Journal of Alloys and Compounds, 211/212 (1994) 438–441
JALCOM 062

Thin film characterization using generalized Lamb waves and harmonic generation

Ph. Richard, G. Gremaud, A. Kulik and O. Behrend

Département de Physique, Institut de Génie Atomique, Ecole Polytechnique Fédérale de Lausanne, CH-1015 Lausanne (Switzerland)

Abstract

A non-destructive method for the local characterization of the mechanical and adhesion properties of a thin film on a semi-infinite substrate is proposed. A numerical inversion procedure based on the simplex algorithm was developed and tested for the dispersion relation of generalized Lamb waves propagating in a thin film on a substrate.

A continuous wave scanning acoustic microscope was used to propagate these surface modes in the specimen and to measure the dispersion curves. This procedure allows the determination of the following parameters of the film: longitudinal wave velocity, transverse wave velocity, density and thickness. Possibilities of this method are studied and tested on several metallic samples.

An application to interface characterization is suggested. By comparison of the position and shape of experimental and calculated dispersion curves for various boundary conditions (delamination, weak and perfect interface) at the interface on the one hand and by observation of the harmonic generation phenomena due to the high non-linearity of weakly bonded interfaces on the other hand, qualitative information on the adhesion of the film on the substrate could be obtained.

1. Introduction

The scanning acoustic microscope (SAM) was first introduced in 1974 [1]. Since then, applications of point-focus-beam acoustic microscopy to quantitative material characterization have been developed extensively through $V(z)$ curve analysis [2, 3]. So far a reliable measure of bond strength has not materialized. Acoustic microscopy studies have shown evidence of complete disbonds [4]. The acoustic microscope allows us to discern the interface differences between a perfect bond and total delamination of the layer. What is not evident is the ability of this method to go further in the local characterization of intermediate bonding conditions. On this particular point some experimental and theoretical demonstrations have indicated the possible utility of surface acoustic waves in the detection of contact defects [5–8]. Generalized Lamb waves appear to be particularly useful in this regard.

The original aspect of our technique is the use of the continuous wave (CW) SAM [9] to excite and measure these surface modes. We have simultaneous access to localized parameters such as adhesion, elastic modulus, density and thickness of the layer through experimental data and numerical inversion of the generalized Lamb wave dispersion equation. The ultrasound is focused to a spot in the micrometre size range, but mapping on a larger scale is also possible. A wide ultrasonic frequency range is required to obtain an accurate measure of the surface wave velocity dispersion relation.

Another important feature for bond quality inspection could be non-linear acoustic microscopy [10]. By working in the transmission mode and measuring the generated second-harmonic acoustic signal (distortion of the waveform) in the focal region, it is possible to observe sharp changes at the boundary between various structures.

2. Theory

2.1. Generalized Lamb wave propagation

In the absence of piezoelectric effects and external forces the wave equation for the displacement in a perfect elastic medium is given by the elastic wave equation

$$\rho_0 \frac{\partial^2 u_j}{\partial t^2} = C_{ijkl} \frac{\partial^2 u_k}{\partial x_i \partial x_l} \qquad (1)$$

where u_j are the displacement amplitudes, ρ_0 is the mass density and C_{ijkl} is the stiffness tensor. At the surface of a uniform half-space a pure Rayleigh (surface) wave propagates with a non-dispersive velocity deter-

0925-8388/94/$07.00
SSDI 0925-8388(93)00062-4

mined only by the elastic constants of the homogeneous solid. This velocity becomes frequency dependent (dispersive) if the half-space is elastically modified by an additional layer for example. For a thin isotropic and homogeneous layer of a material A deposited on a semi-infinite isotropic and homogeneous substrate of a material B the boundary condition that has to be satisfied at the free surface ($x_3=0$) implies a solution of the form

$$u_j=\alpha_j \exp(ikbx_3) \exp[ik(x_1+x_2-vt)] \quad (2)$$

This solution describes generalized Lamb waves [11] localized at the interface $x_3=0$ and propagating along the x_1 axis. To be called surface waves, the quantity b in each term of the solution must be a purely imaginary constant. For this type of surface wave the particle motion under the free surface describes an ellipse lying in the sagittal plane.

2.2. The dispersion relation

The dispersion relation for generalized Lamb waves propagating in a thin layer on a substrate is obtained by solving the elastic wave equation (1) using adequate boundary conditions [11] and a solution of the form (2). This resolution yields

$$\det(M)=0 \quad (3)$$

where M is a 6×6 matrix depending on the characteristics of the layer (transverse and longitudinal wave velocity, density and thickness) and those of the substrate. This condition gives the characteristic dispersion equation of the problem and must be solved by computer calculations.

