LASER GENERATED ACOUSTIC SHORT PULSE PROPAGATION IN INHOMOGENEOUS THIN FILMS AND MICROSTRUCTURES

Jacqueline Vollmann, Dieter M. Profunser, Andreas H. Meier, Jürg Dual

ETH Zürich, Swiss Federal Institute of Technology, CH 8092 Zürich, Switzerland vollmann@imes.mavt.ethz.ch

Abstract

Diffusion processes occurring at surfaces or interfaces of thin film layers are changing the mechanical properties locally. Such diffusion processes like oxidation or migration of atoms of neighboring materials can cause layers having gradually varying mechanical properties - like density, Young's modulus, or shear modulus - perpendicular to the surface or interface.

The growing miniaturization of MEMS devices enlarges the relative size of these layers and thus enhances the importance of phenomena occurring at such material or phase interfaces thus demanding a detailed quantification of its mechanical properties. In this investigation particular interest is drawn on the question how the propagation characteristics of bulk acoustic waves are affected by diffusion layers. The reflection and transmission behavior of bulk acoustic waves encountering a continuum having a spatially dependent sound velocity is discussed based on numerical simulations as well as on experimental verifications.

In contrast to previous work done in this field in which diffusion effects are generally considered as undesirable phenomena, the deliberate realization of microstructures having well defined gradually varying material properties in one or more dimensions represents a goal of this investigation. For metallic thin film multi layers, thermally induced diffusion processes have shown to be an easy and reliable technique for the realization of layered structures having continuously varying mechanical properties within several 10 nanometers.

Among the experimental methods suitable for the in-depth profiling of submicron metallic thin films, providing resolutions of several nanometers, are short pulse laser acoustic methods, Rutherford Backscattering Spectroscopy (RBS), and Glow Discharge Optical Emission Spectroscopy (GDOES). Short pulse laser acoustic methods and Rutherford Backscattering Spectroscopy (RBS) have the advantage to be nondestructive. The short pulse laser acoustic method is described and RBS measurements are presented for verification purposes.

Finally potential engineering applications like micro-machined spectrum analyzers, acoustic isolation layers, and band pass filters, operating at very high frequencies are presented.

Introduction

When a mechanical stress pulse, which is propagating in an elastic medium, encounters a material or phase interface, which generally represents a change of the acoustic impedance, it is split up into a part which propagates further into the new material and another part which is reflected. The ratio of the transmitted and the reflected stress amplitude is depending on the acoustic impedances of the neighboring materials or phases.

Provided that the acoustic impedance change is realized by a step function of infinitesimal extent, the reflection/transmission ratio is independent of the wave length i.e. the frequency of the incident pulse. However this situation changes as soon as the acoustic impedance change takes place in a layer of finite extent. If the thickness of such a layer - which shall be called 'soft acoustic interface' in the sections below has the same order of magnitude as the wavelength of the propagating stress wave, its reflection/transmission behavior becomes frequency dependent.

This phenomenon can be utilized for a geometrical and mechanical interface characterization as well as for a new type of micro mechanical signal filter or spectrum analyzer.

A numerical example shall illustrate the phenomenon:

A multi layer consisting of a pure gold layer of 5 nm thickness, embedded between two 'soft acoustic interface' layers of 15 nm thickness and two pure aluminum layers, is exposed to a two frequency mechanical strain pulse as shown in figure 1.



Figure 1: 'Two frequency' excitation signal



Figure 2: Ratios of densities and dilatational moduli vs. multilayer thickness.



Figure 3: Displacement (color contrast) vs. time and multilayer thickness.

Here the 'soft acoustic layers' are characterized by a harmonic transition of the mechanical properties, like dilatational stiffness and density, from the aluminum value to the corresponding gold value and vice versa.

The layered structure is excited with a strain signal applied at the surface, which consists of two harmonic components (0.1 THz and 0.8 THz) multiplied by a hanning window. The total longitudinal extent of the one dimensional structure amounts to 151 nm. The thickness of the 'soft acoustic interface layer' amounts to 15 nm. The length scale of the diagrams of figure 2 is normalized with the shortest bulk wave length occurring in the entire structure for the given excitational pulse ($\lambda_{\text{bulk Au min}}$ = 4.016 nm) and the time scale is normalized with the corresponding cycle of the highest frequency ($T_{min} = 1.25$ ps). The dilatational modulus is the stress/strain relation of a linear elastic continuum of infinite extent. The frequencies and the thickness of the 'soft acoustic interface layer' are chosen in a way, that the frequency dependence of the reflection/transmission ratio can be demonstrated as shown in figure 3. The length of a bulk wave in aluminum ($\lambda_{bulk Al max}$) at a frequency of 0.1 THz amounts to 64 nm. Thus, for the lower frequency the ratio of the 'soft acoustic interface layer' and the corresponding wave length $d/\lambda_{bulk Al max} = 0.24$ leads to a strong reflection as visible in figure 3. In the case of the higher frequency, $\lambda_{bulk Al min}$ amounts to 8 nm,

higher frequency, $\lambda_{bulk Al \min}$ amounts to 8 nm, d/ $\lambda_{bulk Al \min}$ = 1.9 and the signal is dominantly transmitted. However, even for the 'long' waves the force reflection coefficient R_f (see Equation 1) is given by the material combination and cannot be exceeded. In the case of an ideal aluminum/gold interface of infinite extent, its value is 0.57, indicating that 57% of an initial stress amplitude propagating in the aluminum layer will be heading back after the reflection at the aluminum/gold interface.

