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Determination of the bond strength of some microns coatings using the laser shock technique

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Abstract. High power laser shocks with a 0.6 ns pulse duration have been used to study the debonding of coatings electrolytically deposited on the opposite face of the substrate than the one shocked. Experiments have been carried out on various substrate/coating systems such as stainless steel/copper or nickel and hastelloy X/platinum. Experimentally, a lower intensity debonding threshold has been determined for each of these systems. On the other hand, an upper threshold above which a systematic removal of the coating is obtained has been evidenced. By the numerical simulation of these experiments, a traction range for debonding at the interface has been determined for the three systems. A significant difference for the adhesion levels of these systems has been evidenced using this method. Thus, the possibility to use the laser shock technique as a non destructive adhesion test for coatings of some tens microns is clearly demonstrated.

PACS. 47.40.Nm Shock-wave interactions and shock effects – 68.35.Gy Mechanical and acoustical properties; adhesion – 42.62.-b Laser applications

1 Introduction

In many industrial materials, the reliability of a coating on a substrate is relevant to the adhesive quality of the system. In order to assert a required adhesive quality to these substrate/coating systems, it is necessary to perform adhesive tests. Most of the usual tests are not completely satisfactory because they can be destructive (scratch test), difficult to implement or unable to be applied systematically with a high repetitive rate with reproducible conditions [1–4]. Besides, with most of these tests, it is difficult to determine accurately the traction conditions, depending on the method used. Since the development of high power lasers and their applications, the spallation process induced by laser shocks has been widely studied [5,6] and its application to the debonding of coatings has been initiated [7–12]. It has been applied first to the debonding of very thin coatings [13] or more recently to assemblies of several materials [14,15]. In this study, we have tested the method on three different substrate/coating systems (hastelloy X/platinum, stainless steel/copper and Stainless steel/nickel) with different thicknesses of electrolytic coatings (7.16 μm, 36 μm and 30 μm respectively). The samples have been provided by the companies Chromalloy France for the Pt coating and Sochata for the two other ones. We demonstrate here the ability of short laser shocks to determine the relative adhesive properties of the various coatings studied on their substrates. By using the numerical simulation of shock waves propagation into these systems, by comparison with the experiments performed, we have determined a traction threshold range to induce for each interface in order to debond it. Lastly, the perspectives and possibilities to use this method as a non destructive systematic test for adhesive properties of coatings on substrates is discussed.

2 Principle of laser shock debonding

In most of the work on the topic, the laser shock debonding is introduced as a simple spallation process which is due to the crossing of the incident unloading wave with the reverberated release fan from the free surface. The principle of the generation of the tensile wave is illustrated on a simple modeling of a laser shock. In order to simplify the modeling, we assume the propagation of a square pressure pulse of P_{max} amplitude with a duration τ, making the acoustic approximation for all the waves propagating in the materials. Hence, the different states reached on the space-time diagram (*cf.* Fig. 1a) for a system substrate A/coating B where the shock impedance of B is higher than A's one can be plot on the (p, u) plane (*cf.* Fig. 1b). The schematic stress history extracted

