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## ► To cite this version:

Souhail Amami, Serge dos Santos, Adriana Savin, W. J. Jeong, J.H. Bae, et al.. Acousto-ultrasonics for nondestructive evaluation of epoxy adhesive joint integrity in complex structures. 10ème Congrès Français d'Acoustique, Apr 2010, Lyon, France. hal-00554466

## HAL Id: hal-00554466 https://hal.science/hal-00554466

Submitted on 10 Jan 2011

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# 10ème Congrès Français d'Acoustique

Lyon, 12-16 Avril 2010

# Acousto-ultrasonics for nondestructive evaluation of epoxy adhesive joint integrity in complex structures

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In industry, achieving a perfect adhesive joint is a quite complicated matter. It requires following rigorous procedures for surface preparation to minimize the risks of surface contamination and to increase glue impregnation on both the surfaces to be bonded. The bonding and curing must also be carried out in controlled atmosphere: temperature and humidity must be continually monitored to ensure an optimal curing and to guarantee maximal bonding strength. Once the adhesive joint is cured, few nondestructive testing methods are available to evaluate bonding quality. This article presents three acousto-ultrasonic methods used to detect and localize defects in epoxy adhesive joints: wave attenuation, Lamb wave generated by Hertzian contact, and nonlinear Time Reversal (TR) based Nonlinear Elastic Wave Spectroscopty TR-NEWS methods associated to chirp-coded excitations. Results showing the detectability of defects are presented. An immediate test of its capability to detect scatterers will be practically implemented using approach recently proposed and leading to in-situ epoxy adhesive joint pseudo-tomography.

## **1** Introduction

Over the last few decades, increasing interest has been bestowed on employing nonlinear wave propagation effects, which possess the intrinsic property of enlarging bandwidth frequency of analysis, to detect, at a subwavelength scale (below the ultrasonic wavelength), the early damage in the degradation process leading to mechanical fracture. Higher frequencies are used for resolution improvement and lower frequencies for aging The Nondestructive Testing (NDT) monitoring. community today refers to methods based on analysis of nonlinear effects as Nonlinear Elastic Wave Spectroscopy (NEWS)[1]. NEWS methods have been shown to improve crack detection and might, therefore, also be advantageous in studying complex structures such as bonding properties of the secondary barrier. Symmetry Analysis has been included in new kind of NEWS systemic approaches aimed at employing absolute symmetries like Time Reversal (TR), reciprocity between emitters and receivers, and others, which optimize the determination of nonlinear properties extracted with coded excitations (e.g. pulseinversion PI or chirp-coded processes, etc.)[2,3]. Recent years have seen considerable development (both in numerical- and experimental aspects) of TR based NEWS methods using TR invariance, and reciprocity combined with ESAM and were implemented in practice as the wellknown TR-NEWS methods[4-8]. Local nonlinearity evaluation was originally observed experimentally using TR explorer system developed by Artann the Laboratories[2,8], which exploits reciprocity and PI signal processing. Imaging based on TR-NEWS continues to develop, with new systems being designed to obtain better focus and optimal images.

Using symbioses of these systems, the fundamental experimental results of Symmetry Analysis (TR, reciprocity, chirp-coded excitations, etc.) and NEWS methods, TR-NEWS were integrated into applications for improving identification of nonlinear scatterers, such as bubbles [2], landmines [8], cracks in complex aeronautic materials [9] and these techniques are now widely recognized as extremely reliable [10].

