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Investigations of mechanical properties of microfabricated resonators using atomic force microscopy related techniques

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ABSTRACT

In this article we demonstrate application of atomic force microscopy (AFM) related techniques in characterization of mechanical properties of micromechanical resonators. The investigated structures are a group of doubly clamped beams of 100 nm thick silicon nitride fabricated by low pressure chemical vapor deposition (LPCVD) and lithography with reflective aluminum coating of 10 nm thickness. Width and length of the fabricated and tested structures vary in the range from 3 up to 10 μm and from 20 to 80 μm respectively. In order to determine the structure stiffness force deflection curves were recorded using contact mode (CM) atomic force microscope at the defined resonator position. Moreover the contact resonance (CR) AFM was applied in order to determine the resonance frequencies of the tested microfabricated resonators. Additionally, in order to estimate the stress in the resonator bilayer structure tapping mode (TM) AFM topography investigations were conducted and the recorded topography images analyzed.

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1. Introduction

Microelectromechanical systems (MEMS) and nanoelectromechanical systems (NEMS) provide ample sensing opportunities. Due to small dimension, low stiffness and high operating frequencies excellent attributes and resolutions of mass change and force investigations can be achieved. Many research reports on mass change and force detection in the range of yoktograms (1 yg = 10^{-24} g) [1] and femtonewtons [2] were recently presented. However, it is worth noting, that in today MEMS and NEMS technology not only fabrication of very small devices is complex and difficult but also many procedures connected with metrological (in other words quantitative) investigations of mass change and force are complex and in many cases not solved [3]. In the above mentioned investigations structure stiffness, resonance frequency and quality factor must be precisely determined so that metrological features of the MEMS/NEMS device are known. Only in this way development of appropriate measurement and control environment for the fabricated and being applied NEMS and MEMS structures will be possible. In most solutions the MEMS/NEMS devices are a composition of many thin film structures. In a natural way this makes fabrication of small and thin structures possible but on the other hand, due to the unknown mechanical material properties, causes problems to the device characterization. Additionally, fabrication process influences considerably the final mechanical properties of the developed structures. To the most important fabrication related factors belongs in this case mechanical stress, which is introduced during the deposition and etching of the thin film layers. It vastly modifies the overall structure stiffness, resonance frequency and moreover, its influence is hard to simulate and predict theoretically. In our experiments we investigated doubly clamped silicon nitride/aluminum beams. The low-cost and simple fabrication process made out of these structures useful devices for the mass change and force detection. By variation of the bridge thickness it is possible to fabricate and apply family of MEMS and NEMS devices, which can integrate actuators for electromagnetical or thermal deflection actuation. However, the fabrication process based on silicon nitride deposition and silicon etching introduces stress in the beam structure, which influences the mechanical properties of the silicon nitride/aluminum resonator bridge [4]. Majority of the silicon nitride microresonators are fabricated using low pressure chemical vapor deposition (LPCVD) technology. There are various experimental technologies which were used to characterize the fabricated structures. The most popular are based on application of nanoindenters [5]. More-

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over this technology does not allow to locate the position of investigation with appropriate resolution.

In our opinion the most reliable methods enabling characterizations of mechanical parameters of the MEMS and NEMS resonators are based on local observations of structure shape and resonance frequency measurements. They should usually be performed with nanometer resolution and additionally should enable investigations in the frequency range up to several MHz [6]. From that point of view atomic force microscopy (AFM) based techniques seem to be very attractive in the MEMS and NEMS investigations. They enable to localize the place of test experiments, ensure high detection resolution in wide frequency range. Moreover, due to the observed progress, more and more metrological procedures is available.

In this article we describe AFM related measurements of doubly clamped silicon nitride/aluminum resonators enabling determination of structure stiffness and its resonance frequency. We measured topography of the investigated structure and observed its deflection under defined static load. We recorded also the resonance frequencies of the resonators using reference cantilevers and compared the obtained results with the theoretical ones.

2. Fabrication process

Doubly clamped beam resonators, due to the process and structure simplicity belong the most reliable MEMS and NEMS devices. Bridge structures with thickness of 110 nm, width and length ranging from 3 to 10 μm and from 20 to 80 μm respectively were investigated. The fabrication process started with a (1 0 0) oriented, *n* type, 1–3 Ωcm silicon wafer. After initial cleaning silicon nitride was deposited on the entire silicon wafer. In order to obtain the microbridges with homogenous and well defined thickness silicon nitride layer with thickness *h* of 100 nm were deposited by LPCVD. In this way amorphous material was obtained and this leads to a lower rigidity and stiffness than expected for crystalline silicon nitride. In order to enable structure shape patterning a photosensitive emulsion (Shipley 1805) with 500 nm thickness was deposited (using spin method) on the silicon nitride layer. Next, using direct writing lithography method (the HIMT DWL200 equipment with an argon laser 365 nm) for microbridges shape was defined on the diagonal of the square with a side *a*. Fabrication process of presented structures is a standard MEMS production process and is inexpensive allowing the mass production.

