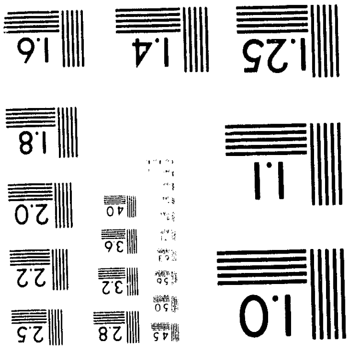
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DOE/ER/13484-7

PROGRESS REPORT NO. 7

for

THIN-FILM CHARACTERIZATION AND FLAW DETECTION

(DE FG02-86ER13484)

prepared by

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1. Project Overview

A. Specific Project Objectives:

The objectives are to determine the elastic constants of thin films deposited on substrates, to measure residual stresses in such films and to detect and characterize defects in thin film substrate configurations.

There are many present and potential applications of configurations consisting of a thin film deposited on a substrate. Thin films that are deposited to improve the hardness and/or the thermal properties of surfaces are of principal interest in this work. Thin film technology does, however, also include high T_c superconductor films, films for magnetic recording, superlattices and films for band-gap engineering and quantum devices. The studies carried out on this project also have relevance to these applications.

Both the film and the substrate are generally anisotropic. A line-focus acoustic microscope, is being used to measure the speed of surface acoustic waves (SAW) in the thin film/substrate system. This microscope has unique advantages for measurements in anisotropic media. Analytical and numerical techniques are employed to extract the desired information on the thin film from the measured SAW data. Recent results include: (1) analytical and numerical techniques for the direct problem and the inverse method, (2) measurements of superlattice film constants, (3) investigation of the effect of surface roughness and (4) measurements of residual stresses in thin films.

Planned future work will be concerned with the detection of flaws, such as delaminations in thin film/substrate configurations.

B. How this project relates to the DOE mission.

Thin films enter in numerous energy producing systems, generally as thin coatings to protect surfaces of components from wear, impact, corrosion and thermal disturbances. It is important that the elastic properties of such films can be determined accurately, that residual stresses can be measured, and that defects such as delaminations can be detected and characterized. The proper functioning of thin films depends on these capabilities. Since the reliability and life extension of energy producing systems is part of the DOE mission, the work on thin films carried out on this project directly fits into that mission.

2. Scientific and Technical Content

A. Schedule of major research activities.

Feb.1/'93 - Jan. 31/'94: Theoretical measurement model of V(z) curve for superlattice thin films, plus related measurements by line-focus acoustic microscopy. Comparison of theoretical and experimental results. Start of measurements of stresses by acoustic microscopy by the use of a stressing stage under the acoustic miscroscope. Preparation of an invited review article to be published in Advances in Acoustic Microscopy (edited by G.A.D. Briggs), MSA.

Feb.1/'94-Jan.31/'95: Theoretical and experimental work for characterization of residual stresses in thin films.

Feb.1/'95-Jan.31/'96: Theoretical and experimental work for characterization of defects and delaminations of thin films.

B. Scientific and technical issues

The scientific and technical issues that are being addressed may be summarized as: (1) the measurement of elastic constants of anisotropic thin films deposited on anisotropic substrates, (2) the measurement of residual stresses in such thin films, and (3) the detection and characterization of flaws and delaminations in anisotropic thin film/substrate systems. The successful resolution of these issues is necessary for fruitful and productive applications of thin-film technology.

C. Experimental and theoretical approach taken

Acoustic microscopy has been used for the characterization of a thin film deposited on an elastic substrate. The technique is based on the measurement of the V(z) curve. The V(z) curve is a record of the transducer voltage output (V) with the variation of the distance (z) between the acoustic lens and the specimen. The SAW velocity can be obtained from the periodic variation of the V(z) curve. The line-focus acoustic microscope allows the measurement of SAW velocity in a single prescribed direction and hence it can be used to measure the near-surface anisotropy of elastic materials. The instrument has been used to measure the anisotropic dependence of SAW velocities on the propagation directions on single crystals, and single-crystal and superlattice thin-films grown on single-crystal substrates.

