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Adhesive bond testing of carbon–epoxy composites by laser shockwave

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Abstract

Adhesive bonding, particularly of composite laminates, presents many practical advantages when compared with other joining methods but its use is limited, since there is presently no non-destructive 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 at high strain rate. Compression waves are generated by a short and powerful laser pulse under water confinement and are converted after reflection on the assembly back surface into tensile waves. The resulting tensile forces normal to the interfaces can cause a delamination inside the laminates or a disbond. The adhesion strength is probed 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 confirmed by numerical simulations show that the proposed method is able to differentiate weak bonds from strong bonds and to estimate quantitatively the bond strength.

1. Introduction

Composite materials, such as carbon–epoxy, are now extensively used in aerospace structures due to the weight reduction they provide compared with metals. Full advantage of composites would, however, be fully realized if the various composite parts of an aircraft structure could be safely adhesively bonded [1]. However, at the present time, although there have been many developments in adhesives, in understanding adhesion and stresses in adhesive joints and developing adequate industrial practices (such as proper surface preparation) [2, 3], adhesive bonding is not judged sufficiently reliable to be accepted for assembling primary aircraft structures without fasteners [4]. What is essentially missing to gain this acceptance is a reliable non-destructive technique for insuring the quality of an adhesive bond, i.e. evaluating its strength, and this is in spite of significant research efforts [5]. Further, if an adequate quality assurance technique for adhesive bonding was available, a large structure could be made more economically by bonding several small parts instead of having it co-cured.

Current industrial practice relies first on the evaluation of the strength of the joint configuration by making coupons

duplicating the actual assembly in the structure. These coupons are then destructively tested by shear, peel, double cantilever or other tests to find the adequate bond strength. Secondly, industry uses non-destructive inspection (NDI) to find flaws such as voids, porosity and disbonds in joint assemblies [6, 7]. These flaws are either located at the interface between the adhesive and one adherent, potentially causing adhesive failure, or throughout the adhesive layer, potentially a cause for cohesive failure [8]. The techniques presently used for composite inspection, x-ray radiography, ultrasonics and more recently thermography [9], are adequate to detect flaws that have some volume but do not have the sensitivity or the contrast mechanism to detect zero or near-zero volume defects, such as weak bonds and in particular the dreadful condition of a kissing bond. Although the definition of a kissing bond varies throughout industry and research community, we will adopt here the definition of an intimate contact between adhesive and adherent but with little adhesion strength [5]. A kissing bond is therefore an extremely weak adhesive bond. Such intimate contact obviously gives no contrast mechanism for x-ray radiography which relies on material density variations for imaging. In the case of ultrasonics, the good mechanical contact makes essentially no difference for the transmission

and reflection of ultrasonic waves the bond being weak or strong. For thermography, the good thermal contact makes the heat diffuse or the thermal waves propagate essentially equally the bond being weak or strong. Therefore NDI techniques generally and currently used by industry cannot detect kissing bonds as well as bonds of any weakness level.

Research efforts have consequently focused on the development of techniques to detect these zero-volume defects such as kissing and weak bonds. Enhancement of the sensitivity of ultrasonics to bond conditions was obtained using a resonance approach (ultrasonic resonance spectroscopy) and some sensitivity to kissing bonds has actually been demonstrated [10–12]. By additional modelling of the resonating structure, the adhesion or specific stiffness parameters introduced with the model were found to correlate with the adhesion strength in the case of weakly bonded metals [13]. Resonance effect is also at the basis of the Fokker bond tester that was first introduced more than 50 years ago and has been widely used in industry. It turns out, however, that the predictions of this instrument or similar instruments were not found sufficiently reliable [5].

Contrast to bond weakness or to a kissing bond can be more reasonably expected if the contact between the adhesive and the adherent is modified by the technique, i.e. if the technique involves applying some stress across the bond. Techniques that fall into this category are shearography, in which the stress is applied by pressure or vacuum [14], laser tapping in which the stress is thermoelastic following laser light absorption [15], vibro-thermography (also called Sonic IR) in which the part is put into strong vibrations [16–18] and non-linear ultrasonics in which high amplitude ultrasonic stress is applied [19]. For some of these techniques, particularly non-linear ultrasonics, there have been claims to sensitivity to kissing bonds. However, the applied stress level is usually much below the bond strength so strong sensitivity to bond weakness cannot be reasonably expected by these techniques. Sensitivity should be, however, expected if the stress level is sufficiently high so that a weak bond is broken apart whereas a strong bond stays intact. Such an approach is not strictly NDI but more a proof test which could be non-invasive if properly calibrated and if the applied stress is below a determined threshold. We are reporting below results obtained on bonded composites with such a proof testing technique which uses laser generation for material stressing.

