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PROJECT FINAL REPORT

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4.1 Final publishable summary report

4.1.1 Executive summary

ENCOMB brought together leading experts in aeronautics research and development from ten European countries. Driven by central problems within the aeronautics industry, ENCOMB aimed to provide advanced non-destructive testing methods for reliable quality assurance of adhesive bonds in CFRP structural components.

Although composite materials are already used in the manufacturing of structural components in the aeronautics industry, an optimised light-weight design for CFRP primary structures cannot be deployed due to the lack of adequate quality assurance procedures for adhesive bonding. The successful implementation of a reliable quality assurance concept will provide the basis for increased use of light-weight composite materials for highly integrated structures. The expected weight savings for the fuselage airframe are up to 15%. The resulting reduction of fuel consumption and hence CO_2 emissions will present a major step towards the greening of air transport.

Therefore, the objective of the ENCOMB project was the development and adaptation of extended non-destructive testing (ENDT) methods for pre- and post-bond inspection of CFRP aircraft structural components in order to establish a reliable quality assurance concept for adhesive bonding.

State-of-the-art (E)NDT techniques have been screened and the most suitable ones taken forward for development and adaptation. As part of this process, five application scenarios have been identified as of primary importance for aircraft manufacturers.

Pre- and post-bond quality assessment has been based on the characterisation of adherend surfaces and adhesive bonds. In order to achieve this high quality samples have been manufactured following strict requirements regarding raw materials, manufacturing and bonding processes to ensure minimal deviation in terms of quality of the samples and enhance the reliability of the developments. The adherend surfaces and adhesively bonded samples have been characterised with conventional laboratory analysis methods and conventional NDT techniques. Extensive mechanical tests have been performed on the bonded samples to determine the influence of sample treatment on their mechanical performance.

The development, adaptation and validation of ENDT technologies for the detection of selected physico-chemical properties of CFRP adherend surfaces and characterisation of the quality of the adhesive bonds has been carried out in two steps. The first comprised a comparison of treated samples with a clean reference as a screening for principal suitability. The second step has been dedicated to optimising those technologies with demonstrated suitability by means of samples with different contamination levels down to threshold levels of insignificant impact on bond strength.

In summary, 31 technologies have been investigated and developed. For each of the application scenarios several techniques have been developed that are able to detect different contamination levels and passed the validation step. Furthermore, several techniques have been developed with good potential to comply with the requirements after further development effort.

The results have been disseminated through 56 peer-reviewed articles so far and a wide range of additional dissemination activities (e.g. conference presentations, leaflet distributions, exhibitions, press releases, newsletters, website).

4.1.2 Summary description of project context and objectives

Project context

The major challenges for the European aeronautics industry, as identified in the ACARE report of the Group of Personalities “European Aeronautics: A vision for 2020” (2001) and further addressed in the two ACARE Strategic Research Agendas (SRA, 2002, 2004), are to provide more affordable, cleaner, safer and more secure air travel. These five high level target concepts (as quoted in SRA 2) can be broken down as: Quality and Affordability, Safety, Efficiency of the Air Transport System, Security, and Environment.

Tightly connected to the environmental aspect is the challenge of structural weight. Structural weight has always been an essential issue since the beginning of aviation history. All aircraft manufacturers have searched for robust but light materials to build their planes. Today, composite materials are the most promising. They have started to become standard for a growing number of parts of aircraft structures because they are far lighter and possess higher specific mechanical properties than the aluminium alloys they replace.

Even though composite materials are already used in the manufacturing of structural components in the aeronautics industry an optimised light-weight design of CRFP primary structures cannot be readily deployed due to a lack of adequate joining technologies.

In principle, adhesive bonding is the optimum technique for joining CFRP structures. For this reason, adhesive bonding has been used in the aeronautics industry for more than 20 years. Currently, the application of adhesive bonding to primary CFRP structures is restricted to the manufacturing of not load-critical structures such as stiffened panels, e.g. co-bonded CFRP stringers on CFRP skin of a vertical tail plane (see Figure 1).

Figure 1: Placement of stringers for a stiffened CFRP skin of the A380 vertical tail plane.

However, in order to exploit the full potential of CFRP for the manufacturing of lightweight structures in the aeronautics industry there is a need to apply adhesive bonding as a joining technology to load-critical CFRP primary structures. However difficulties in assessing the bond quality by non-destructive testing (NDT) limit its use for aircraft structural assembly. In consequence, certification by the regulation authorities is restrictive.

The implementation of reliable adhesive bonding processes by advanced quality assurance is expected to lead to an increased use of light-weight composite materials for highly integrated structures minimizing rivet-based assembly. The expected weight savings for the fuselage airframe are up to 15%. These weight savings have further effects on the size and weight of the engines. From the overall weight savings, significant reductions in fuel consumption and hence CO_2 emissions per passenger-kilometer as well as reduction of the aircraft direct operating costs are expected.

In order to ensure the performance of adhesively joined load-critical CFRP structures, technologies suitable to detect adhesion properties of bonded components are required.

Quality assurance processes for adhesive bonded CFRP primary structures that are not load-critical already exists. Adhesive bonded structures are inspected by means of conventional non-destructive testing (NDT) in order to detect defects like pores, debonds or delamination in the joint area. The materials (e.g. adhesives, prepreg materials) and process parameters (e.g. surface treatment, curing) are also controlled and monitored. In addition, specimens that have been running through the complete manufacturing cycle are tested by non-destructive and destructive methods to identify systematic process failures.

However, the failure of load-critical primary structures affects the safety of the aircraft. Therefore, the requirements on the quality assurance of manufacturing processes are extremely high. Hence, a robust and reliable quality assurance process for adhesive bonding is mandatory.

In principle, the performance of adhesive bonded CFRP components depends on the intensity of operational loads to which the adhesive bond is exposed during in-service aircraft operation, the density and size of defects such as debonds, pores, delamination and the physico-chemical properties of the adhesive bond (see Figure 2). The operational, environmental and mechanical loads are considered in the structural design. Defects in the joint area can be detected by means of conventional NDT. But there are no methods available to test the physico-chemical properties of adhesive bonds.

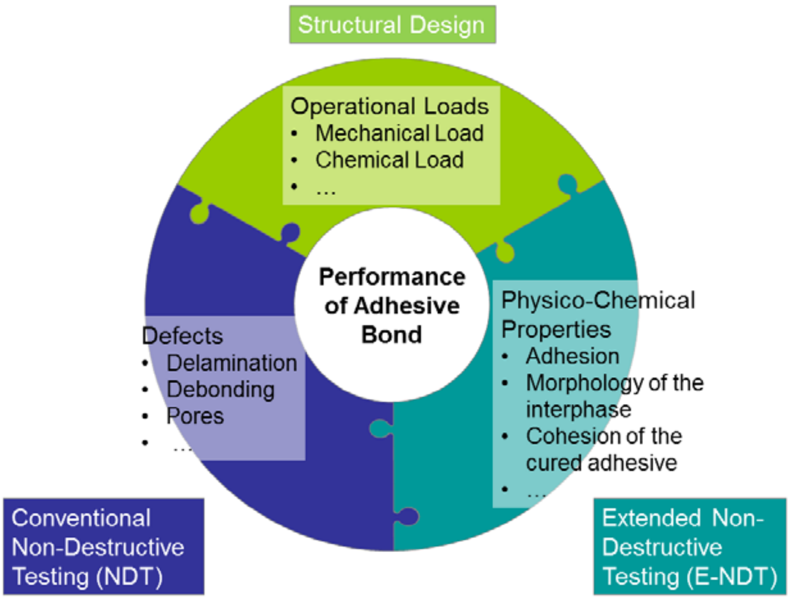


Figure 2: Parameters influencing the performance of adhesive bonds

The properties of adhesive bonds, such as mechanical strength, result from the physico-chemical properties of adherend surfaces and adhesives. The physico-chemical properties of adherend surfaces are affected for example by the degree of contamination or the surface pretreatment. The physico-chemical properties of adhesives depend on a range of conditions, from the curing parameters and age of the adhesive, to the application parameters and ambient conditions. The adhesion, the morphology of the interphase and the cohesion of the cured adhesive are a direct product of these properties and are fundamental to the strength of the adhesive joint. If the physico-chemical properties of adhesive bonds are not sufficient then adhesion failure, weak bonds or bonds that weaken in-service can occur.

Therefore, a novel class of non-destructive testing techniques, classified as Extended NDT (ENDT), are required. The principle of ENDT methods is based on the detection of selected physico-chemical properties which are important for the performance of an adhesive bond.

The focus of the ENCOMB project has been to identify, develop and adapt methods that are suitable for the assessment of adhesive bond quality, CFRP adherend surfaces and adhesives. Therefore detection capabilities and the sensitivity of measuring techniques have had to be tested and improved in order to achieve quantifiable analytical results.

Project objectives

In order to address the challenge to assess the quality of an adhesive bond, the main objective of ENCOMB was the identification, development, adaptation, and validation of ENDT methods for the characterization of adherend surfaces and adhesive bond quality. To this aim, the following aspects have been addressed:

- The study of promising technologies with high and low technology readiness levels from the field of analytical and spectroscopic polymer and surface characterization such as nuclear magnetic resonance spectroscopy, dielectric spectroscopy, infrared spectroscopy, terahertz spectroscopy etc.
- Demonstration of the potential to be suitable for measurements in the field of manufacturing, rework, and (in-service) repair for technologies with low technology readiness level (semi-automated operation, high reproducibility, adequate speed of measurement).
- Extensive evaluation of the testing methods against application scenarios that are important for adhesive bonding of primary structures in the aeronautics industry. Example of this have included the effect of adherend surface contamination, thermal degradation, and the impact of the curing state of adhesives on adhesion properties and overall bond performance.
- The improvement of the detection capabilities and the sensitivity of the measuring techniques in order to assess the quality of adhesive bonds, adherend surfaces, and adhesives. This was tested by quantifiable results which were used for further development or adaption of the techniques.
- The determination of a variety of physico-chemical properties of adherend surfaces and adhesives by the application of ENDT.

Within ENCOMB promising technologies with high and low technology readiness levels have been studied. Though it is not necessary that a high technology readiness level is achieved in the project, the techniques have to show the potential to be suitable for measurements in the field of manufacturing, rework and (in service) repair. They have to operate automatically with high reproducibility and adequate speed of measurements.

The testing methods have to be evaluated regarding application scenarios that are important for adhesive bonding of primary structures in the aircraft/aerospace industry. Important scenarios are:

- Effect of adherend surface contamination on adhesion properties and overall bond performance.
- Effect of thermal degradation of adherend surfaces/adhesives on adhesion properties and overall bond performance.
- Effect of curing state of adherend surfaces/adhesives on adhesion properties and overall bond performance.

The Consortium

In ENCOMB, a multidisciplinary consortium of 14 partners from ten European countries brought together leading experts from top-level European research organizations, universities, and industries active in aeronautics research and development.

Altogether, four commercial companies participated in the project, three of them belonging to the major industrial players in the European aeronautics industry and a major manufacturer of electronic and analytical measurement instruments. The upstream approach followed within this level 1 project was reflected by the participation of recognised research organisations and universities. The participation of major European aircraft manufacturers ensured the relevance of the work through compliance with appropriate application scenarios, technological specifications and thus the greatest potential for the exploitation results.

4.1.3 Description of the main S&T results/foregrounds

ENCOMB was structured in four technical work packages: WP1 – the definition of application scenarios for adhesive bonding of composite structures along with technology requirements, WP2 – the preparation and contamination/degradation of test specimens, WP3 – the development of ENDT methods for characterisation of adhesives and adherend surfaces, and WP4 – the development of ENDT methods for adhesive bond characterisation.

Based on five identified major application scenarios, specimens, test campaigns, and specifications for the development of the ENDT technologies were defined. For the technological development, a two-step test campaign was established. Starting with a campaign to assess the suitability of each technology regarding the application scenarios, two campaigns each targeting a subset of the application scenarios for optimisation and verification of the detection capabilities followed.

WP2 – Sample preparation and characterisation

Within WP2, samples have been prepared with regard to the application scenarios and requirements (see Table 1). The details of the preparation and contamination of the samples have been reported in five peer reviewed publications. To provide reference values for the comparison of the ENDT results, all samples have been characterised by laboratory analytical methods regarding their physico-chemical properties (XPS, XRF, FTIR) and by conventional NDT methods regarding their structural integrity (ultrasonics, radiography). The results of the characterisations by laboratory analytical methods are depicted in Figure 3.

Table 1 Contamination/degradation levels for the five application scenarios.

Application scenario	Contamination level			
	1	2	3	4
Release agent [atom% Si]	2.2±0.3	6.7±0.2	8.4±0.8	10.5±0.3
Moisture [% mass increase]*	0.46 ±0.01	0.84 ±0.05	1.19 ±0.01	1.29 ±0.01
Hydraulic fluid/water [pH]	4	3	2	-
Thermal degradation [°C]	190	200	210	220
Poor curing [°C]	170	160	150	-

*These values only apply to the bonded samples for WP4. The samples for detection of surface contamination (WP3) were incubated by each partner and the contamination degree (% mass increase due to water uptake) of these samples varies slightly depending on the partner.

The mechanical performance of bonded samples has been characterised by the measurement of the mode-I fracture toughness (G_{IC}). The results of the mechanical testing are summarized in Figure 4. The details of the measurements have been reported in the following publications [1-5].

Within WP2, a defined manufacturing process guaranteeing repeatability and comparability of samples, the revision-safe manufacturing and distribution of a large number of samples (900+), and an extensive characterisation for later correlation with ENDT results by laboratory analytical methods, conventional NDT methods, and mechanical testing have been achieved.

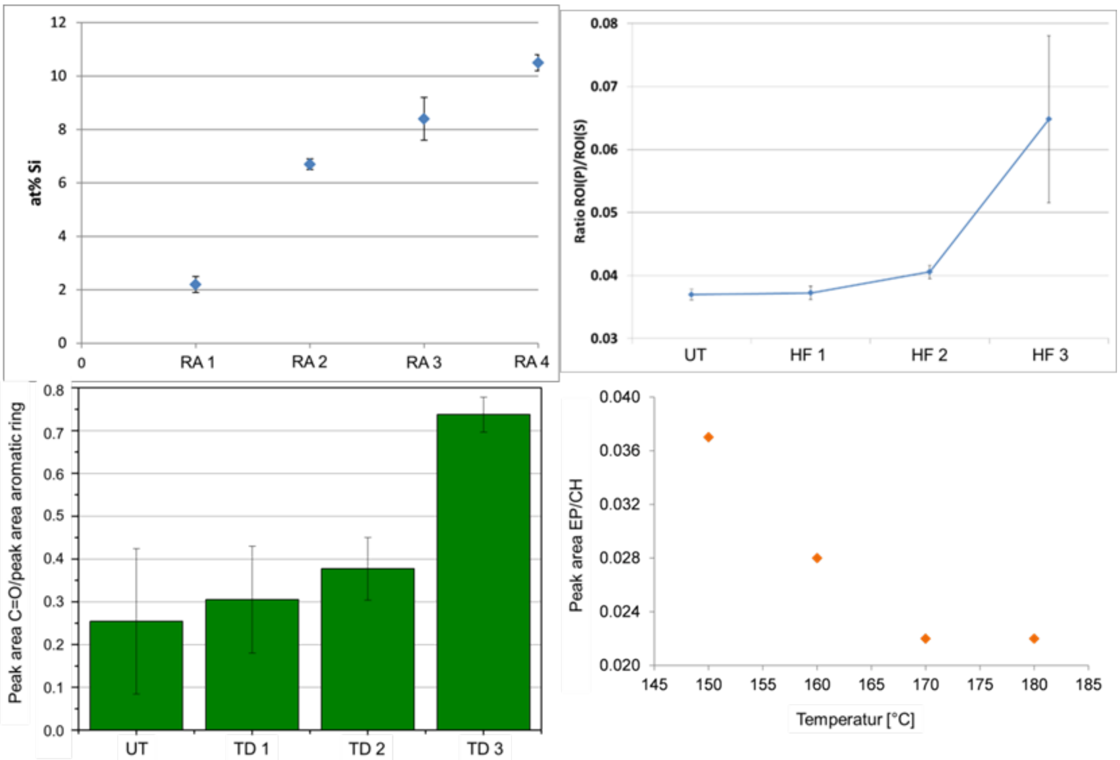


Figure 3: (upper left) Amount of release agent (RA) on the sample surface in atom% Si for different contamination level measured by XPS; (upper right) Amount of hydraulic fluid (HF) on the sample surface specified as the ratio between the phosphorous and sulphur concentrations for different contamination level by XRF; (lower left) Thermal degradation (TD) of the sample surface specified as the ratio between C=O and aromatic ring concentrations for different degradation level by FTIR; (lower right) Poor curing of the adhesive (PC) specified as the ratio between epoxy ring (EP) and CH concentrations for different degradation level by FTIR.

