Non -destructive testing for thin films with picosecond ultrasonic

1. INTRODUCTION

Downsizing and thinning all the electronic parts has always been a trend in our modern era. However, the nanoscience and nanotechnologies were still science fiction in the 60's and the word nanotechnology was used for the first time in 1974. At the same time, the first atomic force microscopes (AFM) and scanning acoustic microscopes (SAM) were developed. Today nanotechnologies represent huge investments - even from governments - and a global market of several thousand of billions of euros.

Non-destructive testing at the nanometric scale is the purpose here. Ultrasounds are widely used in the aeronautics industry or during medical echography. The spatial resolution reached in that case is around the millimeter which is a million time too large when we speak of nanotechnologies. SAM systems benefit from a higher definition thanks to MHz/GHz ultrasounds and the smallest resolution in depth achievable is around the micron.

However, the nanometric world requires another 2 to 3 orders of magnitude below and it can only be reached thanks to THz ultrasounds. These frequencies cannot be generated with standard transductors, that's why this new method is using ultrafast lasers with pulses duration range from 100 fs to 400 fs. This technique is often called picosecond ultrasonics, it has been developed at Brown University in the USA by Humphrey Maris in the mid 80's. Results which are presented here, have been obtained with a JAX, the first photo acoustic imaging system using ASOPS (Asynchronous Optical Sampling) from Neta. The ASOPS systems are pump/probe instruments equipped with ultrafast lasers. Repetion rate of lasers may be various but the amount of energy per pulse on sample have to be monitored to avoid degradation.

2. GENERAL DESCRIPTION

Let's see in detail what happened when the pump laser hits the surface, the most part of the energy is absorbed by the first layers of atoms and converted into heat without damaging the sample (Fig. 1), leading to transient thermoelastic expansion and ultrasound emission. This ultrasound propagates at the surface of the material and also into the depth.

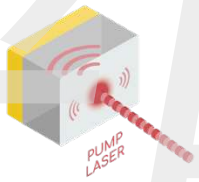

Fig. 1 : Ultrasound generation

The choice of the probe is also important to keep the temporal and the spatial resolution as low as possible, that's why another ultrafast laser is used as a probe (Fig 2.).

The ultrasound is propagating a few nanometers per picosecond through the thin film and at some point, will bounce back partially or completely to come back to the surface when meeting a different medium. Thanks to those high frequency ultrasounds, the resolution in depth is nearly below 1 nm and the lateral resolution is closed to the diffraction limit of the laser's wavelength.

The probe laser is focused on the surface, when the ultrasound hits back the surface, the reflectivity fluctuates locally over time.

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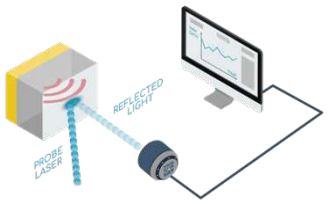


Fig. 2 : Detection of reflectivity changes with photo-detector

In order to detect this variation and study the material, very sensitive detectors are used. The variation of reflectivity is recorded and stored into the computer as a raw data.

The ASOPS systems are pump/probe instruments equipped with ultrafast lasers. Lasers are, in this case, shifted in frequency (Fig. 3). Both lasers are synchronized by a separate electronical unit. The probe arrives slightly after the pump and this delay is extending with time until the whole sampling is over. The elastic answer of the thin film to a pump excitation is too fast to be measured in real time that's why a sampling method is used.

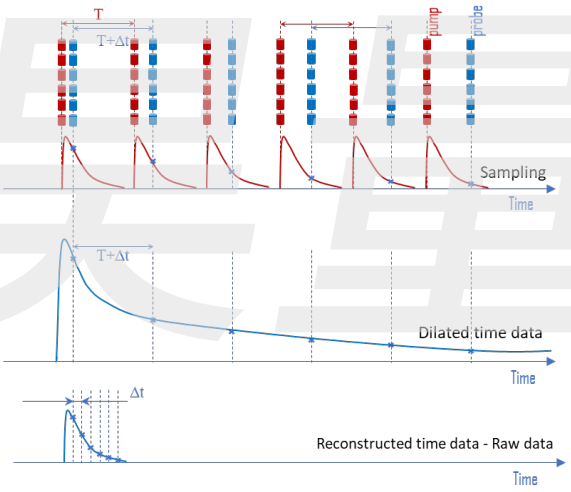


Fig. 3 : Asynchronous pump and probe lasers sampling concept

The measure described above is for one single point. Here with the ASOPS, the measure takes less than a second. It means that by simply scanning point by point all over the surface (Fig. 4), you will get a full map of the studied mechanical parameter in minutes.

