کارگاه های آموزشی مرکز اطلاعات علمی جهاد دانشگاهی

کارگاه آنلاین بررسی مقایسه ای متنون (مقدماتی)

کارگاه آنلاین بررسی نویسی و بایان نامه نویسی

کارگاه آنلاین آشنایی با پایگاه های اطلاعات علمی بین المللی و ترفند های جستجو

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Geometrical parameters measurement of surface functionalized micromachined micro-cantilever using optical method

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Abstract
Micro-cantilever arrays with different dimensions are fabricated by micromachining technique onto silicon \(<1\ 0\ 0>\) substrate. These sputtered gold-coated micro-cantilevers were later surface-functionalized. Scanning electron microscopy (SEM), atomic force microscopy (AFM), and optical diffraction with laser source are employed to characterize the morphology and image measurement of the micro-cantilever arrays, respectively. The spatial resolution produced in the proposed image measurement method is approaching \(1\ \mu\text{m}\), and the repeatable precision is confirmable in nanometers. Compared with conventional AFM and SEM measurement techniques, the proposed method has demonstrated sufficient flexibility, repeatability, and reliability. The experimental results have been analyzed and presented in this paper for MEMS micro-cantilevers. The scanning white light interferometry-based, two-point high-resolution optical method is presented for characterizing micro-cantilevers and other MEMS microstructures. In this piece of work, we investigate the microstructure fabrication and image measurement of geometrical parameters such as length, width, and step height of micro-cantilevers fabricated using bulk micromachining technique onto silicon \(<100>\) substrate.

Keywords: Image measurement, Silicon (100), Micro-cantilevers, Scanning white light interferometry

Background
Accurate measurement of dimensions of microstructures has recently received much attention because of their potential advantages over conventional atomic force microscopy/scanning electron microscopy (AFM/SEM) techniques [1]. A common method to fabricate the micro-cantilevers is to pattern the deposited continuous film using bulk or surface micromachining technique [1,2]. However, these methods are demonstrated perfectly only for submicron micro-cantilever arrays. As the micro-cantilever size decreases to nanometer, interesting behavior may be expected. In particular, reduced micro-cantilever size results in changes of the domain structure [2] and will affect the characterization of the micro-cantilever. One of the other methods to fabricate the nanometer micro-cantilever array is laser micromachining the deposited material onto a silicon substrate [2,3].

Until now, the conventional image measurement technique for planar microstructural properties of micro-cantilever on silicon \(<100>\) substrates have been studied [4]. However, the applicability of optical methods for microstructure arrays is widely established. In this piece of work, we investigate the microstructure fabrication and image measurement of geometrical parameters such as length, width, and step height of micro-cantilevers fabricated using bulk micromachining technique onto silicon \(<100>\) substrate (Table 1).

Step height measurement is required in many fields including semiconductors, microcircuitry, and printing. Small steps are often measured using a profilometer by calculating the least-squares straight line through the data and then identifying the areas above and below this as being step and substrate. The step height is calculated using least-squares fit to the equation: \(Z = aX + b + h \hat{I}\) where \(a\), \(b\), and \(h\) are unknowns, and \(\hat{I}\) takes the value of +1 in the higher regions and −1 in the lower regions. The unknowns \(a\) and \(b\) represent the slope and intercept of the line. The step height is calculated as twice the
Table 1 Dimensions of micro-cantilever: designed and measured using the proposed method

| Serial number | Micro-cantilever | Dimensions | Design | Measurement after micromachining |
|---------------|------------------|------------|--------|----------------------------------|
| 1             | Length           | 200 μm     |        | 184 ± 0.01 μm                    |
| 2             | Width            | 60 μm      |        | 50 ± 0.01 μm                     |
| 3             | Step height      | 200 nm     |        | 180 ± 0.01 nm                    |

value of the third unknown, \( h \). This approach is fine for samples where the flatness of both the step and substrate are good.

Methods

Two procedures were used to fabricate micro-cantilever arrays. Firstly, silicon \(<100>\) substrates were deposited with silicon dioxide using a thermal oxidation procedure [5]. The oxide deposition rate was about 1.5 nm/min and the gas flow rate of 18 sccm. After the oxidation and patterning, the residual silicon was removed by an anisotropic etchant, tetramethylammoniumhydroxide (TMAH) + deionized water, at 80°C for 45 min [6]. Secondly, micro-cantilever arrays were deposited with chrome-gold layer using radio frequency (rf) magnetron sputtering from a gold target at room temperature. The sputtering chamber was firstly pumped down to 180 mTorr. The deposition of chrome was carried out under Ar atmosphere with about 180 mTorr and the gas flow rate of 18 sccm. During the deposition process, the continuous thin film of gold [4] was also deposited onto a silicon substrate under the same condition for the convenience of measuring the film thickness.

