Evaluation of bonding quality in CFRP composite laminates by measurements of local vibration nonlinearity

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Abstract

A conventional way to evaluate nonlinearity in composite laminate plates is based on the second harmonic measurements for propagating Lamb modes. In this paper, a new approach based on measurements of a local nonlinear response of the laminate is suggested and applied to characterizing contaminations of adhesive bonding in carbon fibre-reinforced polymer (CFRP). It is shown that a contaminated boundary layer of the adhesive contributes to an overall nonlinear response of the laminate that enables to evaluate and quantify bonding quality caused by various types and levels of single contaminations. All kinds of single contaminations studied in the context of aviation applications result in enhancement of the nonlinear response of the CFRP laminate which is an indication of deterioration of bonding quality. The enhancement of nonlinearity in weaker bonds was also applied to imaging of the contaminated adhesion areas in realistic airplane components.

1. Introduction

The merits of light-weight, high-strength composite materials characterized by unprecedented performance, energy efficiency, safety, and environmental compatibility are widely recognized and applied in many sectors of economy. To provide manufacturing and maintenance flexibility of high performance composite components the role of technology for joining and consolidating composite parts and other materials is of primary importance. In this context, the need of adequate nondestructive testing (NDT) and evaluation (NDE) of interfacial adhesion which determines reliability of structural bonding has long been considered as a challenge for the NDT/E community.

Conventional ultrasonic NDE instruments used in industry and technology make use of the so-called \textit{linear elastic response} of materials which results in the amplitude and phase variations of the input signal due to its interaction with defects. The nonlinear approach to ultrasonic testing is concerned with \textit{nonlinear material response} related to the frequency changes of the input signal. These spectral changes are caused by nonlinear dynamics of solids which scales from inter-atomic level for perfect materials to meso- and macro-scale nonlinearity for damaged areas (1-3). In many cases, monitoring material nonlinearity reveals directly the vulnerable areas within material or a product with sensitivity far superior to traditional ultrasonic inspection.

A conventional way to evaluate nonlinearity in composite laminate plates is based on the second harmonic measurements for propagating Lamb modes (4,5). The approach proposed in this research makes use of a local nonlinear response of the
adhesive bond. A contaminated bonding layer with weaker adhesion is supposed to have a higher nonlinearity that enables to recognise, evaluate and visualise the difference in bonding quality.

2. Nonlinear NDT: methodology and results

2.1 Specimens preparation

The measurements were carried out for a large set of composite specimens with different bonding conditions. The samples (10x10 cm²) manufactured according to Airbus AIPS 03-02-019 comprised two 8-ply CFRP laminates bonded with an epoxy adhesive layer FM300K from Cytec®. Hexcel M21E® is the material that was used for the composite laminates with the given stacking sequence [0, 0, 45, -45, -45, 45, 0, 0].

Two typical stages during the life of a structural part for which the adhesive properties of a bonding joint could be degraded were considered: the production process and the maintenance and repair scenario. All samples for production scenarios were bonded by using the adhesive FM® 300K (0.2) from Cytec while the specimens for repair scenarios were bonded by using the adhesive FM® 300-2.

For production scenario, the following kinds of contaminants were studied: Release agent (RA) and fingerprint (FP).

Release agent (silicon-based) is used during the molding process to ease the demolding of the parts. The release agent used was FREKOTE® 700NC. It was applied to the surfaces by dip coating before bonding.

Fingerprint is typical for both manufacturing and repair scenarios due to inappropriate handing of the part. Samples were prepared using a standardized salty fingerprint solution (artificial hand perspiration solution) according to DIN ISO 9022-12. This solution contains sodium chloride, urea, ammonium chloride, lactic acid, acetic acid, pyruvic acid and butyric acid in demineralized water. Samples were prepared by applying this solution with the size of a fingerprint to the samples. Various degrees of contamination were achieved by using different dilutions (with demineralized water) of the FP solution: FP1: 10% FP solution, FP2: 50% FP solution and FP3: pure FP solution.

For repair scenario, the De-icing fluid (DI) contaminant was applied. The other two processes that could result in a loss of adhesion due to external influences or errors during bonding process were also studied: Thermal degradation (TD) and Faulty Curing (FC).

