LASER SHOCK WAVES TECHNIQUE FOR DAMAGE THRESHOLD DETERMINATION OF AERONAUTIC COMPOSITE MATERIALS

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Abstract
Laser shocks were performed on two aeronautical composite materials. The use of time resolved image acquisition enabled to study the dynamic behavior of the damage induced by the laser shock wave propagations. Since laser loading is really short, post-mortem analysis were possible. X-Ray radiography, cross section microographies and Interferometric confocal microscopy were used to investigate the laser shock effect on composite material. Main damage dimensions were compared from one material to the other. A difference in the damage threshold to laser shock was lightened.

1 Introduction
This study is part of ENCOMB European project (Extended Nondestructive testing for COmposite Bonds) [1]. The aim of this project is to increase the use of bonded composite materials in aeronautic, in order to replace the current techniques like riveting or bolting. These methods are expensive and not well-adapted to composite materials since complex machining has to be set up (drilling leading to delamination) [2]. Moreover, the use of the bonding technique would enable a significant weight lightening of the aeronautic structures, which means an aircraft consumption reduction. Nevertheless, several implementation problems can penalize the bonding process because the bond quality can be weakened by a bad curing cycle, a surface contamination before bonding, etc. [3]. Moreover, there is no non-destructive technique currently available to quantify the bonding mechanical resistance. Facing this issue, new methods are developed in order to certify the bonding mechanical quality. One of the developed methods is the laser shock wave technique, which can create a short but intense tensile loading at an interface [4]. In some conditions, this technique has already been successfully used to test different bonding strengths of assemblies [5], [6], [7]. Nevertheless, more investigations are necessary in order to optimize the technique and to better understand the associated complex physical phenomena [8]. In particular, the composite material dynamic behavior under laser shock has to be characterized to develop a technique efficient for any kind of material and which
could be transferred to industry. For that, experimental investigation of laser effects on aeronautic composite materials is a first step. Moreover, these results are also linked to shock resistance of composite materials. Indeed, the laser shock conditions can be compared to different shock loadings at very high strain rate or very high impact velocity (icestones, birds). Since the samples can be recovered after the laser shock, it is possible to analyze them and to compare the damage level between samples. In this study, the laser shock wave technique has been used in order to test several carbon/epoxy composite materials. Different diagnostics have been developed to analyze the dynamic response of the composite and to quantify the inside damage levels created by the wave propagation. A damage threshold for shocked samples has been approached by comparing the post-mortem observations.

2 The laser shock wave technique

2.1 Principle

The laser shock wave technique consists in a high power laser irradiation of a surface. When focused on a material, it transforms the surface into an intense plasma gas. The plasma expansion produces a shock wave into the material by the reaction principle. The created incident shock wave is then propagating through the thickness according to properties depending on the material characteristics and geometry. When reaching the sample back face, the reflection of this incident shock wave creates a release wave propagating backward (see in Figure 1). This release wave is crossing the incident unloading wave coming from the front face and initiated by the end of the loading (back to the initial state). Since the laser pulse is really short, the unloading occurs right after the shock wave propagation, which enables the two release waves to intersect inside the material. This crossing of two release waves can lead to a local high tensile stress area which could damage or not the material depending on the laser parameters. The generated tensile stress level is directly linked to the laser shock energy, whereas its location mainly depends on the material properties and the pulse characteristics. Therefore, the defect creation for a given material can be controlled by changing the laser source parameters.

Figure 1. Sketch of the laser shock wave technique principle

2.1 Experimental setup

The laser shocks were performed in two different laboratories. Several power laser sources are available in PPRIME Institute (Poitiers, France). The one used in this study generates laser gaussian pulses whose duration full width at medium height lasts about 25 ns and whose beam energy can be adjusted in the range [0J – 25J]. This laser source is generally used to test thick samples (millimeters) and can create sizeable inside damage (millimeters) in case of composite materials. The laser shocks are made in water confinement configuration.
The LULI (Laboratoire pour l’Utilisation des Laser Intenses, Ecole Polytechnique, Palaiseau, France) runs several high power laser sources. The ELFIE laser source used in this study delivers laser gaussian pulses whose duration full width at medium height lasts 0.35 ps and whose beam energy is tunable in the range [3J – 10J]. The samples are placed inside a vacuum chamber. Windows enable the laser irradiation and the transverse observations (see in Figure 2). The positioning of the samples was chosen in order to present the front face to the laser irradiation, and the cameras were focused on the back face to observe spallation. A power flash lamp (Balcar starflash 3) provides light (400 to 700 nm) during the laser experiment. In order to get several images of one laser shock, three DICAM pro cameras are used on the same optical axis. Beam splitters are placed on this axis to reflect one image in each camera (see in Figure 2). The whole observation system (flash and cameras) is fully synchronized with the laser shock timing. The three cameras can be delayed independendly to picture three different instants of the shock wave propagation inside the tested sample.