The sputtered chrome-gold layer is known to exhibit affinity with thiophenol molecules [7,8]. Considering this fact, we have dip-coated the piezo-resistive micro-cantilevers with 1 μmol thiophenol in ethanol solution for 3 h and then rinsed with ethanol for 2 min. The surface becomes functionalized.

The surface morphology of the micro-cantilever arrays was investigated by the scanning electron microscopy (JEOL 2000, JEOL Ltd., Tokyo, Japan). The grazing incidence laser diffraction, which avoids the effect of substrate to the pattern, was used to study the image measurement of the microstructure. The mechanical properties at the temperature 300 K were measured by atomic force microscopy.

The image measurement of significant parameters of surface-functionalized micromachined micro-cantilevers such as length, width, and step height were obtained using SEEEBREZ® optical multi-sensing system (Quality Control Solutions Inc., Temecula, CA, USA) with laser probe and Taylor-Hobson’s Form Talysurf® 3-D surface profiler machine (Taylor Hobson Precision Leicester, England) with 3-D Telemap Gold 3-D profile software. The coni-spherical stylus with the base radius of 2 μm was used for the contact-mode measurements.

The coordinate measurements were done with SEEEBREZ® optical multi-sensing system with laser probe. This system has an auto focus facility. After the sample was prepared for measurements, the origin of the wafer coordinates was put relatively. Then maximum z-coordinate was fixed with a 633-nm laser beam marker so that the maximum step that can be measured is \( 633/4 = 158.25 \) nm. The measurement of length and width is simple because the two points placed shall be in-plane. However, for the measurement of step height, since any two points shall not be in-plane, we fix one point (maximum \( Z \)) using the marker, and the other point can be placed where the height need to be measured (minimum \( Z \)). The numerical difference between maximum \( Z \) and minimum \( Z \) of the markers (\( \Delta d \)) shall give the step height. The film step height (\( \Delta d \)) is directly proportional to the wavelength of the laser light and is inversely proportional to twice the refractive index of the film being etched. Thus, the film step height, using this proposed method, is measured with the relation in Equation 1 [9],

\[
\Delta d = \frac{\lambda}{2\eta}
\]

where \( \lambda \) is the wavelength of the laser light and \( \eta \) is refractive index of the etched layer (Au, refractive index = 0.181) [9]. We have measured the length, width, and step height using the proposed method, which is relatively easy and accurate. The \( x, y, \) and \( z \) coordinates of any parts of the structure are measured accurately.

Results and discussions

Figure 1 shows the proposed two-point-high-resolution and accurate optical method for image measurement of the micro-cantilever array. Figure 2 shows the SEM micrograph of the micro-cantilever array, free-standing in a trapezoidal anisotropically etched microcavity with a thickness of 200 nm. The micro-cantilever has a wider parameter distribution, where the mean parameters deviation is approximately 10 nm. Further analysis of SEM micrograph shows that there is some etch-product remnant at the bottom of the trapezoidal etch-pit. However, the sidewalls of the etch-pit are smooth. Figure 3 shows the atomic force microscopy micrograph of the sample. It confirms that the film with micro-cantilevers arrays is free-standing in a trapezoidal microwavity. The close observation of AFM micrograph shows clusters of green Y-shaped tentacles of thiophenol molecule adsorbed on Au surface. Further analysis shows that the
crystallite size distributes is in the range of 5 to 10 nm, calculated with the Scherrer formula [10].

To get the etch-depth information, the micromachined sample was kept on the base of a Taylor-Hobson's Form Talysurf® 3-D surface profiler machine. The coni-spherical stylus was reset to its original position after deciding the frame of $x$-$y$ travel. The machine starts building the etch-profile slowly as the coni-spherical stylus moves over the sample. Three-dimensional profile generated using Taylor-Hobson's Talysurf profiler machine of the sample are shown in Figure 4, with the contact mode using coni-spherical stylus moving parallel to the film plane. The image generated using the stylus-based profilometry is in conformity with the scanning electron microscopy. Figures 5 and 6 depict the length and width measurement with SEEBREZ® machine in high accuracy and repeatable precision. The measurements obtained with the proposed optical method are depicted in Table 1.

