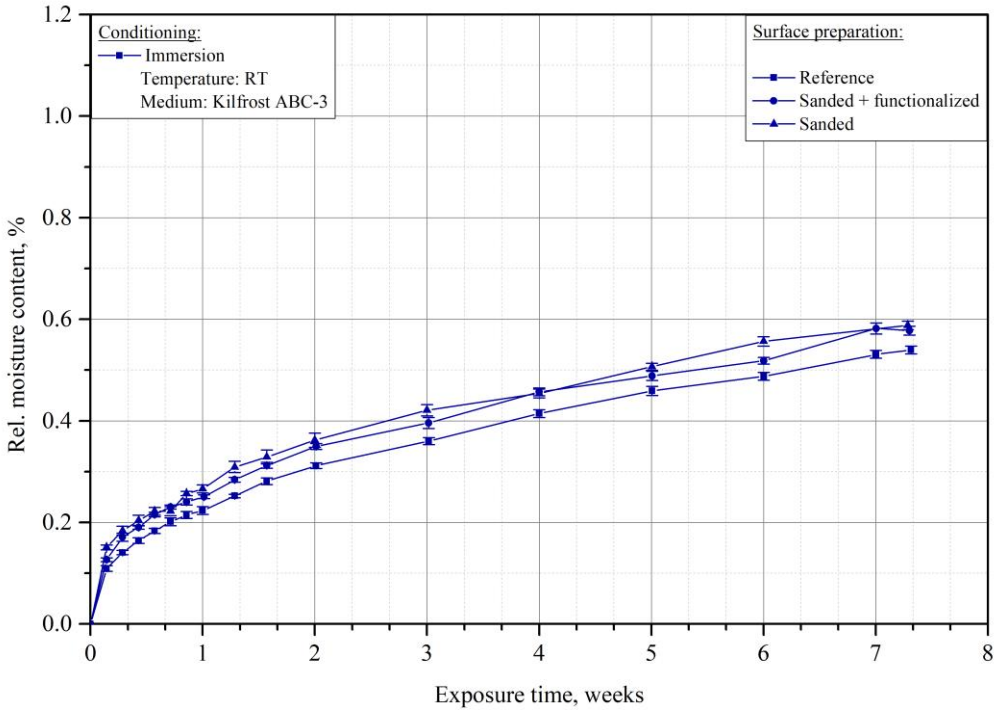

Figure 1. Moisture absorption and standard deviations of the reference- and repair-specimens for conditioning in Kilfrost at RT (mean of 8 specimens). Data until 1 week conditioning from Roeper et al. (2017) [8].

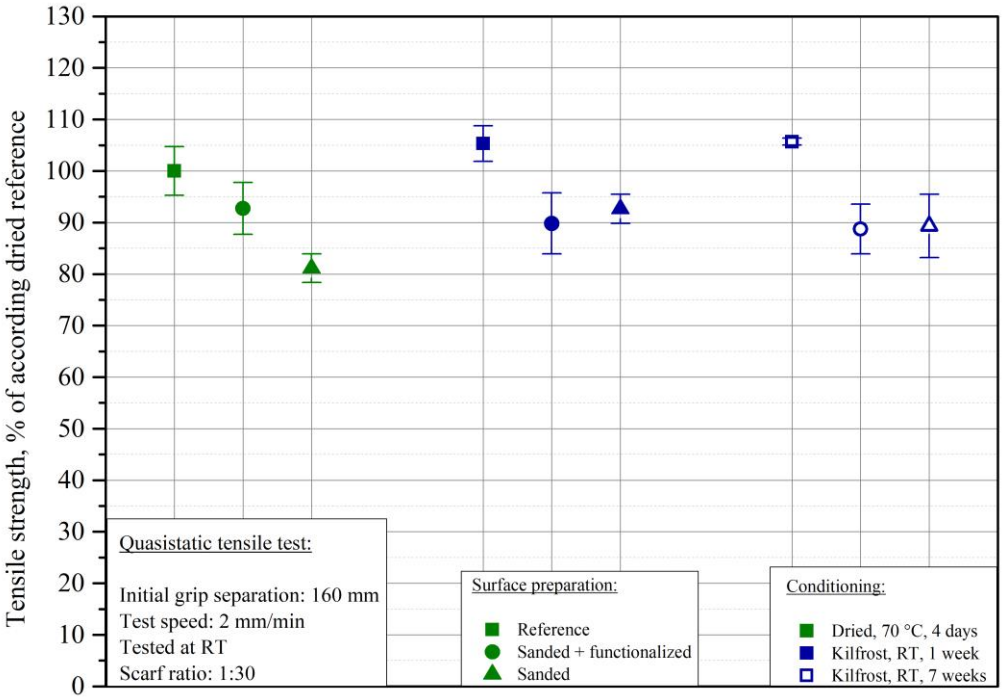

Figure 2. Influence of the environmental conditions on the mean relative tensile strength (normalized to the unrepaired, dried reference) and standard deviations of the reference- and repair-specimens (tested at RT) with sanded as well as functionalized surfaces. Data of the dried specimens and for 1 week conditioning in Kilfrost from Roeper et al. (2017) [8].

