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Ultrasonic Characterization of the Mechanical Properties of Micro- and Nano-structured Thin Films

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Summary

This paper describes three ultrasonic non-destructive methods that are used to characterize the mechanical properties of micro- and nano-structured thin films. Line-focus Acoustic Microscopy, photoacoustic guided wave method and photoacoustic bulk-wave pump-probe techniques are applied to determine the mechanical properties of Diamond-Like Carbon coatings and Cr/Si thin films, and the results are discussed.

Introduction

With the rapid developments in micro- and nano-scale science and engineering, thin film technology is finding applications in a wide range of fields. Of interest to this work are surface hard coatings, which show the capability of modifying the surface properties of a material to achieve improvements in performance and reliability [1]. In order to obtain the desired properties of the coatings, it is important to understand how the properties of thin films are affected by the coating process. It is essential to accurately determine the mechanical and thermal properties of thin films, by which those structures are made. Mechanical characterization of thin films is important for the proper functioning and reliability of thin film devices with the desired functional properties (e.g. integrated circuits, magnetic discs, or electronic devices). Understanding the mechanical and thermal properties of thin films establishes the foundation for reliable product design and thin film growing techniques. To study the mechanical properties of the thin films, many testing methods have been developed. Among these, nano-indentation, micro-beam bending technique and bulge test are most widely used. These techniques are not nondestructive, and usually cannot be used *in situ.*

At Northwestern University, we use ultrasonic methods to characterize the mechanical properties of thin films. The techniques include (i) contact acoustic microscopy using a line-focus acoustic microscope (LFAM); (ii) photoacoustic (PA) guided-wave acoustic microscopy, and (iii) bulk-wave pump-probe tech-nique. In all these techniques, the velocities of bulk and/or surface acoustic waves are measured and are related to the elastic properties of the thin films. In this paper, we will briefly introduce the basic aspects of the experimental and analytical methods. Applications to several micro- and nano-structured films including ultrahard diamond-like carbon (DLC) coatings and Chromium thin films on silicon are described and discussed.

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Experimental Techniques

Line-focus acoustic microscope (LFAM): LFAM has proved to be a highly accurate method to determine the mechanical constants of isotropic or anisotropic films that are on an elastic substrate [2]. LFAM experiments are generally based on measuring the $V(z)$ curve, which is an output of the measured voltage as a function of the distance between the focus plane of the acoustic transducer and the specimen surface that is noted as z. The measurement and analysis of the $V(z)$ curves provides information about the velocities of the transducer generated leaky surface acoustic waves (LSAW) on the sample surface. A simple interference model by ray theory has been established for the determination of characteristics of the LSAW that propagate along the interface between the coupling water and the solid sample. The LSAW velocity is calculated from the periodicity Δz of the interference signal appearing in the $V(z)$ curves and is given by:

$$v_{LSAW} = v_w \Big/ \sqrt{1 - \left(1 - v_w / 2 f \Delta z\right)^2} \quad (1)$$

where v_w is the acoustic velocity in water, and f is the operating frequency of the transducer. The experimental procedure of LFAM is as follows: first the $V(z)$ curves are obtained by adjusting the distance between the transducer and sample surface; secondly the fringe interval Δz is determined by filtering and fast Fourier transform; thirdly v_{LSAW} is measured at different acoustic frequencies so as to get a dispersion curve of the LSAW. The mechanical properties of the thin films are then evaluated by least-square fitting the measured dispersion curves to an appropriate theoretical model by iterating over the material properties.

Guided-wave Photoacoustic (PA) Method: Similar to the LFAM, the PA laser ultrasonic method can also be used to obtain the dispersion curves of surface acoustic waves (SAW) in thin films. It is a more direct and convenient non-contact way of thin-film characterization and many applications have been introduced [3]. To launch a broadband SAW on the sample, a pulsed high power pump laser is line focused on the surface to generate a SAW pulse with a frequency bandwidth that is mainly determined by the laser pulse duration and the focus spot size. The shorter the pulse is and the smaller the spot size, the higher the frequency of the generated acoustic wave. In our work, a regen amplified femtosecond laser is used as the generation laser. A stabilized balanced Michelson interferometer is used for the optical detection of the acoustic pulses. The broadband ultrasonic wave disperses as it propagates along the surface. By measuring the ultrasonic wave train at two different source to receiver distances, the dispersion curve can be calculated after phase deconvolution of the signals. After fast Fourier transform of the two signals, discontinuities for the phase spectra are corrected by phase unwrapping. The phase velocity spectrum is then given by:

$$v_{SAW}(f) = 2\pi f \Delta L / \Delta\phi(f) \tag{2}$$

where ΔL is the spacing between the two measurement positions and $\Delta\phi(f)$ is the phase difference after phase unwrapping. The mechanical properties of the specimen are characterized by the same curve fitting procedure as for the LFAM. Since a single broadband measurement is adequate to generate the entire dispersion curve, the broadband PA method is a more direct technique for thin film characterization as compared to the LFAM.

Bulk-wave pump-probe technique: Another photoacoustic technique using an ultrashort femtosecond laser pump-probe method has been developed to quantitatively characterize the mechanical and thermal properties of thin films [4]. Basically, the pump-probe technique involves the excitation of the sample by a strong pump pulse and monitoring the subsequent relaxation processes by a weaker probe pulse, which is delayed with respect to the pump pulse by means of a variable optical delay line. Typically, the intensity of the reflected probe beam is monitored. The relative variations of the surface reflection coefficient due to the thermal and acoustic response are typically on the order of 10^{-7} to 10^{-5}. Highly sensitive lock-in detection is therefore applied by setting its reference frequency to the modulation frequency of the pump pulse. In our experiments, the laser pulses with a pulse duration of ~100 fs and a repetition rate of 80 MHz are generated from a Ti:sapphire laser. The laser beam is split into two beams of unequal intensity. The intense pump beam is used as a heating source while the lower power beam is used as the detection beam to monitor the change in reflectivity on the sample surface. The probe beam passes through a gold-coated retroreflector mounted on a micro-positioning motorized stage to vary the optical delay length. As the delay path length of the probe beam increases, a time delay is achieved between the arrivals of the pump and the probe pulses. The reflected probe beam, which contains a snapshot of the transient information of the sample surface, is sent into a balanced photodetector and the weak signal is amplified by the lock-in amplifier. By moving the delay stage, the laser induced transient reflectance change on the sample surface is recorded at various times. The ultrashort laser pulse launches bulk acoustic waves of very high frequency normally into the thin films which then reflect off boundaries. These high frequency acoustic echoes are typically found embedded within a large transient thermal signal. Since non-dispersive bulk acoustic waves are measured, the pump-probe technique provides a direct measure of the elastic properties of the thin film.

Ultrasonic characterization of thin films

Cr-DLC Coatings: The first set of films evaluated in this work is ultrahard diamond-like carbon (DLC) coatings, which have been widely applied as the coating material on shaving blades, cutting tools, and wear-resistance components. The Cr-DLC coatings investigated here were deposited on steel substrates and had a range of properties due to changes in deposition process parameters. The structure of the DLC coatings indicates the presence of nanocrystalline clusters, and includes a multi-layered structure of Cr-DLC, Cr, with interpolated layers between the two.

In order to obtain the elastic properties of Cr-DLC coatings, including the Young's modulus, Poisson's ratio and coating density, the LSAW phase velocity dispersion curves for a set of Cr-DLC coatings were measured by the LFAM. The acoustic frequencies range from 140 MHz to 250 MHz. The measured LSAW phase velocity dispersion curves for various Cr-DLC specimens are shown in Fig.1 (scattered dots), where the specimens are labeled as SP1 to SP7. The specimens were made using different recipes, and clearly have differing elastic properties. In order to derive the elastic properties from the measured dispersion curves, a multi-layer structure is proposed as the theoretical model. Below the top layer of the DLC coating, there are two interfacial layers, one pure Cr layer right on the steel substrate and another interpolated layer between the Cr-DLC coating and Cr layer. The well-developed transfer matrix method is used to calculate the theoretical ultrasonic dispersion curves for multi-layered structures [5]. The simplex algorithm proved to be useful for fitting a function of more than one variable [2]. Thicknesses of the DLC coatings were separately measured and used in the calculation as

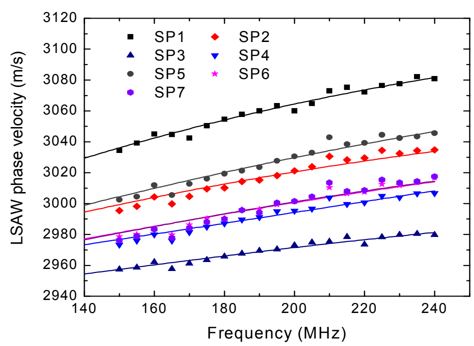

Fig.1 Measured and fitted dispersion curves for Cr-DLC coating samples.

