



# Improving Reliability of Bonded Composite Repair with Advanced Pretreatment and Surface Inspection Technologies

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# ABSTRACT

Technologies for the surface inspection and preparation in the context of a new composite repair process using adhesive bonding that we propose are discussed. This advanced repair process (termed SAFIRRE) aims to improve the robustness of the bonding process by using a combination of surface technologies with superior performance, higher degree of automation and advanced process control over the current state of the art. Examples for those technologies are discussed. In addition directions for future research activities to advance the bonded repair technology are indicated.

# **1.0 INTRODUCTION**

The need to reduce fuel consumption and  $CO_2$  emission has led to a strong increase in the use of carbon fibre reinforced plastics (CFRP) in aerospace structures (civil and military). Therefore efficient repair/overhaul processes for CFRP structures are a prerequisite for a favorable total life cycle cost of an aircraft. For the repair of damaged primary aerospace structures made out of CFRP special challenges exist. In areas with high demands on aerodynamic flushness a bonded patch repair is the most suitable repair solution in many cases. In contrast to riveting a bonded repair does not disrupt the integrity of the structure by hole drilling and it provides a favorable strength-to-weight ratio. However, special attention must be paid to the robustness and the durability of the bonded repair to guarantee the airworthiness of the repaired structure. This paper outlines an approach under development at Fraunhofer IFAM called SAFIRRE to achieve this goal.

## **1.1 SAFIRRE repair process**

The objective of this paper is to provide a vision of a composite repair process based on adhesive bonding for primary aircraft structures (as in large civil aircrafts or high-value military aircrafts) and to foster discussion&collaboration about such a process in the scientific community. We will briefly outline essential steps in this repair process and address future research that we feel is likely needed to achieve this goal.

Although bonded repair has proven excellent structural integrity in long-time survey studies, there is currently no authorized (certified) bonded repair process for primary CFRP structures of large civil aircrafts [1-3]. Some reasons for this situation are: a) there is no method for non-destructive inspection (NDI) available today to guarantee the integrity of the adhesive joint, b) in the past problems occurred because surface preparation and conditions were not adequate and could not be detected, and c) current modelling does not allow to predict "weak bonds" (adhesive failure at the interface).

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Therefore this paper will focus on the role of the composite surface and associated technologies for surface inspection and preparation in the repair process. It is argued that the control of the surface properties is paramount to establish a reliable repair process. Aspects such as damage detection, certification of materials, training of work force, joint design, curing, and post-bonding inspection are not considered in this vision. Of course, for the certification of bonded repair processes these issues need to be addressed, however. But those aspects are out of the scope for this paper.

Assuming that the surface to be bonded to is highly significant in achieving a robust repair process, we propose to eliminate the likelihood of errors (=events affecting the durability of the joint adversely) related to the surface by using technologies for surface preparation/inspection with a high degree of automation and the potential for high-quality surface treatment. Besides automation and superior performance, process control to check and validate the surface properties is thought to be the second element for a robust repair process in addition. Therefore we propose a repair process that relies on the combination of surface pretreatment/cleaning methods with surface pre-bond inspection methods. We termed this approach <u>safe</u>, <u>integrated</u>, and <u>reliable repair</u> process, abbreviated as SAFIRRE. Figure 1-1 outlines this two-key-element approach.



# Figure 1-1: SAFIRRE approach for bonded repair of composite aircrafts with focus on surface quality

In detail, we propose for the pre-bond operations a sequence of (A) contamination detection and surface quality assessment, followed by (B) adequate and corresponding surface pretreatment/cleaning based on the



results from step A, followed by (C) a surface inspection directly prior bonding to validate the effectiveness of the surface pretreatment and to ensure the quality of the surface. For the individual steps A to C it might be necessary to use a combination of technologies, although for cost reasons it is desirable to minimize the number of required technologies.

## 1.2 Technologies for SAFIRRE repair process

We are currently studying the following technologies to be used solely or in combination in the SAFIRRE process:

a) for surface inspection (step A and C)

- x-ray fluorescence (XRF) and FT-IR spectroscopy using mobile and small instruments
- optically stimulated electron emission (OSEE)
- laser-induced breakdown spectroscopy (LIBS)
- aerosol-wetting test developed at IFAM (using automated droplet detection)

b) for surface pretreatment (step B)

- atmospheric plasma
- CO<sub>2</sub> snow treatment
- vacuum blasting
- laser treatment





Figure 1-2: Examples for surface inspection and treatment technologies. (left): mobile FT-IR instrument; (right): automated CO<sub>2</sub> snow cleaning

We are presenting only selected results for these technologies to highlight some important features without aiming to provide a complete picture. Especially we like to point out the challenges in the experiments to validate the technologies for such a repair process.