This technique actually uses a high energy pulsed laser for generating high amplitude compression pulse which propagates through the sample. Upon reaching the free back surface, this pulse is reflected as a tensile pulse that can pry apart the material encountered along its propagation path. Laser shock generation has been applied to the measurement of the bond strength between a thin planar coating on a substrate [20–25], between carbon fibres and their matrix [26], and more recently between cells and bio-materials [27]. Recent work addresses the problem of adhesive bonding of carbon–epoxy composites [28]. In contrast to thin coatings, laser shock waves on these ‘thick’ composite samples do not induce spallation, but delaminations or disbonds within the structure. The laser shock method can be seen as a proof test and the evaluation

is non-intrusive and non-destructive if the bond is good. It has also the advantage of being able to operate on curved samples and shockwave is more conveniently produced than with mechanical impact, explosions [29] or electron beams [30]. The method provides a local measurement of ‘intrinsic’ adhesive strength without mechanical contact [26]. This strength directly relates to the strength produced by chemical bonding between the adhesive and the adherents.

The transient tensile force in laser shockwave testing propagates essentially normal to the interfaces. For this reason, this ‘intrinsic’ adhesive strength should not be directly compared with the strength given by established destructive testing techniques (such as lap shear, double cantilever beam, mixed mode flexure) which solicit differently the bonded assembly [2]. Another characteristic of the laser shock method compared with standard destructive testing is its very high strain rate, so the dynamic measures obtained should be very different from the quasi-static given by established destructive tests. Comparison of the laser shock method with established destructive testing is beyond the scope of this work and could be the object of a future study. The laser shock method is expected to be sensitive to a variety of factors that affect this ‘intrinsic’ strength, such as surface contamination, improper mixing ratio for two-component adhesive, adhesive degradation and improper curing. It should also be noted that we are considering bonds without defects, such as voids, porosity and disbonds. As mentioned above, such defects can be detected by well established NDI techniques, the impact of such defects on the strength value given by the laser shockwave technique being beyond the scope of this work.

In this paper, the laser shock wave technique is further explored for evaluating the bond strength of composite laminates joined by an adhesive layer. Adhesion strength is probed by increasing the laser pulse energy step by step. A good joint would be unaffected under a given stress level whereas a weaker one would be damaged. In the following, the principles of bond strength evaluation will be detailed and applied to bonded composite plates made of carbon fibres embedded in epoxy. The method is made quantitative and *in situ* by optically measuring the sample back surface velocity with an interferometer. The interferometer signals give real-time signatures of well-bonded and disbonded interfaces and are used to obtain an estimate of the bond strength. The disbonds were confirmed by laser-ultrasonic inspection made on shocked samples. A detailed description of laser-ultrasonics can be found in [31]. Results show that the proposed test is able to evaluate quantitatively bond strength at high strain rate.

2. 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 [24, 32], the surface of the material is first covered with an

Figure 1. Setup for laser shock generation.

Figure 2. Time-space diagram of the propagation of a shock wave pulse with duration T . Dark and light grey areas represent, respectively, the compression wave (pressure superior to the average pressure) and the tensile wave (pressure inferior to the average pressure). Dashed and full lines are used for indicating decrease and increase of pressure, respectively.

optically absorbing tape and then with a constraining medium, transparent to the laser wavelength, as illustrated in figure 1. Black electrical tape which is widely available is an efficient option for the absorbing layer. A water layer is used as constraining medium.

The source size (roughly the laser spot diameter) is a few times larger than the sample thickness, with the result that the waves propagating through the material are mostly compressional. Figure 2 shows a diagram of the evolution of a compression shock wave generated at the top surface with a time duration T , propagating through the thickness of a homogeneous plate and finally reflected by the free back surface as a tensile wave. The triangle whose base line is delimited by the instants t_1 and t_2 corresponds to the time and space where the compression due to the end of the incoming wave is balanced by the beginning of the reflected tensile wave. If attenuation mechanisms are neglected and for a typical shock pulse shape, the maximum tensile stress (represented as a white dot) begins at a distance from the back surface given by $DT/2$, and this tensile wave propagates unchanged until the next reflection. Here, $D(z, t) = c + su(z, t)$ is the shock wave propagation velocity, c is the elastic wave propagation velocity, u is the particle velocity and s is the Hugoniot slope parameter. Under our generation conditions, only the tensile stress could induce failure within the laminate or at the adhesive bonded interfaces. It should also be noted that the pressure level is

below the Hugoniot elastic limits (HEL) of all the materials, so that wave propagation is in a weak or elastic shock regime in which the waves still travel approximately at the usual sound velocity [33]. However, under higher laser energy, it is possible to reach the regime of strong shock propagation, or at least elastic-plastic wave propagation.