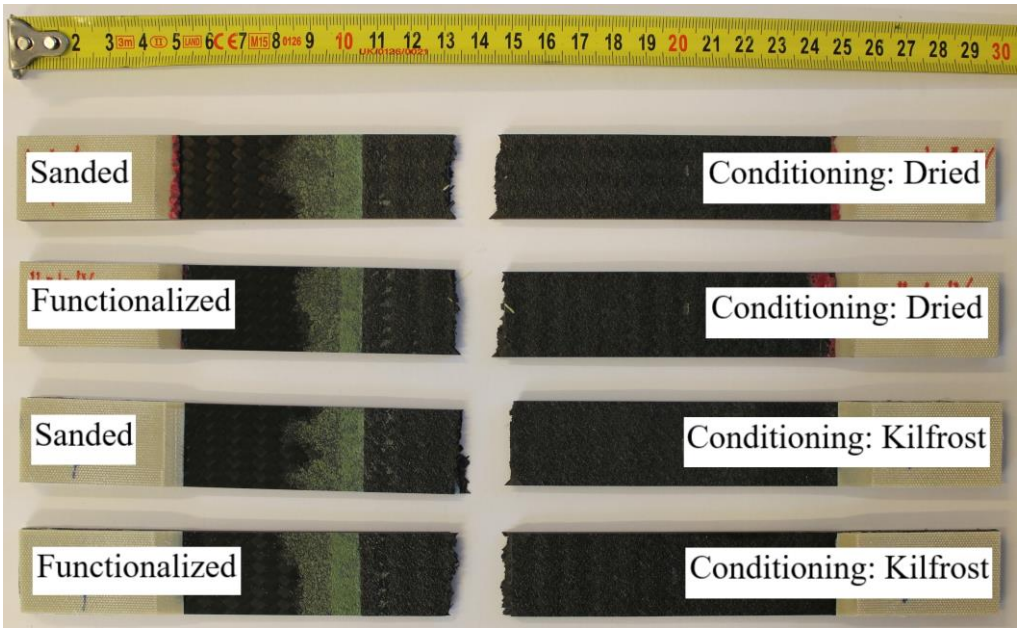

Figure 3. Failure modes (exemplary) of dried repair-specimens and after immersion in Kilfrost for 7 weeks, tested at RT.

According to Figure 4 the relative moisture content did not rise significantly between 7 weeks of exposure to hot/wet conditions (repair-specimens 1.04 %, reference-specimens 0.98 %,) and 14 weeks (repair-specimens 1,06-1,08 %, reference-specimens 1.03 %). Similar to conditioning at RT, the repaired specimens absorbed a higher amount of moisture than the unrepaired reference-specimens. Again, the amount of moisture absorbed was independent of the surface preparation technique.
The increased test temperature (RT to 70 °C) as well as the increased drying period (from 4 days to 7 weeks) did not affect the tensile strength of the dried repaired as well as of the unrepaired reference-specimens as depicted in Figure 5. The tensile strength of the unrepaired specimens remains constant at 100 ± 5.6 % (4 days drying) [8] and 100 ± 3.1 % (7 weeks of drying). The dried repair-specimens´ tensile strength after 4 days of drying reaches 85 ± 5.8 % (functionalized) and 88 ± 2.5 % (sanded) [8]. The specimens dried for 7 weeks show similar results (88 ± 4.6 % for the functionalized and 86 ± 3.9 % for the sanded specimens). Immersion in Skydrol for 6 weeks did not significantly affect the tensile strength of the reference-specimens (102 ± 5.7 %) or the functionalized (89 ± 2.4 %) as well as the sanded (91 ± 3.1 %) repair-specimens [8]. In comparison to these results, the tensile strength after extended immersion for 12 weeks remained at a constant level (103 ± 5.0 % for the unrepaired and 90 ± 2.1 / 88 ± 1.7 % for the repaired samples). Similar results were achieved with the hot/wet-conditioned samples for 7 weeks and for 14 weeks. After 7 weeks the tensile strength of the reference-specimens showed a decrease within the standard deviation range of the dried samples to 97 ± 1.0 % [8], after 14 weeks 101 ± 4.5 % were measured. For the repaired specimens with functionalized surfaces the tensile strength was found to be at 84 ± 4.3 % after 7 weeks [8] and at 89 ± 3.2 % after 14 weeks. The results for the sanded specimens were comparable, 86 ± 3.2 % (7 weeks exposure) [8] and 87 ± 1.5 % (14 weeks exposure). Figure 6 exemplarily shows the failure modes of the specimens tested at 70 °C. All the respective specimens failed by fiber fracture. Due to this type of failure, no significant differences in tensile strength between the two methods of surface preparation technique exists for all conditions and conditioning durations since the respective interfaces did not fail.