Specimen	Recipe	σ	ρ (kg/m^3)	h (μm)	E (GPa)
SP1	13	0.21	2501±25	4.37	72.5±1.0
SP2	17	0.22	2146±20	3.30	55.0±1.0
SP3	18	0.21	2089±50	2.74	45.0±3.0
SP4	24	0.22	2653±55	1.41	92.0±2.0
SP5	25	0.21	2535±50	3.25	67.8±4.0
SP6	25	0.21	2260±35	3.04	56.2±2.5
SP7	25	0.21	2730±60	3.21	64.6±4.0

Table 1 Fitted parameters for Cr-DLC films.

given. The fitted dispersion curves for Cr-DLC coatings are shown as the corresponding lines in Fig.1 and the calculated mechanical parameters are listed in table 1, in which E, σ, ρ and h are the Young's modulus, Poisson's ratio, density and thickness of the coating respectively.

Cr/Si Thin Films: Next, magnetron-sputtered chromium thin films deposited on the <100> silicon wafers were investigated using broadband PA guided wave and bulk-wave pump-probe techniques. The dotted line in Fig.2(a) shows the measured SAW phase velocity dispersion curve for a 308nm Cr thin film on silicon wafer substrate using the guided-wave PA approach. From the experimental dispersion curve measurements, the Young's modulus for the Cr film is calculated as 234±5 GPa and its Poisson's ratio is 0.21 (using the bulk value for its density). The fitted dispersion curve is shown in Fig.2(a).

Bulk-wave pump-probe experiments were also conducted on the Cr thin films. Fig.2(b) shows the thermo-reflectance variation as a function of time (pump-probe delay). An obvious acoustic echo can be clearly distinguished from the thermal decay. The measured longitudinal wave velocity in Cr is estimated around 6620 m/s. The estimated Young's modulus is about 275 GPa, which is higher than the value derived by guided-wave PA method. This difference may come from the uncertainty in the density of the thin layer and also from possible optical penetration in the thin film, and remains to be resolved.

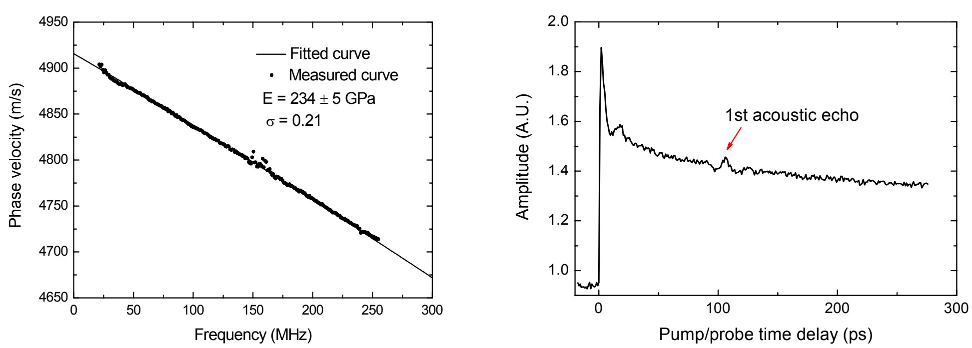

Fig.2. (a) Measured and fitted SAW dispersion curves and (b) transient optical reflectance change signal measured by pump-probe method for the Cr film.

Conclusion

Ultrasonic non-destructive evaluation methods, including LFAM, PA guided wave method and the bulk-wave pump-probe technique, have been used to characterize the mechanical properties of thin films. The photacoustic techniques are non-contact and are particularly attractive for in *situ* applications. Measurements were made on Cr-DLC coatings and Cr/Si thin films, with results that are in good agreement with those in the literature. In ongoing work using these techniques, we are characterizing other material systems including ultrananocrystalline diamond coatings and edge-supported multi-layer thin films without substrates. A comparative assessment of these techniques vis-à-vis nano-indentation results is also underway. Results from these will also be presented at the conference.

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