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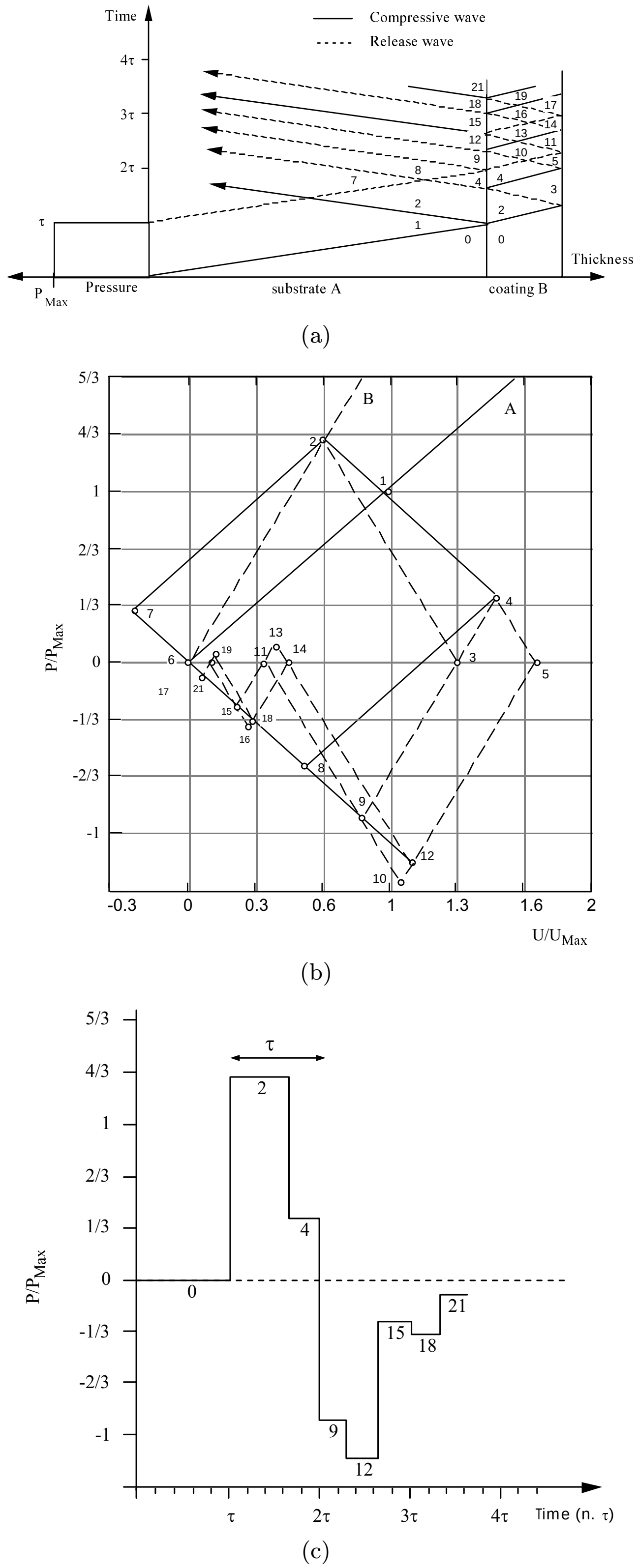

Fig. 1. Space-time diagram of a square pulse propagating in a substrate A with a coating B whose shock impedance is higher than the substrate's one (a), corresponding states on the (pressure-particle velocity) plane (b) and sketch of the stress history generated at the interface (c).

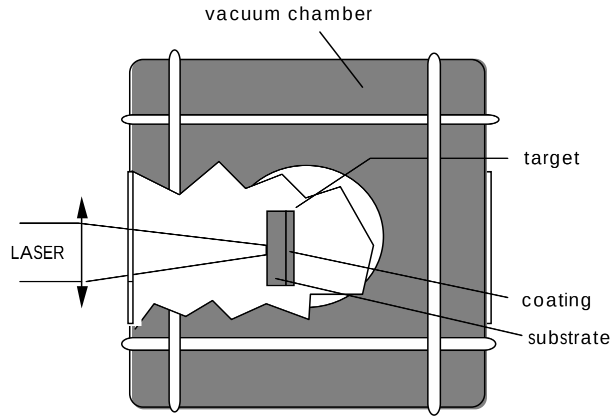

Fig. 2. Experimental set-up used to determine the debonding threshold for various systems.

from the interface (*cf.* Fig. 1c) shows that, the first traction states (namely 9, 12, 15 states) are generated by the impedance mismatch between the coating and the substrate and the reflection/transmission of the unloading wave coming from the front surface of the substrate. From the analysis of this particular case, we see easily that the stress history of the interface and particularly the generation of a tensile state at the interface depend on many parameters such as:

(i) the relative impedance mismatch between the coating and the substrate;
(ii) the thickness of the coating relatively to the shock duration;
(iii) the thickness of the components acting on the hydrodynamic decay during the propagation of the shock waves;
(iv) the shock profile;
(v) the nature and the preparation of the interface acting on the bond strength.

3 Experimental study

3.1 Experimental set-up

The high power laser European Large Scale Facility of LULI[1] has been used as a shock generator with a 600 ps Full Width Medium Height pulse at $\lambda = 1.06$ μm. The 90 mm laser beam is focused on spots of 1 to 2 mm on the substrate's bare surface, at the opposite side of the coating. The target is placed in a vacuum chamber to avoid the laser breakdown into the air at high intensities (*cf.* Fig. 2). For every tested system, a first shot is performed with the maximum available energy (about 80 J) and if the removal of the film is achieved, the debonding threshold laser intensity is investigated by decreasing step by step the incident intensity by interposing attenuators in the beam.