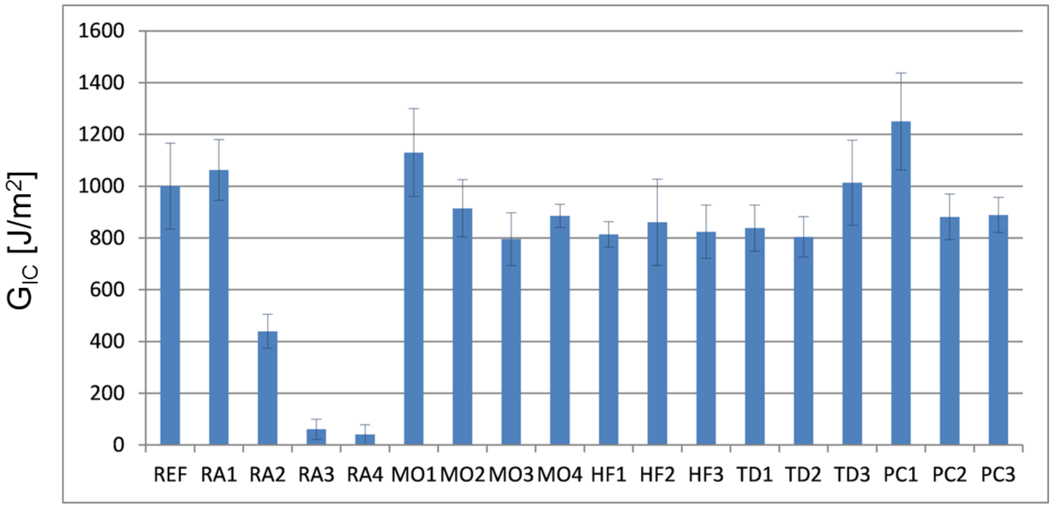


Figure 4: Average G_{IC} value for bonded samples with different degrees of contamination/degradation.

WP3 – Extended NDT for adhesive and adherend surface characterisation

In the following, the results are shortly summarized for each investigated ENDT technology followed by a short conclusion.

Optically stimulated electron emission

The OSEE technique has been investigated to elucidate whether OSEE can be used to differentiate contaminated surface states from clean ready-to-bond ones. Secondly, the development has been focused on the correlation of the OSEE signal with the degree of contamination and the minimum detection thresholds for each contamination scenario.

The investigations and developments clearly show that all contaminated samples exhibit significantly smaller OSEE signal intensities than the samples ready for bonding (reference) (see Figure 5).

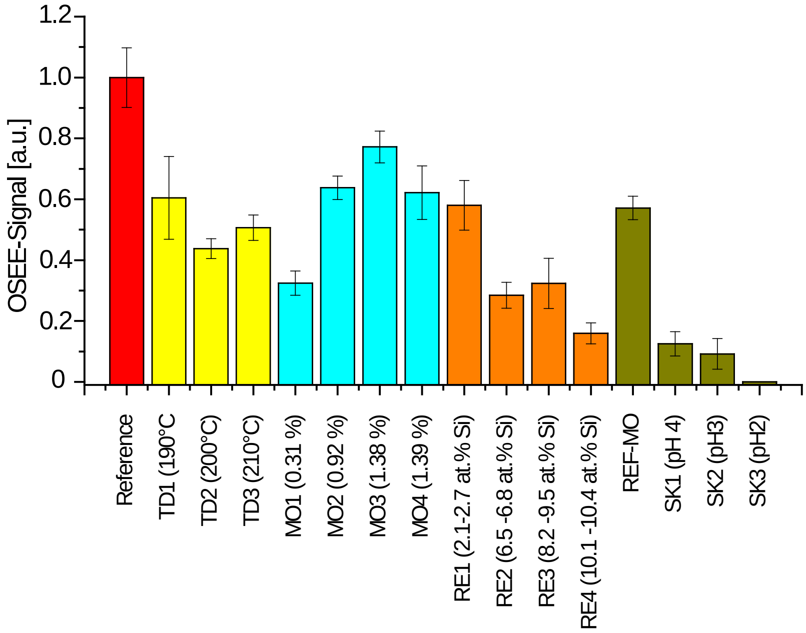


Figure 5: OSEE intensities obtained for distinct CFRP surface states. TD=thermal degradation, MO=moisture uptake, RE=release agent contamination, SK=hydraulic oil/water contamination.

In detail, the results show that (i) OSEE is an appropriate technology for detecting thermo-oxidative effects (thermally degraded samples) which were shown to reduce the strength of adhesive joints produced from correspondingly impacted CFRP adherends; (ii) moisture contaminated samples (MO) exhibit a significantly smaller OSEE intensity than the samples ready for bonding (from a sample covered by a several micrometre thick layer of liquid water no detectable OSEE signal was obtained (data not shown)); (iii) release agent (RE) contaminated samples show significantly smaller OSEE intensities than the reference samples and the intensity strongly depends on the concentration of release agent; (iv) samples contaminated with hydraulic oil (SK) show drastically smaller signal intensities than the reference samples and the water contaminated sample which was used as reference for this scenario (REF-MO). Thus, the OSEE intensity is strongly influenced by the concentration of hydraulic oil in the liquid contaminant.

The results work clearly demonstrate that the OSEE technique is a useful tool for monitoring pre-bond surface quality of CFRP as all contaminated samples could be significantly distinguished from clean reference samples even though it is not possible to draw conclusions from the OSEE signals on the kind of surface contamination.

Laser scanning vibrometry

Vibrometry measurements have been conducted to detect surface contamination in comparison to the clean surface of a reference sample. For the experiments the samples were excited with a piezoelectric transducer using a tone burst excitation signal with 5 cycles. The propagating elastic waves were measured with scanning laser vibrometer working in 1D mode. The results have been analysed using an index proportional to the squared signal averaged over the whole set of measurement points. This index can be associated with the out-of-plane vibration energy. The details of the measurements have been reported in the respective publications.

In Figure 6, the results of the vibrometry measurements for the different sample sets are depicted. To emphasize the sensitivity to surface anomalies the value of the energy (*E)* and its dispersion over the surface represented by the standard deviation (σ) is shown.

The results show that some of the contaminated samples clearly differ from the reference. The method clearly indicates the moisture contaminated CFRP samples (MO). Both chosen parameters (*E* and σ) show a significant decrease in comparison to the reference (Ref). The CFRP samples contaminated by hydraulic oil (HF) showed slightly smaller signal intensities than the reference sample. The thermally degraded samples (TD) do not differ too much from the reference; an overcured sample (OC) was also detected successfully.

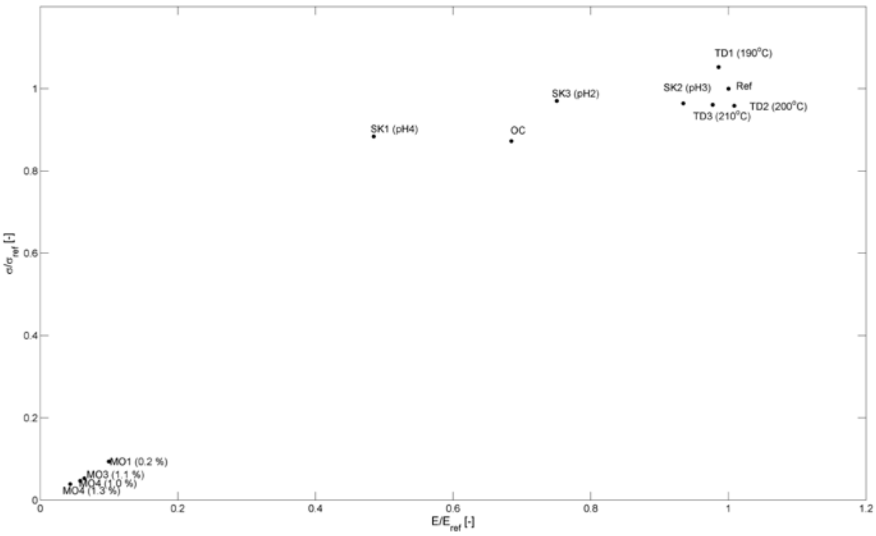


Figure 6: Results for the surface characterization using laser vibrometry.

Infrared (IR) spectroscopy

IR spectroscopy has been employed to investigate the surface state of contaminated samples. The measurement setup, in particular the optical path, has been modified in order to be able to analyse large samples and to perform phase sensitive measurements.

The results show that different contaminations such as hydraulic fluid (e.g. a Skydrol/water mixture can be distinguished (Figure 7). The samples were treated with a hydraulic fluid/water mixture in different dilutions characterised by their pH. A sample contaminated with distilled water was used as reference sample (sk ref).

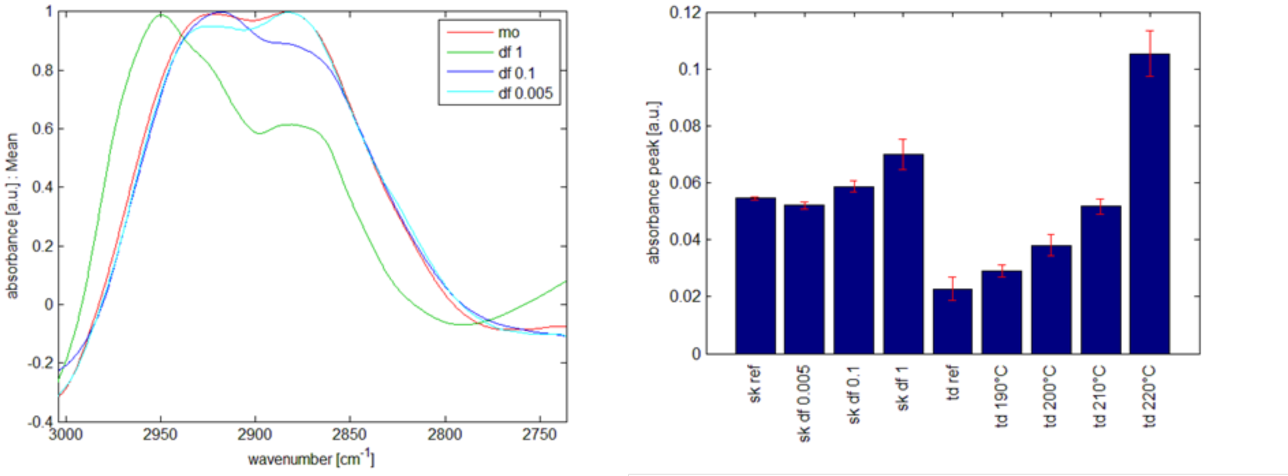


Figure 7: (left) Detail of smoothed, baseline corrected, normalized (maxima) spectral data of samples contaminated with a hydraulic fluid/water mixture of different dilutions (pH2-pH4); (right) Absorbance peaks of hydraulic fluid/water (~2850 cm^{-1}), thermal degradation (~1650 cm^{-1}).

The samples with pH values of 2 and 3 can be clearly distinguished by the IR spectroscopic measurements at wavenumbers between about 1300 and 1100 cm^{-1} and between about 3000 cm^{-1} and 2800 cm^{-1}. The sample with a pH value of 4 cannot be clearly distinguished from the moisture sample due to extremely low concentration of hydraulic fluid (dilution factor of 0.005).

Further, the investigations show that thermal degradation (td) can be distinguished (Figure 7). The oxidation of the samples at different temperatures causes an absorbance peak at about 1650 cm^{-1}. Moisture treated samples can be distinguished using a broadband absorbance feature at 1700 to 1500 cm^{-1} and between 3000 to 3600 cm^{-1}. Chemometric modelling as partial least square regression has been used for spectral interpretation. The CFRP samples contaminated by release agent (RE) show best observable spectral features at about 1260 cm^{-1} and 1100 cm^{-1} (data not shown). They can only be used for prediction of release agent contamination down to a detection limit of 15 atom% Si (10 atom% Si using polarisation dependent IR spectroscopy).

In summery we have demonstrated that IR spectroscopy is a powerful tool to detect different surface contaminations (hydraulic fluid, water, thermal degradation). Up to now distinct detection of release agent at the required low detection limits is not possible.

Aerosol wetting test

The Aerosol Wetting Test (AWT) is a method to characterize the wetting properties of surfaces (Figure 8).The aim within ENCOMB has been to investigate the capability of the AWT method to discriminate between different surface states by their wetting behaviours.

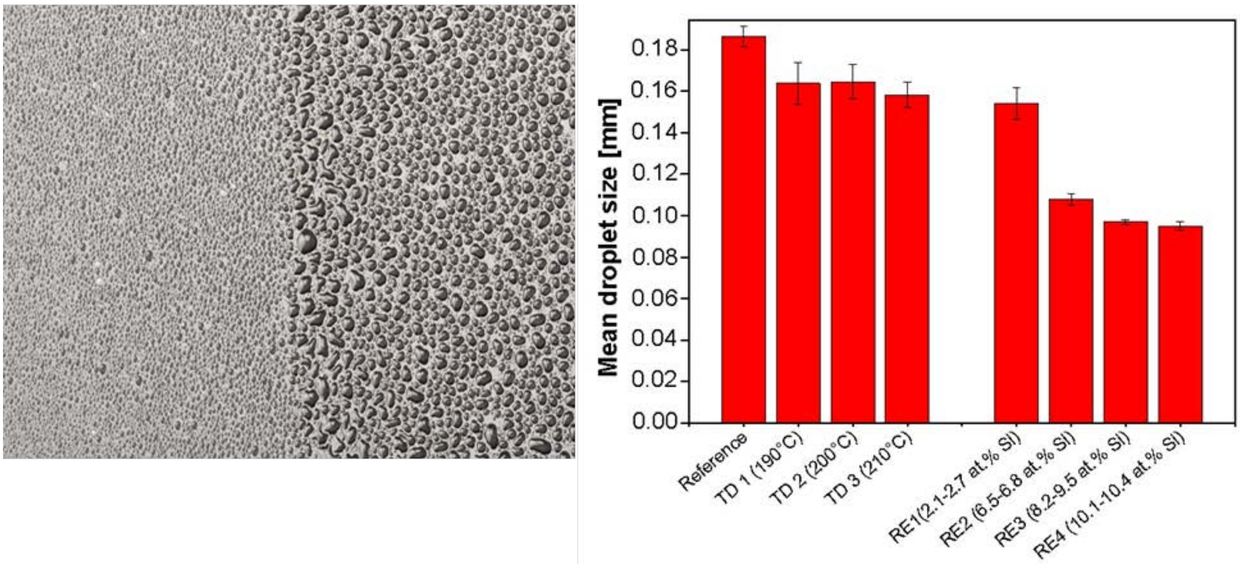


Figure 8: (left) CFRP surface with two different wetting properties. Bad wetting due to contamination on the left side; Good wetting after surface pre-treatment on the right side. (right) Mean drop sizes as results of Aerosol Wetting Test for release agent (RE) contaminated and thermally degraded (TD) CFRP samples. Mean values for reference over 2 samples, all other 1 samples (three measurements per sample).

