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Measurement of Poisson's ratio by means of a direct tension test on micron-sized specimens

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ABSTRACT

A methodology developed for measuring Young's modulus and the full stress–strain curve on micron-sized specimens was extended here to measure Poisson's ratio. A dog-bone type specimen was used within a small loading machine with a maximum load of 5 N. The specimen was fabricated from single crystal silicon (SC-Si) with the specimen gage and loading direction in the ⟨0 0 1⟩ orientation. A silicon on insulator (SOI) wafer was used with a deep reactive ion etching (DRIE) based process. Geometrical parameters of the initial cross-sectional area of the gage were measured by means of image processing on environmental scanning electron microscope (ESEM) images. The test setup also consists of an optical microscope with a monochromatic camera and a data acquisition system. The strains were obtained through the displacement field which was determined by means of digital image correlation (DIC). A speckle pattern was placed on the specimen gage. SC-Si was chosen to study since it is expected that on both the micro and macro-scales, Young's modulus and Poisson's ratio will have the same value. Hence, the accuracy of the method may be examined.

The average value of Young's modulus $E = 131.4 \pm 2.1$ GPa was obtained with the micro-specimens and is consistent with values determined on the macro-scale ($E = 130$ GPa). The average value of Poisson's ratio on the micro-scale was found as $\nu = 0.23 \pm 0.03$ which is lower than the macro-scale value of $\nu = 0.28$. The failure stress was determined to be $\sigma_f = 1.46 \pm 0.10$ GPa. Results for Young's modulus reflect the reliability of the methodology which is suitable for characterization of a large variety of materials exploited in micro-devices for both sensing and actuation. The reasons for the low values measured for ν were investigated through emulations of determining the strains. An improvement in the image acquisition system is suggested.

1. Introduction

Microelectrical mechanical systems (MEMS) are increasingly being used as accelerometers, angular rate, pressure and flow sensors, as well as in digital light processing (DLP) systems and optical communications. In order to design these systems, the mechanical properties of the materials used to fabricate them should be determined from micron-sized specimens. It has been observed that for some materials, some properties are scale and process dependent and differ when measured on micron-sized specimens.

A methodology for directly measuring mechanical properties on the micro-scale was presented in [1]. In that study, measurements were made on SC-Si micron-sized dog-bone specimens with the loading direction in the ⟨0 1 1⟩ orientation. Young's modulus E was found to be 171.9 ± 4.2 GPa and the failure stress was $\sigma_f = 1.40 \pm 0.36$ GPa. Young's modulus on the macro-scale in that direction was found indirectly by acoustical wave speed measurements as 169 GPa [2]. In this direction, Poisson's ratio is approximately 0.07. Hence, with the optical equipment used here, it would not be possible to measure Poisson's ratio.

The methods for measuring mechanical properties on the micro-scale can be divided into two groups: indirect and direct. In particular, those investigations carried out to measure Poisson's ratio are focused upon. Resonance tests are one of the indirect methods. These were used to measure Poisson's ratio of silicon oxide [3], SC-Si [4] and tetrahedral amorphous carbon [5]. Disadvantages included possible heating of the specimens by means of the laser used to cause the vibration, sensitivity to boundary

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conditions, dependence on other material properties, such as density, and necessity to conduct the tests in vacuum or account for friction when they are conducted in air. Bending is another indirect method. This method has been used extensively to measure Young's modulus. It has not been used often to measure Poisson's ratio. It was used in [6] to make measurements on SC-Si. Although this is an easy method to implement, the interference curves, used to measure Poisson's ratio, were not symmetric and may lead to inaccurate results. Moreover, this method may be used only for isotropic materials. Several groups exploited nano-indentation to measure Poisson's ratio. For this property measurement, only isotropic material may be tested. A special specimen was proposed in [7] to measure Poisson's ratio of polysilicon. Nano-indentation in combination with measurement of the curvature of a thin film on a substrate were used to obtain Poisson's ratio of silicon oxide, niobium oxide and tantalum oxide [8]. In carrying out these tests, Young's modulus, Poisson's ratio and the coefficient of thermal expansion were found. In [9], the usual indentation method was extended and simulated by finite element calculations to determine both Young's modulus and Poisson's ratio, simultaneously. Tests have yet to be carried out using this approach. Another indirect method exploited a microscopic hole which was introduced within polysilicon [10] to measure both Young's modulus and Poisson's ratio. Use of an AFM caused difficulty in measuring the small displacements encountered.

