

# THIN FILM CHARACTERIZATION BY PICOSECOND ULTRASONICS ON HIGH CURVATURE SURFACES

## 1. INTRODUCTION

The system JAX designed by Neta uses unique properties of femtosecond pulses and photo-acoustic effects to inspect single thin film and complex multilayers structure.

Historically, acoustic waves were first generated with piezoelectric materials [1]. To increase the spatial resolution of these waves, it is necessary to increase their frequency. Whereas the frequency of the acoustic waves generated by piezoelectric transducers is still limited to a few hundreds of MHz [2], this frequency can extend up to several hundreds of GHz for acoustic waves generated by femtosecond lasers [3]. The classical pump-probe experimental setup was based on a single laser and a delay line to generate and to detect the acoustic waves. To overcome the slowness of the delay line, a new patented setup aimed to generate and detect the acoustic waves with two lasers whose repetition rate is enslaved and slightly different [4]. Technology embedded in JAX is based on this principle.

In this paper we would like to validate possibility of making 100% optical and non-destructive measurement on opaque thin films independently of the shape of the sample. Unlike classical destructive methods, those measurements allow new way to characterize particular area. Results have been obtained on a thin gold layer deposited on a titanium sample that shows a flat and a curved surface and a set of iron samples with different gold layer deposition.

Whether for the gold/titanium curved sample or gold iron flat samples, a standard metallizer have been used for gold deposition process.

## 2. EXPERIMENTAL SETUP

The results presented in this paper have been obtained with the system JAX-M1 whose principle of measurement is illustrated on Figure 1.

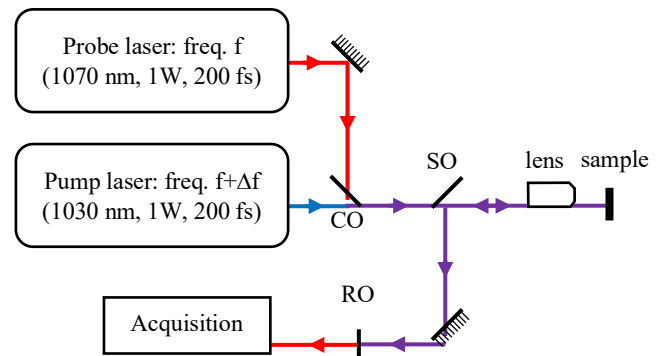


Figure 1. Heterodyne setup principle. CO: combining optics; SO: sampling optics; RO: rejection optics

Thanks to the electronic delay management between the two lasers instead of a mechanical delay line, considering the same signal to noise ratio, heterodyne experiments are faster than homodyne experiments by a factor between 10 and 100 [5].

## 3. RESULTS

### 3.1 Principle of Measurement

A typical measurement is presented in Figure 2.

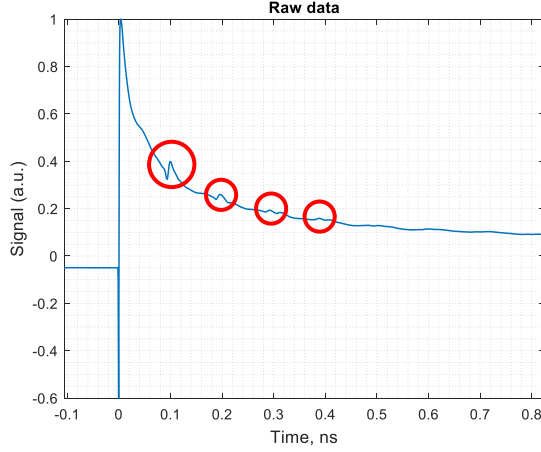


Figure 2. A typical measurement

This signal can be decomposed into three main parts.

At time  $t = 0$  (the coincidence), the energy of the pump pulse is absorbed by the electrons of the lattice of the coating and transferred to the phonons of the lattice. This leads to a brief and high variation of the reflected signal.

The heated area then recovers its equilibrium temperature. This is highlighted by the exponential decrease of the reflected signal called the thermal decay.

Superimposed on the thermal decay is the acoustic part of the signal. Three main kinds of information can be deduced from the acoustic part of the reflectivity measurement:

- when the material is sufficiently thick and opaque (more than about 20 nm for metals), the measurement highlights echoes like the ones surrounded in red in Figure 2.
- when the material is transparent or semi-transparent and sufficiently thick (several hundreds of nanometers minimum), the acoustic waves generated on the substrate or on a thin top absorbing layer (called transducer) propagate in the transparent medium. The acoustic wavefront can then induce local changes in the reflectivity of the material. The successively constructive and destructive interference between the part of the probe that is reflected on the substrate and the one reflected on the moving acoustic wavefront induces Brillouin oscillations with frequency:

$$f_{\text{Brillouin}} = \frac{2 \cdot n \cdot V_{\text{ac}}}{\lambda} \quad (1)$$

with  $n$  the optical index for the probe beam,  $V_{\text{ac}}$  the acoustic velocity and  $\lambda$  the wavelength of the probe beam.

Measuring the Brillouin frequency gives an estimation of the acoustic velocity, hence identifying the material.

- when the transparent layer is sufficiently thin to measure the Brillouin oscillations on the whole observation range or when several transparent materials succeed one after the other, the Brillouin oscillations can show phase shifts. By measuring the duration of a train of Brillouin oscillations, this gives an estimation of the transparent layer thickness.