The investigations have shown that the AWT is suitable to detect release agent contamination (RE) and thermal degradation (TD) of CFRP adherent surfaces (Figure 8). The technique is able to discriminate between the different contamination levels of release agent with a detection limit below 1 at% Si. The information depth is a few nm on CFRP with a measuring area of some cm² with the set-up used. For thermally degraded CFRP specimens temperatures higher than 180°C lead to a smaller mean drop size. With increasing temperatures no significant decrease of mean droplet size is visible. As the AWT is sensitive only for the topmost layer potential changes of the CFRP material at higher temperatures has no influence on the signal.

The technique is not suitable for the detection of moisture (MO) or hydraulic fluid/water (HF) contaminations on CFRP surfaces (not shown). The reason is that water is used for the droplet generation. This is one fundamental limitation of the AWT; it is only suitable for non-aqueous contaminations.

Within the project, it has been demonstrated that only a short drying is necessary and by using ultra-clean water there is no chemical modification of the CFRP adherent surface. This makes the AWT a truly non-destructive test method.

Portable Handheld FTIR spectroscopy

A portable mid-infrared Fourier transform spectrometer (Agilent Technologies 4100) has been used to examine the contaminated samples. As main sample interface the diffuse reflectance has been used resulting in diffuse reflectance IR spectra (other interfaces have been tested for suitability with the diffuse proving to be most appropriate for the CFRP samples).

CFRP materials are complex composite materials with equally complex spectra with each FTIR diffuse spectra containing over 1000 discrete absorbance points. The results clearly show spectral changes due to the treatment type (Figure 9, reference CFRP spectra black line in A, blue in B, red in C & D) Each application scenario leads to different regions of the spectra being affected.

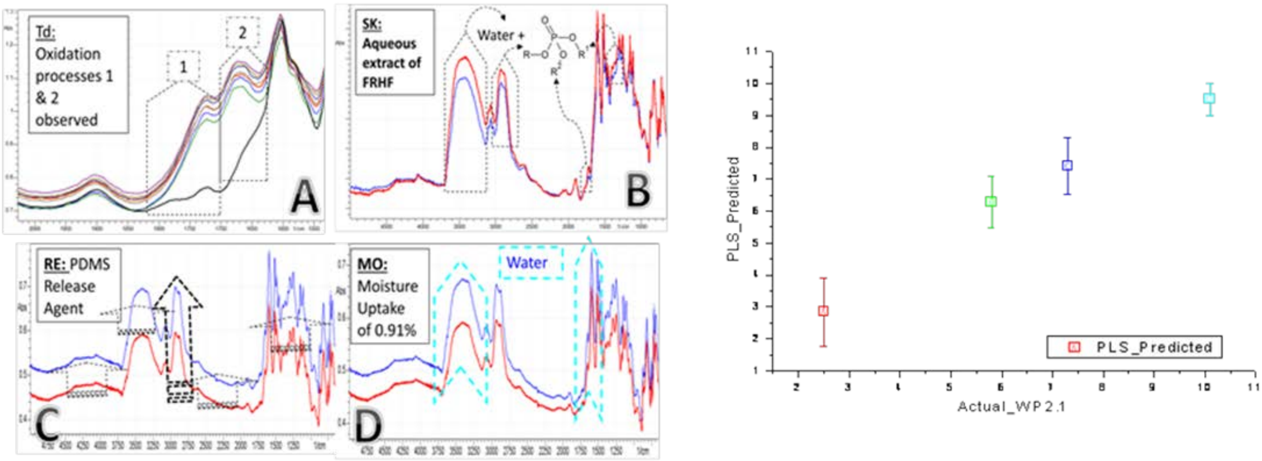


Figure 9: (left) Results of the FTIR analysis - A=Td (thermal degradation), B=SK (hydraulic fluid extract), C=(release agent) & D=MO (moisture uptake); (right) PLS for % release agent (RE) of actual vs. predicted.

To develop a viable PLS model that accounts for the CFRP surface variances correlated to the treatment, a measurement scheme of many spectral positions measured per sample has been established. Multivariate PLS models have been created, calibrated, validated and verified for each of the scenarios. Exemplarily in Figure 9 (right) the actual vs. predicted release agent treatment model is depicted. The error bars indicate the spread of the predicted values which fit well with dip-coating and how the imparted layer will vary with concentration.

Clearly handheld FTIR mid infrared spectroscopy is selective as different spectral regions change. That can be directly related to the treatment and therefore the overall chemical species can be examined by the IR beam. In addition to the selectivity of the diffuse mid-IR enables, we could demonstrate that the sensitivity allows for quantitative models where the variance can be correlated with the degree of treatment.

Laser induced breakdown spectroscopy

The aim of the work has been to determine whether LIBS (scheme of the technology is shown Figure 10 left) is a suitable method for the differentiation between contaminated and ready-to-bond CFRP surface states. In a second step, the investigations have been focused on the correlation of the emission signal intensities with the concentration of the surface contamination.

For data evaluation, mean values of 30 (RE scenario) or 60 (SK scenario) single measurements were calculated as well as the 95 % confidence interval. The results are shown in Figure 10 right. For the release agent scenario (RE), silicium is the key element and Si/C ratios are plotted. For the hydraulic fluid scenario (SK), the phosphorus/carbon ratio has been used. No results for the thermal degradation (TD) and moisture uptake (MO) scenario are shown because in these scenarios no changes in the elemental composition (which is detected by LIBS) occur.

For the RE samples, it is shown that LIBS is suitable to detect all tested contamination levels. The relative signal intensities correlate with the amount of release agent on the surface allowing the quantification of the contamination. For the CFRP samples contaminated with an aqueous hydraulic oil extract (SK), LIBS is able to detect and distinguish conditions SK3 and SK2 from the reference (REF-MO) which is contaminated with pure water. The low contamination level of sample SK1 cannot be differentiated from the reference sample REF-MO.

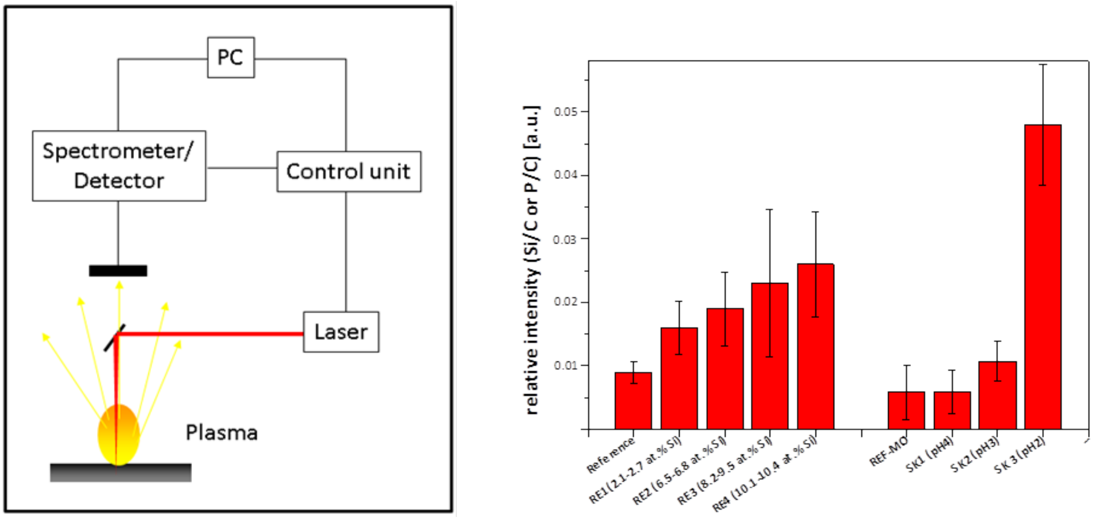


Figure 10: (left) Scheme of LIBS device with main components and functionality; (right) Relative signal intensities obtained from LIBS measurements for distinct CFRP surface states. RE=release agent contamination, SK=hydraulic fluid/water contamination.

For the two scenarios release agent and hydraulic fluid, we could demonstrate that LIBS shows great potential for the identification of surface contaminations. Furthermore, quantification is possible for almost all tested contamination levels. Due to the element-specific evaluation, detection and quantification of multiple contaminations is expected to be successful.

THz/GHz Polarization-frequency reflectometry (PFR) method

For the development of the PFR method, the sample surfaces have been scanned by an incident THz beam with various polarizations and frequencies. The change of the reflection coefficient modulus caused by defects on the sample surface have been recorded and analysed. Using the PFR method the influence of various contaminations on the CFRP surface reflectivity has been detected by the comparison between the reflection coefficients of contaminated and untreated areas of the CFRP.

Examples of the spatial distribution of the reflection coefficient of the CFRP samples for the investigated contamination scenarios are shown in Figure 11. The spatial distribution of the reflection coefficient for contaminated CFRP samples demonstrates a pattern of random spots. Therefore for a more reliable detection of contaminations it is necessary to analyse large areas of the sample surface.

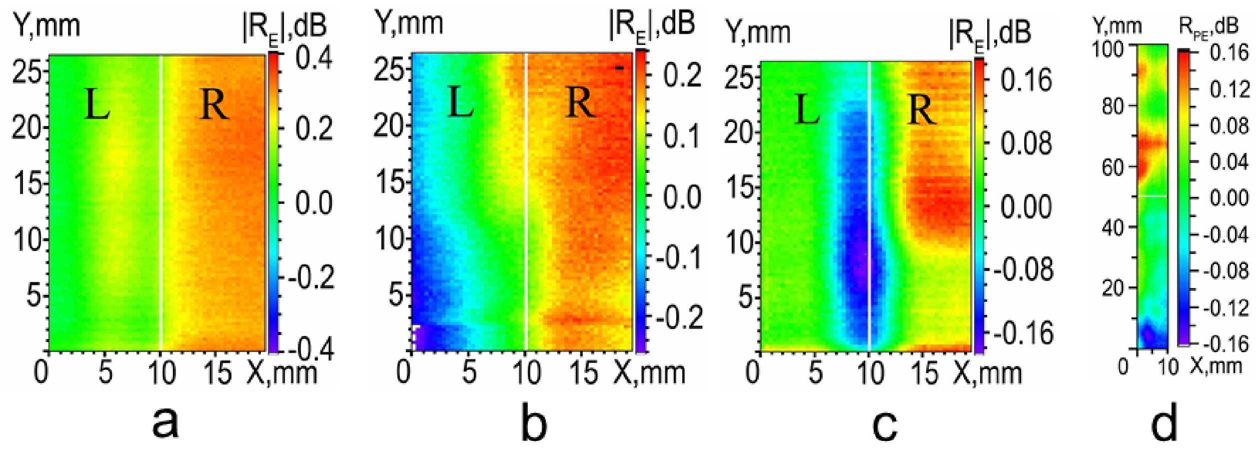


Figure 11: The examples of spatial distribution of the reflection coefficient of contaminated CFRP samples: a) moistening; b) release agent; c) thermal degradation; d) hydraulic fluid. In case of a), b) and c) the contaminated area is right half (R), and in case of d) the contaminated area is top half of the sample.

The measurements of the spatial distributions of the reflection coefficient for untreated and contaminated CFRP samples have demonstrated that the PFR method allows detecting thermal degradation, moisture, release agent and hydraulic fluid surface contaminations if the samples are irradiated by a E-polarized incident beam and the irregularity of untreated sample surface reflection coefficient does not exceed 0.05 dB.

As the reflection coefficient from an untreated CFRP sample does not exceed 0.25 dB to 0.3 dB for the E-polarized beam incidence and a contamination increases the value of the reflection coefficient, the change of its value cannot exceed 0.25 dB to 0.3 dB. Thus, despite the fact that PFR method allows detecting these contaminations, such small changes of the reflection coefficient does not allow to estimate the level of contamination reliably enough and to determine the kind of surface contamination. It should be noted that the PFR method may detect CFRP degradation at high temperatures between 260 ºC and 300 ºC, i.e. close to the CFRP destruction temperature, but we cannot consider the PFR method as suitable for detection of CFRP thermal degradation.

THz/GHz Quasi-optical method of internal reflection reflectometry

The quasi-optical method of internal reflection reflectometry (QOIRR) has been employed to investigate the contaminated samples. The technique uses a quasi-optical beam for oblique probing of the CFRP sample surface through the isosceles of a triangular prism located at a distance *h* from the sample surface (Figure 12).

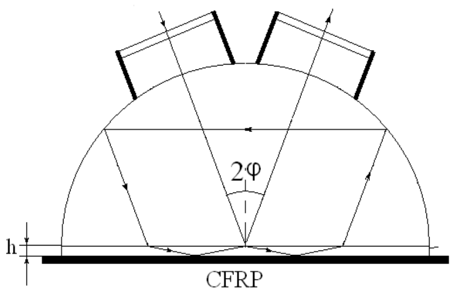


Figure 12: Interferometer-resonator for the QOIRR method.

The detection of contaminations has been performed by the change of the depth of the interference minimum of the waves reflected from the internal boundary of the quartz half-cylinder and the sample surface, when irradiating untreated (for calibration) and contaminated samples. It has been found that any kinds of contaminations cause reduction of its value.

Three samples with different contamination level of hydraulic fluid have been studied. The change of the interferential minimum depth has been from 4 to 9 ±1.5 dB and its value depends on concentration of the hydraulic fluid in the solution. The release agent contamination has also been detected reliably as the change of the interferential minimum depth was from 3 to 5±1.5dB. For the thermal degradation scenario, it has been found that a temperature impact exceeding 290°C can detected reliably. The study of CFRP samples contaminated with moisture in presence of a thin layer of liquid water on the surface has shown a considerable change of the interferential minimum depth for the PE-scenario of irradiation. However, the change of the depth became minimal when water had evaporated from the CFRP surface. In case of the PH- or SH-scenarios the incident wave can get into subsurface layer of the CFRP sample. However, in this case the detection of moisture is not possible unambiguously because of the background signal caused by the inhomogeneity of the CFRP structure.

We have demonstrated that the QOIRR technology is very perspective for the CFRP quality control during its manufacturing as in most cases it allows to distinguish reliably enough contaminated samples from untreated ones. Currently the method does not allow to define the kind of contaminations.

