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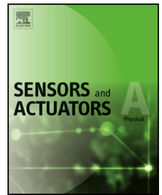
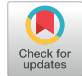


Si micro-cantilever sensor chips for space-resolved stress measurements in physical and plasma-enhanced chemical vapour deposition



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ABSTRACT

The control of extrinsic and intrinsic mechanical stresses in thin films is crucial. Stresses can limit the film performance e.g. by stress-induced delamination or undesired bending of film/substrate combinations; however, stresses can also be used to obtain functionality. Thus, understanding of stress-inducing mechanisms, correlations of stress with film synthesis parameters and controlling the sign and amplitude of stresses in thin films is important and a facile and reliable stress measurement method is necessary. Here, a stress measurement chip is presented which is based on the measurement of the residual overall film stress by a film-substrate combination curvature-based measurement technique. The novel Si-based cantilever sensor chip can measure residual stress in films from a few nanometers thickness up to several microns. Moreover, the sensor chips are applicable for determining the coefficient of thermal expansion, and for examining the film thickness homogeneity over large areas in a deposition system. They can be applied in physical vapor deposition and plasma-enhanced chemical vapor deposition processes with different geometrical and process-related boundary conditions. Exemplary results which were obtained with the sensor chips are discussed to demonstrate their easy applicability, accuracy, versatility, reliability, the thickness dependence of the residual stress and the homogeneity of SiO_x films as well as the residual stress and the thermal expansion values of Al-Cr-N films.

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1. Introduction

Thin films and coatings are used to improve the efficiency, reliability and durability of a wide range of engineering applications by providing unique combinations of chemical, magnetic, electrical, optical, mechanical or tribological properties. Stress and strain can modify these performance-relevant properties [1–3]. In many cases, the influence of stress on the film-substrate combination is unwanted, due to potential film failures through delamination or cracking. In some cases, strain is used to tailor e.g., electronic, optoelectronic or mechanical properties, e.g. compressive stress can enhance the strain tolerance of thin films [4,5]. Stress evolution is related to the microstructure of the growing film, which in turn strongly depends on the deposition parameters [6]. Stress is classified in intrinsic and extrinsic stress. Intrinsic stress arises during film growth and comprises partially superimposing processes, including heteroepitaxy, island coalescence, grain growth, impurity incorporation, defect formation, structural and morphological evolution, phase transition, momentum-transfer effects from impinging energetic particles, etc. A common source for extrinsic stress in thin films is the mismatch of the coefficients of thermal expansion (CTE) of the film and the substrate, which leads to thermal stresses when a temperature difference between processing and application exists.

There are different approaches to determine stress or strain in thin films, e.g. the $\sin^2\psi$-method using x-ray diffraction (XRD) [7,8], reflection high-energy electron diffraction (RHEED) [9] or Raman-spectroscopy [10]. With XRD the lattice strains in crystalline films can be measured and converted into stresses, if the elastic constants of the film are known. It is challenging to determine the

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mechanical stress of very thin films. Especially for amorphous or nanocrystalline films the stress analysis using XRD is not suitable. Residual stress can be determined by measuring the curvature of a film-substrate combination. The advantage of this method is that the residual stress can be calculated without knowledge of the mechanical film properties. If the film is sufficiently thin ($\sim 1\%$ of substrate thickness) only the film thickness, substrate thickness and its Young's modulus need to be known. The superiority of a curvature-based stress measurement technique in comparison to other methods is justified by its robustness, simplicity of operation, long-term measurement stability and sensitivity. This method relies on a mechanical model for the relation between substrate curvature and film stress, proposed by Stoney [11]. This model is sufficiently accurate when meeting several indispensable conditions. The accuracy and limits of Stoney's formula have been discussed comprehensively [12–14]. The relation between stress and substrate curvature is still an active area of research, both for fundamental science of film growth and for the development of MEMS technologies [15–38]. To determine the thermomechanical behavior of thin films the residual stress in a film can be measured on a substrate in dependence of temperature. This is also based on curvature change measurements, induced by a thermal bending of a film/substrate combination [39,40]. With this technique, it is possible to measure stiffness, yield stress and the CTE of a film. Furthermore, it can also provide information on the temperature at which a phase transformation takes place or more generally on any process that changes the dimensions of a coating (e.g. densification [41]).