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 $u(0, t)$ (see figure 1). A detailed description of the interferometer can be found in [34]. Then a simple relationship is used to relate this velocity to the stresses within the sample. The pressure $P(z, t)$ at any instant t and at a depth z inside the plate is equal to the sum of the pressures produced by the wave propagating towards positive z and the one propagating towards negative z . Since the impedance $Z = P/u = \rho D$ is defined not only as the ratio between the pressure and the particle velocity but also as the product of ρ the material density and the propagation velocity, it gives

$$P(z, t) = \rho(D \cdot u^+(z, t) + (-D) \cdot u^-(z, t)), \quad (1)$$

where $u^+(z, t)$ and $u^-(z, t)$ are, respectively, the particle velocities of waves propagating towards positive and negative z . The minus sign comes from the wave propagation towards negative z . The z -axis origin is taken at the back surface where velocity measurements are made and z is oriented positive from the front loading surface towards the back surface. Under the assumptions of 1D propagation in a homogeneous material, no attenuation and an adiabatic process, this equation becomes, at the free back surface and after the propagation of $u^+(z, t)$ and the retro propagation of $u^-(z, t)$ [35],

$$P(z, t) = \frac{1}{2} \rho D (u(0, t + |z/D|) - u(0, t - |z/D|)). \quad (2)$$

The factor $1/2$ comes from the total reflection ($u^+(0, t) = u^-(0, t)$) of the wave at the free surface: $u(0, t) = u^+(0, t) + u^-(0, t)$. The last equation gives the pressure at any depth, and during the time of the experiment. The longitudinal rupture threshold is then given by the following equation:

$$P_{\text{rupt}} = \frac{1}{2} \rho D (u(0, t^i) - u(0, t^i - |2z_{\text{rupt}}/D|)), \quad (3)$$

where t^i corresponds to the time of the velocity signal at which the damage is identified and z_{rupt} the distance of the damage from the back surface. t^i appears on the back surface with a delay of $|z_{\text{rupt}}/D|$ from the time t_{rupt} at which rupture occurs. In the next paragraph, it will be shown that the shock propagation is elastic for the material studied. The consequence of this fundamental point is that the velocity signals obtained with different laser energies should all be superimposed when normalized, except the signal in which a disbond signature appears. The way to obtain t^i is thus to increase gradually the laser energy until obtaining the disbond and to compare the normalized velocity signals. The depth of the rupture z_{rupt} is measured by post shock laser ultrasonic inspection. Figure 3 gives a schematic representation of the procedure. The time at which the two curves differ is t^i and $\frac{1}{2} \rho D u(0, t^i)$ corresponds to the stress imposed at the joint by

Figure 3. Schematic representation of velocity signals of the back surface. The black and grey signals represent, respectively, a signal slightly above and below damage threshold. Grey signal has been normalized to the black signal maximum.

the incoming part of the wave. Knowing z_{rupt} , it is easy to find the stress $-\frac{1}{2}\rho Du(0, t^i - |2z_{rupt}/D|)$ imposed by the outgoing part of the wave.

For multi-ply composite structure, the propagation is altered by the transmission/reflection of the waves between the different layers, leading to an incorrect evaluation of the tensile stress inside the material. The propagation is also affected to some extent by material anisotropy. Since the source has a finite size, diffraction effects cause the waves to differ from the ideal case of plane waves propagating normally to the plies. It implies a discrepancy between the real pressure imposed on the joint and that given by equation (2). In what follows, the validity of this equation will be tested and then some corrections will be made to evaluate more accurately the stress within the material.

Although the purpose of this work is primarily to measure the bond strength between two carbon–epoxy laminates, we will also address the strength between the plies of the laminate since in the case of a strong bond the laminate may yield before the bond.

3. Materials properties

The composite laminates were obtained by curing a stack of 4 or 8 carbon fibre plies pre-impregnated with epoxy (Cytec 5276-1). In each ply, the fibres (G40-800 – 24K) are unidirectional. The total thicknesses of the 4- and 8-ply are about 0.72 mm and 1.35 mm, respectively. The orientation of the plies is $[0/90]_s$ and $[0/45/90/-45]_s$ for the 4- and 8-ply plates, respectively. Figure 4 shows the sketch of a 4-ply composite laminate. It consists of an alternate succession of pure epoxy layers (white and thin layers) and carbon–epoxy layers (dark grey layers). The diameter of the carbon fibres is estimated to be around $5\mu\text{m}$ and the volume fraction of carbon fibres is 70% on average. X-ray tomography shows that the ply thickness varies from 160 to $180\mu\text{m}$ and epoxy layer thicknesses are about $15\mu\text{m}$. The three internal epoxy layers are called ep_1 , ep_2 and ep_3 . Given that the plies are co-cured, the epoxy matrix has a quite homogeneous high strength, even between the plies. The composite sample

Figure 4. Sketch of a 4-ply laminate. It consists of an alternation of pure epoxy layers (white and thin layers) and carbon–epoxy layers (dark grey layers) with carbon fibres oriented along 0° (black lines) and 90° (black spots). The three internal epoxy layers are indicated by ep_1 , ep_2 and ep_3 .