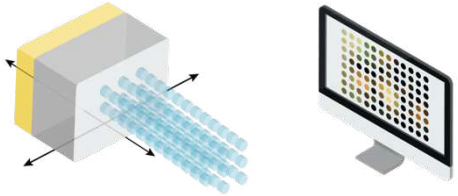
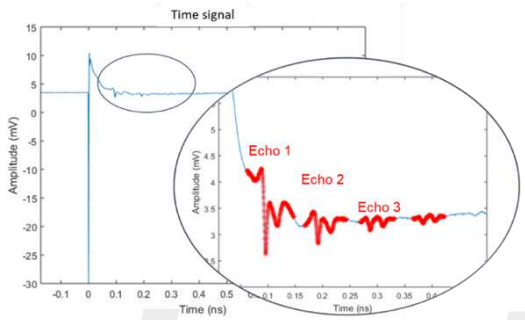


Fig. 4: Mapping of sample's thickness

3. THICKNESS MEASUREMENT

Many destructive measurements exist to evaluate thickness (SEM or calo tester) but time of whole measurement process, sample's shape, angular tolerances may be issues. Hopefully, picosecond ultrasonic may solve a lot of those problems.

For instance, if your interest is in the thickness of a thin film (th), you can easily retrieve an accurate value by measuring the time between two echoes of the ultrasound at the surface of the sample (Fig. 5).



$$th = \frac{\Delta t \times Vl}{2}$$

Where th is the thickness of the thin film,
Δt is time between 2 following echoes,
Vl is the speed of sound into the thin film material,

Different thin films (single or multi layers), from 1 nanometer to 20 nm have been measured with this brand-new picosecond ultrasonic imaging system: Ag, W, Cu, Au, Al, Ti, Rb, TiN, TiO2, SiO2, Si3N4, ITO, Si, P-Si, TiCn-Al2O3, Al2O3…

Repeatability of the measurement is also a key point for non-destructive test, and it have been calculated at 0,1% for a metallic layer of 5 nm.

The industrialization of such an innovative and complex as JAX is giving an easy access to new information. Since a punctual measurement takes a few milliseconds, it is easily feasible to measure all over the surface of the sample and get a full mapping of the thickness.

In the example below (Fig. 7), the sample consists of a 500 µm silicon substrate and 255 nm sputtered tungsten single layer. The scanned surface is approximately 1.6 mm x 1.6 mm and the lateral resolution in X-Y is 50 µm, 1089 points in total. The aim was to map the thickness of the tungsten layer.

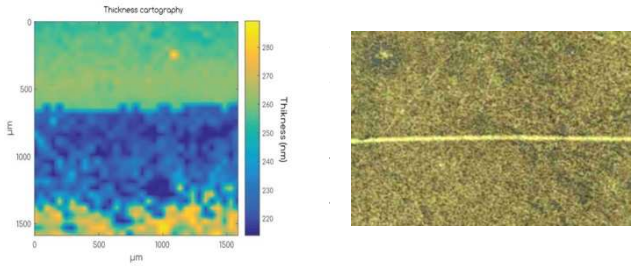


Fig. 7: Example of sample thickness mapping with JAX / optical view of the sample

A large scratch is being highlighted at the surface, but the average thickness remains in the range of 250 nm. The total time of mapping is various, according to the number of measurements, resolution and speed of translation stage.
We just saw that single layer thin film thickness measurement is pretty straight forward. If you are dealing with more than one layer the raw data is much more complex to read. However, it is possible to model the sample and to compare the simulated signal to the actual measure with an incredible fit.