This study presents an acousto-ultrasonic (AU) methods aimed at detecting bonding defects during construction of the secondary barrier. The localization possibilities of AE has been also associated to TR-NEWS method which also uses the time domain approach for crack identification. A symbiosis of both methods has been also proposed as an possible improvement of crack identification with pseudo-tomography feature [11, 12]

## 2 Experimental feasibility

#### 2.1 Sample preparation

Samples were designed to reproduce the behavior of a real insulation structure as close as possible. Samples with dimensions of 300 x 230 mm were cut from an insulation panel, composed of 170mm thick RPUF covered with a rigid triplex sheet. A 200x110 mm supple triplex sheet was then glued on the surface of the rigid triplex, using the same epoxy glue used in the manufacture of the secondary barrier of LNG carrier tanks[13,14]. A sketch of the manufactured specimen is presented in Figure 1. The supple triplex is composed of three elements: glass cloth/aluminum foil/glass cloth, assembled by rubber-

based adhesive layers, whereas the rigid triplex is composed of two thin glass-resin plates and an aluminum foil.



Figure 1 : Defected manufacturing: An artificial air bobble is manufactured by gluing two discs of vinyl on the both sides of a ring of the same material. It is then inserted into the epoxy layer.

To simulate air bubbles of different sizes, artificial defects were placed in the adhesive joint. Many defectmanufacturing procedures were tested. The following procedure was chosen because it offered better defectdimension repeatability. Air bubbles were simulated using a thin enclosure manufactured as presented in Figure 2. A vinyl ring and two vinyl discs of the same diameter were assembled using 3M cyanoacrylate glue. During the sample-manufacturing step, the previously prepared enclosure was placed on top of a fresh epoxy layer before laying the supple triplex sheet. The epoxy joint was then cured under the same environmental conditions (temperature and humidity) using the same pressing pad used in the manufacture of the real secondary barrier of MIII CCS.



Figure 2 : The real position of the artificially debonded regions in the multilayered plate and the triangulation

# 2.2 TR-NEWS for nondestructive evaluation of epoxy adhesive joint

The objective to develop an in-situ experimental set-up guided our choice to test standard new and innovative AU methods that employ the recent developments in advanced instrumentation. Complexity of the tested structure prompted the use of one of the TR-NEWS methods to detect air bubbles. Nonlinear Time Reversal Acoustics is now an efficient technique employed for improving localization of nonlinear scatterers using Time Reversal properties. After the TR process, the acoustic waves are retro-focused to locally induce high acoustic pressure producing a nonlinear response (Figure 3). This method is more efficient for more complex and reverberating media. NEWS with TR procedure is recognized as a useful tool for non-destructive testing (crack detection) of various solid materials, such as complex composites. The technique of detecting cracks in epoxy adhesive joints with TR-NEWS will be accepted as an original application among other validations performed until recently in landmines [8] and echodentography [15], and will be used for validation in numerous new simulation studies [16-17].

The immense complexity of adhesive joints generates several types of acoustic waves identified as longitudinal, edge longitudinal, edge shear, head and surface. Surprisingly, these linear contributions and mode conversions prove an advantage for the TR-NEWS approach as it is more efficient in a complex medium. Furthermore, as the TR-NEWS approach uses nonlinear signatures, all these complex- but linear effects will be replaced by the code-based signal pre-processing approach described below

# 2.3 Advanced signal processing : chirp-coded pulse excitation

To increase the signal-to-noise ratio and improve extraction of the nonlinear signature derived from the scatterers, the chirp-coded TR-NEWS method [9] was employed using the Artann Laboratory acquisition system. TR-NEWS improvement was achieved by coded excitation using chirp frequency excitation [18-20] and validated for NDE imaging. Its ability to separate second order harmonic components regardless of the transmitted bandwidth makes Pulse inversion (PI) techniques a highly successful signal processing tool in second harmonic imaging.

The success of PI is not restricted to medical imaging where multiple pulses are transmitted in a manner such that adjacent pulses are inverted relative to their neighboring pulses, but also to any characterization of second order nonlinear systems in the time domain. Over the last decades, higher order nonlinearities seem to yield more information on various applications where contrast agents associated with chirp-coded signal processing are employed for improving contrast to tissue signals.

The impulse response needed for the TR-NEWS method was evaluated using the correlation between a chirp-frequency coded signal in the 400-600 kHz frequency bandwidth and its AU response emanating from the medium.