Figure 5. Back surface velocity signals measured under 1200 mJ (grey curve) and 800 mJ (black curve) laser shock pulse energy on a 4-ply laminate.

shows strong anisotropy indicated by a longitudinal velocity c_L of 3100 m s^{-1} in the z direction normal to the plies and of 8300 m s^{-1} along the 0° direction of a single ply (along the fibre direction). Adhesive paste Hysol® EA9394 was also used to bond the composite laminates together.

4. Results for a composite laminate only

Experiments have been first performed on laminates only to find the inter-ply damage threshold. Comparisons between the measured and calculated signals are used to develop a better understanding of the weak shock or elastic wave propagation in this material. Equation (2) is then refined using numerical modelling in the case of this layered medium and used to calculate the disbond threshold. The damage mechanism is also discussed.

4.1. Experiments below the damage threshold

The first set of tests was performed on a 4-ply laminate, as sketched in figure 4. Figure 5 shows the back surface velocity

Figure 6. Back surface velocity signals measured (black and noisy curve) and simulated (grey curve) for a 400 mJ laser pulse in a 4-ply laminate.

signal as a function of time for different laser pulse energies. The first sharp peak at about $0.35 \mu\text{s}$ corresponds to the arrival of the compression wave (L) followed by a reduction of the velocity due to rarefaction. The second and third peaks at about $0.85 \mu\text{s}$ and $1.45 \mu\text{s}$, denoted as $3L$ and $5L$, correspond to this compression wave after propagating over three and five times the thickness, respectively. The small echoes between these two peaks are due to reflections between the plies. The shapes of the velocity signals obtained with different laser energies are almost identical when normalized with respect to the maximum. The small differences can be explained by the tape and by the thickness variations of the different layers. Nevertheless, it is a proof that the shock regime is elastic and that the strong compression waves generated do not modify the material properties. As expected in the elastic shock regime, the shock velocity D is approximately equal to the elastic velocity c and no Hugoniot step is observed. Generation and propagation of the high amplitude waves are in fact non-invasive since the elastic limit for the carbon fibre is well above the rupture threshold of the epoxy and the epoxy rupture is itself brittle at high strain rate, without any prior plastic deformation. This is confirmed by applying many shocks at the same place without observing any change.

Figure 6 shows the back surface velocity signal as a function of time for a 400 mJ laser pulse as well as the signal obtained by a numerical simulation performed using LS-Dyna software. The two signals are in good agreement regarding arrival times and amplitudes. The geometry used is the one sketched in figure 4. Since the laser spot size to sample thickness ratio is large and the measurements are limited to the z -velocity, the carbon-epoxy plies can be represented in the simulation as isotropic plates. The different ply orientations are then not included and the tape is also not taken into account to simplify the simulation. While the tape transmits almost all the initial compression stress, a reflected tensile stress is enough to detach the weakly bounded tape. A delay of about 50 ns corresponding to the initial propagation through the tape is then required to superimpose the signals. Since the tensile wave detaches the tape, no reverberation through the tape is experimentally observed. Tape detachment could also be a cause of the poor amplitude agreement at the $3L$ peak and

Table 1. Material properties.

	ρ (g cm $^{-3}$)	c (m s $^{-1}$)	E	ν
Epoxy	1.26	2600	3.95	0.4
Carbon-epoxy	1.66	3200	10.8	0.35
Hysol EA9394	1.55	3100	3.99	0.45

Figure 7. Radial $g(r)$ and time $f(t)$ profiles of the simulated loading in normalized amplitude.

thereafter. The dynamical behaviour of the solid is taken to be elastic and non-linear effects are not included in the simulation. The elastic constants used are summarized in table 1.

A simple normal force model is used for representing the laser-material interaction. The radial $g(r)$ and time $f(t)$ profiles of the initial pressure loading are shown in figure 7. The pressure applied is given by $P(r, t) = \alpha g(r) f(t)$, where α is a proportional factor. $\alpha = 100 \text{ MPa}$ was used to obtain the agreement shown in figure 6. Since the processes and parameters (such as the breakdown threshold, the heating of the plasma by the laser, the degree of ionization of the plasma, the screening Debye length inside the plasma, the water cooling and the acoustic impedance of the materials) involved in generation and expansion of the confined plasma under water are very difficult to know precisely [36], the laser-material interaction is simply modelled from experimental knowledge. It was found that the velocity varies linearly with the laser energy and since the laser radial profile is Gaussian, $g(r)$ is taken as a truncated Gaussian axisymmetric profile. Truncation occurs at the radius at which the power density falls below breakdown. The loading time profile $f(t)$ is determined by taking into account the three regimes mentioned in [32]. But rather than solving the equations presented in this reference,

Figure 8. (a) Experimental setup and (b) time profiles of the signal recorded behind the black tape (black signal). The profile shown in figure 7(b) has been superimposed in grey for comparison.

