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# QUANTIFICATION OF THE ADHESION OF METAL THIN FILM INTERFACES BY PICOSECOND ACOUSTICS

Alejandro Vital Juarez<sup>1</sup>

Arnaud Devos<sup>1</sup>

Jean-Michel Desmarres<sup>2</sup>

<sup>1</sup> IEMN, Villeneuve d'Ascq, Lille

<sup>2</sup> CNES, Toulouse, Lille

alejandrovitaljuarez.etu@univ-lille.fr, arnaud.devos@univ-lille.fr

## ABSTRACT

We present a picosecond ultrasonic measurements of the modification of the adhesion between a thin film and its substrate that results from the presence of a pollution at the interface. The bonding is studied through measurements of the acoustic reflection coefficient at the interface.

The Colourful Picosecond Acoustics (APiC) method uses a femtosecond laser in a pump probe scheme which makes it a non-destructive method able to create a map of the variation of the bonding over the area of the interface.

## 1. INTRODUCTION

The study of the bonding at the interface between thin layers is of great importance in the fabrication of space mirror. With the purpose of ensuring the good quality of the material, a large number of methods have been developed to characterize the adhesion of thin films to substrates [1]. Among this methods, CNES uses the scotch tape or the Centrifugal Adhesion Testing Technology (CATT) which give either a qualification or a quantification of the adhesion energy [2–4]. A great disadvantage, which is shared by all this methods, comes with the destruction of the sample. Picosecond ultrasonics has been used to map the adhesion changes in a same sample by studying the acoustics reflection coefficient at the interface [5] and has the advantages of been non-destructive.

In this work, we apply the ultrasonic technique to the study of the modification of interfacial bonding that results from the pollution of the surface of the substrate. We want to demonstrate the capacities of the APiC method not only to give the quality of the adhesion at the interface, but also to map its weakness causes by any pollution present between the layers.

We make the choice to leave a fingerprint on the surface of the substrate before adding a thin layer, representing the pollution which compromises the adhesion. We then measure the acoustic reflection coefficient at the interface, demonstrating the impact of the fingerprint in the adhesion by reproducing its form.

## 2. COLOURFUL PICOSECOND ACOUSTICS

Picosecond Acoustics is a non-destructive testing method using the generation and detection of a very high frequency acoustic wave by ultra-short laser pulses. The generation

of such acoustic wave obtained by thermoelastic effect depends on the length of the absorption of light by the material. The acoustic wave propagates inside the studied sample and provides information about the elasticity and the thickness by measuring the time of flight of each echo detected at the surface. Detection is then done by a photo-elastic effect causing a variation in optical reflectivity, allowing to obtain the difference in optical intensity at the surface [6].

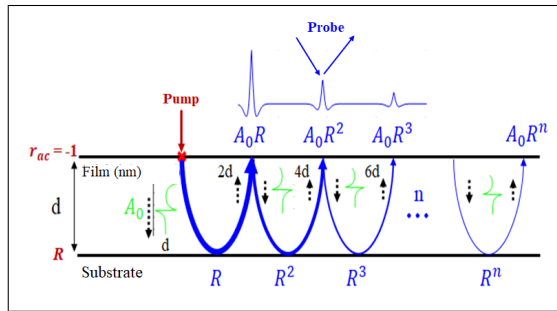
By modifying the value of the laser wavelength (Pump-Probe) it is possible to reinforce the detection of the echoes to decrease the thermal background and more. This idea gave birth to the APiC a non-destructive testing method which is used for measurements of thicknesses and elastic properties in complex stacks of thin films by creating a nanoscale SONAR. By following the transmission of acoustic waves and their reflection at the interface between two layers, it is possible to detect an adhesion anomaly [7].

## 2.1 Acoustics measurements

The principle of the experiment can be seen in Fig. 1 with the reflection of the successive echoes inside the material. The light source comes from a laser operating at 800 nm, with a pulse duration of about a hundred femtoseconds and a repetition rate of 80 MHz. The laser spot focus onto an area of 1  $\mu\text{m}$  diameter with an energy of a few  $nJ$ . The pump raises the temperature of the film and sets up a thermal stress which will change the pressure inside the material, creating the acoustic pulse. When an echo reaches the surface of the sample, the optical reflectivity of the film changes by a small amount  $\Delta R(t)$ . The time-delayed probe focused into the same area of the sample surface illuminated by the pump and detects the optical changes produced by the acoustical vibration. The successive echoes arrive at the surface with a time of flight which value depends on the acoustic velocity inside the material and its thickness. They continue to propagate until the sample stops vibrating and gets back to its original states.

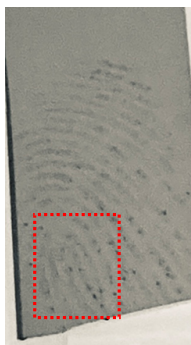
## 3. FINGERPRINT MAPPING

Measurements are mostly made on nickel layers on different types of substrate deposited by either evaporation or sputtering. By changing the deposition conditions, the adhesion, which is determined by the extraction of the



**Figure 1:** schematic diagram of the experiment and pulses in the material

reflection coefficient, will change. Fig. 2 shows a picture of the fingerprint on the silicon. The red square is the area where the acoustics measurements are done. We choose to



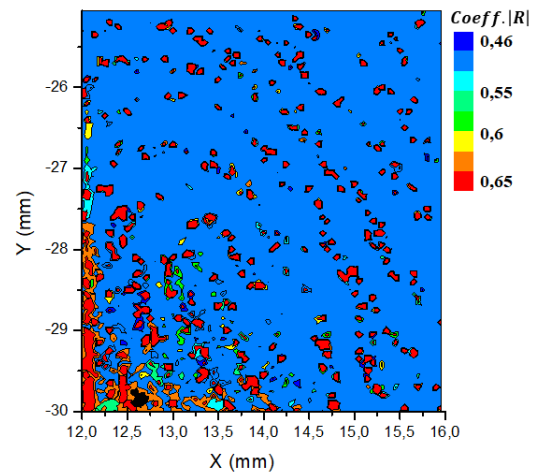
**Figure 2:** Picture of the sample : visible fingerprint between the substrate and the thin layer

do the measurements in the middle of the fingerprint, so we can have a clear map of the digital mark. Also, as the print has clearly left a mark of the finger's ridges and valleys, we expect to reproduce the figure by mapping the area where adhesion became weaker during the sputtering. This allow us to show, not only the importance on the adhesion of having a clean substrate but also the capacity of the APiC method to detect flaws on the sample. The mapping area is a  $4 \times 5 \text{ mm}^2$  rectangle, the laser does one measurement every  $50 \mu\text{m}$ .

Fig. 3 shows the result of measurements by giving a map of the decrease coefficient value. A bad bonding creates a bigger reflection of the energy on the interface which means a higher absolute value of the coefficient. By comparing the acoustic cartography with the picture of the fingerprint, we notice that the great values go perfectly with the shape of the fingerprint left in that area. It is safe to assume that there was a weak bonding in the area due to pollution.

#### 4. CONCLUSION

We have shown the capacity of the APiC to find the weak bonding on the sample by mapping with precision every corrupted area. This study represents the first step of many others adhesion studies with a polluted substrate. As the fingerprint is visible on the sample after sputtering, the



**Figure 3:** Map of every area with a corrupted bonding due to pollution: Acoustic cartography

next step would be to detect adhesion problems which are untraceable by optic methods. We believe that this capacity can be widespread into more complex and specific situation, in which only an acoustic wave would be able to detect the variations.

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