# 2.0 EXPERIMENTAL DETAILS

The laminates were made from prepreg HexPly® UD/M21/35%/268/T800S from Hexcel with a nominal cured ply thickness of 0.262 mm. The laminates were cured at 180 °C for two hours. A film adhesive Hysol EA 9695 .050 PSF NW from Henkel with a 121 °C cure was used. Mechanical testing for Fig. 3-1 employed a test in close agreement with the withdrawn DIN EN 6066 defining a specimen for a tensile test of a scarfed bonded joint. The bonded joint was made of two already cured and scarfed laminates and the adhesive film subjected to tensile tests. This approach allows to concentrate on the effect of contaminations and their removal by proper surface pre-treatment by looking at the base material only. The reference samples in this figure have a surface as it was obtained after mechanically scarfing followed by an organic solvent wipe. The so prepared specimens were also the starting point for contamination procedures and subsequent pretreatment processing. The scarf ratio of the adherents was about 1:16. Results shown in Fig. 3-5 were obtained by standard lap shear testing. As the results show, moisture can have a significant effect on bond strength. For all bonding experiments presented in this paper we did not measure or control specifically the so-called pre-bond moisture content of the specimens. The specimens were processed as-received and as-stored. Under our lab conditions we expect little variance of moisture content between the samples. But obviously, more precise control of moisture content in such experiments is preferred.

The mobile XRF unit was a Xl3t from Niton (ThermoFischer Scientific). For surface pretreatment the following tools and corresponding process parameters were used (Table 2-1).

pretreatment tool	process parameters
atmospheric plasma (AP): plasma jet PFW10 from Plasmatreat GmbH (Germany)	process gas: air; treatment speed: 10 m/min; distance to substrate surface: 6 mm
laser: Nd-YAG laser Clean CL 250 from Clean- Laser System GmbH (Germany)	pulse frequency from 10 to 40 kHz, maximal pulse energy of 23 mJ and the pulse width about 100 - 300 ns
CO <sub>2</sub> cleaning: snow blasting machine SJ-25 from CryoSnow GmbH (Germany)	treatment speed: 10 m/min; distance to substrate surface: 5 cm
vacuum blasting: machine from GP Anlagenbau (Germany)	blast material: corundum EKF100 with a typical particle size of $150 \ \mu m$

#### Table 2-1: Pretreatment tools and parameters

# 3.0 RESULTS AND DISCUSSION

The model scenario for repair in our research assumes a damage in a monolithic epoxy-based CFRP laminate structure that is repaired with an adhesive patch using a scarfed geometry. Contaminations inside the laminate and on top of the surface are likely but unknown.

## 3.1 Influence of contaminations on bond strength

For the selection of the required surface inspection technologies and in case that it is not possible to remove



contaminations fully from a surface it is important to understand which contaminations at what concentration affect bond durability. The major challenge in such lab experiments is to quantify the contamination and its distribution across the surface. A second challenge is to establish lab contamination procedures with a high degree of accuracy and precision that allow to repeat and compare experiments. Those challenges have to be overcome to specify with high reliability the requirements on both surface pretreatment and surface inspection methods.

Fig. 3-1 presents the change of bond strength when differently contaminated surfaces are bonded in comparison to a non-contaminated reference surface. Due to the above mentioned challenges we refrain from quantifying the amount of contamination. Contamination by water, hydraulic fluid, and deicer has been achieved by dipping the specimens for a defined time in the respective liquid. Hand lotion was applied manually by a brush.



Figure 3-1: Bond strength after bonding to differently contaminated surfaces. The graph also shows the influence of ageing the bonded specimens before testing. The samples with deicer show an adhesive failure mode. All other samples have a complex failure mode with a cohesive failure mode partially within the adhesive layer and the laminate. The ageing seems to affect the failure mode only slightly.

In these experiments contaminations due to a runway deicer had the most prominent effect on bond strength. As expected ageing strongly influences the bond strength. So choosing the right testing&ageing conditions is also of major importance in the assessment of the effect of contamination on bond durability.

## **3.2** Contamination detection

To evaluate the surface inspection technologies it is necessary to evaluate detection limits and to find marker signatures in the analytical signal for automated evaluation. Again a major challenge in these experiments is the preparation of samples with defined quantity of contaminations. Another challenge arises when a mixture



of contaminations exist in/on the laminate.