In contrast to the importance of thin film stresses and related properties, their measurement is not commonly performed and is usually restricted to special experiments. To enable an easy applicable and accurate measurement method for thin film stresses both in scientific and industrial environments, we present a micro-machined Si-based cantilever sensor chip for space-resolved quantitative stress measurements appropriate for applications in physical vapor deposition (PVD) and plasma-enhanced chemical vapor deposition (PECVD) systems with different geometrical and process-related boundary conditions. The stress sensor chip is characterized by universal applicability, high accuracy and resolution, easy handling, good reproducibility and versatility (e.g., as an indicator for film quality assurance in industrial environment). Next to the stress sensor an efficient method (digital holographic microscopy) for the determination of the curvature was adapted [42]. The use of this new methodology was verified in exemplary experiments, which are discussed in this paper. (I) Nanoscale SiO_x films with different thicknesses were deposited on the sensors and the residual stresses were evaluated with respect to resolution and reproducibility. Furthermore, the sensors were applied to investigate the spatial homogeneity of SiO_x films in a PECVD reactor. (II) To demonstrate a further application of the micro-cantilever stress sensors, Al-Cr-N films were sputtered using HPPMS at different substrate bias voltages. The film substrate combinations were thermally cycled and in addition to the residual stresses, the measured CTE values were correlated to the synthesis parameters. Moreover, the accuracy of the sensors was evaluated by comparing the residual stresses of a-C:H films deposited at different bias voltages on Si-sensor chips and single crystalline Si-samples (20 mm × 20 mm × 0.36 mm).

2. Materials and method

2.1. Film stress measurement system

Coated single crystalline Si micro-cantilever beams are used to determine the residual stress σ_f by measuring the change in beam curvature before (R_0) and after deposition (R). The curvature change is directly proportional to the residual stress in the film and is given by a modified Stoney equation [11]:

$$\sigma_f = \left(\frac{Y_s}{1-\nu_s}\right)\frac{t_s^2}{6t_f}\left(\frac{1}{R}-\frac{1}{R_0}\right) \quad (1)$$

Y_s and ν_s are the Young's modulus and Poisson's ratio of the cantilever beam material, t_s and t_f are the thicknesses of the substrate and the film respectively. The equation provides a reasonable accuracy if the film substrate combination meets certain necessary conditions [12–14]. The curvature radius and the topography of the cantilever beams are simultaneously captured by a custom-designed digital holographic microscopy (DHM) set-up (Fig. 1a). The image and data analysis methods can be found in [42]. A diode laser beam (10 mW, 660 nm, Coherent Inc., Santa Clara, CA, USA) is spatially expanded by an objective serving as a beam expander after being attenuated by an intensity filter. The expanded beam is split by a beam splitter (50:50) into two coherent beams, the object beam and the reference beam. The beams are reflected by a mirror and an object (e.g. the stress sensor) respectively, pass the beam splitter again and interfere with each other. The interference pattern (hologram) is captured by a digital CCD camera (PIKE F-421, Allied Vision Technologies GmbH, Stadtroda, Germany) without any additional focusing lens (2048 × 2048 pixels, pixel size $7.4 \times 7.4\,\mu m^2$). To obtain the intensity and phase information of the object beam from the recorded hologram a numerical reconstruction method, based on Fresnel transformation, is used. Fig. 1c shows a reconstructed image of a coated micro-cantilever stress sensor. The reconstructed data from a hologram image contains the intensity as well as the phase information of the light reflected from the sensor chip. The reconstructed intensity shows the 2D picture (Fig. 1c) of the sensor and the unwrapped phase image reveals the surface topography and 3D surface profile of the area of interest (Fig. 1b). As the information derived from DHM depends on the light reflected from the sample surface, a sufficient reflectivity is required. The radius of curvature (ROC) is fitted by using the least squares approach. The resolution (z axes) of the unwrapped topography is on the order of 20 nm. The DHM setup is equipped with a computer-controlled x-y translation stage and a LN_2-cooled heater (T_{max} = 240 °C). The complete imaging process (<5 s) from acquisition to analysis is automated and optimized for different high-throughput measurements based on curvature mapping [42]. The curvature technique is well established and was used in numerous experimental studies to determine the film stress [43–49].

2.2. Fabrication of the micro-cantilever sensor chips

The micro-cantilevers were designed with the aim to allow for robust and accurate stress measurements in different kind of vapor deposition set-ups. Therefore, the chips were designed with a size of 7.4 mm by 6.55 mm. This is small enough to put several sensors at different locations on a wafer or in a substrate holder carousel. Fig. 2c shows the dimensions of the cantilevers. The length of the cantilevers ranged from 0.8 to 3.6 mm. The reason for this design is that with one chip, different orders of magnitude of stress can be measured: if the stress is low a relatively long cantilever is better for obtaining a good measurement of the ROC, if the stress is high shorter cantilevers are advantageous. This is important for a quick evaluation of stress in unknown materials and deposition systems. The distance between the cantilevers is minimized to 10 μm, which allows their use in PECVD processes. The small gap prevents backside coating of the stress sensors. The sensor chips were batch-fabricated using photolithography and Si micromachining technologies. We obtain 96 sensor chips from one four-inch diameter wafer, with 100% yield. Fig. 2a shows the main process steps: First a 100 mm Si wafer (100) is coated on both sides with a thermal

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