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Bulk-wave and guided-wave photoacoustic evaluation of the mechanical properties of aluminum/silicon nitride double-layer thin films

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Abstract

The development of devices made of micro- and nano-structured thin film materials has resulted in the need for advanced measurement techniques to characterize their mechanical properties. Photoacoustic techniques, which use pulsed laser irradiation to nondestructively induce very high frequency ultrasound in a test object via rapid thermal expansion, are suitable for nondestructive and non-contact evaluation of thin films. In this paper, we compare two photoacoustic techniques to characterize the mechanical parameters of edge-supported aluminum and silicon nitride double-layer thin films. The elastic properties and residual stresses in such films affect their mechanical performance. In a first set of experiments, a femtosecond transient pump–probe technique is used to investigate the Young's moduli of the aluminum and silicon nitride layers by launching ultra-high frequency bulk acoustic waves in the films. The measured transient signals are compared with simulated transient thermoelastic signals in multi-layer structures, and the elastic moduli are determined. Independent pump–probe tests on silicon substrate-supported region and unsupported region are in good agreement. In a second set of experiments, dispersion curves of the A_0 mode of the Lamb waves that propagate along the unsupported films are measured using a broadband photoacoustic guided-wave method. The residual stresses and flexural rigidities for the same set of double-layer membranes are determined from these dispersion curves. Comparisons of the results obtained by the two photoacoustic techniques are made and discussed.

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Keywords: Photoacoustics; Materials characterization; Thin films

1. Introduction

Thin film structures are important components of many micro-devices and micro-electromechanical systems (MEMS). Thin film and coating materials show unique mechanical and thermal properties, and are finding applications in advanced micro- and nano-technologies [1–4]. In many MEMS applications, thin membranes are critical to the proper performance of micro-devices. Furthermore, thin film properties vary over a wide range depending on fabrication conditions and parameters. For instance, silicon nitride with its low-wear property and high stiffness, is one of the most commonly used materials in the fabrication of MEMS structures such as cantilever tips. However, low-pressure chemical vapor deposition (LPCVD) fabricated silicon nitride films have large differences in stiffness and residual stress compared with those fabricated with plasma enhanced chemical vapor deposition (PECVD) [5,6]. Therefore, reliable and convenient methods to evaluate the mechanical properties of thin films are necessary.

The mechanical properties of thin films can be investigated using tensile tests, bulge tests, nano-indentation tests,

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surface Brillouin scattering, scanning force microscopy, and acoustic microscopy, etc. [7–12]. All these techniques are either destructive or are not suited for *in situ* testing. In contrast, photoacoustic techniques launch very high frequency ultrasound via rapid thermal expansion in materials using ultrafast pulsed laser illumination, and as such they are noncontact and nondestructive. The mechanical properties of thin films can be measured by analyzing acoustic waves that propagate in these structures. The high temporal and spatial resolution of photoacoustic techniques enables *in situ* nondestructive characterization of mechanical properties of ultrathin films.

Two types of photoacoustic methods can be used to investigate thin films: bulk ultrasonic wave and guided-wave technique, respectively. The technique using a femtosecond laser to launch longitudinal acoustic waves with extremely high frequency (higher than gigahertz) using transient photothermal reaction is sometimes called picosecond ultrasonics or femtosecond transient pump–probe spectroscopy [13–15]. This method uses two coherent femtosecond laser beams (drawn from the same laser source) with unequal intensity to measure the transient optical reflectance and/or transmittance change on a picosecond time scale. A strong pump beam is used for photoacoustic generation, and a time-delayed weaker probe beam is used to obtain a series of snapshots of the transient optical reflectance change, typically using lock-in detection schemes. Such femtosecond pump–probe techniques have been widely applied to measure the transient thermal and acoustic phenomena of various types of materials, such as metals, semiconductors, nano-particles and thin films with sub-micron thickness [13–18]. Different models have been developed for femtosecond ultrafast laser heating to theoretically analyze femtosecond transient experiments on solid materials [19–21].

Guided acoustic waves including surface acoustic waves (SAWs) and Lamb waves are also intrinsically related to the mechanical properties of layered materials [22]. The Young's moduli, Poisson's ratios and other physical parameters of the thin film materials strongly affect the dispersive nature of guided waves. Ultrafast laser generated broadband SAWs have been applied to characterize thin films and hard coatings with thicknesses ranging from hundreds of nanometers to tens of microns [23,24]. Photoacoustic guided waves have also been used in the determination of the modulus and residual stress of freestanding thin films using Lamb waves generated by the narrowband transient grating (TG) method [25,26].

In this paper, we present the applications of two photoacoustic methods, i.e., femtosecond pump–probe spectroscopy and photoacoustic guided-wave technique, for characterization of the mechanical parameters of edge-supported aluminum and stoichiometric silicon nitride (Si_3N_4) double-layer thin films [27]. Such edge-supported thin film structures that are only a few hundred nanometers thick and a few hundred microns wide form an integral part of many MEMS devices such as mirror arrays and pressure sensors. In a first set of experiments, the Young's moduli of the thin films are measured using the femtosecond pump–probe technique. Experimental transient photothermal and photoacoustic signals are compared with theoretically simulated waveforms, which are calculated using the photothermoelastic transfer matrix method [28]. Good agreement in predicting acoustic echoes is achieved between simulations and experiments. In a second set of experiments, the broadband photoacoustic guided-wave method is used to measure the phase velocity dispersion curves of the A_0 Lamb wave mode on freestanding double-layer thin films. The residual stresses and flexural rigidities are simultaneously determined. The main purpose of this paper is to investigate the consistency of the measured mechanical properties of thin films using two different but related photoacoustic techniques by comparing the independently measured flexural rigidities of thin membranes. This is essential because thin film properties reported in the literature vary widely and it is important to ensure that these variations are intrinsic to the material and not an artifact of the measurement tool used.

2. Sample fabrication

Thin film specimens were fabricated using standard microfabrication processes. The first step in the fabrication of the double-layer films was to deposit a thin layer of low-stress stoichiometric silicon nitride, Si_3N_4, on the $\langle 100 \rangle$ silicon wafer using LPCVD process at a temperature of 800 °C and a pressure of 205 mTorr. The gases used to deposit Si_3N_4 films were mixtures of ammonia NH_3 and dichlorosilane SiH_2Cl_2 with a ratio of 1:5. The gas flow rate and content ratio were varied to study the influence of fabrication conditions on the material properties of the films. Silicon nitride films with thickness ranging from approximately 200 nm to 400 nm were deposited. After growing the different sets of silicon nitride films, metal depositions were made on the wafers using *E*-beam evaporation system. Thin layers of 100 nm of chromium were evaporated to cover both sides of the wafer. The chromium on the unpolished side of the wafer was used as a metal etch mask during the fabrication of the freestanding silicon nitride membranes. The chromium on the polished side of the wafer was used as protective layer for the silicon nitride during further processing steps.

The second step was to pattern the unpolished side of the wafer. A uniform thin layer of positive photoresist was spun on the wafer. The photoresist was patterned by exposing rectangular patterns to UV light. The photoresist was then developed and the exposed chrome was chemically etched to expose the silicon nitride layer. The chromium then acted as a metal etch mask for the next step when the exposed silicon nitride was dry etched using a reactive ion etching (RIE) system. The silicon nitride was etched until the silicon on the unpolished side was exposed. The exposed silicon was then anisotropically etched using KOH with an etch rate of 1 μm/min. Then the wafer was