Optical fibre sensors

Optical fibre sensors based on single fibre Bragg gratings and arrays have been fabricated and integrated into CFRP bonded joints. Figure 13 shows the schematic and a micrograph of the manufactured specimens. Finite element simulations have been performed to analyse the hygrothermal response of the bonded composite plates: Figure 13 (right) shows a typical distribution of strain in the embedded optical fibre along the optical axis for a thermal loading of ΔT=80 °C.

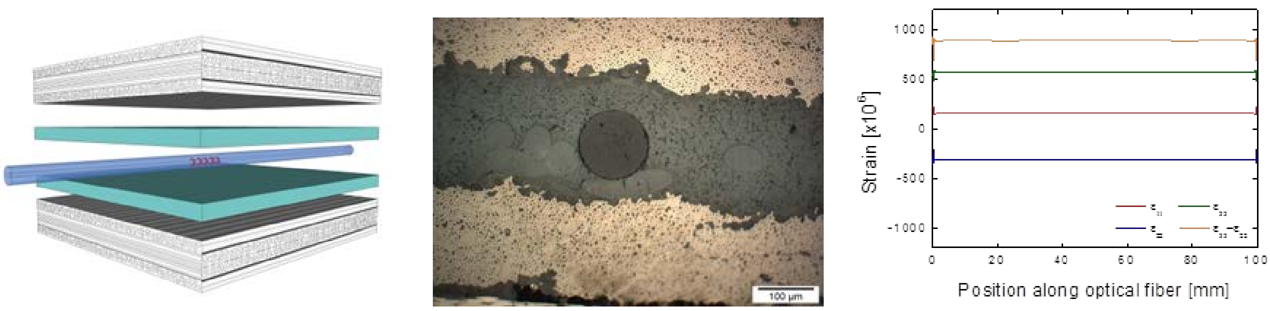


Figure 13: (left) Schematic of the sample: adhesive with FBG between two biaxial CFRP plates; (middle) Micrograph of the optical fibre placed in the bonded specimens; (right) Optical fibre strains in the sandwich (FE simulations).

The temperature sensitivity of the fibre before and after integration into the bonded joints is shown in Figure 14. The integration into the structure leads to two different Bragg resonance peaks separated by 270 pm at 20 °C. The difference is due to differential strains perpendicular to the fibre axis ($\varepsilon_{33} - \varepsilon_{22}$) as shown in Figure 13 (right).

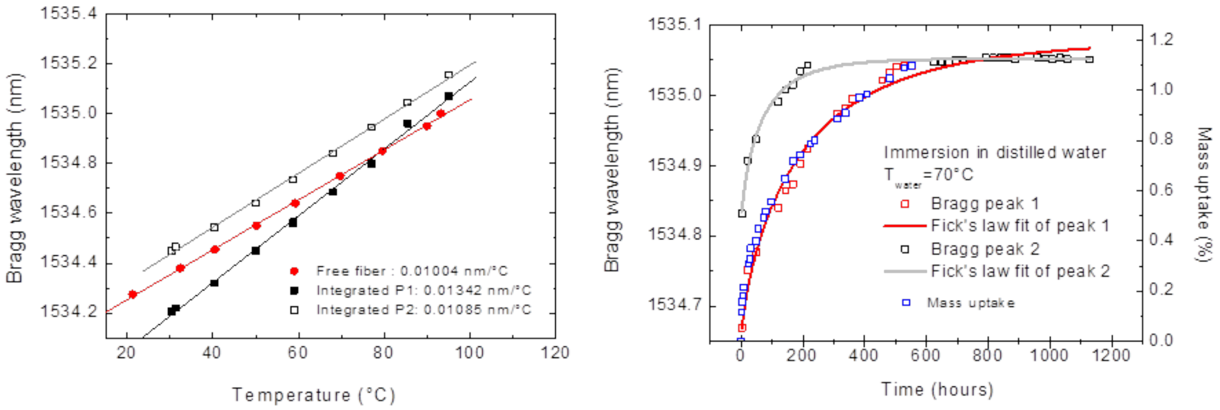


Figure 14: (left) Thermal response of the free and embedded fibre Bragg grating. Inset: simulation of strains in the bond length; (right) Evolution of the Bragg peak due to water uptake of the composite laminate at 70°C.

Thermal load changes the FBG peak positions and reduces the birefringence (Figure 14 left). The FBG thermal response and hence the thermal expansion of the fibre before and after integration is quite similar, i.e. there is only a small mismatch in the thermal expansion coefficient between the fibre and the bonded structure. Figure 14 (right) shows the wavelength response and mass uptake during water diffusion at 70 °C. Again the initial birefringence disappears with time. One of the Bragg peaks follows the water uptake with a wavelength sensitivity of 0.359 nm/weight% and can thus be used to quantify the water uptake in the bonded joint.

Considering a typical wavelength resolution of 10 pm, we have demonstrated a water uptake as small as 0.03 weight % (40 pm). Moreover, as both Bragg peaks depend on temperature and swelling differently it should be possible to separate them.

Electronic nose technology (E-Nose)

The E–Nose technology has been investigated for the development of fast detection, discrimination and quantification of hydraulic fluids, release agents and moisture contaminations at different levels. In order to increase desorption of volatiles coming from the CFRP surface, the basic E-Nose structure has been modified with an IR emitter designed to slightly heat surface under analysis. The results have been obtained by the adaptation of a hybrid sensors array composed of 2 MOX sensors, 1 electrochemical sensor, 1 PID and an Ion Mobility Spectrometer.

Exposed to complex mixtures, the E-Nose sensors array produce an informative dynamic response that, processed to extract relevant response features, can be used to train a multivariate pattern recognition algorithm (e.g. a neural network). The latter process builds a knowledge that is used to recognize the distinct fingerprint of a surface contaminant in the application scenarios. Exemplarily, the results for the detection of release agents contaminated surfaces are depicted in Figure 15 (right side).

Performance Indexes at different Reliability Reject Thresholds values			
Classification Accuracy	78.5%	84.5%	90%
False Positive Rate	20%	8%	6%
False Negative Rate	6%	0%	0%
Reject Rate	21%	35%	45%

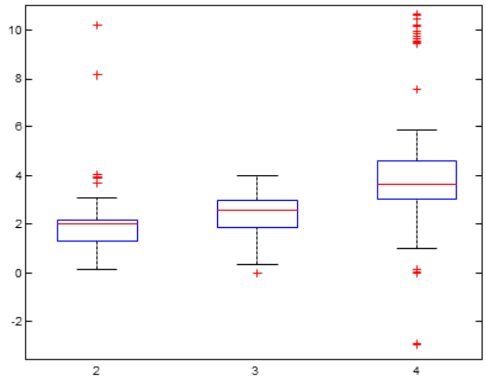


Figure 15: (left) Overall Performance Indexes, (right) Box plot of output signal from hydraulic fluid contamination quantification trained neural network.

As shown in Figure 15 (left side), classification accuracy, computed as percentage of correctly classified samples ranges from 78% to 90% in the considered threshold values range. This accounts for the electronic nose being capable not only to identify the contaminated samples but also to identify the main contaminant with interesting performances. In particular it has to be underlined that false negative rate can be set to as low as 0%, accepting an 8% rate of false positives. This is particularly important considering the unbearable potential cost of a contaminated sample misclassification.

Analysis of wrongly classified samples shows that chances of misclassification are increased at low contaminant concentrations especially for moisture contaminated samples. Apart from hydraulic fluids, it seems that the current version of E-Nose is capable to only qualitatively assess the level of contamination. Although it has to be considered that further improvements are expected as newly adapted, focused versions of the NDT tool are under development.

The project results demonstrate that the E-Nose technology has the capabilities for the development of a rapid, first line NDT tool for identification of contaminated CFRP panels with contaminant discrimination capability.

Dual-band active thermography

Dual band active thermography experiments have been performed using an infrared camera which has two sensitive elements one over the other for each pixel. The elements are sensitive in the mid-wave infrared (MWIR) at 4.4-5.2 µm and in the long-wave infrared (LWIR) at 7.8-8.8 µm, respectively. A strong flash tube is heating the adherend surface of the CFRP, generation a transient temperature rise (Figure 16 left). The IR camera records the radiative response of the test object as a function of time. In particular for short times, the radiation can be expected to reflect the infrared emission properties of species at the adherent surface in the two available spectral bands. Besides that, the radiative response is determined by the thermal effusivity of the material, which is a function of its thermal conductivity, its specific heat capacity and its density. Volume changes, for example due to thermal damage or moisture uptake, will change the thermal effusivity and should therefore be detectable in the camera signal.

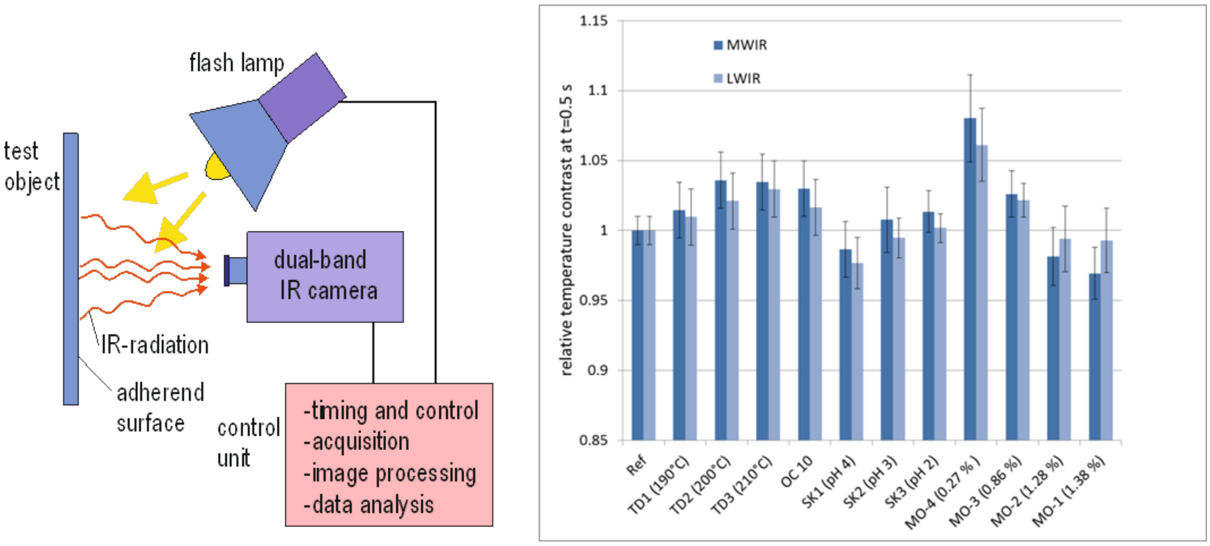


Figure 16: (left) Scheme of the dual-band active thermography; (right) Relative thermal contrasts for different surface and volume states in two spectral ranges. TD=thermal degradation, OC: over-curing, SK=hydraulic oil/water contamination, MO=moisture uptake.

The results show that characteristic IR spectral features of the contaminants in the project did not fit well with the available (and unchangeable) spectral bands of the camera. For the lower contamination levels, there is no detectable spectral contrast. The results of infrared spectrometry suggest that this could be improved using cameras with variable spectral filters. Better results have been achieved when the effects of contaminants on the effusivity have been analysed in terms of a relative thermal contrast compared with a reference sample. In Figure 16 (right) the main results for the different types of contaminants are summarized. Thermal damage and the contamination with hydraulic fluid show weak effects with some significance. The contamination with release agent did not show any effect.

The present work clearly demonstrates that active thermography can detect moisture levels of down to 0.5 wt%. The sensitivity to moisture is larger in the MWIR band. Active thermography can be applied contact-free and on curved surfaces.

Laser induced fluorescence

As the Laser induced fluorescence (LIF) allows to analyse large objects, the technique has been used to investigate the different application scenarios. LIF spectra have been recorded using a laboratory system with the following specifications: excitation source - Nd:YAG, 6 ns pulse, 1064 nm laser; detector - 0.3 m monochromator equipped with 600 g/mm grating and ICCD detector.

The investigations show that a narrow band with a maximum at 432 nm and a strong fluorescence in a wide band with maximum at 600 nm is observed for an excitation at 532 nm. The intensity of the fluorescence changes significantly for the different samples. Based on the spectroscopic data obtained at 532 nm laser excitation, the intensity of fluorescence (A), the fluorescence lifetime (T), and the fluorescence decay time (t) have been determined and are have been used to differentiate the samples depending on the type of surface contamination.

Firstly, the CFRP samples covered with a silicone-containing release agent (RE) have been investigated. The results do not indicate any differences. In the second stage the CFRP samples contaminated by hydraulic oil (SK) were analysed. The results for fluorescence lifetime (T) indicate a decreasing value of T with decreasing pH value of the hydraulic fluid solution (Figure 17 left). The same behaviour is observed for the decay time (t). The fluorescence intensity seems to be insensitive to contamination with hydraulic fluid. In the last stage of research thermally degraded samples (TD) have been investigated. Three different temperatures have been considered. With increasing temperature the intensity increases (Figure 17 right). Fluorescence lifetime and decay, however, do not allow distinguishing the TD samples.

The obtained results indicate that the analysis of the fluorescence at 532 nm laser excitation allows discriminating samples contaminated with hydraulic fluid as well as thermal degraded samples.

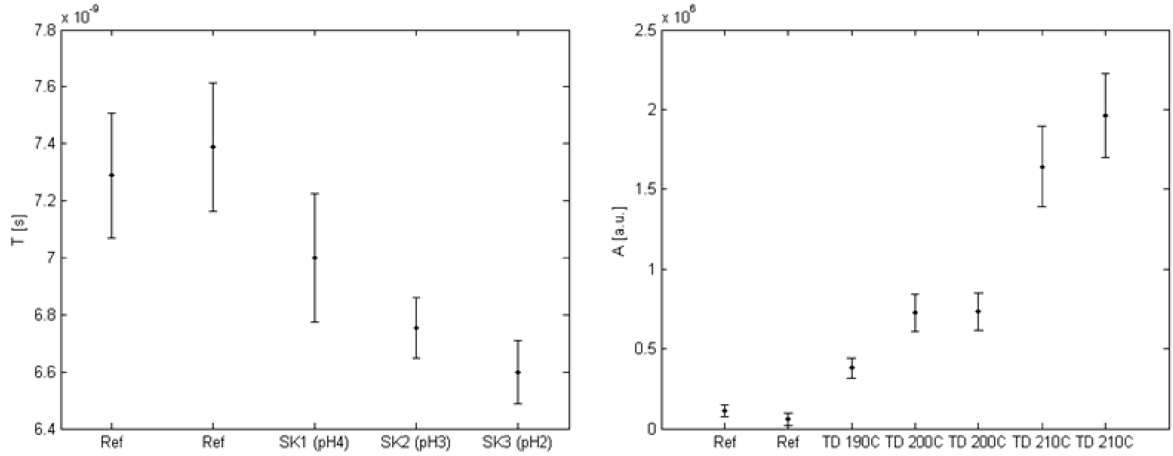


Figure 17: (left) Fluorescence lifetime calculated for reference samples and CFRP samples contaminated by hydraulic oil (SK); (right) Fluorescence intensity calculated for reference samples and thermally degraded CFRP samples (TD).

THz technology

THz technology has been used to investigate the different application scenarios. Many THz systems work with pulsed, broadband THz radiation and time-resolved detection schemes (TDS). A typical THz-TDS setup is shown in Figure 18.

