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Surface Acoustic Waves in Thin Films Nanometrology

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Abstract: Thin films nanometrology is an emerging field in nanoscience as the synthesis, processing and applications of nanostructured thin films require an in-depth knowledge of their elastic constants. The elastic energy of a surface acoustic wave propagating in a solid medium, is concentrated at the interface between the solid and air (or a sufficiently rarified medium); consequently, high frequency surface acoustic waves with sub-micrometer wavelengths are an extraordinary tool for a qualitative and quantitative elastic characterization of thin films. In this article, a short review is presented to describe the main ultrasound techniques based on surface acoustic waves for thin films characterization and to highlight the probing limits of acoustic nanometrology.

Keywords: Surface Acoustic Wave, Acoustic Microscope, Photoacoustics, Thin Films

1. Introduction

In the realm of nanotechnology, the realization of thin films and nano-structured thin films systems plays an important role for their industrial applications (e.g. devices integrating electrical and mechanical functionality on the nanoscale, optical coatings, displays, physical and chemical sensors, photovoltaic cells, batteries, etc.). Thin films may also be used for protection of substrate materials against corrosion, oxidation and wear, or to reduce friction or electrical resistance, as well.

The quantitative study of the mechanical properties of thin films is a main issue especially in the microelectronic industry. As elastic properties of a thin film are strongly affected by its thickness, techniques are being developed for thin film characterization [1]. Semiconductor industry, for example, strongly relies on nano-indentation measurements for the mechanical characterization of thin films: while having high lateral resolution, this is a destructive technique that is not reliable if the film is thinner than 500 nm. Moreover measurements are influenced by the elastic parameters of the film substrate and the choice of the substrate is imposed by the material hardness [2].

Surface acoustic waves are dispersive waves offering an important tool for non-destructive thin films metrology because, during propagation in thin films, wave velocity, vibration amplitude and phase are strongly influenced by film thickness and elastic constants.

2. Surface Acoustic Waves

Surface acoustic waves (SAWs), also known as Rayleigh waves, are elastic waves propagating over a plane boundary between semi-infinite solid and a sufficiently rarified medium (air or a liquid, for instance) [3-6]. They consist in a longitudinal and shear displacement coupled together and travelling at the same velocity; the two components are in phase quadrature so that the polarization locus is elliptical; in particular, the displacement vector rotation is counterclockwise (retrograde) at the material surface and clockwise (progressive) beneath the surface [3,4,7,8].

The vibration amplitude of SAWs is highest at the surface of the solid and decay exponentially within the material; at a depth corresponding to few wavelengths, the vibration amplitude decreases to $1/e$ the value it has on the surface of the material. This distance to the material surface is defined as the penetration depth of SAWs: the higher the frequency the more the elastic energy of SAWs is concentrated in a thin layer starting at the sample surface. This makes SAWs propagation very sensitive to surface elastic characteristics and to micro structural gradients close to the surface; consequently SAWs are a powerful nondestructive tool for the characterization of thin films/substrate configuration in which the film to be tested could be much thinner than the penetration depth of the wave.

Whereas for a completely homogeneous sample the wave velocity is constant, Rayleigh waves propagating on a thin film deposited on a substrate are dispersive because wave velocity is function of both the frequency and the layer thickness and the elastic parameters.

There are several methods for SAWs generation, but when experimenting with thin films some physical constrains arise (e.g. reduced lateral dimensions, absence of surface loading, thin films anisotropy, etc.) reducing the choice of applicable methods. Only two of them can be exploited for thin films characterization: acoustic microscope and picosecond photoacoustic.

3. Acoustic Microscope

Acoustic microscopy is basically a pulse-echo technique where SAWs play a central role in both the intensity and the phase of the reflected signal. The heart of the acoustic microscope is the acoustic lens, made of sapphire, Fig.1 [9-12].