De-icing fluid is relevant to the repair environment: Residues of potassium formiate of the de-icing fluid on the outer surface of an aircraft may end up in the repair areas and finally lead to an adhesive failure of the bonding. The de-icer used was SAFEWAY® KF from CLARIANT. It was diluted with demineralized water to obtain solutions with the following concentrations in percent of volume: 2% (DI1), 7% (DI2), and 10% (DI3). The solution was applied to the surfaces by dip coating (aqueous solution) and dried in the oven for 2h at 40°C.

Thermal degradation can cause local overheating and damage of resin in CFRP. For different degrees of thermal degradation the samples were stored for 2h at the following elevated temperatures: 220°C (TD1), 260°C (TD2), and 280°C (TD3).

Faulty Curing: Inappropriate use of the adhesive or wrong curing cycles may lead to a loss of the adhesive properties in both manufacturing and repair scenarios. An IR
spot light was used to perform the adhesive curing, and the temperatures used were as follows: 120°C (FC1), 140°C (FC2), and 160°C (FC3).

For each type of contamination in both scenarios, a set of three specimens of every level of contamination was prepared for reliable statistics. Along with other three reference specimens (REFR and REFP) (non-contaminated adhesive) for each of the scenarios a total number of the specimens to be tested amounted to 60.

2.2 Testing technique and results

The nonlinear technique is based on the local generation of high amplitude vibrations and detecting the higher harmonics in the excitation area. Commercial piezo-actuators (isi-sys) with a frequency response extended from low kHz into high kHz range (above 100 kHz) were applied in the experiments. The actuators are driven by a CW voltage generated by the HP 33120A arbitrary waveform generator and are vacuum-attached to one of the sides of the specimen while nonlinear vibrations are measured on the opposite side of the specimen. To measure and analyze the frequency content of the vibrations generated locally in the excitation area a scanning laser vibrometer (SLV, Polytec 300) operating in the vibration velocity mode with maximum frequency bandwidth of 1.5 MHz was used.

The dynamic range of the SLV measurements (100-120 dB) is well beyond the level of nonlinear frequency components. In the experiments, the vibration amplitude was measured to be in the range of (4-5)10⁻⁸ m so that a local strain developed in the excitation area is ≈10⁻⁵. This strain is sufficient for manifestation of noticeable local nonlinearity in composite materials.

To avoid an impact of reflections on the local vibration in the excitation area, the edges of specimens were covered with dissipative material and rather high vibration frequency was chosen (49 kHz). With these precautions, the spectrum and the temporal pattern of the vibrations are measured directly in the acoustic source area (a circle with ≈5 mm radius) where no plate wave propagation is involved yet.

Each value of the fundamental frequency vibration velocity \( v_0 \) and the higher harmonic components \( v_n \) measured in the probing area was used for evaluation of the nonlinear ratio \( N_i = \frac{\sum v_n^2}{v_0^2} \). An average value of the nonlinear ratio in the probing area was then calculated \( N = \frac{\sum N_i}{m} \) (where \( m \) is the number of measurements) and the standard deviation of the results was estimated as \( \Delta N = \sqrt{\frac{\sum (N_i - N)^2}{m(m-1)}} \).

The measurements were repeated in various points over the central part of the specimen to provide the relative error \( \Delta N/N \leq 10\% \).

The nonlinear ratio \( N_i \) is a part of the vibration energy \( \sim v_0^2 \) converted into the higher harmonics \( \sim v_n^2 \) so that it clearly quantifies material nonlinearity. To avoid the influence of the amplitude-dependent effects in estimation of the nonlinear ratio \( N \) the amplitude of the input voltage of the transducer was kept constant (20 V) over the course of measurements. As a result, in all the specimens measured the fundamental vibration amplitude was virtually constant within \( \sim 10\% \) deviation.
The results of $N$ measurements are plotted in Figs. 1-7 in the way to trace the impact of the contamination level (the number right after the contamination notation (1, 2, 3)) for a particular specimen number (-1, -2, -3):

1. According to Figs. 1-2, the reference specimens (without contamination of the adhesion layer) reveal the minimal values of $N \approx 3$. The contamination of adhesive noticeably enhances the nonlinearity for all specimens by 1.5 - 2 times. The maximum nonlinear ratio $N$ was obtained in TD 3-2 specimen ($N = 50 \pm 6$, outside the scale in Fig. 3). In this specimen, the $N_i$ values were found to depend on the position of the measurement point. This fact, along with anomalously high value of the nonlinear ratio indicates the presence of local delamination in the specimen induced by thermal activation.