For excitation of the pulsed THz radiation, a femtosecond laser pulse is used. One part of the laser beam illuminates a THz emitter (e.g., a photo-conductive antenna). The emitted THz radiation is focused on the sample; the transmitted pulse is re-collimated and focused onto a THz detector (e.g., photoconductive switch). For time-resolved detection of the THz signal, the second part of the laser beam illuminates the detector. By moving a delay-line in the optical path of the laser beam, the time delay between excitation and detection of the THz pulse is varied, and the THz pulse is sampled in a time-resolved way. The THz spectrum is obtained by Fast Fourier transformation of the time-resolved signal. From the phase and the amplitude information, the frequency-dependent THz material parameters (refractive index and absorption coefficient) can directly be obtained.

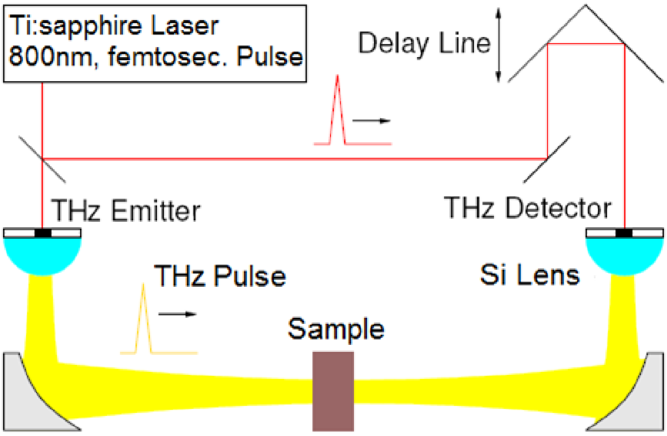


Figure 18: Schematic of a THz-TDS setup.

The results of the experiments show that a measurement of the samples in transmission is not possible as the THz radiation is completely blocked by the conducting carbon fibres. In reflection geometry, measurements have been possible. However, the detection of the contaminations is not possible as they are not responding to the THz signal.

Optical coherence tomography

Optical coherence tomography (OCT) is a purely optical, non-destructive, non-invasive, and contactless high -resolution imaging method, which allows the acquisition of one-, two-, or three-dimensional depth -resolved image data of (sub)surface regions in situ and in real -time.

Within the project, the OCT images have been acquired with a spectral-domain OCT system, which is equipped with a multi focal-length probe head. With such a probe head it is possible to easily switch between different imaging optics, and thus, to change the lateral resolution and depth of focus of the probing beam.

The samples have been measured parallel and normal to the fibre direction. However, a significant difference cannot be observed between the samples related to the corresponding application scenarios.

Conclusion

Within WP3, ENDT technologies for the characterisation of CFRP adherend surfaces have been selected, adapted, and validated with regard to their potential to satisfy the application scenarios and requirements. Four different scenarios have been investigated in the test campaigns: Hydraulic fluid/water and release agent contamination, moisture in adherends and heat damage of adherends.

In summary, 19 ENDT technologies have been tested. The results have shown that (i) the hydraulic fluid/water contamination is detectable by 7, (ii) release agent contamination by 4, (iii) moisture in CFRP adherends by 7, and (iv) heat damage of adherends by 4 technologies. Several techniques show good results for the detection of different contamination levels. Five technologies have not been further investigated as they did not comply with the project requirements.

WP4 – Extended NDT for adhesive bond characterisation

In the following, the results are shortly summarized for each investigated ENDT technology followed by a short conclusion.

Nonlinear ultrasound

The aim of this study has been to determine if Nonlinear Ultrasonic techniques are capable of detecting changes in bond strength resulting from contamination. Specifically whether changes in a bond contaminated with release agent could be detected.

Within the project, we have used ultrasonic arrays to produce both linear and nonlinear images which can be compared to determine their sensitivity to contamination. Here multiple independently addressable elements are used to inspect the interior of the specimen. These are used to directly image the interior of the specimen at the excitation frequency and through transmission measurements are made at the harmonic frequency to allow the nonlinearity to be determined. Full matrix capture and the total focussing method are used to produce images of the interior of the specimens. The basic experimental configuration is illustrated in Figure 19 (left).

As illustrated all tests are carried out in water and there are two arrays used, one above and one below the specimen. The array below has a centre frequency twice that of the top array. Ultrasonic array images are produced using back scattered signals in the top array and nonlinear images produced as a result of interaction with the weak bond are generated from measurements of the through transmission case. Measurements were made along the specimens at 12 locations (covering 90% of the volume) so the resulting data represents the trends seen across the complete specimens.

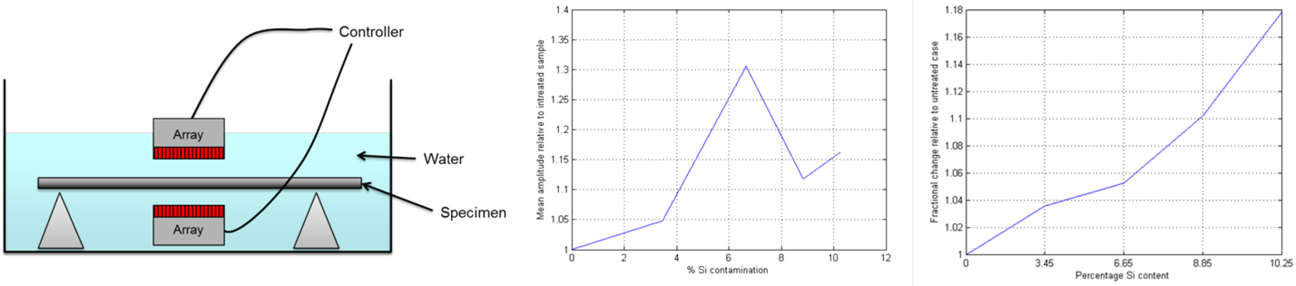


Figure 19: (left) Basic experimental arrangement; Changes in measured properties from linear measurements (middle) and nonlinear harmonic generation (right).

Firstly the linear response has been examined. The mean amplitude of the signals at the bondline is evaluated by taking the mean of all image pixels at the bond depth. This is then plotted against the level of contamination present in the bond at manufacture, best described by the levels of silicon contamination. The resulting performance is shown here in the middle plot in Figure 19. The results show an increasing trend indicating a clear change from the undamaged state. However there are significant anomalies when attempts are made to determine the specific level of contamination.

The result of the nonlinear measurements depicted in the right hand plot of Figure 19 show the measured energy at the harmonic frequency relative to the undamaged specimens. In contrast to the linear measurements these show a clear monotonically increasing trend with increasing levels of contamination, suggesting good sensitivity of nonlinear measurements to this contamination.

Both approaches investigated here have shown sensitivity to contamination being able to detect changes from the base uncontaminated condition. This is better in the nonlinear case; however the measurement is significantly more complex. If the requirement is simply to detect that there is contamination the linear measurement is likely to be adequate, with the added benefit that it will produce an image of the bondline to determine if there are any gross material failures in addition to the changes resulting from contamination.

LASAT technique

In the frame of the ENCOMB project, the LASAT (Laser Shock Adhesion Test) technique has been used to load the contaminated bond interfaces, and thus to evaluate their strength compared to a reference.

The technique is based on the generation of high tensile stresses caused by laser induced shock waves. For that purpose, a high power laser beam is focused on the composite surfaces. The irradiation results in a dense plasma which rapidly expends and creates a shock wave in the material (Figure 20 left). The shock propagation induces tension in the material (Figure 20 middle). The level of tensile stresses is directly correlated to the input pressure meaning the laser intensity. Knowing the level of stresses, a good adhesive bond would not fail under a given loading, when a weak bond would. The main challenge has been to generate the correct amount of stresses on the right position for the given assembly.

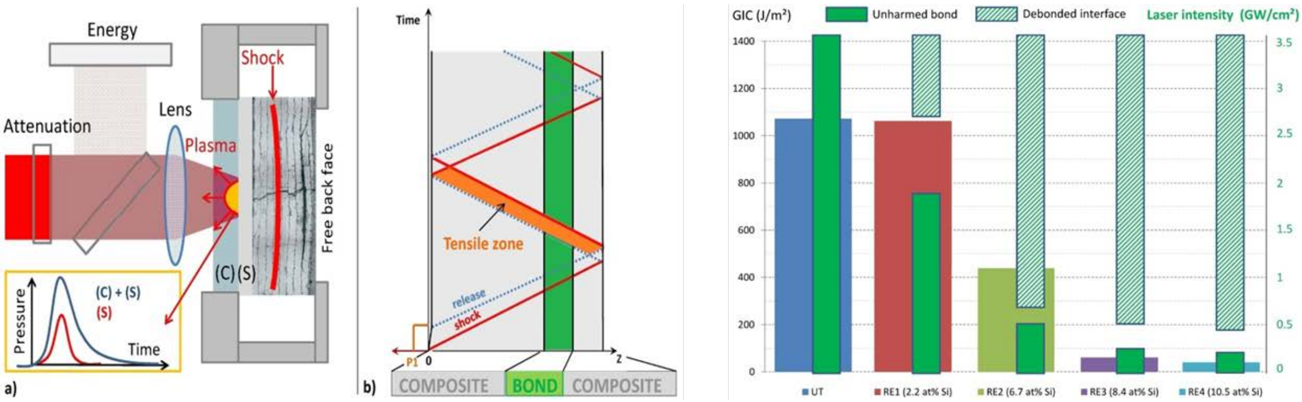


Figure 20: (left) Principle of a shock wave generation by laser in a composite target; (middle) Time/position diagram showing the LASAT principle; (right) Comparison between the LASAT technique results, the GIC measurements and the contamination degrees in case of the release agent scenario (LASAT thresholds has been evaluated thanks to ultrasounds testing).

Within the project, the first step has been to understand the composite dynamic response under laser shock loading. Experiments and numerical simulations have been carried out. Based on the results, we have performed laser shocks on bonded composite using various laser intensities and determined the debonding thresholds of the different bonded scenarios.

The results show that in each case, discrimination between the reference and the contaminated samples is possible. Moreover, a correlation between the contamination degrees and the debonding threshold has been evidenced in particular for the release agent scenario as shown in Figure 20 right. Partially, similar results are found for the other applications scenarios.

The obtained results have also highlighted an optimization problem: the shock parameters have not been correctly adapted to the bonded composite tested. The developed numerical models have thus been used to identify different optimized configurations.

With the performed work a better understanding of the laser shock phenomenon on composite and bonded composite have been achieved. The results clearly demonstrate the great potential of the technique to evaluate the bonding strength of composite assemblies. Further, the optimization of the technique has been investigated. New shock configurations have been found to perform a more efficient test.

THz/GHz reflectometry

For the quality control of the adhesive bonding and in particular to detect air gaps in the adhesive layer of the bonded CFRP sample, we have applied the Quasi-optical method of internal reflection reflectometry (QOIRR).

The technique uses a quasi-optical beam for oblique probing of the CFRP sample surface through the isosceles of a triangular prism located at a distance *h* from the sample surface (Figure 21). The prism is made as a half-cylinder with input and output hollow dielectric beam guides sliding along its lateral cylindrical surface. The prism and the CFRP surface forms a multipass interferometer-resonator in which multiple interaction of the incident beam with the sample at grazing angles of incidence takes place and considerably increases efficiency of the interaction between electromagnetic wave and the sample.

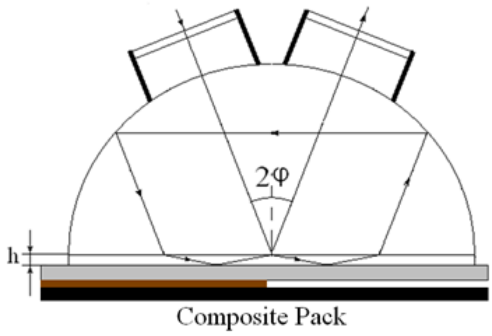


Figure 21: Interferometer-resonator for QOIRR method.

The detection of the quality of the adhesive bonding has been performed by the change of the depth of the interference minimum of the waves reflected from the internal boundary of the quartz half-cylinder and the composite material when irradiating the sample with adhesive bonds of high quality (for calibration) and the sample having adhesive bonds with defects.

The experimental work has been performed in the frequency range of 0.11-0.225 THz for various cases of orientation of the polarization plane of the incident beam with respect to the plane of incidence and the direction of the carbon fibres.

The results reveal that it is not possible to detect defects in the adhesive bonding of the sample at high enough potential of installation (65 dB). The reason is that the CFRP is a multi-layered lattice of parallel conductive carbon fibres located with a step much smaller than the wavelength and with the filling factor close to unity. Such a lattice is partially transparent for the wave polarized orthogonally to the fibre direction and almost completely reflects the wave polarized parallel to the fibre direction. However, the CFRP samples used in the project consist of three orthogonally oriented fibre layers which prevent a penetration of electromagnetic waves into the adhesive bond.

Further, we have analysed the applicability of the QOIRR method for the quality control of an adhesive bond if one of the substrates is transparent in the sub-THz frequency range. A possible composite material for that purpose is glass fibre reinforced plastics (GFRP) which has a dielectric permeability constant of ~3.8 and low losses. We have manufactured adhesive bonded samples consisting of GFRP and CFRP substrates. The results of the measurements show that the change of the interferential minimum depth is 3-4dB when sample is irradiating with a beam polarized parallel to the plane of incidence.

In summary the results show that the QOIRR method does not allow the analysis of adhesive bonded CFRP substrates in the 0.11-0.225 THz frequency range because of high attenuation of electromagnetic waves in the material. However, this method can be used for the nondestructive testing of adhesive bonding when one of the substrates is transparent in sub-THz frequency range.

Laser ultrasound

Laser ultrasound (LUS) has been used to characterize the strength of the adhesively bonded CFRP samples. In principal, a pulsed laser with a wavelength compatible to the absorption behaviour of CFRP is used to excite US waves; a pulse duration of about 50ns provides a broadband US spectrum which is evaluated by a long-pulsed Nd:YAG laser and an interferometer.

The experiments have been performed with bulk waves in pulse-echo (reflection) and through transmission configuration. Furthermore line excitation has been investigated in order to induce surface and Lamb waves. The acquired data has been analysed by bandpass or Savitzky-Golay filtering, Fourier or wavelet transformations and different averaging algorithms.

First the fundamental capability of laser ultrasound has been demonstrated by detecting the location and depth of manually induced delaminations (Figure 22 left/middle).

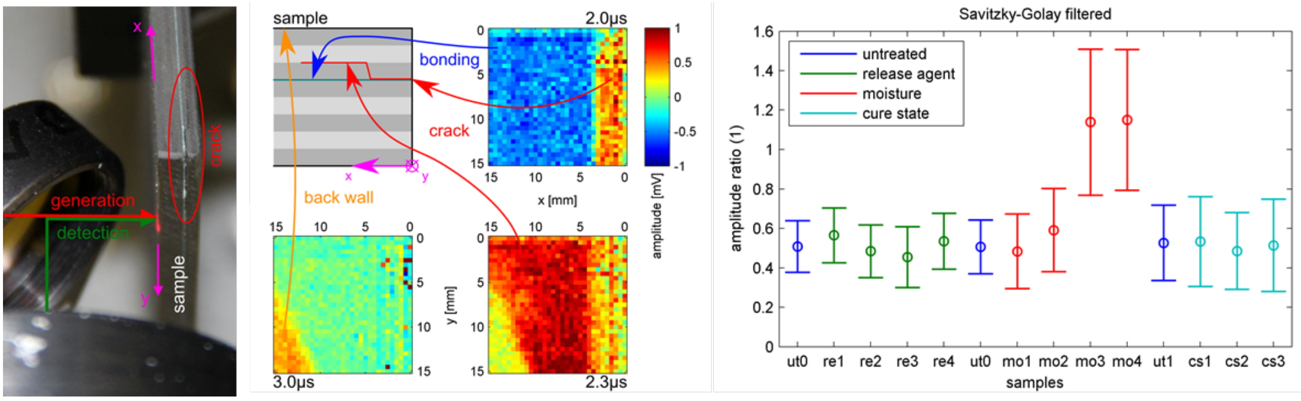


Figure 22: Reflection LUS setup (left) and measurement results (middle) indicating the location and depth of a manually induced delamination (red); Amplitude ratio of bond line and back wall reflection echoes filtered with Savitzky-Golay algorithm (right).