As far as we are concerned regarding this white paper, we will focus on the first point mentioned above. Knowing the acoustic velocity in the layer, the detection of the arrival time of acoustic echoes gives a direct estimation of the layer thickness:

$$e = \frac{V_{\text{ac}} \cdot \Delta t}{2} \quad (2)$$

with  $e$  the layer thickness,  $V_{\text{ac}}$  the acoustic velocity and  $\Delta t$  the arrival time of the first echo or the difference in arrival time between two echoes. Usually, as the time of the coincidence is less easy to determine than pointing echoes, the difference in arrival time between two echoes is more accurate.

Moreover, the system Neta JAX-M1 gives relevant results that are remote and non-destructive, even if the sample is tilted with a tolerance that is about  $\pm 5^\circ$ .

### 3.2 Results for a Gold Layer Deposited on a Ti Sample

The shape of the titanium sample on which a gold layer has been deposited is presented in Figure 3.

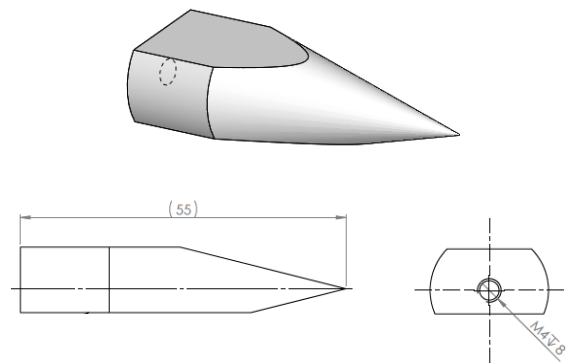


Figure 3. Drawing of the sample

The shape of this sample allows two kinds of measurement. First, measurements on a flat surface will give an estimation of the reproducibility; second, measurements on a conical surface will show how the measurements are reliable, even for curved surfaces.

### 3.2.1 Results on a flat surface

Four measurements have been performed on the flat part of the sample. The results are synthesized on Table 1, considering a sound velocity in gold equal to 3 240 m.s<sup>-1</sup>.

Measurement	1	2	3	4
First echo (ps)	11.3	11.3	11.8	10.3
Second echo (ps)	23.6	23.1	24.6	22.6
Thickness (nm)	19.9	19.1	20.7	19.9

Table 1. Thickness measured on the flat surface

The above results give a mean thickness and a standard deviation equal to 19.90 nm +/- 3.3 %.

These measurements show a very low standard deviation, hence proving a very good reproducibility.

### 3.2.2 Results on a conical surface

Different measurements have been performed all along a line that begins at the junction between the flat part and the conical part of the sample (for point 1) and ends at the tip of the conical part (for point 6).

Measurement	1	2	3	4	5	6
1° echo (ps)	9.3	8.4	8.8	8.4	8.4	7.9
2° echo (ps)	20.6	19.2	18.2	18.2	18.2	16.7
Thickness (nm)	18.3	17.5	15.1	15.9	15.9	14.3

Table 2. Thickness measured on the conical surface

A global trend is easily observed for the first six measurements: the closer to the tip, the thinner the layer. This is consistent with the deposition process. Under a certain thickness, the measurement could not give an exploitable result so easily.

Nevertheless, these results show that measurements are still possible for a gold layer on titanium whose thickness is as low as around 15 nm.

### 3.3 Results for a Gold Layer on flat iron samples

Some complementary results have been obtained regarding gold layers deposited on four iron samples. The deposit process is sputter deposition with different deposit times in order to vary the gold layer thickness. The results are synthesized in Figure 4.

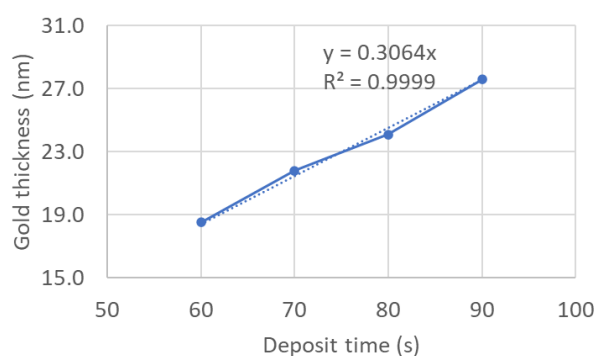


Figure 4. Gold thickness as a function of the deposit time

As expected, measurements show that the gold thickness  $e_{Au}$  (in nm) is proportional to the deposit time  $t_d$  (in s):  $e_{Au} = 0.3064.t_d$ . As the coefficient of determination  $R^2$  is very close to 1, this proves how reliable are the results obtained with the system Neta JAX-M1.

### 3.4 Examples of already tested materials

The results above prove the relevance of our system to characterize the thickness of a thin gold layer deposited on a titanium sample even curved.

Moreover, until now, other valuable results were obtained on the following samples:

- TiCN coating on a Al<sub>2</sub>O<sub>3</sub> substrate
- TiN coating on a Ti substrate
- Ag and Ag-ITO coatings on a glass substrate
- The following coatings on Si wafers: Al, SiO<sub>2</sub>, TiN/SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>, TiO<sub>2</sub>...

## 4. CONCLUSION

This article showed how our system can offer numerous possibilities for the mechanical characterization of materials. In particular, measurements showed relevant results for opaque layers as thin as 15 nm.

By detecting the arrival time of acoustic echoes that make a round trip in an opaque thin layer, the layer thickness is easily deduced knowing the sound velocity in the material.

These measurements are easy, fast as well as reliable. A typical measurement only needs about one second. This paves the way to the characterization of materials in many application fields.

## 5. REFERENCES

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