3. Experimental procedures

The SAM we used in our measurements is built using a commercial wide angle acoustic lens and a network analyser (with harmonic measurement option) that produces and detects the continuous wave ultrasound [9]. The S_{11} parameter (complex ratio between the reflected and the incident wave amplitude) for the transducer–lens–water–sample system is measured. By scanning the spherical lens–sample distance, we obtain the continuous wave $V(z)$ curve. We generate the fast Fourier transform (FFT) of this curve to separate the standing waves in water and the surface acoustic wave (SAW) at the interface between water and sample. From this measurement it is possible to obtain the SAW velocity. The experimental dispersion curves were determined in the frequency range from 120 to 300 MHz in 5 MHz increments.

The film used for the elastic constant determination was a gold layer 0.84 μm thick deposited by sputtering on a fused quartz substrate 1 mm thick. For the adhesion study we used a nickel film 3.2 μm thick deposited by electroplating on an aluminium alloy substrate 5 mm thick, with a very thin copper adhesive layer in between.

4. Results and discussion

4.1. Local elastic constant determination

We perform $V(z)$ measurements as a function of working frequency over the whole bandwidth of the lens and calculate the SAW velocity. For the previously described gold film the first higher mode was excited and measured. Figure 1 shows the measured data of the dispersion curve (●) and the fitted theoretical curve (top curve); the agreement is seen to be excellent.

Using the numerical inversion algorithm with the transverse wave velocity in the layer and the thickness and density of the layer as regression parameters, we obtain from these data the Young modulus E, the Poisson coefficient ν and the thickness h of the gold layer. The determined material constants are summarized in Table 1. The problem of convergence of the regression algorithm is discussed by Behrend *et al.* [12].

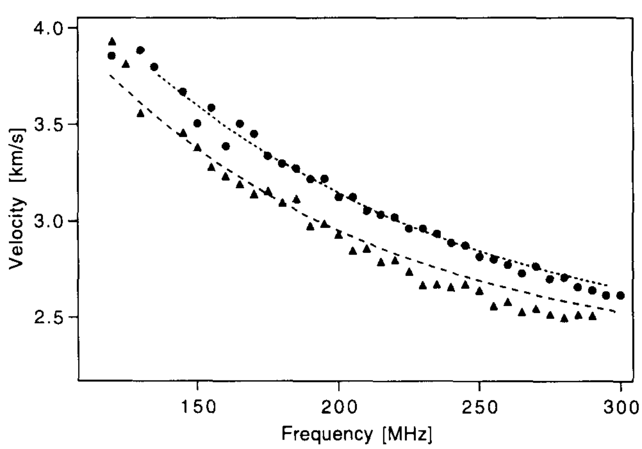

Fig. 1. Experimental (●) and calculated dispersion curves for an Au film 0.84 μm thick on fused quartz. The top curve is fitted with the regression parameters (for elastic constant determination). The second experimental curve (▲) is for the same materials with a very thin Cu intermediate layer, while the corresponding (bottom) curve is the dispersion equation resolved for an Au film 1.05 μm thick.

TABLE 1. Determined elastic constants and thickness of gold film

	E (GPa)	ν	h (μm)
Film	78.88	0.42	0.90 (+6.8%)
Bulk [13]	78.0	0.44	–

Surface values slightly different from the bulk values are used as input values for the adhesion characterization. The layer thickness is generally difficult to obtain with sufficient precision. In this way it is interesting to notice that the thickness is the parameter most modified by the regression (+6.8% of the nominal thickness). This regression technique can also give the local elastic constants of bulk material (substrate) using pure Rayleigh wave propagation.

4.2. Adhesion characterization

If the perfect bonding condition is not met, the SAW velocity is perturbed and the dispersion relation is modified. Knowing this, it is possible to extract useful information on adhesion by simply analysing the correlation between the experimental dispersion curve and the calculated dispersion relation from (3) for a given layer thickness and boundary condition.

Figure 2 presents the experimental data (●) and the calculated theoretical dispersion curve (top curve) corresponding to this case (with the surface values for the layer, its nominal thickness and the bulk values for the substrate). The other two curves are calculated for the weak adhesion case (middle curve) and for total decohesion of the layer (bottom curve). The weak adhesion condition corresponds to discontinuity of the shear stress across the interface. By comparison of the position and shape of the experimental and calculated dispersion curves, using various models for the boundary conditions, we can deduce that this nickel layer presents very good bonding to its substrate. This is mainly due to the substrate preparation and the deposition technique. Besides, we know by a destructive technique that this case corresponds to very good bonding quality.