The bulge test was used by quite a few authors [11,12]. It was implemented mainly to measure Young's modulus and the residual stress. It has also been used to measure Poisson's ratio with more than one membrane geometry. Measurements were made on silicon nitride [11,12], 3C-SiC [13] and a two layer membrane made of silicon nitride and silicon oxide [12]. Vlassak and Nix [11] argued that because of the sensitivity of the measurements, many tests are required in order to obtain accurate results for Poisson's ratio. Accurate measurements were not obtained for ν in [13] and in [12] large scatter was reported. Another indirect method is that of acoustic waves. It was used in [2] to measure mechanical properties of SC-Si and germanium on the macro-scale. This method was used on the micro-scale to measure mechanical properties of gold [14], niobium nitride, vanadium nitride and titanium nitride [15], and silicon oxide [16]. All of the groups made use of lasers in their measurements and depend on the accuracy of measuring the distance and time between signal excitation and pick-up.

Next, the direct methods are described. Although in all cases, a form of dog-bone specimen based on ASTM E 8M [17] was fabricated, the specimens differ from their macro-scale counterparts in order to deal with the handling of a small specimen. With this method, a full stress–strain curve may be obtained, so that Young's modulus, Poisson's ratio, the failure stress and strains are determined. For ductile materials, the yield stress, ultimate stress and strain hardening behavior may be obtained. The difficulty with this method is related to fabrication of a small specimen, accurate measurement of the cross-sectional area of the gage, proper alignment of the gage with respect to the load line, accurate measurement of the small loads and measurement of the strains. This method was used in [18–33]. Materials such as polysilicon, SC-Si, gold, titanium, aluminum, nickel, platinum, nitonol (shape memory alloy), silicon oxide, titanium nitride covered with thin layers of SC-Si, diamond-like carbon on a substrate of SC-Si, and 3C-SiC were tested. In the majority of investigations only Young's modulus was measured. Poisson's ratio was measured in [19,25–27,30,32,33]. It may be noted that some strain measurement techniques require materials with a crystal structure [32], while others require surface topography [24]. For those involving scanning, such as, atomic force microscopy (AFM) and X-ray diffraction (XRD), the test must be interrupted to carry out a scan. In fact, use of an AFM was complicated because it required a very stable temperature.

The aforementioned challenges in measuring Poisson's ratio in micron-sized specimens have led to development of a method using optical microscopy which is employed in this investigation. In Section 2, the methods used to measure Young's modulus, Poisson's ratio, as well as the failure stress and strain are presented. Computer generated images of tensile tests involving small rotations are described in Section 3. In addition, the possibility to improve the accuracy of the transverse strain by magnifying the images was examined. Test results are presented in Section 4. The major aim of this study is to explore the possibility of measuring Poisson's ratio by means of a direct tension specimen and optical microscopy.

2. Methods

A universal methodology to be used for accurate characterization of mechanical properties for a wide variety of materials was presented in [1]. In that study, Young's modulus, the fracture stress and strain, as well as the full stress–strain curve for SC-Si specimens in the ⟨0 1 1⟩ direction were obtained. Fabrication was carried out using photolithography by means of deep reactive ion etching. This material was chosen since it is expected that on both the micro and macro-scales, Young's modulus will have the same value. Hence, the accuracy of the methodology was examined.

The method was extended here to measure Poisson's ratio ν and to examine the feasibility of measuring this parameter with the given test set-up. Poisson's ratio is defined as

$$\nu = -\frac{\epsilon_{yy}}{\epsilon_{xx}} \qquad (1)$$

where ϵ_{xx} and ϵ_{yy} are the in-plane axial and transverse strains, respectively. Since, in accordance with macro-scale measurements, $\nu = 0.07$ when both x and y in Eq. (1) are in the ⟨0 1 1⟩ directions, it was decided to carry out these experiments in the ⟨0 0 1⟩ direction. Young's modulus and Poisson's ratio have been measured by acoustical methods on the macro-scale [34] in the ⟨0 0 1⟩ direction as $E = 130$ GPa and $\nu = 0.28$.

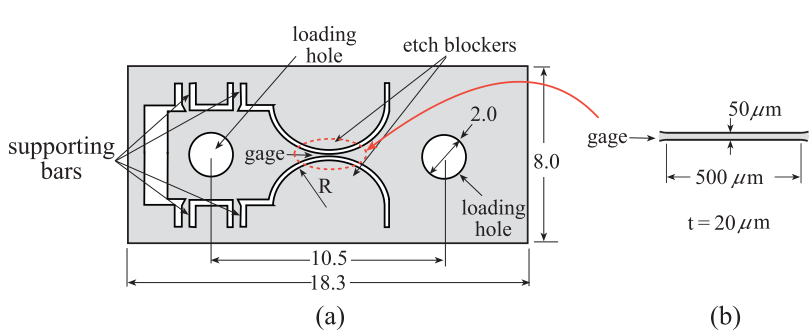

Fig. 1. (a) Dog-bone specimen and frame; dimensions in millimeters. (b) Gage; dimensions in microns (t = thickness).