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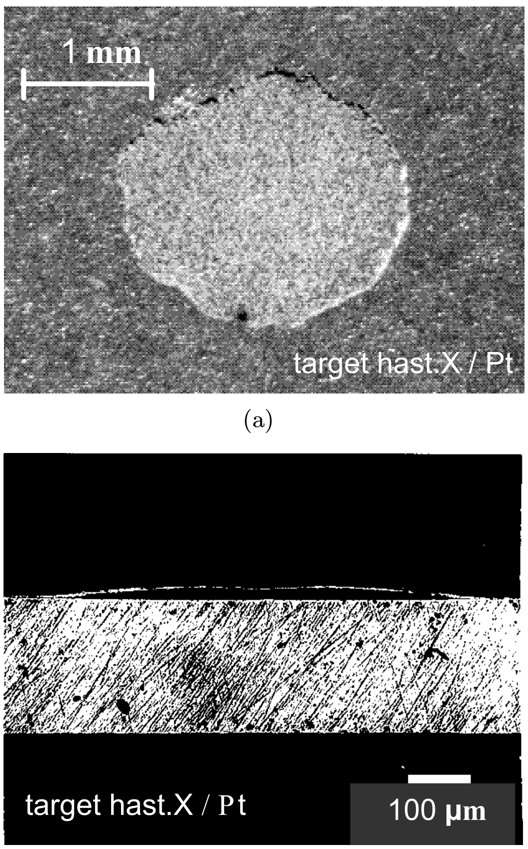
Table 1. Mechanical parameters and shock waves data used for the different materials involved into the tested systems (density: ρ_0, parameters of the linear relationship between the shock speed U_s and the particle velocity u: $U_s = C_0 + su$, Grüneisen parameter: Γ, yield strength: Y_0, bulk modulus: G).

	ρ_0 (g/cm^3)	C_0 (m/s)	s	Γ	Y_0 (GPa)	G (GPa)
Hastelloy X	8.2	4 820	1.49	2	0.6	90
Stainless steel	7.8	5 935	1.474	1.735	0.6	77.5
Copper	8.93	3 933	1.5	1.99	0.32	47.7
Nickel	8.2	4 119	1.806	2	1.078	79.2
Platinum	21.449	3 680	1.46	2	0.32	61.5



(a)

(b)

Fig. 3. Observation of the coated surface of the substrate with a totally removed film on a size corresponding to the laser spot (a), an incipient coating removal on a cross-section (b).

Doing so, different stages of the film removal are observed according to the following classification illustrated by Figures 3a and 3b:

- complete removal of the coating (marked □ on the next figures) (*cf.* Fig. 3a);
- incipient removal of the coating, with a small part expelled (marked △);
- bulging of the film, which has to be observed on a cross-section of the sample to detect the level of debonding, meaning that we are close to the debonding threshold (marked ⋄, *cf.* Fig. 3b);
- the sample remains intact, no decohesion is observed (marked ○), meaning that we are below the debonding threshold and that the generated traction has been too low to provoke any removal.

3.2 Description of the tested systems

Three different systems of interest for the companies Sochata and Chromalloy-France have been tested:

- substrates of hastelloy X with an electrolytic coating of 7.16 μm of platinum;
- substrates of Z10CNW17 stainless steel with an electrolytic coating of 36 μm of copper or 30 μm of nickel.

The thickness of the substrates was never superior to 400 μm in order to insure a planar configuration for shock waves propagation with a focused spot of 1 or 2 mm diameter. The different parameters used for simulating shock waves propagation into these assemblies are listed in Table 1. For hastelloy X, these parameters couldn't be found into literature. So, a set of these parameters has been determined to fit by simulation free surface velocity measurements of bare hastelloy X plates submitted to laser shock loading.

