Adhesive bond strength evaluation in composite materials by laser-generated high amplitude ultrasound

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Abstract. Adhesive bonding of composites laminates is highly efficient but is not used for joining primary aircraft structures, since there is presently no nondestructive inspection technique to ensure the quality of the bond. We are developing a technique based on the propagation of high amplitude ultrasonic waves to evaluate the adhesive bond strength. Large amplitude compression waves are generated by a short pulse powerful laser under water confinement and are converted after reflection by the assembly back surface into tensile waves. The resulting tensile stresses can cause a delamination inside the laminates or at the bond interfaces. The adhesion strength is evaluated by increasing the laser pulse energy until disbond. A good bond is unaffected by a certain level of stress whereas a weaker one is damaged. The method is shown completely non invasive throughout the whole composite assembly. The sample back surface velocity is measured by an optical interferometer and used to estimate stress history inside the sample. The depth and size of the disbonds are revealed by a post-test inspection by the well established laser-ultrasonic technique. Experimental results show that the proposed method is able to differentiate weak bond from strong bonds and to estimate quantitatively their bond strength.

1. Introduction
In aerospace, adhesive bonding of structural components presents many practical advantages when compared to other joining methods. However, its use for primary structures is impeded by the absence of reliable nondestructive methods that can ensure the integrity of the joint.
Laser shock generation and spallation have been studied for the measurement of the bond strength between a thin planar coating on a substrate [1], [2]. Recent work addresses the problem of adhesive bonding of structures made of carbon-epoxy composite [3].
In this paper, the laser shock wave technique is further explored for evaluating the bond strength of composite laminates joined by an adhesive layer. The principles of bond strength evaluation will be first detailed and then applied to bonded composite plates made of carbon fibers embedded in epoxy.

2. Composite laminate properties
The composite laminates were obtained by curing a stack of 4 carbon fibre plies pre-impregnated with epoxy (Cytec 5276-1). In each ply, the fibres (G40-800 – 24K) are unidirectional, and the orientation of the plies is [0/90]s. The total thicknesses of the 4-ply is about 0.72 mm. Given that the plies are co-cured, the epoxy matrix has a quite homogeneous high strength, even between the plies. The composite sample shows strong anisotropy indicated by a longitudinal velocity of 3100 m.s⁻¹ in the z
direction, normal to the plies, and of 8300 m.s\(^{-1}\) along the fibre direction of a single ply. Adhesive paste Hysol® EA9394 was also used to bond the composite laminates together.

3. Principle and experimental approach
A powerful Q-switched Nd:YAG laser which delivers optical pulses of 8 ns duration and up to 2 J energy at 1064 nm wavelength is used to induce shock waves or very high amplitude ultrasonic waves in the sample. The laser beam is focused to a spot diameter of about 4 mm. To avoid surface damage and to increase the ultrasonic wave amplitude [4], the surface of the material is first covered with a black electrical tape and then with a constraining water layer, as illustrated in figure 1. Under our generation conditions, it should be noted that the pressure level is below the Hugoniot Elastic Limits (HEL) of all the materials, so that wave propagation is in the elastic regime [5].

![Generation Laser](image1)

![Figure 1: Schematic setup for laser shock generation.](image2)

![Figure 2: Schematic representation of velocity signals at the back surface.](image3)

The source size is a few times larger than the sample thickness, with the result that the waves generated at the top surface are mostly compressional plane waves. When propagating through the thickness of a homogeneous plate, they are reflected by the free back surface as a tensile wave. Close to this surface, almost no stress is imposed because the compression due to the end of the incoming wave is balanced by the beginning of the reflected tensile wave. If attenuation mechanisms and diffraction are neglected, the maximum tensile stress begins at a certain distance from the back surface, and propagates unchanged until the next reflection. For multi-layer structure, the propagation is altered by the transmission/reflection of the waves at the different interfaces. Only the tensile stress can induce failure within the laminate or at the adhesive bonded interfaces.

To quantitatively evaluate the stresses inside a sample, an optical velocimeter based on a Fabry-Perot solid state etalon was developed and used to monitor the back surface velocity \(u(t)\) (see figure 1). A detailed description of the interferometer can be found in reference [6]. Then a simple relationship is used to relate this velocity to the stresses within the sample. In particular, the rupture threshold is given by the following equation [7]:

\[
P_{\text{rupt}} = 0.5 \rho c T_0 z u(t')^2 - T_1 z u(t')^2 - 2 \tau , \quad \text{with} \quad \tau = \int_0^{z_{\text{rupt}}} z/c \, dz
\]

(1)

where \(t'\) corresponds to the time on the velocity signal at which the damage is identified. It is delayed by \(\tau\) from the time \(t'_{\text{rupt}}\) at which rupture occurs. \(z_{\text{rupt}}\) is the distance of the damage from the back surface, measured by post shock laser ultrasonic inspection [8]. \(c z\) is the propagation velocity through the sample in the \(z\) direction and \(\rho c\) is the acoustic impedance of the ultimate bottom layer. \(T_0(z)\) and \(T_1(z)\) stand for the product of all the pressure transmission coefficients between the free surface and the position \(z_{\text{rupt}}\) and according of the wave propagation direction. Since shock propagation is elastic for the material studied, the velocity signals obtained with different laser energies should all be superimposed when normalized, except for the signal in which a disbond signature appears. The
way to obtain $t'$ is thus to increase gradually the laser energy until obtaining the disbond and to compare the normalized velocity signals. Figure 2 gives a schematic representation of the procedure.