Figure 2. Experimental setup used for transversal observations. Three DICAM PRO cameras are synchronized to picture three different time states of the target under a laser shock.

3 Experimental results

3.1 Time resolved measurements

A thin T300/914 unidirectional composite sample was shocked using ELFIE laser source (LULI). The main laser shock parameters are given in Table 1, where $D_{foc}$ is the laser focalized diameter on target. Intensity can be calculated using equation (1).

$$ I = \frac{4 \cdot E}{\Delta t \cdot \pi \cdot D_{foc}^2} \quad \text{with} \quad \begin{cases} E : \text{laser energy} \\ \Delta t : \text{pulse duration} \\ D_{foc} : \text{focalized diameter} \end{cases} \quad (1) $$

The experiment setup presented in the previous section was used to picture several moments of the damage growth under the laser shock in this composite. Results are presented in Figure 3. On the left side of each picture, the composite sample can be observed with a small tilt on the dedicated rack. Fibers are oriented vertically. At time 0, the laser shock is produced on the sample front face. After one microsecond, a thin part of the material starts to grow due to the

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness (µm)</th>
<th>Energy (J)</th>
<th>Duration (ps)</th>
<th>$D_{foc}$ (mm)</th>
<th>Intensity (PW/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3.7</td>
<td>400</td>
<td>3.87</td>
<td>0.35</td>
<td>2</td>
<td>0.35</td>
</tr>
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</table>

Table 1. Laser shock parameters measured for a T300/914 composite sample (S3.7).
tensile stresses generated by the laser shock wave propagation. It is a thin slice of material, meaning a few fibers still taken into the epoxy matrix like a thin composite ply. At time 2 µs, the first fiber breakages have already occurred as proved by the space between the two growing slopes. Two microseconds later, a lot of small parts composed of a few fibers only start to separate from the sample. The ejection has started and the damage becomes significant.

![Figure 3](image)

Figure 3. Time resolved observations at different instants for unidirectional composite material T300/914 – 400µm thick

These observations enable to better understand the consequences of a laser shock on a composite material. This loading case is extremely short and intense, so it is a bit far from real impact case. Thus, other loading cases are also presented in the next section on thicker composite samples.

3.1 Post-mortem analysis

3.1.1 Laser shock experiments

Two aeronautical composite materials are compared. The first one is the T300/914 unidirectional composite material, already used in the previous section. It is composed of several pre-impregnated plies of carbon fibers and epoxy matrix (approximately 150 µm thick). It is referred as S3. The second one is a T800/M21 unidirectional composite, qualified by aeronautical companies as an enhanced version of T300/914. Indeed, it is still made of carbon fibers and epoxy matrix, but thermoplastic nodules are added in the M21 matrix in order to increase the shock resistance of the material. The pre-impregnated plies are thicker (about 250 µm). It is referred as S4. One series of each material has been investigated using laser shock waves. The laser shock parameters used in PPRIME Institute are given in Table 2. In order to compare the two material responses, the same four shocks were performed on each material (see in Table 2). After the laser shocks, the samples were recovered from the experimental setup to be analyzed using several post-mortem diagnostics such as X-Ray radiography, Interferometric confocal microscopy, and optical microscopy.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness (mm)</th>
<th>Energy (J)</th>
<th>Duration (ns)</th>
<th>Dfoc (mm)</th>
<th>Intensity (GW/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>0.66</td>
<td>27.37</td>
<td>4</td>
<td>0.19</td>
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</tbody>
</table>

Table 2. Laser shock parameters used for T300/914 composite samples and T800/M21 composite samples
3.1.2 X-Ray radiography

The X-ray radiographies of the two series are presented in Figure 4. The fiber direction is horizontal and the samples are ordered from left to right in the laser intensity level decreasing order. The main damage in plan dimensions are revealed in white by the zinc iodide inserted using a specific solution beforehand. A direct correlation can be made between the damage extent and the laser intensity for each material. For damaged samples, a large rupture in the fiber direction can be observed. The flexural component of the loading is responsible for that rupture since unidirectional composites are quite weak in that direction. For the highest intensity values, the laser shock wave propagation has also created delamination in the samples, as shown by the white elliptical shapes. The anisotropic characteristics of the damage are shown by these elliptical shapes, in spite of the axisymmetric loading and according to the composite anisotropy.