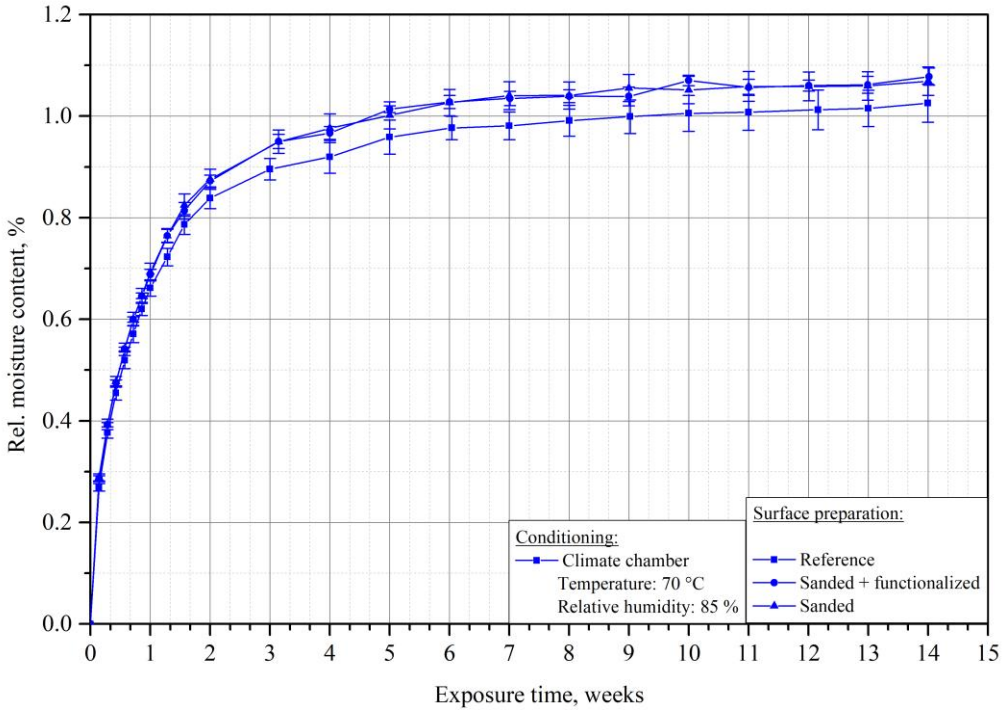

Figure 4. Moisture absorption and standard deviations of the reference- and repair-specimens for conditioning under hot/wet conditions (mean of 8 specimens). Data until 7 weeks conditioning from Roeper et al. (2017) [8].