$f(t)$ is simply taken as the sum of three half-Gaussians: one increasing half-Gaussian for the heating phase and two delayed decreasing half-Gaussians for the adiabatic cooling and the tail.

In order to determine the amplitude, width and delay of the Gaussians, experiments were performed with the setup shown in figure 8(a). The sample was replaced by a 4 mm thick plate made of cured epoxy. The generation conditions (laser duration, spot size) are the same as for the experiment on the composite samples, except for a laser pulse energy of 1 J. Black tape and water confinement were used as before. A very thin layer of gold was deposited on the front surface underneath the black tape to increase its reflectivity. In such a configuration, the velocimeter response is sensitive to the two surface velocities V_0 (back free surface) and V_1 (top surface behind black tape), as well as the photoelastic effect produced by the stress wave propagating inside the epoxy volume. The signal measured by the velocimeter is given by the equation below:

$$y(t) = \frac{\lambda_0}{2} G^{-1}(\Delta I / I_{\text{tot}}) \cong I_1 / I_{\text{tot}} \left(n_1 V_1 + (n_0 - n_1) V_0 - \frac{1}{2} q_{11} n_1^3 \int \frac{\partial \sigma_{zz}(z, t)}{\partial t} dz \right) + I_0 / I_{\text{tot}} n_0 V_0, \quad (4)$$

where λ_0 is the optical wavelength of the detection laser, G^{-1} is the inverse function giving the optical frequency shift for an intensity variation ΔI at the output of the Fabry–Perot velocimeter. $n_0 = 1$ and $n_1 = 1.7$ are, respectively, the optical indices of air and epoxy, $q_{11} = -15 \times 10^{-12} \text{ m}^2 \text{ N}^{-1}$ is the epoxy stress optical coefficient, I_0 , I_1 are the intensities collected from, respectively, the back free surface and the top surface and $I_{\text{tot}} = I_0 + I_1$. Using a sufficiently thick epoxy layer, light collection from the back surface is minimized and the motion of this surface is delayed, so that equation (2) can be reduced to

$$\frac{y(t)}{n_1} \cong V_1 - \frac{1}{2} q_{11} n_1^2 \int \frac{\partial \sigma_{zz}(z, t)}{\partial t} dz. \quad (5)$$

The photoelastic contribution is evaluated to be around 13 m s^{-1} for a stress having the profile mentioned in figure 8(b) and a maximum of 200 MPa and is then not considered.

The experimentally measured velocity signal $y(t)/n_1$ which represents the initial pressure loading is shown in

Figure 9. Comparison between the theoretical stress σ_{zz} obtained directly from the simulation and the evaluations of σ_{zz} by equations (2) and (6) at the epoxy layer ep_3 depth.

figure 8(b) together with the theoretical profile of figure 7(b). Good agreement is observed, which shows that the loading is well represented by the model.

A further step is to predict the stress level at various plies within the laminate from the measurement of back surface velocity. Since the numerical model well represents the experimental data at the back free surface, the simulated stresses should also correspond to the experimental stresses at any depth. The result is then compared with the value predicted using the back surface velocity and applying equation (2). As shown in figure 9, an error of about $\pm 15\%$ is found for the maxima tensile stresses at the epoxy layer ep_3 depth. This discrepancy is explained by the limitation of equation (2) which applies only for a homogeneous medium and not for a layered medium in which multiple reflections occur. It can then be demonstrated that equation (2) can be refined by introducing the transmission coefficients of the ultrasonic wave between the layers:

$$\sigma_{zz}(z, t) = \frac{1}{2} \rho(0) D(0) \left(\frac{1}{T_0(z)} u(t + t^*) - T_1(z) u(t - t^*) \right). \quad (6)$$

$T_0(z)$ and $T_1(z)$ stand for the product of all the pressure transmission coefficients between the free surface and the position z and according to the wave propagation direction and

Figure 10. Back surface velocity signals measured for laser shock pulse energies above damage threshold in the 4-ply laminate.