To characterize the bond line quality with bulk waves, an amplitude ratio of the bond line echo and the back wall echo has been calculated. In Figure 22, the results for Savitzky-Golay filtered data are plotted for untreated (ut), release agent contaminated (re), moisture treated (mo) and poorly cured (cs) samples. The different contamination scenarios are undistinguishable from the untreated reference samples (blue) except for two samples from the moisture scenario (mo; mass uptake of more than 1.2 wt%). These measurements are potential outliers and have to be considered with care because the bond line echo is barely distinguishable from noise leading to unreliable amplitude ratios. These uncertainties are also indicated by the increased error bars. An in depth evaluation of the results revealed that the shape of the back wall echo has changed significantly (not shown here), which has not been explained yet.

In conclusion, the work has been shown that the laser ultrasound technique has the capability to detect delamination but with the current configuration information about the bond strength cannot be obtained.

Active thermography using optical excitation

A thermal testing technique with optical heating and an evaluation technique in the time domain have been used for the inspection of adhesive bonded samples. The technique evaluates the thermo-physical properties of the adhesive bond in order to determine if the adhesion quality has been affected by a contamination. Variations in material properties affect the thermal conductivity, specific heat capacity and the density and hence the effusivity of the material is changed.

The experimental setup is shown in Figure 23. The excitation has been performed using a flash light or alternatively, using a LED arrays of 64 single lamps delivering 40 W. For the evaluation of the thermal response directly after the excitation and up to 5 sec after excitation, an IR camera with a 3.6 - 5.1 µm focal plane array and 320 x 256 pixels and a resolution of 25 mK has been used.

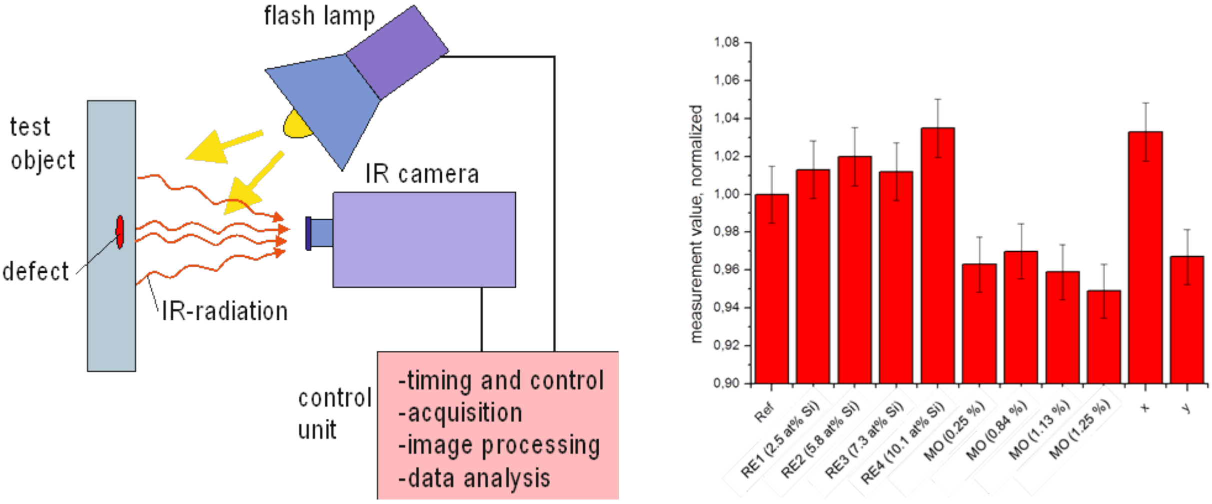


Figure 23: (left) Scheme of the active thermography with optical excitation; (right) Normalized measurement values of all samples at t = 5s, with release agent (RE) contaminated, moisture contaminated (MO) and unknown specimens X and Y. The standard deviation represents 1.5% from the reference value.

The results obtained with active thermography for the characterisation of adhesive bond states reveal clear trends for the detection of an adhesive bond affected by release agent or moisture. Figure 23 (right) shows all results for measurements with an evaluation of the thermal response 5 seconds after excitation in order to get the thermal information from a the bondline region at 1.5 mm depth.

The results show a clearly increasing signal with the amount of residual release agent (RE). In contrast, the results reveal a significant decrease of the signal with increasing moisture content (MO) in the composite substrate. This decrease of the signal is already significant for a moisture content of 0.45 wt.%. This effect is against the expectations as a weaker adhesive bond is assumed to reduce the thermal conductivity and so, give a stronger signal than a reference with a good adhesive bond. Other effects such as plasticisation of the composite substrate could be responsible for this observation.

Unknown specimens (X and Y in Figure 23) used for validation purposed have been successfully discriminated from the reference and attributed to the RE and MO application scenario based on the average signal registered.

Laser scanning vibrometry

Vibrometry measurements have been investigated to detect weak bonds due to pre-bond contamination of one of the surfaces and poor curing of the adhesive.

The experiments have been conducted with scanning laser vibrometer working in 1D mode (Figure 24). During the measurement the sample surface is probed by a laser beam measuring elastic waves propagating in the samples. Due to Doppler Effect the vibrometer measures the displacement velocity along the laser beam. The sample is excited with a piezoelectric transducer using a tone burst excitation signal with 5 cycles.

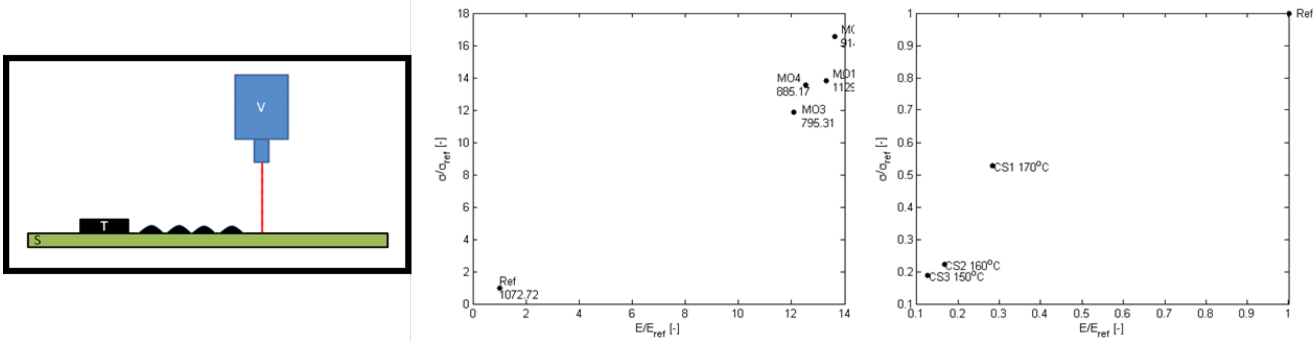


Figure 24: (left) Measurement scheme for vibrometry technique, (middle) Results for moisture contaminated bonds characterization using laser vibrometer; next to the sample symbol the mean G_{IC} value is shown; (right) Results for poorly cured bonds characterization using laser vibrometer; next to the sample symbol the curing temperature is shown.

To analyse the measurement signals, we have employed the following signal processing procedure: at each measurement point an index proportional to the squared signal is calculated. This index is proportional to the squared voltage and can be associated with the out-of-plane vibration energy. The calculated index is averaged over the whole set of measurement points.

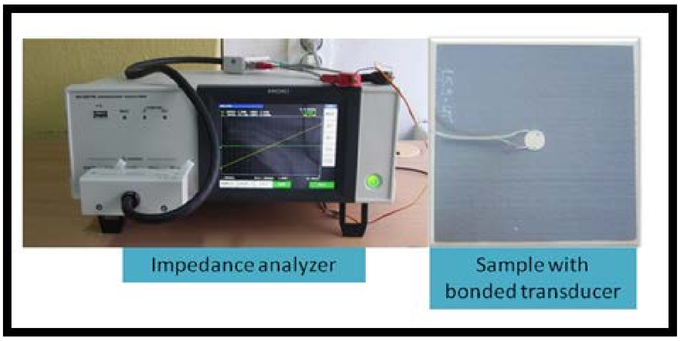
In Figure 24, the results of the vibrometry measurements for the moisture contaminated and poorly cured samples sets are depicted. To emphasize the sensitivity to weak bonds the value of the energy I and its dispersion over the surface represented by the standard deviation (σ) is shown.

The results show that moisture contaminated samples (MO) clearly differ from the reference. Both chosen parameters (E and σ) show a significant increase in comparison to the reference (Ref). The poorly cured samples (CS) also differ from the reference. Moreover, the difference to reference is increasing with decreasing curing temperature. In the case of the release agent contaminated bonds (not shown) a significant difference to the reference has not been observed.

Electromechanical impedance

Electromechanical impedance (EMI) has been investigated to detect weak bonds due to pre-bond contamination of one of the surfaces and poor curing of the adhesive.

The principle of the technique is the measurement of basic electric parameters of a piezoelectric transducer (resistance, reactance, impedance, phase angle). The sensor is attached to the host structure and supplied by a low voltage source (Figure 25). Due to electromechanical coupling the electric impedance spectrum of the transducer is modified by the presence of the host structure. The appearance of additional resonance peaks, their frequency shift or magnitude change can be interpreted as an indicator of defects in the host structure.



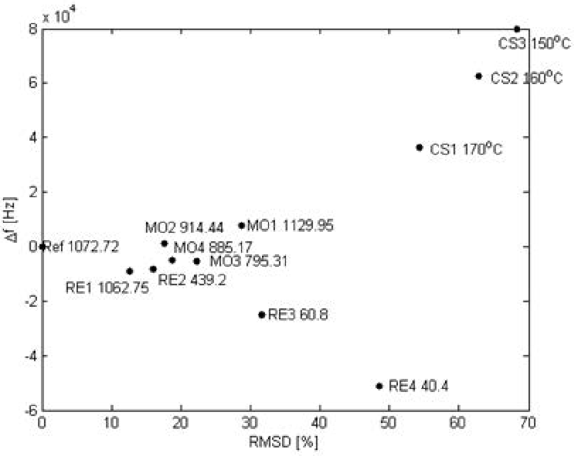


Figure 25: (left) Measurement setup for the EMI technique; (right) Results for the characterization of adhesive bonds (next to the sample symbol the mean G_{IC} value or curing temperature is shown).

Within the research, we have analysed the changes in conductance (G). In order to quantitatively asses the results a Root Mean Square Deviation (RMSD) index and the shift of the peak maximum have been used.

For the samples with a release agent pre-bond contamination, the resulting weak bonds have been successfully detected (Figure 25 right). The measured maximum of the conductance shifts left for all samples. Further, the samples with a weaker adhesive bond, the bond strength decreases from sample RE1 to RE4, show a monotonously increased RMSD value. Moreover, the frequency shift increases also indicating the decreasing quality of the adhesive bond. The exception is the RE1 sample that does not differ from the reference sample in terms of the determined G_{IC} value.

In the case of moisture contaminated samples (MO), for all samples a difference to the reference has been found. Unexpectedly, the samples with a higher degree of contamination (MO2 to MO4) differ less from the reference than the sample with the lowest degree of contamination (MO1). The reason may be connected to the large standard deviation of the G_{IC} values and mixed failure mode.

For the samples with a poorly cured bondline (CS) the results also show an increase of the RMSD index and a frequency shift with decreasing curing temperature.

Ultrasonic frequency analysis

The basis of this technology is the conventional ultrasonic inspection by means of the pulse-echo method. However, in contrast to the conventional technique, we have investigated the frequency band to characterize different bond line defects by their influence on the frequency spectrum obtained by Fast Fourier Transform.

If the frequency response of the material is analysed in the region that has a kissing bond, considerable differences in the dynamic response in the form of changes of frequency and amplitude of the peaks and the disappearance of modal frequencies of vibration are expected. The methodology is divided in three steps (Figure 26): (i) a conventional ultrasonic signal is recorded; (ii) a defined part of the time-of-flight signal is transformed into to the frequency domain; (iii) the frequency spectra is determined; depending on the expected contamination scenario, changes of the bandwidth, the peak or the centre frequency are determined.

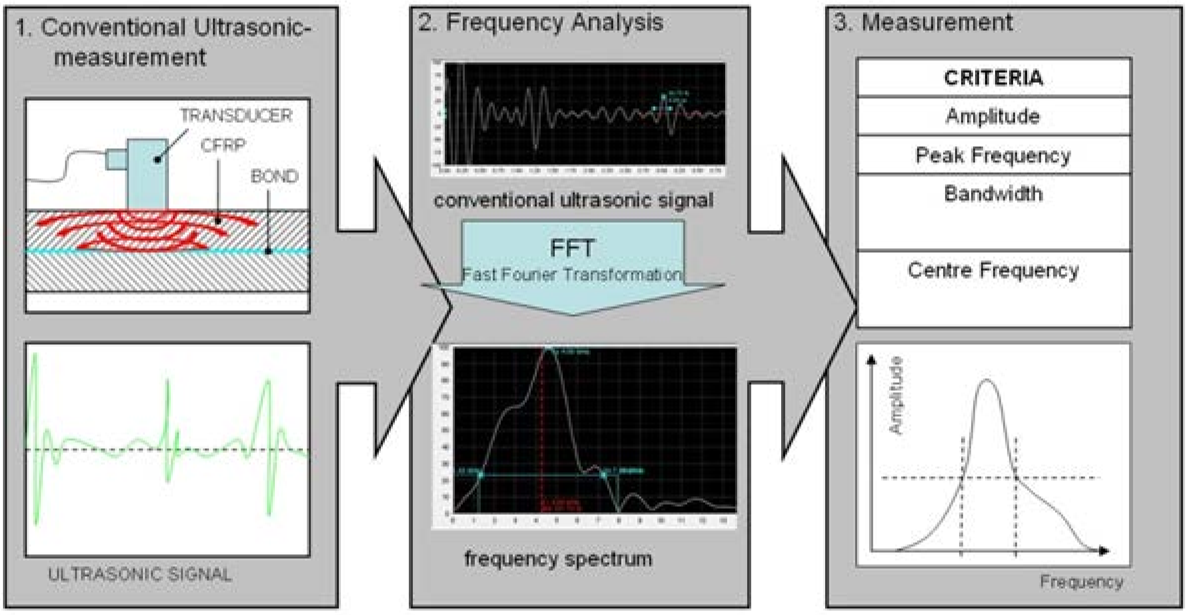


Figure 26: Process steps of the ultrasonic frequency analysis.

In the frame of the project, we have characterised three different contaminated CFRP sample sets to examine the ability of the method to detect the contaminations and to separate between different degrees of contamination.