4. Application to bonded laminates

To show that the technique is able to distinguish a strong joint from a weak one, bonded laminate assemblies (4-ply laminate on top of another 4-ply laminate) were prepared to present two areas with different adhesion strengths. All surfaces of the laminates were first cleaned by a solvent wipe. Then, only the half of the laminate’s surfaces was treated by a corona discharge, a technique which is well known to improve adhesion [9]. Finally the laminates were assembled with their treated surface face to face. The dimensions of the samples were about 30 mm x 40 mm. The bond thickness all along the joint was measured to range between 90 and 160 µm.

Before laser shock testing, samples were non-destructively tested by laser-ultrasonics to detect delaminations or voids in the bond. Then, to distinguish the areas of different adhesive strengths, several shocks were applied over the entire surface, beginning with a “low” laser energy level and were followed by another laser-ultrasonic inspection of the whole sample. This procedure was repeated for increasing values of laser energy. Since the disbonds or delaminations occur from the reflection at the back surface, the laser-ultrasonic inspections were always made from this back surface. Figure 3 presents the B&C-scans representation of the signals obtained from the laser-ultrasonic inspections before and after shocks. The laser shock energy started at 400 mJ and was increased up to 1200 mJ by step of 200 mJ. No void is noticed in the pre-shock C-scan of the figure 3(a), and no disbond either is observed for 400 mJ (the C-scan at 400 mJ is identical to the one shown in figure 3(a)). The dashed rectangle delimits the weak area (without corona discharge). Disbonds are observed in this area at 600 mJ and becomes more evident when laser energy is increased to 1000 mJ, figure 3(b). B-scans indicate that the disbonds have occurred at the joint depth. All the shocks realized on the weakly bonded area have produced disbonds and almost no disbond is observed in the strong area. When laser energy reaches 1200 mJ, all the new delaminations observed are found to take place between the second and third laminate plies from the back surface (figure 3(c)). The epoxy matrix damage threshold is thus lower than the joint strength in the good bonded area.

Figure 3. Typical C-scan obtained after shocks realized with laser energies (a) up to 400mJ, (b) from 600 mJ to 1000mJ, and (c) from 1200 mJ. Some C-scans are associated with their B-scans obtained along the dashed line. The lines pointed respectively by $t_b$ ($t_j$) correspond to the reflection of the wave on the shock loading surface (on the disbonded joint interface). The encircled multiple echoes in (c) are reflections at the disbonded interface between the 2nd and 3rd ply.

Figure 4 shows the normalized velocity signals obtained below (400mJ), just above the weak joint damage threshold (600mJ) and just above the epoxy matrix damage threshold (1200mJ). In fact, the maximum of these signals are respectively about 50 m/s, 80 m/s and 150 m/s. Until the disbond signature, the signals are almost identical, which confirms again the elastic regime of wave propagation and that damage results from a brittle behavior of the material under high strain rate deformation. When applying the equation (1) to these signals, we obtain a value of about 340 MPa for the epoxy matrix strength and about 150 MPa for joint strength in the weak bonded area.
Figure 4. Normalized velocimeter signals produced at 400mJ (grey), at 600mJ (black) and at 1200mJ (point). The arrows indicate signatures of the matrix and adhesive ruptures.

5. Summary & Perspectives
A method based on laser shock waves combined with laser-ultrasonic inspection has been used to evaluate quantitatively the bond strength of carbon fibre composite laminates. It has been shown that the shock waves propagate only under the elastic regime, and that damage results of a brittle behavior of the material under high strain rate deformation. These observations confer to the method a quality of non invasive proof test and allow the adhesion strength to be measured quantitatively. The method requires first the identification of a damage signature in the back surface velocity signal and then the evaluation of the damage depth by laser-ultrasonic inspection. The inter-ply bond strength within the laminate was evaluated to about 340 MPa. Then two laminates were bonded in such way that half of their joint presents a weak strength and the other half a strong strength (comparing to the inter-ply bond strength). The test technique actually reveals the two different bonded areas. The strength of the strong bonded area was found to be at least equal than the one between the plies whereas the weakly bonded area showed a strength of about 150 MPa. The encouraging results provided by this work are an incentive for further development of the technique in view ultimately of its use for certifying adhesive bonding of primary aircraft structures.

Acknowledgements
The authors would like to thank Martin Lord and Christian Néron for their assistance in instrumentation for all aspects of this project. We also would like to thank Andrew Johnston, Richard Cole and Julieta Barroeta Robles of the Institute of Aerospace Research of NRC for providing the laminates and for useful information and discussions on composite materials and adhesive bonding. This work is part of the collaborative project SATAC between the National Research Council of Canada and the Centre National de la Recherche Scientifique of France.

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