$t^*(z) = \int_0^z |z_0/D(z_0)|dz_0$. These refinements still suppose a one-dimensional wave propagation, in which pressure and stress are equal. But, since simulations are three dimensional, $\sigma_{zz}(z, t)$ better represents the right-hand side of the equation. The result given by equation (6) is shown in figure 9. The extrema values coincide with the theoretical simulated values. The in-between discrepancies come from all the waves that are reflected between plies without reaching the back surface and also from diffraction.

4.2. Experiment above the damage threshold

The results for laser pulse energies well above the damage threshold are shown in figure 10. All the measurements were made at different locations on the sample. The signals obtained at 1300 and 1350 mJ present signatures very distinct from the one presented in figure 5. At 1350 mJ, the disbond signature is identified at about $t^i = 480$ ns. The following small oscillations with a constant period and decreasing amplitude correspond to reverberations within one ply. At 1300 mJ (laser power density of 1.3 GW cm^{-2}), damage is identified but at about $t^i = 620$ ns.

Figure 11(b) shows a laser-ultrasonic amplitude C-scan image obtained on the 4-ply laminate after laser shocks. Note that laser-ultrasonic inspection is performed in the thermoelastic regime, without the tape or water confinement used for producing shockwaves or high amplitude ultrasonic waves. The sample in-plane dimensions are about $50 \text{ mm} \times 50 \text{ mm}$. The damage is a crack localized inside the epoxy layer ep₁ when laser energy is greater than 1350 mJ and inside the epoxy layer ep₂ when laser energy is between 1200 mJ and 1300 mJ, as shown in the A-scans of figure 11(a). X-ray tomography images made on the section of the shocked samples confirm that disbonds occur inside the bulk of the epoxy layers and then are identified as a cohesive rupture. Because layered media exhibit tough and weak parts, the damage produced is different from the spallation encountered in homogeneous media.

Once the location of the damage had been identified, $t^*(z)$ is calculated and the damage threshold is determined from

equation (6) applied on the velocity signals shown in figure 10. The calculated threshold is about 340 MPa, wherever damage occurred. Note that this dynamical tensile limit is much higher than the usually accepted static tensile limit for this material [37], but the laser shockwave experiments are done at a much higher strain rate than usual mechanical tests.

These results are in good agreement with the fact that the damage threshold is reached closer to the back surface when loading increases, as can be understood from the sketch of figure 12. The two signals represent schematically the velocities for pressure loadings just above and far above damage threshold in a homogeneous medium. Since the propagation regime is essentially elastic, the two signals are proportional before the rupture signatures and reach different peaks. The slope ($\Delta u/\Delta t$) of the release part of the signals is proportional to the pressure loading and also is equal to $\Delta u_{\text{rupt}}/2\tau$, where $\tau = z_{\text{rupt}}/D$. This implies that the damage depth (z_{rupt} from the back surface) is inversely proportional to loading pressure. In the case of the composite material, rupture is observed at the interfaces ep1 or ep2 according to the loading (ep1 for the higher pressure).

5. Results for bonded laminates

Having measured the tensile strength of the composite laminate itself, the technique is now applied to the strength evaluation of bonded laminate assemblies. To study the influence of the sample thickness, several assemblies were inspected. The case of a 4-ply laminate on top of another 4-ply laminate is in the following referred to as ‘4/4’ assembly. The other assemblies are a ‘4/8’, its symmetrical counterpart ‘8/4’ and a ‘8/8’ assembly. The first figure refers to the number of plies of the laminate upon which laser shock loading is applied.

To show that the technique is able to distinguish a strong joint from a weak one, samples were prepared to present two areas with different adhesion strengths. All surfaces of the laminates were first cleaned by a solvent wipe. Then, only half the laminate surfaces was treated by a corona discharge, a technique which is well known to improve adhesion [38]. Finally the laminates were assembled with their treated surface face to face. The dimensions of the samples were about $30 \text{ mm} \times 40 \text{ mm}$. The bond thickness all along the joint was measured to range between 90 and $160 \mu\text{m}$. Some tests realized on a pure cured paste sample show that its tensile strength is higher than 340 MPa, so that the interfaces between the paste and the laminates should present the lowest strength of the whole assembly.

The procedure to measure the strength of the joint is similar to the one explained in section 2. 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 11. Laser-ultrasonic amplitude scan of the laminate after laser shock for different laser pulse energies. (a) A-scans from the regions indicated in the C-scan (b).

Figure 12. Schematic representation of velocity signals obtained in a homogeneous medium for pressures above $P^>$ and much above $P^>>$ the damage threshold. The ruptures take place at $z^>$ and $z^>>$, respectively, where $z^> > z^>>$.