$$R_{f} = \frac{Z_{Au} - Z_{Al}}{Z_{Al} + Z_{Au}}$$
(1)

MICROMECHANICAL REALIZATION OF 'SOFT' ACOUSTIC INTERFACES

 $Z = c_p \rho$

Whereas the numerical example presented in the introduction follows some kind of an 'if we could' approach, the sections below deal with the micro mechanical realization on the one hand side and with quantitative in-depth profiling methods on the other hand. The material combinations and the dimensions of the specimen are chosen in accordance with the parameters for an optimal detectability of a short pulse laser acoustic set up which was developed at the Center of Mechanics, ETH Zürich. The light matter interaction of this set up is optimized for aluminum thin films and therefore at least the surface layer needs to be aluminum. Several methods and techniques have been tested and compared in order to obtain a more or less defined continuous transition of the mechanical properties of one layer to the properties of the neighboring layer within a few 10 nm. Among the methods are an electron beam vapor deposition chamber with two beams and targets which can individually be controlled, an alternating sequence of various layers of different thicknesses, ion beam implantation, and thermally induced diffusion. For the problem outlined above, thermally induced diffusion turned out to be the best solution since this method allowed the realization of series of varying thicknesses by varying the temperature. A standard specimen consisting of a vapor deposited 20 nm Au layer embedded between two 60 nm Al layers on a Al₂O₃ substrate is chosen. A series

of specimen has been exposed to $300^{\circ}C / 200^{\circ}C / 100^{\circ}C$ in a vacuum oven during 30 minutes. In parallel, a series of reference specimen consisting of 140 nm Al films on Al₂O₃ substrates has been exposed to the same temperature cycles in order to exclude potential effects of the grain structure or the optical surface reflectivity on the photo acoustic detection.

IN-DEPTH PROFILING METHODS

The majority of the measurements for the analysis of the acoustic properties of interfaces between neighboring metallic layers presented in this investigation, has been carried out on a short pulse laser acoustic set up. For verification purposes and in order to receive more detailed information about the buried gold layer and its diffusion into the aluminum layers, Rutherford Backscattering Spectroscopy (RBS) measurements have been performed on the series of multilayers which has been exposed to the different temperature cycles.

Short pulse laser acoustics

The laser acoustic method presented below, works in the thermoelastic region which means that there is no ablation and therefore the technique is non destructive. A short laser pulse (800 nm, 70 fs), the pump pulse, is absorbed at the metallic thin film surface and is initiating an elastic pulse which propagates into the film. Echoes, occurring at discontinuities of the acoustic impedances, are heading back to the surface and are causing a slight temporary modification of the optical reflectivity. The optical reflectivity at the surface is scanned versus the relative time shift to the initial pump pulse with a probe pulse, which is created by a partly reflecting mirror and which follows a different path in order to allow the control of the time shift between the excitational and the detecting pulse. The experiment is repeated constantly at a repetition rate of 81 MHz while the time shift is changed. The method was first presented by Thomsen et.al. [1],[2]. A detailed analysis of the light-matter interaction for the excitation as well as for the photo acoustic detection is given by Profunser et.al. [3]. The main challenge lies in the reduction of optical and electronical crosstalk between the excitational and the detecting signal path. Therefore two modulation frequencies, cross polarization, and balanced photo detection is used. A detailed description of the set-up and of the signal processing is given by Vollmann et.al. [4]. The diagram of figure 4 shows two independently measured reflectivity curves compared with a simulation (top). The nominal thicknesses of the multilayer are: 60 nm Al / 20 nm Au / 60 nm Al on a Al_2O_3 substrate.



Figure 4: Two photo acoustic measurements of a 60 nm Al / 20 nm Au / 60 nm Al multilayer on a Al_2O_3 substrate (nominal values) compared with a numerical simulation.