The moisture contaminated samples show a strong change of the frequency spectra, in particular a change of the bandwidth and the amplitude of the signal. The measurement of the release agent contaminated samples also shows a change of the bandwidth. But a significant change of the amplitude of the spectra is not observed. The results for the curing state of the bond line indicate a strong change of the amplitude of the conventional ultrasonic signal. However, significant change of the frequency spectra that suggest variations of the bond strength are not observed.

In conclusion, the results demonstrate that a weak bond affects the frequency spectra of an ultrasonic signal. However, changes of the properties of the CFRP material affect the measurements too. Though the influence is smaller than the changes caused by a weakened bondline, the nature of the change is the same. This might affect the results of this testing method and limit its applicability.

Industrial laser ultrasound

The objective of the performed work has been to investigate the capability of the Laser ultrasound method (LUS) to discriminate different levels of bonding quality.

The technology requires two lasers, one dedicated to the ultrasound generation and a second one for the detection of ultrasound. The ultrasound generation is based on thermo-elastic effect: the laser beam is absorbed on the top surface and creates a fast increase of the temperature. This consequently induces a mechanical stress that generates acoustic waves propagating toward the material. The ultrasonic detection is performed with the combination of a long-pulse laser very stabilized in frequency and an optical interferometer. The acoustic wave that is reflected on the top surface induces by the Doppler Effect a frequency shift of the detection laser. The interferometer converts the frequency modulation into an intensity modulation that is easily detected with a photo-sensor. The technology is well adapted to the detection of inclusion, delamination and porosity.

Within the project, two LUS systems (LUIS, LUCIE) that are industrial demonstrators devoted to the inspection of large and complex CFRP parts have been used working in a pulse-echo mode (Figure 27 left).

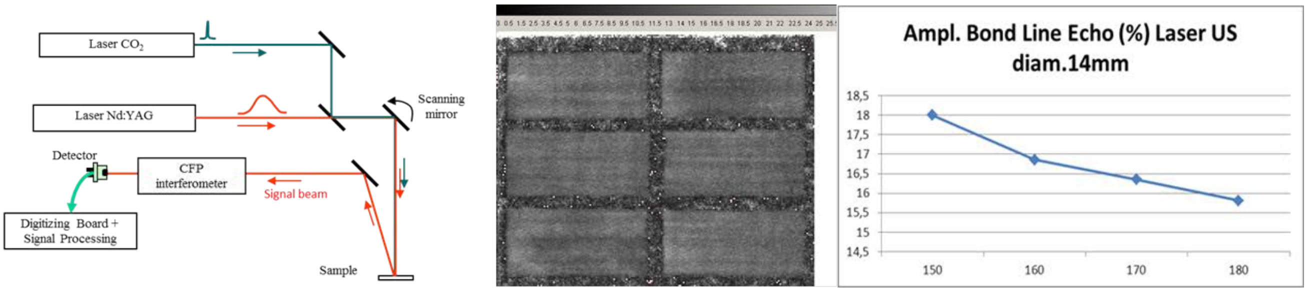


Figure 27: (left) Scheme of the Laser Ultrasonics inspection system; (middle) Amplitude C-scan (%); (right) UT Amplitude for PC samples.

For the application scenario “curing state”, Figure 27 shows the amplitude C-scan of the bond line echo (middle) and the statistic results vs curing temperature (right). A clear correlation between the amplitude of bond line echo and the curing temperature is observed.

Similar results have been found for the samples contaminated with moisture: the amplitude decreases with the level of contamination (not shown). Also there is a good correlation between the G_{1C} results for the contaminated samples and the results from the ultrasonic measurements.

For the sample set contaminated with release agent (not shown), a correlation between the amplitude of the bond line echo and the level of the contamination was not observed.

Conclusion

Within WP4, ENDT technologies for the characterisation of CFRP adhesive bonds have been selected, adapted, and validated with regard to their potential to satisfy the application scenarios and requirements. Three different scenarios of weak bonds have been investigated in the test campaigns: weak bond due to release agent or moisture contamination and poor curing of the adhesive.

In summary, 11 ENDT technologies have been tested. The results show that (i) weak bond due to release agent is detectable by 5 technologies; (ii) weak bond due to moisture by 3; and (iii) poor curing of the adhesive by 4 technologies. Several techniques show good result for the detection of different contamination levels. One of the initial 11 technologies was not employed later test campaigns as it proved not suitable.

Summary

Within the ENCOMB project, 19 extended NDT methods for pre-bond surface quality assurance have been tested and optimized. Up to 10 of them have been developed to reliably detect individual common contaminants down to critical threshold values. 12 extended NDT methods have been investigated and optimized for weak bond detection. Weak bonds due to pre-bond release agent or moisture contaminations have been detected by 6 technologies, poorly cured bonds by 4.

A validation step concluded the ENCOMB developments and has been passed by 5 of the surface-sensitive ENDT methods and by 3 of the bond quality assessment techniques.

The results are summarised in the following Figure 28.

Adherend surface quality

METHOD	PARTNER	Step 1 Scenarios RA	MO	HF	TD	Step 2 Potential for detection of RA	MO	HF	TD	VALIDATION
X-ray fluorescence spectroscopy	IFAM	-	-	✓	-	used as reference methods				
Infrared spectroscopy	IFAM	-	✓	✓	✓					
Reflectometry / Ellipsometry	IFAM	-	-	-	-					
Laser scanning vibrometry*	IMP PAN	✓	✓	✓	✓	○	•	○	○	Fail
Optically stimulated electron emission	IFAM	✓	-	✓	✓	••	•	••	•	Pass
Infrared spectroscopy	RECENDT	✓	✓	✓	✓	•	••	••	••	Pass
Aerosol wetting test	IFAM	✓	-	✓	✓	••	○	•	•	Fail
Portable Handheld FTIR spectroscopy	AGILENT	✓	✓	✓	✓	••	••	••	••	Pass
Laser induced breakdown spectroscopy	IFAM	✓	-	✓	-	••	○	••	○	Pass
THz/GHz reflectometry	IRE NASU	✓	✓	✓	-	•	•	•	○	Pass
Optical fibre sensors*	EPFL	-	✓	✓	-	N/T	••	••	N/T	N/T
Electrochemical impedance spectroscopy*	IFAM	-	✓	✓	-	N/T	•	N/T	N/T	N/T
Electronic nose technology	ENEA	-	-	✓	-	•	•	••	N/T	Fail
Dual-band active thermography	IZFP	-	-	-	-	○	•	•	•	Fail
Laser induced fluorescence	IMP PAN	-	-	-	✓	○	○	••	••	Fail
THz technology	RECENDT	-	-	-	-					
Optical coherence tomography	RECENDT	-	-	-	-					

Adhesive bond quality

TECHNIQUE	PARTNER	Step 1 Scenarios RA	MO	PC	Step 2 Potential to detect weak bonds caused by RA	MO	PC	VALIDATION
Active thermography using ultrasonic excitation	EADS -D	-	-	-				
Terahertz technology	IRE NASU	-	-	-	○	○	○	N/T
Linear Ultrasound	UnivBris	✓	-	-	••	N/T	○	Uncertain
Nonlinear ultrasound	UnivBris	✓	-	-	•	N/T	○	Pass
LASAT technique	CNRS	✓	-	-	••	•	•	Pass
Laser ultrasound	RECENDT	-	-	-	○	○	○	Fail
Active thermography using optical excitation	IZFP	✓	✓	-	•	•	N/T	Uncertain
Laser scanning vibrometry*	IMP PAN	-	✓	-	○	•	••	Fail
Electromechanical impedance*	IMP PAN	✓	✓	✓	••	•	••	Fail
Ultrasonic frequency analysis	EADS-D	-	✓	-	•	••	○	Pass
Laser ultrasound	EADS IW F	-	-	-	○	•	•	Fail
Active thermography (for T_g analysis)	IFAM	-	-	-				

Figure 28: Overview of ENCOMB results; ✓: Clear detection of contaminant, differentiable from reference surface state, -: No differentiation from reference state, ••: High, •: Low, ○: No, N/T: Not Tested, *: With structure integrated sensor.

4.1.4 Potential impact (including the socio-economic impact and the wider societal implications of the project so far) and the main dissemination activities and exploitation of results

In order to extend the use of adhesive bonding for load-critical CFRP primary aircraft structures quality assurance processes for ensuring the performance of the adhesive joints have to be developed. The ENCOMB project has targeted this objective by identifying, screening, developing, and adapting NDT methods for the characterization of CFRP bonded structures, the characterization of the CFRP adherend surface state before bonding and the state of the cured and uncured adhesive.

Within the project major innovations with a strong impact on future manufacturing and repair of aircraft structures have been achieved, namely:

- Analytical techniques for characterizing adherend surfaces and for the assessment of the quality of adhesive bonds.
- Knowledge about the effect of different contaminations on the adhesion properties and the overall bond performance according to the selected application scenarios.
- Algorithm(s) that will provide bonding guidelines.
- Quality assurance concept(s) for adhesive bonding of primary structures in aircraft manufacturing and in-service environment.

The impact of the technological and scientific results of ENCOMB is summarized in the following figure.

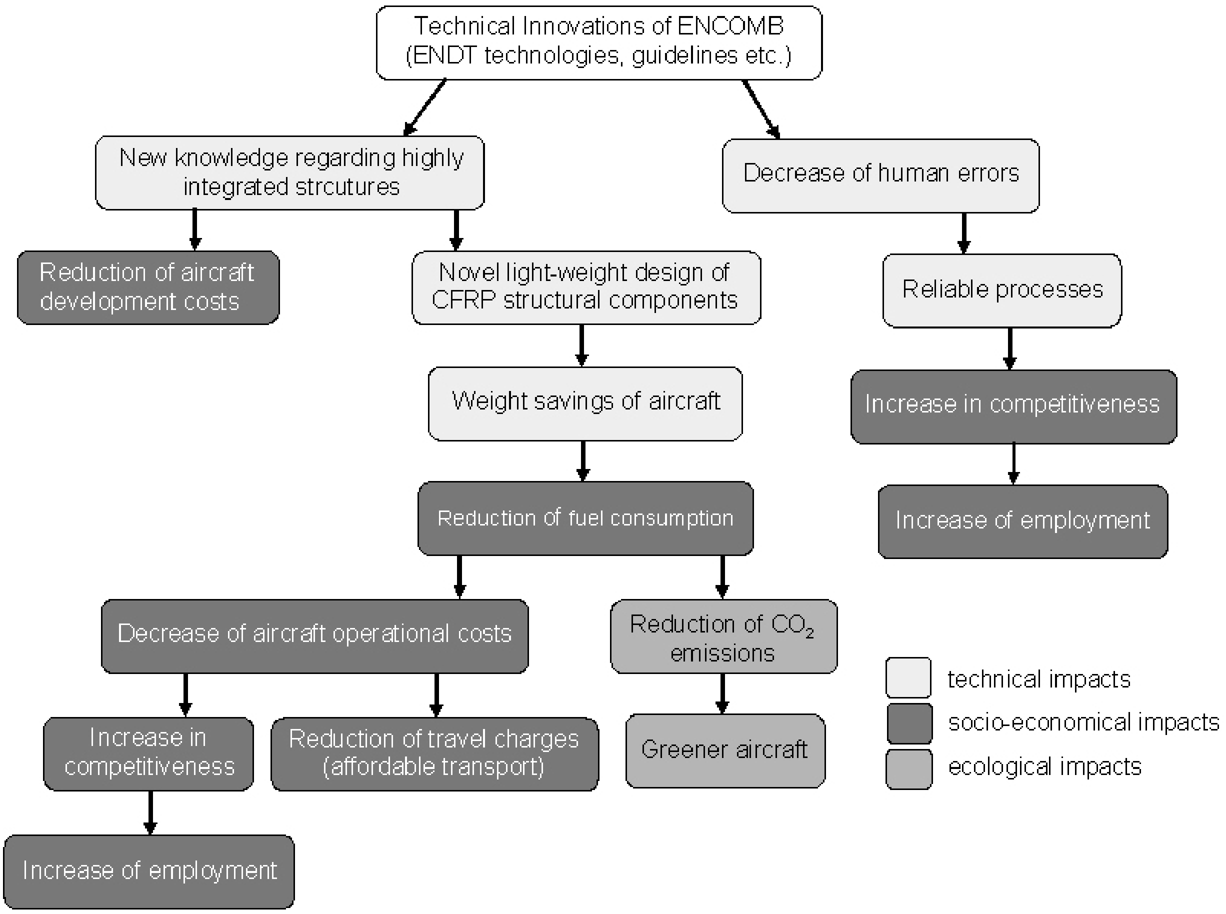


Figure 29: Technical, environmental and socio-economic impacts of ENCOMB.

Fostered by the development of robust and reliable adhesive bonding processes and the related quality assurance techniques, the increased use of advanced light-weight composite materials reinforces the competitiveness of Europe's aircraft industry and European aircraft operators and thus employment is secured or even increased.

In the year 2011, the European aerospace industry employed 479.600 persons[2], Europe's airline industry employed approximately 390.000[3] persons. Both the aircraft production and airline operation industries are active in the most globalised markets; therefore the conservation and future expansion of employment will necessitate a high levels of competitiveness. The project results will play a key role in addressing this issue, both by generating added product value for aircraft manufacturers and reduced costs for operators.

On a socio-economic level, the achieved innovations will contribute to a reduction of fuel consumption which is directly connected to the operational cost of an aircraft leading to more affordable air transportation.

Lower fuel consumption has further significant ecological and societal benefits through reduced emission of CO_2 and other toxic gases. Beside the ecological issue, there are societal demands for the reduction of energy consumption in transport and a higher ecological awareness from citizens and governments on the effect that uncontrolled growth can have on the environment. Aeronautics stakeholders have proactively complied with these demands by establishing roadmaps providing perspectives for the greening of air transport. The Advisory Council for Aeronautics Research in Europe (ACARE) has elaborated a strategic research agenda listing ambitious goals for the Aeronautic Industry in Europe. One of them is a 50% reduction of CO_2 emissions per passenger/km, i.e. a halving of the fuel burnt (based on the status in the year 2000). This shall be achieved through a 15-20% reduction from the engine side, 5-10% from optimised air traffic management (ATM), and 20-25% from structures. Just recently even more ambitious goals have been elaborated by the High Level Group on Aviation Research and published by the European Commission in "Flightpath 2050 –Europe's Vision for Aviation", which aims even for a 75% reduction of CO2 emissions (with the same baseline in the year 2000) by the year 2050.

The results of this project, in particular the novel ENDT technologies, quality assurance processes and the scientific progress regarding adhesive bonding, directly facilitate the implementation of adhesive bonding processes for highly integrated CFRP structures. The expected weight savings for the fuselage airframe are up to 15%. These weight savings have further effects on the size and weight of the engines. This represents an important contribution towards the greening of European air transport.

Outside the civil aircraft sector, the implementation of ENDT technologies will be a promoter for new applications in different European industrial sectors that make use of fibre reinforced lightweight structures (e.g. transportation, wind energy industry). This can help other industries to improve their competitiveness and market share, a requirement for further employment increase in Europe.

Further the project results will have the following technical impacts (Figure 29):

(i) Novel light-weight design of CFRP structural components resulting from the obtained knowledge about assembly of highly integrated CFRP structures. If reliable bonding processes (by using ENDT technologies) are available a huge increase of lightweight structural composite components is expected which is as outlined above directly connected to weight savings.