4. MULTIPHYSICS

Picosecond ultrasonic with JAX can lead to a wide range of measurements, which is already a great source of information if we stick to thickness and adhesion - and get even more from the raw data such as thermal information or critical mechanical parameters. In the industry, thickness and adhesion are the main concern at all steps of the manufacturing process, particularly if you work in thin film field. The acoustic picosecond technique is already used in-line for wafer inspection, which shows its maturity and yet confidentiality. But when you chat with several experts of thin films, they will all agree to tell you that:

- Thickness is a key parameter
- Adhesion is always a problem
- Non-destructive measurement is a fine improvement
- Faster is better
- Imaging is awesome

The standard procedures for adhesion characterization are applicable only on flat and large samples, and they are destructive. When it comes to 3D samples or samples you cannot destroy, if you want to check the adhesion on a very small surface, the laser is the only solution. With picosecond ultrasonics and JAX, an easy way to check the relative bonding quality is to check the attenuation of acoustic transmitted layer per layer. Ultrasound at high frequency have a very poor transmission in air and specific acoustic signature so it can lead easily to find out defects in adhesion. Adhesion can now be verified inline all over the sample during every step of the manufacturing process.

Thermal conductivity is the parameter representing the heat conducting capability of a material.

Thin films, superlattices, graphene, and all related materials are of broad technological interest for applications including transistors, memory, optoelectronic devices, MEMS, photovoltaics and more. Thermal performance is a key consideration in many of these applications, motivating efforts to measure the thermal conductivity of these films. The thermal conductivity of thin film materials is usually smaller than that of their bulk counterparts, sometimes dramatically so. Compared to bulk single crystals, many thin films have more impurities which tend to reduce the thermal conductivity. Besides even an atomically perfect thin film is expected to have reduced thermal conductivity due to phonon leakage or related interactions.

Using pulsed lasers is one of the many possibilities to measure the thermal conductivity of a thin material. The time-domain thermo-reflectance (TDTR) is a method by which the thermal properties of a material can be measured. It is even more suitable for thin films materials, which have properties that vary greatly when compared to the same materials in bulk.
The temperature increase due to the laser can be written as follow:

$$\Delta T(z) = (1 - R)\frac{Q}{C(\zeta \mathrm{A})}\exp\left(-\frac{z}{\zeta}\right)$$

Where R is the sample reflectivity,
Q is the optical pulse energy,
C is the specific heat per volume unit,
A is the optical spot area,
ζ is the optical absorption length,
z is the distance into the sample,

The voltage measured by the photodetector is proportional to the variation of R, it is possible then to deduce the thermal conductivity.

In the industry the detection of surface acoustic wave (SAW) is used to detect and characterize cracks. The same principle can be used at nanoscale. When the pump laser hits the surface, the ultrasound generated is actually made of two distinct waves modes, one propagating in the bulk, which is called longitudinal (see Fig. 1), one traveling along the surface, it's called the Rayleigh mode.

The surface wave is very sensitive to the presence and characteristics of the surface coatings, even when they are much thinner than the penetration depth of the wave. Young Modulus can be determined by measuring the velocity of the surface waves.
The propagation velocity of the surface waves, c, in a homogeneous isotropic medium is related to:

- the Young's modulus E,
- the Poisson's ratio ν,
- the density ρ

by the following approximate relation $c = \frac{0.87+1.12\nu}{1+\nu}\sqrt{\frac{E}{2\rho(1+\nu)}}$

When using an industrial ASOPS system to measure and image the SAW, the pump laser is fixed (Fig. 8) and always hitting the same spot. The probe is measuring its signal around the pump laser thanks to a scanner installed in the instrument.

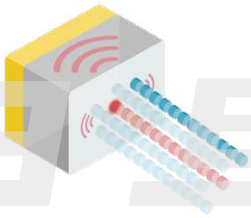


Fig. 8: Surface acoustic wave detection

5. FUTUR CHALLENGES

We had a quick overview of some applications and parameters that can be measured with an industrial picosecond ultrasonic imaging system like JAX. Of course, it was not exhaustive, we could think for instance of adding Brillouin scattering detection in transparent material and more.

Today this technology is moving from the margin to the mainstream. The academic community already recognizes this non-destructive technology as truly operational and able to deliver reliable and accurate measurements. For industrial applications, those systems will most certainly begin to replace standard systems in the short term and to fill the gap of ultrasonic inspection at nanometric scale.

Besides it is easily nestable in the production line while some other instruments are meant to remain research devices because they require much more care, vacuum pumps, complex settings etc.

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