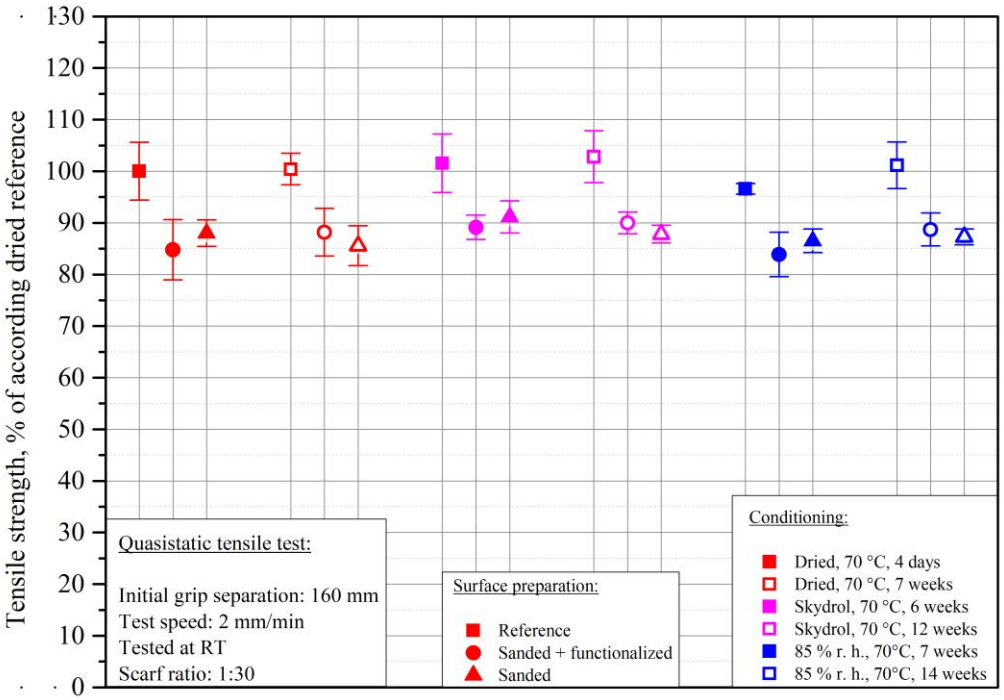

Figure 5. Influence of the environmental conditions on the mean relative tensile strength (normalized to the unrepaired, dried reference) and standard deviations of the reference- and repair-specimens (tested at 70 °C) with sanded as well as functionalized surfaces. Data of the dried specimens (4 days drying), for 6 weeks conditioning in Skydrol and for 7 weeks under hot/wet conditions from Roeper et al. (2017) [8].

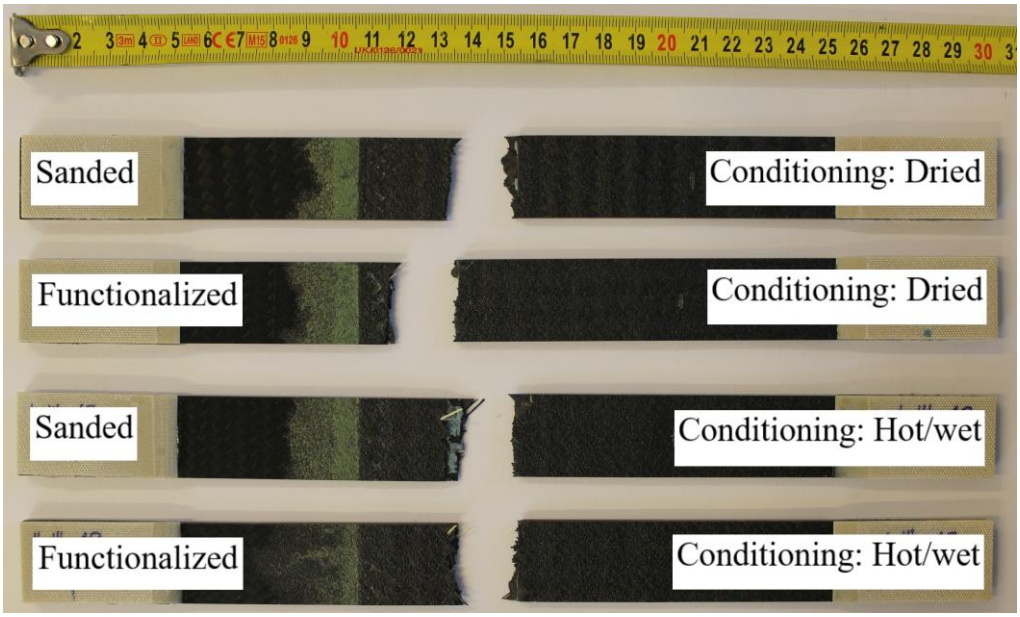

Figure 6. Failure modes (exemplary) of dried repair-specimens and after hot/wet conditioning for 14 weeks, tested at 70 °C.

3. Conclusions

Within this paper the influence of extended conditioning periods under aircraft relevant environmental conditions, specifically of a de- and anti-icing fluid (Kilfrost ABC-3), a hydraulic fluid (Skydrol LD-4) as well as of hot/wet conditions (70 °C / 85 % r. h.), on the tensile strength as well as on the failure modes of aircraft repairs were reviewed. Tensile tests have been conducted on repair- as well as on unrepaired reference-specimens at RT and 70 °C, respectively. Dried specimens have been tested at the respective temperatures as well. Additionally, the influence of two methods for surface preparation within the repair process, sanding and a corona combined with a wet chemical functionalization technique, have been investigated.
In summary, an influence neither of the exposure conditions nor of the exposure time could be investigated for both specimen types. Since the failure mode was fiber failure in all cases, the influences of the two different surface modification techniques could not be studied. Therefore, a second study concerning the influence of selected environmental conditions on repair-specimens with a steeper scarf angle has been conducted and will be published in the near future.

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