5.1. Shock waves in the '4/4' assembly

Figure 13 presents the B- and C-scans obtained from the laser-ultrasonic inspections before and after shocks on the '4/4' assembly. The laser energy started at 400 mJ and was increased up to 1200 mJ by step of 200 mJ. The B-scans are obtained from the signals presented in figure 14(a). Nevertheless, the differences between disbonded and bonded areas can be sometimes very small, as shown in this figure. The C-scans are consequently plots of the amplitude of the spectral peak located between 3 and 4 MHz, which was found very sensitive to bond damage: see figure 14(b).

No void is noticed in the pre-shock C-scan of figure 13(a), and no disbond either is observed for 400 mJ (the C-scan at 400 mJ is identical to that shown in figure 13(a)). The dashed rectangle delimits the weak area (without corona discharge). The first disbands are observed in this area at 600 mJ in figure 13(b). Dashed circles have been superimposed to indicate locations where disbands are observed. The disbands become more evident when laser energy is increased to 1000 mJ in figure 13(c) and B-scans indicate that the disbands have occurred at the joint depth. Almost all the shocks

realized on the weakly bonded area have produced disbands. When laser energy reaches 1200 mJ, all the new delaminations observed are found to take place between the second and third plies from the back surface (figure 13(d)) meaning that the inter-ply damage threshold was reached.

Figure 15(a) shows the velocity signals obtained below (400 mJ) and just above the damage threshold (600 mJ). The grey signal contained in the dashed rectangle is then normalized to the black signal maximum and presented in figure 15(b). The arrival times of the compressional (longitudinal) wave which has crossed one and three times the total assembly thickness are, respectively, denoted L^1 and $3L^1$. $L^1 2L^4$ represents the arrival time of the compressional wave having an additional back and forth propagation inside one of the 4-ply laminates. Finally the tensile wave due to an additional reflection inside the (Hysol) joint thickness reaches the back free surface at the time denoted as $L^1 2L^4 L^H$. The event indicated by $L^1 2L^4 L^{H*}$ is for the same reflection but when disbond occurred. The rupture is observed at the interface closer to the front loading surface (top joint interface). This is seen well in figure 15(b). Until the disbond signature, the signals are almost identical, which confirms again the elastic regime of wave propagation.

Here also, simulation helps us to check the validity of the interpretation. Figure 16 shows the normalized simulated back surface velocities with and without a disbond. In this case, the signals are completely identical before disbond signature. Disbonding is modelled as an adhesive rupture in which only the ties at the interface are broken. During such rupture, the high amplitude tensile wave separates the two planar surfaces at the interface between the adhesive and the adherent. After some time, since almost no plastic deformation has occurred, mechanical contact is expected to be restored. In this case we are producing a real kissing bond with good mechanical contact but no mechanical strength. This explains the weak damage signature obtained with laser-ultrasonic inspection technique and the need to make a careful analysis. The damage is unlike the cohesive damage produced in the epoxy between the plies of the laminate indicated in section 4.2. In that case there was a permanent cracking that is easily detected by ultrasound in the time domain.

Figure 13. Laser-ultrasonic inspection of the '4/4' assembly, C-scan obtained before (a) and after shocks at a laser energy of 600 mJ (b), 1000 mJ (c) and 1200 mJ (d). Some of the C-scans are associated with their B-scans obtained along the dashed line. The lines pointed by t_b correspond to the reflection of the wave on the free top surface. The events indicated by t_j correspond to the reflection on the disbonded interfaces. The encircled multiple echoes in (d) are reflections at the disbonded interface between the second and third ply.

(This figure is in colour only in the electronic version)

Figure 14. Laser-ultrasonic inspection of the '4/4' assembly: (a) displacement signals in black and grey of, respectively, a disbonded and a bonded area and (b) their respective spectra.

Figure 15. (a) Velocimeter signals produced at 400 mJ loading in grey and at 800 mJ loading in black. (b) Zoom on the black signal comprised in the dashed rectangle with the grey signal normalized to the black signal maximum.

Equation (6) is applied to the evaluation of the bond strength of the weakly bonded area of the '4/4' assembly and gives a value of about 150 MPa, less than half the bond strength between plies. The strength of the strong joint cannot be evaluated because it is higher than the inter-ply bond strength.

5.2. Shock waves in the other assemblies

Figure 17 presents the C-scans obtained from the laser-ultrasonic inspections before and after shocks with the '4/8' and '8/4' assemblies. In the case of the '4/8' assembly the

Figure 16. Simulated back surface velocities with (black curve) and without (dashed grey curve) damage at the top joint interface. Simulation was performed for a 100 μm thinner sample than the one used for the experimental data shown in figure 15 so that the comparison with experimental results is only qualitative.