In Fig. 1(a) geometry of the microbridge structure is shown. The microbridge hangs over the etched pyramid, which is formed by the (1 1 1) crystal walls. As a result of developing step the positive photoresist was removed in exposed regions. Then a reactive ion etching (RIE) process was applied to etch the silicon nitride layer in opened windows. KOH anisotropic etching, in which the etch rate of (1 0 0) planes is much higher than the etch rate of (1 1 1) phase, was used to relieve bridge structure. The fabrication process was accomplished with sputtering of 10 nm layer of aluminum ensuring the proper light reflection. This will enable application of optical technologies in detection of the microresonator deflection. The described technology enabled fabrication silicon nitride/aluminum doubly clamped bridges with thickness of 110 nm, width and length ranging from 2 to 10 μm and from 20 to 80 μm respectively (Fig. 1(b)). In comparison to other solutions [7], the described procedure is simple and enables rapid fabrication of the MEMS and NEMS devices.

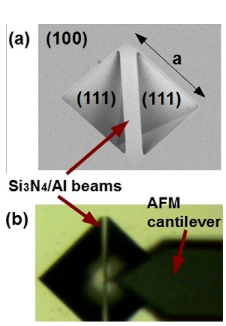

Fig. 1. Silicon nitride/aluminum microresonators: (a) SEM images with selected crystallographic planes; (b) picture from an in-built optical microscope during AFM investigations.

Mechanical parameters of the doubly clamped beams are described by the Euler–Bernoulli beam theory [8]. Indicating the dimensions and the material properties of the structure it is possible to determine the stiffness *k* of the structure and its resonance frequency *f*. The stiffness *k* of a doubly clamped elastic beam with rectangular cross-section, for point loading at the beam's center is expressed as

$$k = 32 \cdot E\frac{w \cdot t^3}{l^3}, \tag{1}$$

where *E* is the elastic modulus, *t* is the thickness, *w* is the width and *l* is the length of the beam. The fundamental resonance frequency *f* of a doubly clamped beam varies linearly with the geometric factor t/l^2 according to the simple relation

$$f = 1.05\sqrt{\frac{E}{\rho}}\frac{t}{l^2}, \tag{2}$$

where ρ is the mass bridge density [9].

Detection of the microresonator resonance frequency proved to be very accurate and reliable method in the mass change and force interaction investigations. However, application of the written above formulas, enabling design of the measurement environment, is quite limited. This results from not only the stress in the silicon nitride structure but also uncertainty of the beam geometry definition (especially uncertainty of the beam thickness, which in most sensitive structures can be in the tens of nanometers range). The mechanical resonator parameters depend also on the silicon nitride Young's modulus, which can vary in very wide range. Additionally, the geometrical structure of the fabricated bridge is much more complicated than the ideal one, which takes into account only the homogenous rectangular beam cross-section. Moreover the unknown mechanical properties of the metallization layer contributes to the effective stiffness in a manner that is hard to predict.

3. Experimental details

In our experiments a commercial atomic force microscope – Veeco Nanoman system with a NanoScope 5 controller was used. Reference silicon probes NANOSENSORS™ AdvancedTEC™ (thickness 4.6 μm, width 45 μm, length 160 μm, nominal value of force constant 45 N/m and resonance frequency 335 kHz) were applied. In contrast to standard AFM cantilevers in the AdvancedTEC™ sensors the probe protrudes outside the spring beam. It this way the microprobe can be placed on the investigated microresonators with precision at the optics integrated with the atomic force microscope (Fig. 1(b)). This specific cantilever was chosen in order to measure the stiffness of the microresonator. In this case, the cantilever spring constant should not be much smaller than the sample spring constant. Unless, by performing force-distance spectroscopy the stiffness of the cantilever and not the sample spring constant is recorded [10]. The stiffness of the cantilever beams used in our experiments was determined by calculation of the thermomechanical noise of the cantilever [11]. The accuracy of determination cantilever spring constants for the thermal noise method is ~15% [12].

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