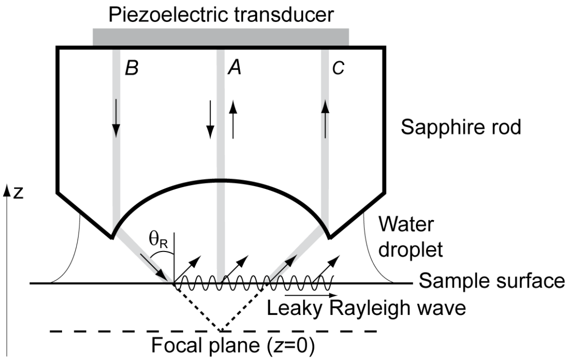

Figure 1. *Sketch of the acoustic lens and of the simplified ray optics model to show the physical origin of the V(z) curve.*

A radio frequency tone burst containing a single radio frequency is applied to the piezoelectric transducer fixed at the top of the lens. The transducer converts the radio frequency signal into an ultrasonic plane wave propagating along the lens axis toward the surface of a spherical cavity that has been carefully ground and polished in the lens body. A coupling liquid is placed between the lens and the sample to transmit the acoustic wave: water is most usually used. Due to the acoustic velocity mismatch between sapphire and water, the plane waves crossing the sapphire-water interface will be refracted into a spherical waves converging onto the focal point of the lens, which is generally placed beneath the sample surface.

Using a simple ray model [13-15] the normal ray A is reflected by the sample surface towards the transducer but, if the aperture of the lens is wide enough, a Rayleigh wave can be excited propagating along the sample surface: that happens for rays, like ray B, incident at a Rayleigh angle $_R$ given by the Snell's law $\sin\theta_R = v_w = v_R$ where v_w is the wave velocity in the water and v_R is the Rayleigh velocity that depends on the Poisson's ratio of the sample material [3.

Really, as the surface of the sample is in contact with the fluids, while propagating, Rayleigh wave leaks energy into the fluid generating longitudinal waves propagating (at Rayleigh angle) toward the lens where, therefore, a ray C, symmetrically placed with respect to the incident ray B, travels back to the transducer.

The electrical signal delivered by the ultrasonic transducer, V, in the time interval when it acts as a receiver through the inverse piezoelectric effect, is due to the interference between the plane wave represented by the ray C, and the acoustic waves directly reflected from the sample surface, represented by the ray A. This results, as can be seen in Fig. 2, in a set of interference fringes observed in the $V(z)$ function.

According to the ray theory, the phase difference between the two waves is a function of the defocus, Δz, (by convention $z = 0$ refers to the focal plane of the lens) and is given as follows:

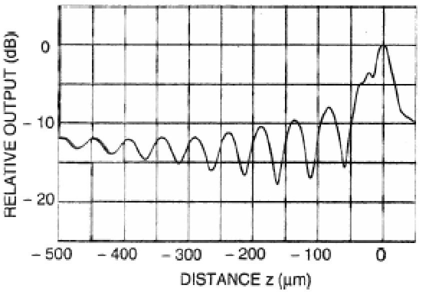

Figure 2. *V(z) curve for a gold film on fused quartz substrate at 190 MHz. (From J. Kushibiki, T. Ishikawa and N. Chubachi, , Appl. Phys. Lett., 57, 1990, pp. 1967-1969. With permission).*

$$\Delta z = 2z\left[k_w\left(1 - \frac{1}{\cos\theta_R}\right)\right] + k_R \tan\theta_R \tag{1}$$

wher k_w and k_R stand for the wavenumber in the water between the lens and the sample and for surface wave, respectively. A phase change of 2π in relative phase difference corresponds to a peak interval in the $V(z)$ function. Using Eq. (1) and Snell's law, it follows that

$$\Delta z = \frac{v_w}{2f(1-\cos\theta_R)} \tag{2}$$

f being the frequency of the elastic wave. Eq.(2) can be rewritten in a the form that explicitly expresses the surface acoustic velocity, v_R, as function of Δz:

$$v_R = \frac{v_w}{\sqrt{1-\left(1-\frac{v_w}{2f\Delta z}\right)^2}} \tag{3}$$

This simple ray optics model allows quantitative measurement of the phase velocity of surface acoustic waves from the $V(z)$ curve, and characterization of acoustic properties of materials. In particular, thickness measurements of thin films can be carried out using dispersion characteristic of surface acoustic waves [16,17]. By measuring $V(z)$ at a certain frequency and finding through the Eq. (3) the corresponding Rayleigh velocity, the layer thickness can be deduced from the theoretical dispersion relation of the SAWs. This technique can be applied with high accuracy without the need of standards [18]. The only drawback is that is difficult to measure the larger layer thickness as multi SAWs modes are excited into the structure; moreover the presence of a coupling liquid between the acoustic lens and the sample surface, in some cases, could change the chemical characteristic of the film.

SAWs velocity and attenuation in thin films can be obtained from the periodic variation and the decay of the *V*(*z*) curve; if mass density is known, elastic constant can be subsequently obtained by using an appropriate technique to best fit theoretical velocity obtained from the experimental measured *V*(*z*) curve. With a spherical acoustic lens, Rayleigh waves are generated by a point-source on the surface of the sample: this experimental configuration is well suited only for the characterization of homogeneous thin films. For anisotropic films, cylindrical lenses have been developed to create a line-source of elastic waves: in this case, Rayleigh waves propagates in a specific direction, normal to the focal line, and it is possible to measure velocities and elastic constants in anisotropic elastic thin films, where the direction of wave energy is not always parallel to *k*-vector [19].

In particular, Achenbach and coworkers have used the Rayleigh waves generated by an acoustic microscope with cylindrical lenses for determining the elastic constants of anisotropic films deposited on anisotropic substrates from the *V*(*z*) measurements [20]. The technique is based on an inversion procedure in which best estimates of the elastic constants are put in a theoretical model of *V*(*z*) to calculate velocities and amplitude of the leaky Rayleigh wave. The values of the elastic constant are then compared with those experimentally measured. The difference is used to adjust the elastic constants and the process is repeated until convergence by least square method is obtained. The presence of stress can also be measured as stress modify sound velocity through third-order elastic constants [21]

4. Picosecond Photoacoustic

SAWs can also be generated through the photoacoustic effect [22]. To generate a stress pulse, the simplest method is to direct a short pulse of light from a power laser at the surface of an optically absorbed material. The light is absorbed within a certain absorption length that depends on the material parameters. The absorbed electromagnetic energy is transformed into heat in the region where the radiation is absorbed and, according to the repetition rate of the pulse, the temperature of this region periodically changes, causing pressure variations that propagate within the sample body and at its surface. If the time duration of pulse is very short, SAWs are generated as the source of acoustic waves is confined at the sample surface. If the laser beam is focused by a cylindrical lens, a line thermoelastic source is produced on the surface of the specimen that generates directional wide-band SAWs [23].

The surface displacement caused by SAWs propagation can be detected in non-contact mode by a laser interferometer at different distances from the laser focus line yielding the amplitude and the phase spectra of the waveforms. From the phase spectra the phase velocity can be calculated for all the frequencies included in the waveforms thus obtaining the dispersion curves of the propagating surface modes. The elastic parameters of the material could be calculated by fitting the dispersion curves deduced from a theory of SAWs dispersion. The technique is very sensible to micro-structural variations near the sample surface and, in case of thin films, it is possible to measure both the mass density and the Young's modulus of the film [24]. Compared to scanning probe microscopy, the technique has a poor lateral resolution due to the dimension of the ultrasound line-source, but it is a completely nondestructive measurement method of some material parameters.