Figure 17. Post-shock C-scan laser-ultrasonic inspection of the '4/8' (a), (b) and '8/4' (c), (d) assemblies: upto 1100 mJ (a), (c) and at 1500 mJ laser energy shocks (b), (d).

shocks were generated on the 4-ply laminate and the laser-ultrasonic scans were performed from the 8-ply laminate side. For the '8/4' assembly, this was the opposite: the shocks were generated on the 8-ply laminate and the laser-ultrasonic scans were performed from the 4-ply laminate side. The diameter of the laser shock spot was increased to about 6 mm in order to keep the laser spot diameter to assembly composite thickness ratio constant. The wave propagation is thus in the same regime of diffraction as the '4/4' assembly. Up to a laser energy of 1100 mJ (figures 17(a) and (c)), no disbond is observed. The first disbands are observed at 1300 mJ and cover almost all the weakly bonded area at 1500 mJ (figures 17(b) and (d)). Temporal ultrasonic signals indicate that the disbands occur at the joint depth. Because of the distance from the back surface, the observation of the disbands is easier from the 4-ply back surface than from the 8-ply back surface. Disbonded areas appear also wider but the energy needed for their creation is the same.

Figures 18(a) and (b) show, respectively, the experimental back surface velocities with and without disbond in the '4/8'

and '8/4' assemblies. The signals obtained below damage threshold are normalized to the maxima of the signals obtained above damage threshold. Actually, the maxima of the grey signals are, respectively, about 96 m s^{-1} in figure 18(a) and about 62 m s^{-1} in figure 18(b). The instants t^i at which the disbands occur are more difficult to identify than previously because only a magnification of the reflected echo amplitude is expected. They are identified at about $1.52 \mu\text{s}$ in figure 18(a) and $1.15 \mu\text{s}$ in figure 18(b). Nevertheless, the disbond signature is merely recognized at later time. Particularly in the '8/4' case, where the waves trapped in the 4-ply composite give rise to strong reverberation. According to equation (6) the averaged bond strengths on all the weakly bonded areas were still, respectively, evaluated at about 140 and 150 MPa for the '4/8' and '8/8' assemblies.

Figure 19 presents the C-scans obtained from the laser-ultrasonic inspections before and after shocks with the '8/8' assembly. The diameter of the laser shock spot was about 8 mm. No disbond is observed below a laser shock energy of 1500 mJ (figure 19(a)). Then the disbands cover almost all the weakly bonded area at 1500 mJ (figure 19(b)). Temporal ultrasonic signals indicate that the disbands occur at the joint depth. The shock velocities signals (figure 20) show that the time at which the disbond signature becomes visible is about $1.82 \mu\text{s}$. In fact the maximum of the grey signal is about 65 m s^{-1} . According to equation (6) the bond strength is still evaluated at about 140 MPa. In this case, as in the case of the '4/8', the identification of the disbond signature from the velocity signal is not straightforward. Alternatively, the bond strength and the rupture time t_{rupt} can be obtained using equation (2) (with the delay calculated from the depth of the identified rupture) and are found to correspond to the point of maximum traction.

Small difference of damage threshold had been found between the weakly bonded areas of the different assemblies. Since the damage is situated further from the back surface in '8/8' and '4/8' assemblies, the discrepancy can be explained by the attenuation, which was not taken into account in the stress evaluation.

6. Summary and 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 in the result of damage under high strain rate deformation the material shows brittle behaviour. These observations confer to the method the quality of a non-invasive proof test and allow the strength measurement to be made quantitatively. The validity and precision of the measurement have been validated by simulation. 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. Laminates without bonds were first tested and the inter-ply bond strength within the laminate was evaluated to about 340 MPa. Then series of two laminates were bonded in such a way that half of their joint presents weak

Figure 18. Velocimeter signals obtained (a) with the '4/8' and (b) with the '8/4' assemblies for laser energies above (black signals) and below (grey signals) the damage threshold. The grey signals have been normalized to their respective black signal maxima.

Figure 19. C-scan laser-ultrasonic inspection of the '8/8' assembly: pre- and after shocks at 1300 mJ laser energy (a) and at 1500 mJ laser energy (b).

Figure 20. Velocimeter signals obtained with the '8/8' assembly for laser energies above (black signal) and below (grey signal) the damage threshold. The grey signal has been normalized to the black signal maximum.

strength and the other half strong strength (compared with the inter-ply bond strength). When tested with the technique, the assemblies reveal effectively the two different bonded areas. The strength of the strongly bonded areas was found to be at least equal to the strength between the plies, whereas the weakly bonded areas showed a strength of about 150 MPa, significantly less than the inter-ply strength. The measured strengths are larger than the usual static values because of the high strain rate measurements. Correlation between the static and dynamic values is expected but needs to be demonstrated.

The encouraging results provided by this work are an incentive for further development of the technique in view of ultimately using it for certifying adhesive bonding of primary aircraft structures.

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