3.3 Experimental results

For the different systems tested, the level of damage produced *versus* the incident laser intensity on the bare surface of the substrate are plotted in Figure 4. For every sample tested, the thickness of the target has been optimized to be the most accurate possible for the interpretation. For each of the three tested systems, by decreasing the incident intensity, we observe an upper threshold above which the debonding of the film is systematically achieved and a lower threshold below which no damage at all is produced. For a given target configuration, with a short laser pulse, when the incident intensity applied to the substrate decreases, the amplitude of the shock arriving at the interface decreases, and thereby, the traction amplitude generated after these compressions too. The thickness of the samples can also act on the stress history and this has to be related to the bond strength. In order to estimate the shape profile of the traction induced at

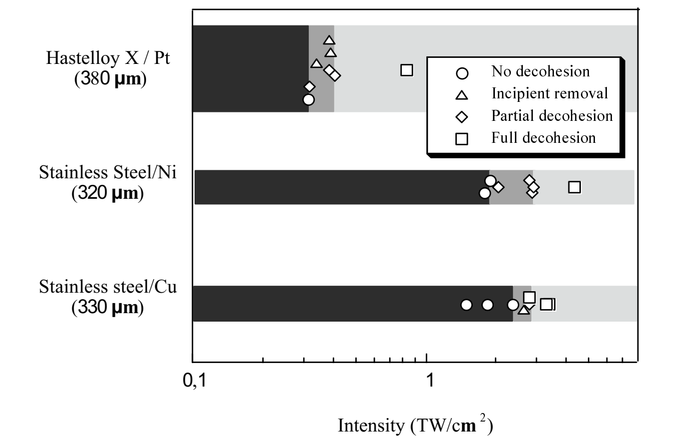


Fig. 4. Experimental observation of the damage produced on the tested systems *versus* the incident laser intensity.

the interface for all the experiments performed, numerical simulations of shock waves propagation into these systems have been run.

3.4 Numerical simulation

The numerical simulation of experimental debondings has been performed using two codes:

- FILM code [16] developed at LULI and simulating the laser matter interaction. Giving as input the incident intensity applied to the substrate and the time profile of the energy deposition, this code computes the corresponding mechanical loading applied to the target in terms of a pressure profile;
- this profile is used as input for the SHYLAC code [17] which simulates the 1D propagation of the loading into the substrate/coating described by an elasto-plastic constitutive law with a Mie-Grüneisen equation of state. From this simulation, we draw the stress history yielded at the interface during the experiment (*cf.* Fig. 5, phase I) which is generally a compression stage followed by a traction. The maximum value of the traction generated in the experiment can be read on these simulations (*cf.* Fig. 5, phase II) and reported on a diagram along with a reminder of the damage observed on the simulated experiment. Hence, the lower and upper threshold values for the traction at the interface can be directly read. This principle has been applied to the three systems and the peak values for each experiment are plotted in Figure 6.

4 Discussion

The black zone corresponds to the incident flux values for which no damage is observed. From this figure, we can see how the laser shock debonding method could be used as a non destructive test for the quality of adhesion for manufactured pieces. For example, in the case of a stainless steel/copper system, if we want to insure an as good