Fig. 3-2 shows the X-ray fluorescence (XRF)-spectra of a CFRP surface contaminated with different quantities of a hydraulic fluid (Skydrol). By using the ratio of the areas of the sulphur peak (contained in the CFRP laminate) and the phosphorous peak (only found in the hydraulic fluid) we found that it is possible to establish a linear relationship between the analytical signal and the surface contamination and to derive a detection limit for a specific laminate thickness and the assumption that no other contaminations are present.

The determination of detection limits in combination with the knowledge which concentrations of a contaminant affect bond durability is considered to be essential for reliable process control.



Figure 3-2: (left): XRF spectra of contaminated CFRP surfaces; (right): relationship between marker signal (ratio of P and S -related peak area) and the contamination concentration. A detection limit of approx. 500 mg/m<sup>2</sup> of skydrol is established under the mentioned assumptions.

## **3.3** Surface pretreatment

For the selection of the appropriate surface pretreatment methods it is necessary to evaluate their effectiveness for contamination removal. Again, here the major challenge is the analytical assessment of the contamination before and after pretreatment. Fig. 3-3 presents the removal of de-icer contamination from a surface using a  $CO_2$  snow system with a fully automated robotic system. It can be seen that more than 1 pass with the  $CO_2$  treatment is necessary to remove the de-icer (or at least the K ions which are major component of the de-icer). To establish a reliable pretreatment, process data as those are mandatory to assure a clean surface has generated. In addition, in principle, such experiments have to be performed for all levels and types of contaminations. A way to reduce the number of experiments might be to use a chemistry-based approach for clustering contaminations according to their chemical behavior and influence on bond strength and durability. In terms of repair cost and adaptation to curved aircraft surface it is also necessary to have efficient automation tools that allow for such pretreatment processes without manual interference.





Figure 3-3: Semi-quantitative level of de-icer contamination on a CFRP surface as a number of treatment passes using a CO<sub>2</sub> snow system. As analytical marker the K peak derived from a XPS analysis is used.

Similar experiments are on-going for the other pretreatment methods, but still in an early stage. Preliminary results seem to indicate that laser treatment tends to ablate material from the surface and will remove contaminations fully at sufficient intensity (fluence). In contrast atmospheric plasma treatment can not always remove contaminations fully, but modify them and make them 'adhesion-friendly'.

## 3.4 Surface state and bond strength

For the final surface inspection (step C in SAFIRRE) it is necessary to establish the (sufficient) removal of contaminations and that the surface properties (such as roughness and chemical composition) are adequate for a reliable repair. It is therefore necessary to understand how different surface states affect the bond strength and durability. Fig. 3-4 compares SEM images from an uncontaminated CFRP surface having received different surface pretreatments. It can be seen that the methods generate very different surfaces states with different extent of fiber exposure and chemical composition.





Figure 3-4: SEM micrographs of a CFRP surface having received different surface pretreatments. In addition the surface concentration of oxygen from a XPS analysis is shown. (reference has been sanded)

It is interesting to see how these different surfaces affect the bond strength. Fig. 3-5 displays the lap shear strength of non-contaminated CFRP surfaces having received the individual surface pretreatments. Due to the nature of the testing procedure we observe for all samples a cohesive failure mode within the laminate. The surprisingly moderate influence of the surface state (topology and chemistry) on the bond strength can be very likely related to the nature of the used adhesive film. The adhesive seems to be capable to build up similar adhesion forces to all surfaces. Research how adhesive chemistry and surface properties are affecting bond durability is still needed to reduce the number of experiment to fully validate such an approach. In addition, predictions on the bond durability using different ageing protocols is very difficult.



Figure 3-5: Bond strength after ageing (1000h @ 70 °C) of bonded CFRP samples having received different surface pretreatments. Lap shear testing was performed at room temperature and measured bond strength values vary by approx. ±2 MPa for each set.



# 4.0 FUTURE RESEARCH AND OUTLOOK

Bonded repair is a very promising approach for the repair of composite aircraft components that requires a thorough understanding of the durability of adhesive joints and its influencing factors. Using adequate surface pretreatment and surface inspection methods with a high a degree of automation and superior performance it should be possible to ensure a reliable repair process. We highlighted some of the challenges in the development of such a process. To achieve the objective therefore, in our view, some of the most urgent research fields and funding needs are

- development of lab contamination procedures that represent a realistic (qualitative and quantitative) scenario for the assessment of inspection and pretreatment methods
- validation of bond testing methods & protocols that are best suited to assess the quality (strength and durability) of the adhesive joint
- chemical clustering of contaminations (towards their effect on affecting bond quality) to reduce the number of experiments
- to establish in the EU a collaborative research group similar to the FAA Joint Advanced Materials and Structures Center of Excellence (JAMS) [4] in the US
- and to strengthen research cooperation between North America, Oceania and Europe of the relevant bodies.

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