2. For the contaminations types TD, RA, FP, DI, and FC material nonlinearity increases with the increase of the level of contamination (Figs. 3-7). Since nonlinearity enhances due to “softening” of the material, the increase in $N$ is an indication of “weakening” of the adhesive bonding.

3. For each type of contamination, the values of $N$ change noticeably with variation of the contamination level that indicates the sensitivity to the changes in the thin boundary layer between the adhesive and the adherends. According to the measurement results, the sensitivity of the nonlinear method is, therefore, sufficient to recognize the effect of contamination on the adhesive bonding.
2.3 Testing of bonding quality in realistic aviation components

The enhancement of nonlinearity in weaker bonds shown above was also applied to mapping and imaging of the contaminated adhesion areas in realistic airplane components. The components tested are shown in Fig. 8. Component N1 belongs to the production scenario with a contaminated part of the lower stringer (di-icing liquid + a few fingerprints, DI+FP). The CFRP patch in component N2 illustrates a repair scenario when a part of the patch is contaminated in a similar way. The exact location of the contaminated areas was not known before the experimental testing.

A piezo-stack transducer and high-power supply (Branson Ultrasonics) provided μm-range displacements at the fundamental frequency of 20 kHz in the source area (nonlinear regime). According to the above, the higher harmonics are supposed to be generated locally in the contaminated areas (the stringer in sample N1 and the patch in component N2) with the efficiency dependent on the adhesion quality. To visualise the nonlinear areas the spectrum of the local vibrations over the specimen surface was probed with a laser vibrometer and the colour-coded distributions of the higher harmonics were mapped.
In the experiment with specimen N1, the excitation transducer was positioned symmetrically in regard of the stringer to be tested (Fig. 9). Such a position provided symmetrical insonation 20 kHz field of the stringer in question as shown in Fig. 9.

The second higher harmonics measured in specimen N1 demonstrates quite different distributions shown in Fig. 10: the higher efficiency of the local nonlinear generation in the left hand-side of the stringer is clearly seen and identifies a contaminated area with lower bonding quality.

![Figure 8](image8.png)

**Figure 8.** The specimens tested: 80x80 cm² CFRP component (N1) with 2 stringers (production scenario, left); 100x80 cm² CFRP component (N2) with repair patch (20 cm diameter, right).

![Figure 9](image9.png)

**Figure 9.** Distribution of the fundamental frequency (20 kHz) ultrasonic amplitude along the stringer length in specimen N1.

The second higher harmonics measured in specimen N1 demonstrates quite different distributions shown in Fig. 10: the higher efficiency of the local nonlinear generation in the left hand-side of the stringer is clearly seen and identifies a contaminated area with lower bonding quality.

![Figure 10](image10.png)

**Figure 10.** Distribution of the second higher harmonic (40 kHz) along the stringer length in component N1.
In component 2, the fundamental frequency field visualised in Fig. 11 shows a rather conventional standing wave pattern over the whole area of the repair patch. On the opposite, the higher harmonic fields measured in the patch shown in Fig. 12 demonstrate quite different nonlinear field distributions. They reveal a strong enhancement of the nonlinearity and therefore lower bonding quality in the same (the right hand-side and lower) parts of the patch for the 2d and 3d higher harmonics (Figs. 12, a, b).

**Figure 11.** Fundamental frequency field excited in the repair patch of specimen N2.

**Figure 12.** The second harmonic (40 kHz, top) and the third harmonic (60 kHz, bottom) fields in the repair patch of specimen N2.
3. Conclusions

1. The nonlinear measurements revealed an increase of nonlinearity caused by contamination of adhesive layer for majority of types of single contaminations. For each type of contamination, the nonlinearity changes noticeably with variation of the contamination level that indicates the sensitivity to the changes in the thin boundary layer between the adhesive and the adherends. According to the measurement results, the sensitivity of the nonlinear method is, therefore, sufficient to recognize the effect of contamination on the adhesive bonding.

2. For the contaminations types TD, RA, FP, DI, and FC material nonlinearity increases with the increase of the level of contamination. Since usually nonlinearity enhances due to “softening” of the material, the increase in $N$ is an indication of “weakening” of the adhesive bonding.

3. The enhancement of nonlinearity in weaker bonds was also applied to mapping and imaging of the contaminated areas in realistic airplane components. The accuracy of the positions of the weaker bonded areas identified and visualised with the nonlinear approach was confirmed by the manufacturer of the specimens.

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References