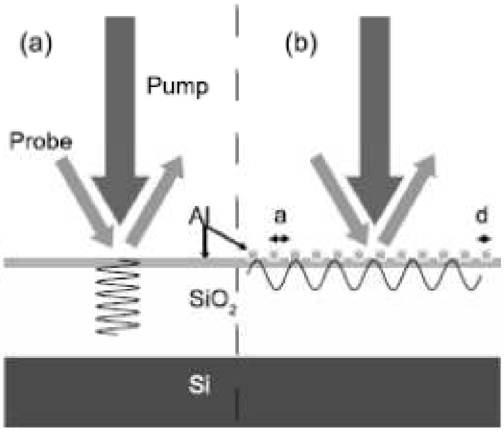

Figure 3. *Schematic diagram of a pump-probe experiment for a 600 nm thick SiO*$_2$ *film deposited on a silicon substrate. (a) off the grating and (b) on the grating (a=400 to 800 nm, h=28 nm, and d=200 nm) (From P. A. Mante, J. F. Robillard and A. Devos, Appl. Phys. Lett., 93, 071909, 2008. With permission).*

Resolution and efficiency of picosecond photoacustics in the generation of SAWs in thin films can be increased using a nanostructured, two dimensional metallic grating deposited in a small region of the thin film. The grating acts as a narrow band interdigital transducer generating, by thermo-acoustic effect, SAWs with a very short wavelength given by the spacing of the grating [25]. The impulsive expansion of the grating due to the absorbed electromagnetic laser light from a power laser (the pump), causes the simultaneous generation of longitudinal wave that travels down into the film and is reflected back from the interface with the substrate. The detection of the shortest wavelength SAWs and longitudinal waves, requires a comparably short wavelength probe. This is obtained with another low power pulsed laser (the probe), which is time delayed with respect to the pump, and is focused onto the sample surface where it is diffracted by the dynamic surface deformation. A classical pump-probe experimental scheme for measuring simultaneously longitudinal and Rayleigh wave velocity, is shown in Fig. 3.

The longitudinal sound velocity v_l and the mass density ρ are related to the Young's modulus E and the Poisson's ratio ν according to [26]

$$v_l = \sqrt{\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}}. \tag{4}$$

This means that the E and ν cannot induced from a measure of v_l and ρ . Anyway, using Victorov's approximation [27]

$$\frac{v_R}{v_t} = \frac{0.718-(\frac{v_t}{v_l})^2}{0.75-(\frac{v_t}{v_l})^2} \tag{5}$$

the velocity of shear wave velocity, v_t, can be deduced from longitudinal and Rayleigh wave velocity. In this way the values of Young's modulus and Poisson's ratio can be obtained because the transverse wave velocity is expressed by the following equation [24]:

$$v_t = \sqrt{\frac{E}{2\rho(1+\nu)}}. \tag{6}$$

The feasibility of such a technique has been experimentally demonstrated, for example, with a complete mechanical characterization of 600 nm thick SIO_2 film [26]. This technique is limited to isotropic thin films and the presence of the deposited nanostructures does introduce a loading on the film, which modify the SAWs velocity somewhat. This effect can be taken into account by adjusting the properties of the modeled thin film until the experimental observed velocity dispersion is recovered yielding a more precise determination of the elastic moduli [29].

Recently, by using coherent extreme ultraviolet light detection, the mechanical properties of thin films with a thickness well under 100 nm, have been measured [30].

5. Conclusions

In conclusion, a short review has been presented describing the two main non-contact ultrasound techniques based on SAWs for thin films characterization: acoustic microscope and picosecond photoacustics. Both the techniques allow the Young's modulus and the Poisson's ratio of isotropic thin films to be measured. Using a cylindrical lens, acoustic microscope can be used also for testing the mechanical properties of anisotropic thin films, although resolution is limited by the maximum usable frequency (≈ 1 GHz at room temperature) due to the strong attenuation of the reflected signal. Picosecond photoacustics using a ultraviolet laser light as a probe beam, can confine the measurements of the elastic moduli in a layer of a thickness < 50 nm, but it requires a nanostructured metal array to be deposited on the thin film, introducing a (small) loading to the films and it can hardly be applied on polymeric thin films.

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