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[2] European Association of Aerospace Industries, Key Facts and Figures 2011, Sep. 2012
[3] AEA NEWS, Issue 5, 2012

(ii) A reduction of maintenance costs (e.g. by reducing the time for in-service repair) ensuring reliable (in-service) repair by adhesive bonding at the same time.

(iii) Reduced aircraft development costs by the achieved knowledge and expertise regarding highly integrated structures with an optimum combination of advanced composite materials eliminating and minimising the number of join/assembly.

(iv) The decrease of human error during production, maintenance, and repair which directly results in more reliable processes and increased safety of air transportation.

Main dissemination activities

The main dissemination tools used and activities performed during the project for providing information to the relevant aeronautic industrial and academic community include:

- The ENCOMB public website,
- The ENCOMB leaflet, poster and roll-up banner which includes information of the project progress, main achievements and final results of the project, and
- The implementation of numerous ENCOMB related dissemination activities (e.g., publications in European / International Journals, Presentations in International Conferences, newsletters, poster displays and leaflet distributions) addressing the full range of potential end users and uses of the ENCOMB results, including research, commercial, investment, environmental, setting standards, skills and educational training.

ENCOMB website

The project website (http://www.encomb.eu/) designed and maintained up to date by EASN-TIS is the main dissemination tool for communicating the progress and the important research results produced within the project to the wider public (see Figure 30). The ENCOMB public website has been constantly updated with the progress of the project mainly including the work performed and the significant results produced within the project.

Figure 30: Screenshot from the "Dissemination" and "Latest News" section of the website (http://www.encomb.eu/)

In addition, a section entitled “Dissemination” dedicated to the inclusion of all performed ENCOMB related dissemination activities has been established and was constantly updated with the publishable information (i.e., the author(s) name(s), titles of the disseminated materials and events and a reference link – if available) of all performed dissemination activities, i.e., scientific publications, conference presentations, workshop presentations, poster presentation, etc. The ENCOMB related dissemination materials are kept in the respective archive in the project’s private area in order to enable the Commission to trace each ENCOMB related dissemination activity.

Further, the “Dissemination” section of the website includes all dissemination material created from the beginning of the project. In particular, this webpage enables the visitor to view and download the ENCOMB poster and leaflet, as well as the project roll-up banner and the updated and final versions of the leaflet, poster and roll-up banner, which includes the latest and final information about the progress and achievements of the project.

ENCOMB leaflet poster & banner

During the project, a leaflet (Figure 31) as well as a project poster and banner (Figure 32) have been created. The final version of the leaflet, poster and banner include information about the final results and achievements of the project throughout its duration. A number of copies of the project leaflet and poster have been distributed to all consortium members in order to be used as dissemination means at conferences, workshops, seminars and any other event that the ENCOMB partners might participate in the future. The banner is kept on the Coordinator premises and is available for use by any partner who intends to perform any project related dissemination activity. The final version of all project related dissemination materials, (i.e. leaflet, poster and banner) is available for download from the ENCOMB public website (flyers: http://www.encomb.eu/flyers and poster/banner: http://www.encomb.eu/posters).

Figure 31: Final version of the ENCOMB leaflet (external and internal side).

ENCOMB section in the EASN newsletter

The distribution of news, achievements and progress to a large number of scientists and professionals throughout Europe was performed through the EASN newsletters. This initiative started at the beginning of the project and continued throughout its duration. In particular, the project developments and achievements were included in the EASN newsletter issues dating July 2011, December 2011, July 2012, December 2012, March 2013, July 2013, November 2013, April 2014 and links to the project website were provided for further information.

ENCOMB results will be published in the July 2014 issue of the EASN newsletter as well with information about the overall achievements of the project and final results.

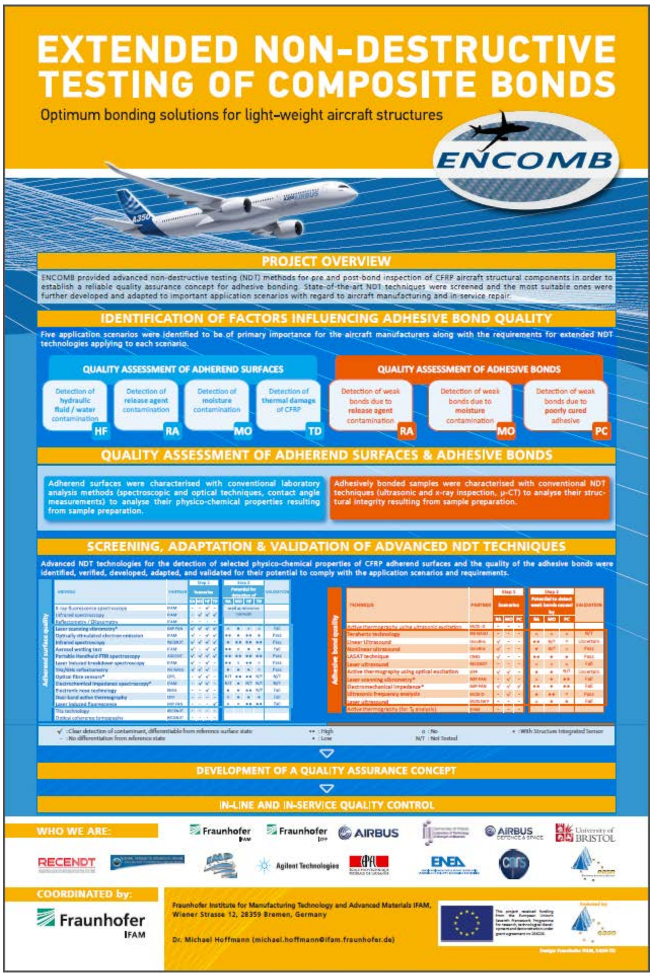


Figure 32: Final version of the ENCOMB poster (left); Final version of the ENCOMB banner (right).

ENCOMB video

The main objective of the video is to present the activities and results of the ENCOMB project by visualising specific tests and other research activities performed by the partners. This video will not only be used as a tool for informing the wider public about the activities and results achieved within the project but it will also be used as an advertising tool by each partner for the work performed within the project beyond its duration.

The content of the video is based on the sequence of the research activities performed within the project. In the beginning some general information regarding the project and its state-of-the-art is included followed by a brief description of the activities carried out in each work package. The video is focused on the presentation of the technologies tested for both adherend surface and adhesive bond quality which are the main research activities of the project and is publically available through the ENCOMB website.

Scientific publications

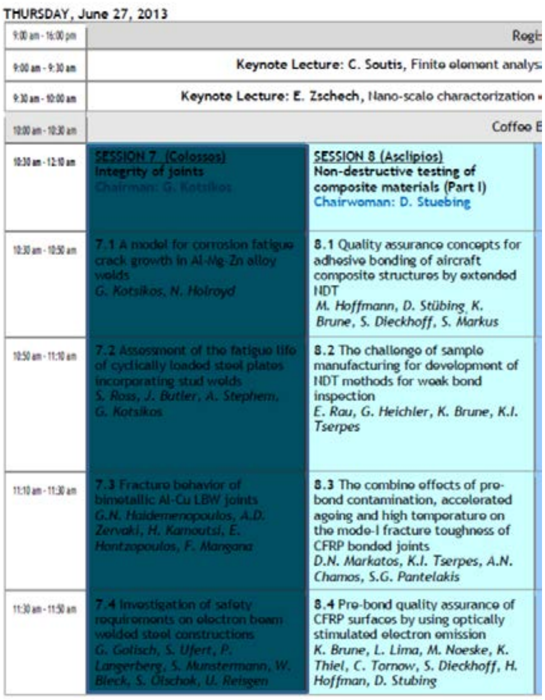
Throughout the project, the results have been made available to the scientific community in peer reviewed scientific publications. At this point it is worth highlighting the high number (56) of peer reviewed scientific publications already published in international journals and conference proceedings. In addition, 17 peer reviewed scientific publications are planned to be published (either in journals or conference proceedings) after the completion of the project. Through these publications (both performed and foreseen), the ENCOMB results have already been and will be made publically known to a large number of scientists.

Conference sessions

During the project, results and achievements have been presented to the scientific community by two dedicated ENCOMB sessions at the following conferences/workshops:

- The 3rd International Conference of Engineering Against Failure, 26-28 June 2013, Kos, Greece

  "The scope of the Conference has been to attract interdisciplinary work dedicated to the design against and prevention of engineering failure. Works are expected to cover a number of different technological areas including Aeronautics, Construction, Automotive, Bioengineering, Recycling, etc."
  (http://ltsm.mead.upatras.gr/lab/lang_en/conference/view/2)



THURSDAY, June 27, 2013

9:00 am - 16:00 pm	Regi	
9:00 am - 9:30 am	Keynote Lecture: C. Soutis, Finite element analys	
9:30 am - 10:00 am	Keynote Lecture: E. Zschech, Nano-scale characterization	
10:00 am - 10:30 am	Coffee B	
10:30 am - 12:10 am	SESSION 7 (Colossos) Integrity of joints Chairman: G. Kotsikos	SESSION 8 (Asclipios) Non-destructive testing of composite materials (Part I) Chairwoman: D. Stuebing
10:30 am - 10:50 am	7.1 A model for corrosion fatigue crack growth in Al-Mg-Zn alloy welds *G. Kotsikos, N. Holroyd*	8.1 Quality assurance concepts for adhesive bonding of aircraft composite structures by extended NDT *M. Hoffmann, D. Stübing, K. Brune, S. Dieckhoff, S. Markus*
10:50 am - 11:10 am	7.2 Assessment of the fatigue life of cyclically loaded steel plates incorporating stud welds *S. Ross, J. Butler, A. Stephem, G. Kotsikos*	8.2 The challenge of sample manufacturing for development of NDT methods for weak bond inspection *E. Rau, G. Heichler, K. Brune, K.I. Tserpes*
11:10 am - 11:30 am	7.3 Fracture behavior of bimetallic Al-Cu LBW joints *G.N. Haidemenopoulos, A.D. Zervaki, H. Kamoutsi, E. Hontzopoulos, F. Mangana*	8.3 The combine effects of pre-bond contamination, accelerated ageing and high temperature on the mode-I fracture toughness of CFRP bonded joints *D.N. Markatos, K.I. Tserpes, A.N. Chamos, S.G. Pantelakis*
11:30 am - 11:50 am	7.4 Investigation of safety requirements on electron beam welded steel constructions *G. Golisch, S. Ufert, P. Langerberg, S. Munstermann, W. Bleck, S. Olschok, U. Reisgen*	8.4 Pre-bond quality assurance of CFRP surfaces by using optically stimulated electron emission *K. Brune, L. Lima, M. Noeske, K. Thiel, C. Tornow, S. Dieckhoff, H. Hoffman, D. Stubing*

Figure 33: Excerpt from the conference program highlighting the dedicated ENCOMB session.

- 3rd EASN Association International Workshop on Aerostructures, 9th until 11th October 2013, Milan, Italy

  "More than 120 participants from Industry, research organizations and Academia attended the Workshop and a total of 90 presentations were performed in 3 parallel sessions. In addition, 36 scientific papers have been published in the Workshop proceedings, some of which will be selected and submitted in scientific journals. Moreover, scientific results from 27 running research projects were presented thus accenting the Workshop as a major European dissemination event for new knowledge and emerging technologies related to aerostructures." (http://www.easn.net/workshops/1/20/)

Summary

The performed dissemination activities can be classified into the following categories: Scientific publications in journals or conference proceedings, Conference/Workshop presentations, Web-based project information, Press releases, Flyer distribution, Poster display, Project related video, Presentations, Exhibitions, and Thesis. The following figure (Figure 34) illustrates the breakdown of the different categories of dissemination activities as reported by the consortium.

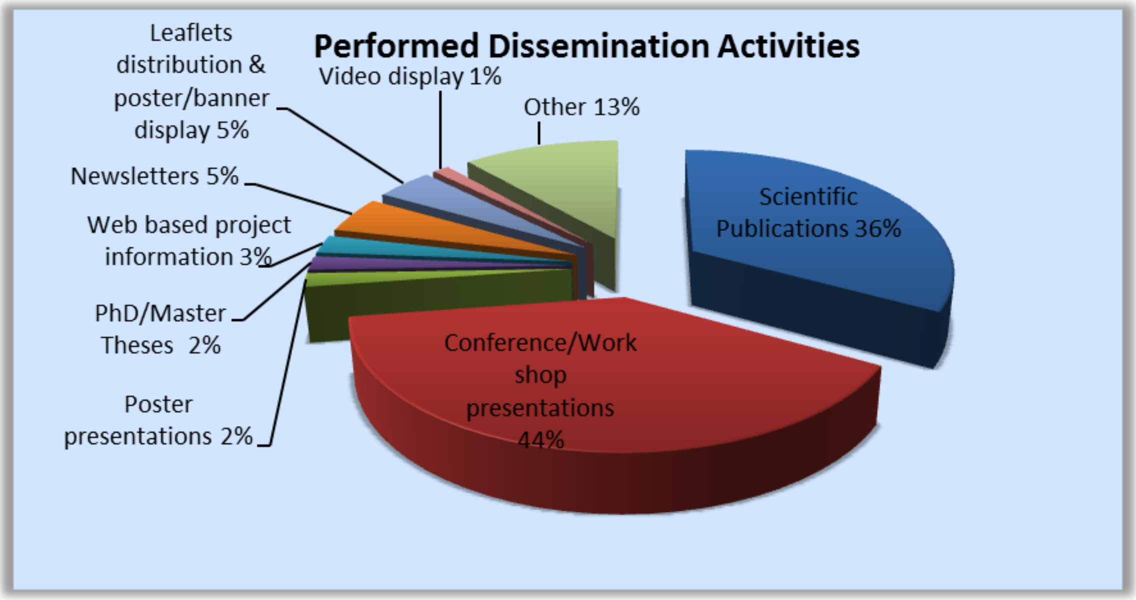


Figure 34: ENCOMB performed dissemination activities.

The majority of the dissemination activities are Conference/Workshop presentations (44%) and scientific publications (36%). These figures reflect the strong representation of universities and research organisations in the consortium. It is worth mentioning that the majority of the scientific publications have been performed in international highly-ranked, peer-reviewed scientific and technical journals. Further to that, other dissemination activities involved: ENCOMB related articles in newsletters (5%), leaflets distribution and poster displays (5%), web based project related information (3%), ENCOMB related PhD/Master theses (2%), Poster presentations (2%), video display in websites and/or conferences (1%) and other dissemination activities, i.e. application notes, internal working groups (13%).

Exploitation of results

In addition to the dissemination activities, the consortium also performed a number of exploitation activities. These activities have been reported in the PUDK and fall into the following categories: Patent, General advancement of knowledge, and Commercial exploitation of R&D results.

The participation of the major European aircraft manufacturer in the consortium makes the rapid acceptance and approval of the new developments and exploitation of project results for aeronautic applications very likely. Hence, it is expected that the project will contribute significantly to the growth and competitiveness of the European aeronautic industry. It is worth mentioning that all consortium partners spread information about the developed technologies between each other so as to ensure full exploitation. Interaction between consortium members enabled the exploitation of specific technologies through the leading industrial participants by incorporating new technologies into future products.

Based on the exploitation activities performed by the partners, two commercial exploitable foregrounds of R&D results and five general advancements of knowledge have been generated within the project.

4.1.5 Project website

http://www.encomb.eu