For bad bonding the experimental and calculated dispersion curves are well known using pure Lamb wave propagation. In this case the layer vibration modes are the same as for a free layer. Nevertheless, the intermediate bonding case (weak adhesion) could not have been emphasized; as far as we know, there are no specimens or thin film deposition processes corresponding to this mathematically defined condition.

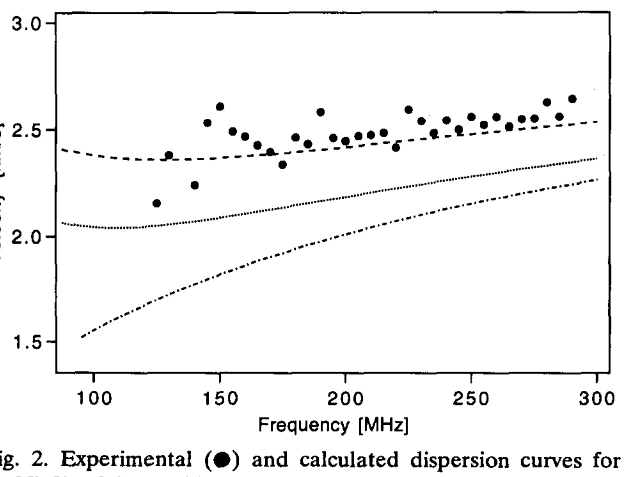

Fig. 2. Experimental (●) and calculated dispersion curves for an Ni film 3.2 μm thick on Al alloy with a Cu (adhesive) layer in between. The top curve is calculated for a perfect interface, the middle curve for a weak interface and the bottom curve for total delamination of the layer.

Finally, if the layer thickness is not well known, it is more difficult to obtain adhesion information (see Figure 1, bottom curve). The differences due to a thicker film, a lower adhesion quality and the influence of the interface layer are still not well established.

5. Conclusions

The metrology mode of the continuous wave scanning acoustic microscopy method was successfully applied to determine the elastic constants and thickness of a gold layer on a fused quartz substrate. The Young modulus was found to be slightly higher than the bulk value for gold, while the Poisson coefficient was found to be somewhat lower than its bulk value.

In addition, the developed method can provide important information on the bond strength between a metallic layer and a substrate. The good adhesion case was emphasized with a nickel layer on aluminium.

Dispersive generalized Lamb waves represent an ideal diagnostic tool for the investigation of interface and adhesion defects in thin film structures, while the harmonic generation capability at the interface can provide direct local adhesion information from the focal region of the acoustic lens.

Acknowledgment

This work was supported by the Priority Program on Materials Research of the Board of the Swiss Federal Institutes of Technology.

References

1 R.A. Lemons and C.F. Quate, *Appl. Phys. Lett., 24* (1974) 163.
2 A. Briggs, *Monographs on the Physics and Chemistry of Materials*, Vol. 47, *Acoustic Microscopy*, Oxford University Press, Oxford, 1st edn., 1992, p. 104.
3 J.G. Morales, R. Rodriguez, J. Durand, H. Ferdj-Allah, Z. Hadjoub, J. Attal and A. Doghmane, *J. Mater. Res., 6* (1991) 2484.
4 G. Gremaud, A. Kulik and S. Sathish, *Europhys. News, 22* (1991) 167.
5 R.D. Weglein and A.K. Mal, *Ultrason. Symp. Proc. IEEE, 2* (1987) 823.
6 J. Kushibiki, T. Ishikawa and N. Chubachi, *Appl. Phys. Lett., 57* (1990) 1967.

7 L. Adler, M.D. Billy and G. Quentin, *J. Appl. Phys., 68* (1990) 6072.
8 P.-C. Xu, A.K. Mal and Y. Bar-Cohen, *Int. J. Eng. Sci., 28* (1990) 331.
9 A. Kulik, G. Gremaud and S. Sathish, in H. Schimizu, N. Chubachi and J. Kushibiki (eds.), *Acoustical Imaging,* Vol. 17, Plenum, New York, 1989, p. 71.
10 R. Kompfner and R.A. Lemons, *Appl. Phys. Lett., 28* (1976) 295.
11 B.A. Auld, *Acoustic Fields and Waves in Solids,* Vol. 2, Wiley, New York, 1st edn., 1973, p. 97.
12 O. Behrend, A. Kulik and G. Gremaud, *Appl. Phys. Lett., 62* (22) (1993) 2787.
13 G.W.C. Kaye and T.H. Laby, *Tables of Physical and Chemical Constants and Mathematical Functions,* 14th edn., Longman, London, 1973, p. 68.