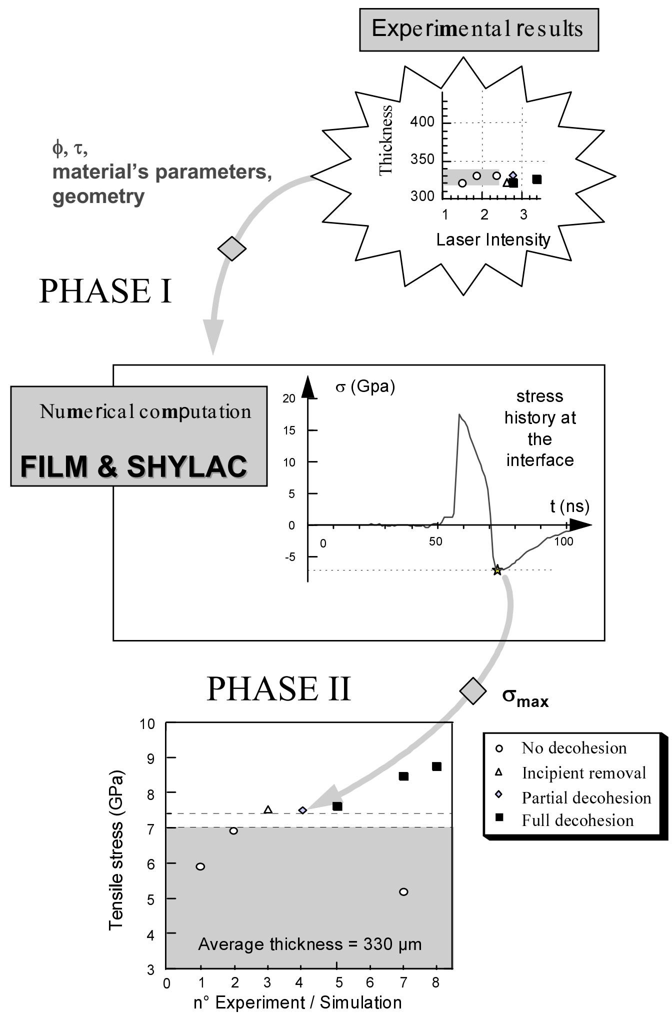


Fig. 5. Sketch of the principle of determination of the traction peak values at the interface by numerical simulation of the experiments using FILM and SHYLAC codes.

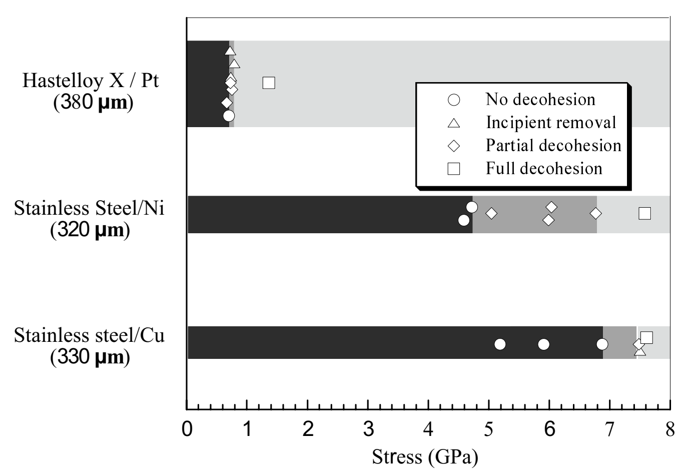


Fig. 6. Peak values of traction generated at the interface of systems determined by numerical simulation and corresponding damage observed experimentally on the coating allowing to read the debonding threshold of each system.

adhesion quality as the tested sample for the next manufactured pieces, one has to irradiate these latter with an intensity just below the threshold (2.5 TW/cm^2 for 0.6 ns laser pulses). If the quality is good enough, no damage on the coating will be produced (just a superficial burning on the spot size diameter on the substrate); and if the coating adhesion has a too poor quality, it will be expelled. So, by this test, the manufactured piece will be damaged only in the case when it does not satisfy the required quality, realizing hence a non destructive debonding test.

By this method, we reveal the different levels of adhesion σ_{co} for the three kinds of systems:

- for stainless steel/copper, 6.9 GPa $< \sigma_{co} <$ 7.5 GPa;
- for stainless steel/nickel, 4.7 GPa $< \sigma_{co} <$ 6.7 GPa;
- for hastelloy X/platinum, 0.7 GPa $< \sigma_{co} <$ 0.8 GPa.

These values, apparently very high compared to those provided by usual tests, are so because of the dynamic feature of the traction generated during very short times. More experiments have to be carried out to be able to know whether a simple cut-off criterion [18] for debonding with these values could be applied, or if the dynamics involved in these configurations have to be taken into account. A limitation to this test with such short laser pulse is the thickness of the sample which has to be rather low (within 1 mm range) to generate enough traction at the interface.

5 Conclusion

In this study, we have shown the ability of 0.6 ns laser shocks to produce the debonding of coatings of nickel or copper on stainless steel, and platinum on hastelloy X. The experiments performed exhibit a specific intensity threshold for every of these systems. This threshold could be used for performing a systematic non destructive test on manufactured pieces in the same configuration as the one of these experiments to attest an equivalent interface strength quality. The numerical simulation of all the experiments allowed us to determine the peak traction threshold produced at the interface to realize the debonding. A very large scale of debonding values ranging from 0.7 to 7.5 GPa have been found for the three systems, confirming the experimental observation of the adhesion level.

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