A two-dimensional profile to recover the depth information is depicted in Figure 7. Etch profile is very important in assessing the etch-uniformity, sidewall smoothness, and etch-depth. Etch-profile also infers the shape, slope, and etch-depth of the microcavity, in which the micro-cantilevers are free-standing.
the micro-cantilevers are free-standing. It is obvious from the inspection of etch-profile of the trapezoidal microcavity that the sidewalls of this anisotropically etched trapezoidal microcavity are smooth, since its profile is not jagged. However, the bottom of the anisotropically etched microcavity is not smooth, since the profile line is jagged containing hills and dales. The inspection of the etch-profile and scanning electron micrograph confirm that the sidewalls are smooth and at the bottom of the trapezoidal microcavity, there is some etched product reminiscent.

Figure 3 Atomic force microscopy micrograph of surface-functionalized micro-cantilever on silicon<100> surface. The small pointer indicates the out-of-the-plane micromachined micro-cantilever. The wide pointer indicates the trapezoidal microcavity in which the micro-cantilevers are free-standing. The curved arrows indicate the displacements (bending directions) of micro-cantilever due to residual thin-film stress.

Figure 4 3-D surface profiles (silhouettes) of micro-cantilever the x-, y-, and z-axes (microns) are shown by the arrows. The origin is placed relatively.
In summary, micro-cantilever arrays were successfully fabricated on silicon <100> substrate using bulk micro-machining technique and deposited with a chrome-gold layer using the rf magnetron sputtering method. These micro-cantilever arrays were surface-functionalized using $1 \mu$mol thiophenol in ethanol solution for 3 h and after rinsing with ethanol solution for 2 min. The micro-cantilever surface becomes functionalized for mass sensing. The length, width, and step height measurements of micro-cantilever are obtained with the noncontact method. We propose a high-resolution and accurate two-point optical method for noncontact measurement of step height, which is a novel method.

Where surface tension in the inks or paints causes either a rounding or dimpling of the ‘step’ and the stresses caused by the curing process can cause distortion in the substrate, the curvature of the substrate might well be sufficient to prevent the use of the simple least-squares line fit. The step and substrate areas are then treated as line segments, which allow the curvature of the substrate to be removed and result in a straight-line representation of the substrate. The step heights are calculated from this line in the areas adjacent to each step.

The inspection of the etch-profile and scanning electron micrograph confirm that the sidewalls are smooth and at the bottom of the trapezoidal microcavity, there is some etch product reminiscent. With this substantial conclusion, we propose a high-resolution accurate method for the exact measurement of length, width, and etch-depth of the micromachined micro-cantilever.
Further applicability of this method can be extended for the measurement of significant parameters of other out-of-the-plane MEMS structures.

The principal limitation with use of this proposed method is that the minimum probe point separation must be 158.25 nm and, hence, shall limit the measurement of features less than 158.25 nm. The minimum number of points would reduce the signal-to-noise ratio that may be offset by the ability to average several scans. The rapid scans would also reduce the sensitivity of the instrument to certain environmental effects, such as mechanical drift due to temperature and vibration.

Figure 6 Length measurement of micro-cantilever. (After placing the origin relatively and fixing Zmax, we place the laser probes at any two points (probe points number indicated in square) for device/feature measurements (the original images obtained are having low resolution as is obvious and cannot be further improved).

Figure 7 Etch-profile of micro-cantilever.
Competing interests
The authors declare that they have no competing interests.

Authors’ contributions
PRA conceived the study. ASK has carried out the design, fabrication and analysis part of this study. Both authors read and approved the final manuscript.

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Acknowledgements
The authors acknowledge the Microelectronics Group, Nanoelectronics Centre, Department of Electrical Engineering, Department of Chemistry, Department of Physics, Suman Mashruwala Micromachining Laboratory – Department of Mechanical Engineering, Indian Institute of Technology Bombay, Powai, Mumbai and Precision Engineering Division, Bhabha Atomic Research Centre, Trombay, Mumbai, India.

Received: 8 October 2012 Accepted: 8 October 2012
Published: 17 October 2012

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Cite this article as: Kurhekar and Apte: Geometrical parameters measurement of surface functionalized micromachined micro-cantilever using optical method. International Nano Letters 2012 2:25.
کارگاه های آموزشی مرکز اطلاعات علمی جهاد دانشگاهی

کارگاه آنلاین بررسی مقابله ای متن (مقدماتی)

پروپوزال بررسی نویسی و پایان نامه نویسی

کارگاه آنلاین آشنایی با پایگاه های اطلاعات علمی بین المللی و ترفند های جستجو