Each curve represents an average of 50 individually measured curves. The parameters of the numerical simulation of figure 4 do not exactly correspond with the nominal values and are determined by Rutherford Backscattering Spectroscopy measurements which are presented in the following section. For lack of a more sophisticated approach, the mechanical properties like the dilatational modulus and the density of the 'diffusion alloy' consisting of 80% Au and 20% Al, are calculated by a linear interpolation of the values of the corresponding components. Two effects are governing the shape of a reflectivity change curve versus the time shift between the pump pulse and the probe pulse, which are presented in figure 4: The dominant effect is the initial jump of the reflectivity change caused by the local heating at the surface. Proportionally to the heat conduction into the surrounding area, the reflectivity change decays nearly exponentially. This is due to the fact, that the heating pump pulse is much shorter than the thermal relaxation time. Superimposed to this thermally caused effect, one can see the periodic alternation of the optical reflectivity change which is caused by the stress pulse echoes reaching the surface. In contrast to the numerical example presented in the introduction (see figure 3), an acoustic pulse, which consists of two frequencies only cannot be realized with the present photo acoustic set up. The strain pulse, created by a pump laser pulse for the parameters used in the photo acoustic set-up, is calculated according to Profunser et.al. [3]. So in order the demonstrate the dependence of the reflection/transmission ratio on the wavelength/'soft acoustic interface thickness' ratio experimentally, the thickness of the 'soft acoustic interface' needs to be varied. This has been realized by the exposure of a series of equally manufactured specimen to different temperature treatments, which leads to different grades of thermally induced diffusion.



Figure 5: Photo acoustic measurements of four 60 nm Al / 20 nm Au / 60 nm Al multi layers on a Al_2O_3 substrate (nominal values) which were exposed to different temperatures in order to 'smoothen' the acoustic interfaces by thermally induced intermetallic diffusion.

Figure 5 shows the photo acoustic measurements of three standard specimen which were heated up to 100° C / 200° C / 300° C, in comparison with the measurement of the untreated specimen previously shown in figure 4. The measured reflectivity change curves of the thermally treated specimen clearly indicate that the broadening of the diffusion zone i.e. the broadening of a the 'soft acoustic interface' suppresses the acoustic contrast. One can also see, that the echo which occurs at the Al/Al₂O₃ substrate interface remains detectable even after the thermal treatment of 300° C.

Rutherford Backscattering Spectroscopy

In order to verify the hypothesis of a thermally induced intermetallic diffusion, Rutherford Backscattering Spectrometry (RBS) measurements are performed at the PSI / ETH Laboratory of Ion Beam Physics, Zürich.



Figure 6: Atom concentration vs. depth calculated from RBS measurements for an untreated and a 300° C treated standard specimen (60 nm Al / 20 nm Au / 60 nm Al multi layers on a Al₂O₃ substrate).

A detailed description of the method is given by W.K. Chu et.al. [5]. Therefore, quantitative depth profiles of the elemental composition of the target can be obtained. The method is nondestructive and a depth resolution of 2 nm can be obtained in the near surface region. Figure 6 shows the Al and Au atom concentrations versus depth of a standard specimen in the untreated case and after a 300°C exposure. It turns out, that even for the untreated specimen an Au concentration of 100% is not achieved in the middle layer. The profiles presented in figure 6 are used for the numerical simulation of the bulk wave propagation shown in figure 4 and correspond very well with the photo acoustic measurements.

CONCLUSIONS, OUTLOOK, AND POTETIAL APPLICATIONS

Frequency dependent reflection/transmission phenomena of bulk waves propagating perpendicular to the surface of metallic thin film multi layers are presented based on an numerical simulation and on photo acoustic measurements. Steps towards a quantitative, nondestructive, photo acoustic determination of intermetallic diffusion effects at a resolution of few nanometers have been demonstrated. The numerically presented hypothesis, that the acoustic interface between neighboring Au/Al layers can be 'smoothed' by thermally induced diffusion processes, has been reinforced by quantitative Rutherford Backscattering Spectroscopy measurements and by photo acoustic experiments. Future directions of the on-going research project are the extension to two dimensional wave propagation phenomena occurring at 'soft acoustic interfaces', the improvement of the resolution of the photo acoustic experiments, and the realization of prototypes of engineering applications like micro mechanical band pass signal filters and spectrum analyzers.

REFERENCES

- C. Thomsen et. al., 'Surface Generation and Detection of Phonons by Picosecond Light Pulses', Physical Review B, Vol. 34, No 6, 1986, pp. 4129-4138.
- [2] C. Thomsen, H.J. Maris, J. Tauc, 'Picosecond Acoustics as a Non-Destructive Tool for the Characterization of very thin Films', Thin Solid Films, Vol. 154, 1987, pp. 217-223.
- [3] D.M. Profunser, J. Vollmann, J. Bryner, J. Dual, 'Measurement and simulation of the laserbased thermo-elastic excitation and propagation of acoustic pulses for thin film and MEMS inspection', SPIE Proceedings, Vol. 4703, 2002.
- [4] J. Vollmann, D.M. Profunser, and J. Dual, 'Sensitivity improvement of a pump-probe set-up for thin film and microstructure metrology', Ultrasonics, Vol. 40/1-8, pp. 757-763, Elsevier Science, Amsterdam, 2002.
- [5] W.K. Chu, J.W. Mayer, and M.A. Nicolet, 'Backscattering Spectrometry', Academic Press, 1978.