As shown in [15], this pseudo-impulse response can be used in the TR-NEWS method for focusing ultrasounds at the location of the receiver (spatial focusing). Nevertheless, the expected focused signal will contain additional echoes emanating from the nonlinear signature that causes perturbation in the focusing process.



Figure 3 : Schematic process of TR-NEWS with the virtual transducer concept. The initial (a) broadband excitation  $T_x(t)$  propagates in a medium where additional echoes coming from interfaces and scatterers in its response  $R_x(t)$  could be associated to a virtual source  $T_x^{(2)}(t)$ . By applying reciprocity and time reversal process to  $R_x(t)$ , the time reversed excitation  $T_x(t) = R_x(-t)$  produces the new response  $R_x(t)$  with a spatio-temporal focusing at z=0; y=0,  $t=t_f$  and symmetric side lobes (d) with respect to the focusing

#### 2.4 TR-NEWS experimental set-up

A specific TR-NEWS set-up was modified for application in epoxy adhesive joints (Figure 4). The specimen under test was analysed by means of an acoustic transducer conditioned to apply high-level excitations with a 150A100B Scientific Research 150W amplifier.



Figure 4 : TR-NEWS experimental set-up for the AU imaging of the bonding defect in the secondary barrier. The transducers are laid in contact on the surface triplex

Signals generated were amplified to enable the transmission of sufficient ultrasound energy to induce nonlinear behaviour. This TR-NEWS set-up was based on locally calibrated measurements that were previously calibrated with a Polytec PI vibrometer of sensitivity approx 20 mm/s/V. The arbitrary waveform generator and acquisition device ( $f_s$ =10 MHz), manufactured by Artann

Laboratories was calibrated and adjusted using a 14-bit NI PXI-5122 device. The chirp-coded ultrasonic excitation  $c(t) = \cos (2 \pi f(t) t)$  was applied at the 500-kHz central frequency transducer placed in contact with the specimen, between the artificially bonded defect of 2cm diameter (Figure. 2). The time varying frequency f(t) covered the bandwidth [400-600] kHz.

## **3 TR-NEWS imaging of the defect**

For each element of the 16-element NDT probe, TR-NEWS focusing was performed simultaneously at time t=1.65 ms for all elements shown in Figure 5. The time reference enables defining of a time origin that could be converted to spatial reference at the NDT probe element position (Figure 6). As is usual for TR-NEWS, additional echoes are generated by additional nonlinear scatterers, which could be spatially identified in the range [1.65; 1.85] ms after focusing with the TR-NEWS. With a 2D plot, it was possible to localize the scatterers in a greylevel colormap scale (Figure 7), which could be integrated into the experimental configuration set-up.



Figure 5 : Time evolution of the 11 TR-NEWS focused coda received with the 16-element NDT Vermon probe (5 element where out of order). The normalized (with respect to 2048) focused spot around 1.65 ms is not dependent of the element, but the additional response stating from 1.65 ms to 1.9 ms is dependent of the element number



Figure 6 : Configuration of emitter-receiver system around the damaged region (see Figure 2) with the NDT transducers kept in contact with echographic coupling gel (distance are in cm)







### 4 Conclusion

The TR-NEWS imaging approach was compared with standard AU-based methods. This enabled the identification of the scatterers (defects) with a selfreferenced method. The time reference emerging from the intrinsic properties of TR-NEWS was used to create selfreferenced images in the space domain and yields for the identification of scatterers in a complex composite aeronautic sample with epoxy adhesive joints. In fact, the feasibility of using the TR-NEWS proved that the advantage of this method is that it calls for no specific knowledge of the complexity of the specimen, unlike for classical AU imaging like B-Scan. It bridges practical in situ applications with an extended version of the proposed TR-NEWS set-up using noncontact laser-based excitation and reception devices. An immediate test of its capability to detect scatterers will be practically implemented using the ESAM-DORT approach recently proposed[12] and lead to in-situ epoxy adhesive joint pseudo-tomography.

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