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Evaluation of residual stresses in thin films by critical buckling observation of circular microstructures and finite element method

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Abstract

An approach for evaluating residual stresses in thin films by critical buckling observation of circular microstructures is proposed, by which the states of residual stresses can be distinguished directly from the observed critical buckling patterns and their magnitudes can be estimated with finite element method after the critical etching length is measured. For practical operation, three samples were prepared by surface micromachining technique and a specially designed video system was set up for *in-situ* monitoring the whole process during the sacrificial layer etching. Then, measurements of residual stresses were performed and the results were compared with those obtained from micro rotating structures. As a result, the approach is proved to be relatively simple, both compressive and tensile residual stresses with wide range of amplitude can be evaluated by just using a single appropriately designed circular microstructure.

Keywords: Residual stresses; Thin films; Critical buckling observation; Circular microstructures; Finite element method; Sacrificial layer etching

1. Introduction

Thin film materials are attractive for the fabrication of microsensors and microactuators in microelectromechanical systems (MEMS) fields. However, as structure sizes continue to shrink, material properties which were previously acceptable can cause problems [1]. One such concern is the residual stress, generally induced during thin film deposition. High residual stress, no matter compressive or tensile, can adversely affect structure performances, yield and service lifetime. As a result, various methods for evaluating residual stresses in MEMS thin films have been developed in the last two decades [2–3], among which critical buckling observation of clamped-clamped microbeams array for compressive residual stress and microrings array *for tensile*, both proposed by Guckel et al. [4–5], is most widely used. However, for surface micromachining applications, evaluation of residual stresses using these microstructures is usually executed after the sacrificial layer is etched completely and drying process has been finished.

In fact, as the etching progresses, buckling of microstructures can also be observed even still in the etching solution. X. Zhang et al. [6–7] reported a few interesting findings on this subject of polysilicon microbeams during the sacrificial layer etching. In this paper, *in-situ* buckling observation of circular microstructures, often used for studying the release-etch mechanism in surface micromachining [8–9], was carried out during the sacrificial layer etching with a specially designed video system similar to X. Zhang's. Then, an approach for evaluating residual stresses was proposed and practical measurements were performed on three samples prepared by different surface micromachining processes. The results were compared with those obtained from commonly used micro rotating structures [2,10–11].

2. Experimental preparations

Schematic diagram of circular microstructures is shown in Fig. 1, and Fig. 1b is the cross section view in A–A direction. The principal process flows for fabricating these microstructures are illustrated in Fig. 2. Firstly, as shown in Fig. 2a, silicon substrate is coated with a sacrificial layer material and the

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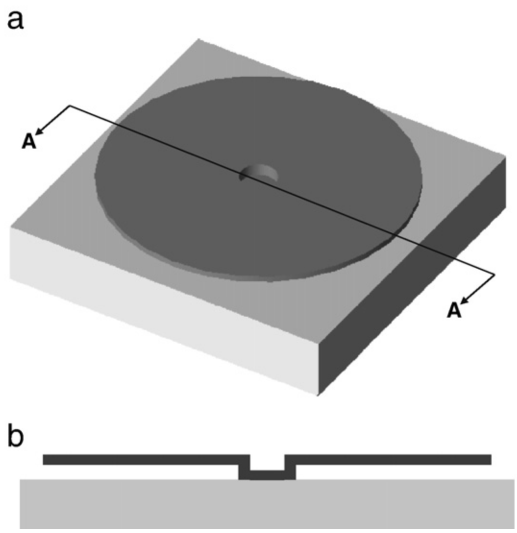

Fig. 1. Schematic diagram of circular microstructures.

anchor areas are formed. Then, the structural layer is deposited and patterned (Fig. 2b). Finally, selective etching of the sacrificial layer creates the free-standing micromechanical structures such as the cantilever beam shown in Fig. 2c.