![Figure 4. X-Ray radiographies of T300/914 and T800/M21 unidirectional composite samples – Four different laser shocks of various intensities were performed on each series](image)

Differences between T300/914 and T800/M21 materials can be noticed in Figure 4. Even if both materials weren’t damage for the lowest intensity level, the T300/914 samples are globally more damaged than the T800/M21 samples. The delaminations seem wider, and the ruptures are more obvious on T300/914 samples. This could be confirmed by optical micrographies.

3.1.3 Optical micrographies

Cross section observations perpendicularly to the fibers were made (see in Figure 5). For all the presented micrographies, the 4 mm diameter laser impact area is represented on the bottom of each sample; it is meaning the front face. They are separated in two by the axisymmetric axis of laser shock loading which is also defining the symmetric plan of the samples since they are all unidirectional. On one side only, the observed damage as delaminations or cracks are highlighted by red lines. On both materials, the damage is located close to the back face, around 250 µm deep. This is due to the pulse duration of the laser source used as previously explained. One more time, a direct correlation between the laser intensity level and the damage extent can be observed. Long transverse cracks due to the flexural component of the loading can also be seen on the micrographies. They are more obvious for T300/914 samples than for T800/M21 samples, especially for the highest
intensity value. In that loading case, the T300/914 sample was broken in two by the laser shock, when the T800/M21 sample was still in one part. But the laser irradiation is also responsible for the delamination which can be observed on almost all the microographies. Delaminations and associated transverse cracks are due to the local tensile stresses induced by the laser shock wave propagation. In case of the highest intensity value, the T300/914 sample has been peeled off, and a few plies were ejected under impact. The corresponding T800/M21 sample is completely delaminated too, but all the plies remained on the sample (see the first two images in Figure 5). In case of the lowest intensity value, the T800/M21 is not delaminated, but the T300/914 is on a small distance. The microographies confirm the observations made by X-Ray radiography: T800/M21 material seems stronger.

![T300/914 unidirectional samples](image1)

![T800/M21 unidirectional samples](image2)

**Figure 5.** Comparison of the inside damage for two set of samples: T300/914 and T800/M21 – All the samples were shocked with various intensity laser irradiances.

### 3.1.4 Interferometric confocal microscopy (ICM)

Interferometric confocal microscopy can provide complementary information. In case of laser shocks, it can be used to measure the back face deformation due to spallation or delamination. The two series have been measured and compared using this technique (see in Figure 6). A zone of 9 mm per 9 mm is measured. The back face deformation amplitude is given in micrometers by the color scale. In case of the most damage T300/914 sample, the area where ejection occurred wasn’t measured (too deep). These measurements and the other ones previously presented are consistent. In order to compare the two materials, profiles orthogonal to the fiber direction and centered on the observed elliptical shape were extracted (see in Figure 7). For the two materials, a correlation between the back face deformation and the laser intensity level can be made. As expected, when the laser intensity increases, the back face deformation is more important. In case of T300/914 samples, the spallation is also visible by the blank in the curve (see in Figure 7).
Figure 6. Interferometric confocal microscopy on T300/914 and T800/M21 unidirectional composite back face samples – The back face deformation amplitude in µm is given by the color scale.

Figure 7. Back face deformation measurements by ICM for two set of samples: T300/914 and T800/M21 – All the samples were shocked with various intensity laser irradiances.

Except for the mid energy level, the back face deformations are more important in case of T300/914 samples. This observation can be summarized by plotting the maximum deformation height against the laser intensity values (see the graph in Figure 8). It enables to evidence a difference in damage threshold to the laser shock between the two composite materials used in this investigation. In addition to the cross section observations and to the X-Ray radiographies, this last analysis justifies the enhanced shock resistance of the T800/M21 compared to the T300/914 characteristic. Thus, the laser shock wave technique could be used as a way to compare the shock resistance of materials.
4 Conclusions
Laser shocks were performed on two aeronautical composite materials: T300/914 and T800/M21. The use of time resolved image acquisition enabled to study the dynamic behavior of the damage induced by the laser shock wave propagations. X-Ray radiography, cross section observations and interferometric confocal microscopy were used to investigate the laser shock effect on composite materials. Main damage dimensions were compared from one material to the other by use of these different diagnostics. The laser shock wave could be used as a way to differentiate the damage threshold of monolithic materials, but also of bonded materials, whose achievement could change the assembly of aeronautical structures.

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