In our most recent work a theoretical measurement model for the V(z) effect has been developed by following the Fourier optical approach, in which the V(z) curve is considered as a Fourier integral over the product of characteristic functions of the acoustic lens and the reflectance function of the fluid-loaded specimen. When the characteristic functions have been determined, the V(z) curve can be directly related to these functions and to the reflectance function of the specimen through a Fourier-type integral. Accurate characteristic functions are essential for the calculation of V(z) curves by this approach. Attenuation in the coupling fluid, the angular dependence of the transmission by the antireflection coating on the lens surface and the actual focal length must be carefully taken into consideration. The V(z) measurement model has been validated by comparison of its results with measurements.

With known elastic constants and mass density of the substrate and known mass density of the superlattice, three independent elastic constants of a superlattice thin film have been determined from the inversion of the experimental SAW dispersion data. The inversion procedure consists of seeking a set of the constants that minimizes the sum of the squares of the deviations between measured and calculated velocities of SAW's propagating in the films deposited on substrates. The iterative calculation for minimizing the sum is carried out by a systematic function minimization algorithm known as the simplex method.

D. Importance of solving the problem

Thin films to improve the surface properties of components are going to be applied very extensively now that the techniques to deposit such films are being perfected. The capability to predict the performance of thin films requires information on mechanical properties. The work on this project is important because it develops techniques to determine thin film properties.

3. Project Output

A. Major recent accomplishments on the current grant

V(z) curves for a line-focus acoustic microscope have been calculated in terms of the characteristic functions of the acoustic lens and the reflectance function of the fluid-loaded specimen. More accurate expressions for the characteristic functions of the acoustic lens have been developed by taking account of attenuation in the coupling fluid, of the angular dependence of transmission by the antireflection coating on the lens surface, and by making a better estimate of the focal length. The reflectance function has been calculated for anisotropic layers deposited on anisotropic substrates. The calculated V(z) curves have been compared with measurements for isotropic and anisotropic materials, and layered anisotropic materials. For thin film/substrate configurations of known elastic properties, the surface acoustic wave velocities obtained from the theoretical and the measured V(z) curves have been compared for the full range of directions of wave propagation, and excellent agreement has been observed.

The measurement model for the V(z) effect has next been applied together with an inverse method based on the simplex method to obtain unknown elastic constants of thin films from the measured speeds of Sezawa modes in thin-film/substrate systems. Results have been obtained for homogeneous nitride films and transition-metal nitride superlattice films.

B. Bibliography of publications emanating from this project.

B.1 Y.C. Lee, J.D. Achenbach and J.O. Kim, "Acoustic Microscopy Measurements to Correlate Surface Wave Velocity and Surface Roughness," Review of Progress in Quantitative Non-Destructive Evaluation, Vol 12B (edited by D.O. Thompson and D.E. Chimenti), Plenum Press, New York, 1993, pp. 1791-1797.

B.2 Jin O. Kim, Jan D. Achenbach, Paul B. Mirkarimi and Scott A. Barnett, "Acoustic-microscopy measurements of the elastic properties of TiN/(V_xNb_{1-x})N supperlatices," Physical Review B, 48, No. 3, pp. 1726-1737, 1993.

B.3 J.D. Achenbach, J.O. Kim and Y.-C. Lee, "Measurements of the Elastic Constants of Superlattice Films by Line-Focus Acoustic Microscopy," Proc. 11th Symposium on Energy Engineering Sciences, CONF-9305134, Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831

B.4 Yung-Chun Lee, Jin O. Kim and Jan D. Achenbach, "V(z) curves of layered anisotropic materials for the line-focus acoustic microscope," J. Acoust. Soc. Am, 94, No. 2, pp. 923-930, 1993.

B.5 Jin O. Kim and Jan D. Achenbach, "Determination of Elastic Constants of Superlattices Using Line-Focus Acoustic Microscopy," Proc. ASME 93 WAM Symposium on Dynamic Characterization of Advanced Materials, to appear.

B.6 Yung-Chun Lee, Jin O. Kim and Jan D. Achenbach, "Stress Measurement by Line-Focus Acoustic Microscopy," Review of Progress in Quantitative Non-Destructive Evaluation, Vol 13 (edited by D.O. Thompson and D.E. Chimenti), Plenum Press, New York, 1994, to appear.

B.7 Yung-Chun Lee, Jin O. Kim and Jan D. Achenbach, "Measurement of Elastic Constants and Mass Density by Acoustic Microscopy", Proc. 1993 Ultrasonics Symposium, IEEE, in press.

B.8 Y.C. Lee, J.O. Kim and J. D. Achenbach, "Stress Measurements by Acoustic Microscopy," submitted for publication.

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