In the experiments, 4” n-type (100) silicon wafers were used as substrates. And three samples with different states of residual stress in structural layer were prepared, as shown in Table 1. All the sacrificial layer oxides were deposited by the decomposition of tetraethoxysilane (TEOS) in a low pressure chemical vapor deposition (LPCVD) furnace at 685 °C, *33.33 Pa* and O_2 flow rate of 200 sccm. Then, two types of structural layer were also LPCVD deposited, polysilicon for compressive residual stress and silicon nitride (Si_3N_4) for tensile. Polysilicon was deposited at 620 °C, *30 Pa*, and using silane as a source gas of 250 sccm, while Si_3N_4 at 785 °C, *33.33 Pa*, and using ammonia and dichlorosilane as source gases of 150 sccm and 10 sccm respectively. Thick-

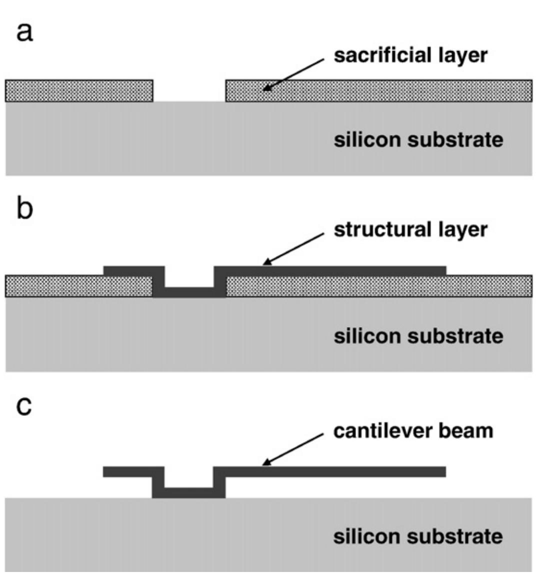

Fig. 2. Principal process flows for fabricating circular microstructures.

Table 1
Three samples prepared by different surface micromachining process

No.	State of stress	Sacrificial layer/thickness [μm]	Structural layer/thickness [nm]
1	Compressive	LPCVD TEOS oxide/~3.00	LPCVD polysilicon/312
2		LPCVD TEOS oxide/~0.75	LPCVD polysilicon/312
3	Tensile	LPCVD TEOS oxide/~0.75	LPCVD Si_3N_4/409

ness of the structural layer imposes great effects on the results. Consequently, accurate measurement of it for each sample is necessary. In our study, scanning electron microscopy (SEM) was adopted for the purpose.

Before etching, an annealing at 950 °C for one hour in a standard diffusion furnace in the nitrogen atmosphere was performed. Wet etching of the sacrificial layer was conducted in a 5:1 buffered hydrofluoric acid solution at room temperature (22 °C). For *in-situ* monitoring the critical buckling patterns of circular microstructures during the etching, a specially designed video system was set up, as shown in Fig. 3. The etching can be stopped at will to freeze the temporal buckling patterns for SEM.

3. Buckling observation and analysis

As the sample is put into the container with fresh etching solution, the sacrificial layer starts to be etched immediately. Fig. 4 presents a simplified model for the etching. R and t represent the radius and thickness of the structure, respectively. And δ denotes the etching length, which increases with the etching time. When etched, peripheral portion of the structure becomes free-standing and residual stress in it is released.

Fig. 5 shows three characteristic evolutions of buckling patterns of a circular microstructure during the etching for polysilicon structural layer. At the beginning, the free-standing circular microstructure has enough stiffness to withstand the released stress loading when δ keeps a relatively small value. Therefore, it still remains flat observed in the microscope (Fig. 5a). As the etching time increases, more part of the structure is freed and its total stiffness gets smaller correspondingly. As a result, for a given residual stress, there exists a critical time when

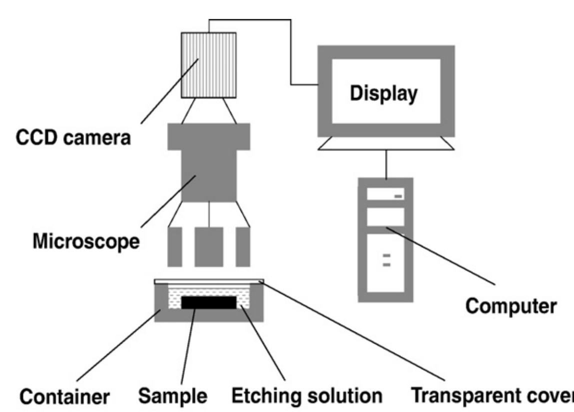

Fig. 3. Experimental device for *in-situ* critical buckling observation.