Sensors and Actuators A 278 (2018) 48–59

Contents lists available at ScienceDirect

Sensors and Actuators A: Physical

journal homepage: www.elsevier.com/locate/sna

Biaxial flexure testing of free-standing thin film membrane with nanoindentation system

Takashi Ozaki*, Tomoyuki Koga, Norio Fujitsuka, Hiroaki Makino, Hiroshi Hohjo, Hiroaki Kadoura

Toyota Central R&D Labs., Inc., 41-1, Yokomichi, Nagakute, Aichi, Japan

ARTICLE INFO

Article history:
Received 7 December 2017
Received in revised form 18 May 2018
Accepted 19 May 2018
Available online 19 May 2018

Keywords:
Biaxial flexure test
Flexural strength
Nanoindentation
Polysilicon
Finite element analysis

ABSTRACT

The advent of microelectromechanical devices has increased the demand for biaxial flexure testing at the micro- and nanoscale. However, testing at these scales is challenging owing to difficulties in manipulating very small samples and applying highly symmetric biaxial loads to them. In this study, we developed a facile technique for on-chip biaxial flexure testing. The principle of the technique was inspired by ball-on-ring biaxial testing commonly employed for macroscopic flexure analysis of ceramics. In our technique, the specimen is tested as a circular membrane fixed at its edges to a substrate. The center of the membrane is pushed from above by a rounded conical nanoindenter (NI) until the membrane fractures. Since this method does not require microscale specimen manipulation involving the securing of microscale components and/or samples by external fixtures, the test procedure is relatively easy to perform. In addition, the load and displacement curves obtained using the NI are high resolution, enabling precise strength evaluation and application to very weak structures such as nanoscale membranes. To demonstrate the test system, we designed and fabricated polysilicon membranes with diameters of 20 μm and thicknesses of 80 nm. The most likely source of error in the system is misalignment between the indenter and the membrane center. Accordingly, its effect was numerically analyzed. The results showed that a misalignment of less than 3.0 μm causes a 0.32% error in the first principal stress. Since the NI tester can easily attain this alignment accuracy, highly accurate stress estimates can be achieved. The measured fracture strengths of the polysilicon membranes were fitted to the Weibull distribution, revealing an average strength of 8.11 ± 0.31 GPa and a shape parameter of 13.9 ± 5.4, both of which agree well with the results from previous research. The fracture origins were observed near the center of the membranes. These results confirm the viability of our concept.

1. Introduction

The need to evaluate the mechanical properties of materials at the micro- and nanoscale has increased because of the growing market for microelectromechanical system (MEMS) devices and the emerging nanoscale devices. In particular, evaluating fracture strength and estimating product lifetime are very important for product design. However, this is challenging because evaluating these properties requires same-scale testing. Specifically, fracture phenomena are affected by scale [1–7] and fabrication process [8–11]. The scale-effect has been known since the 1990 s. For example, Tsuchiya et al. [1], Sharpe et al. [2], and Namazu et al. [3,4] reported that fracture strength increases with decreasing specimen length or width, and Jadaan et al. [5] investigated the relationship between specimen strength and surface area. Furthermore, Reedy et al. reported that fracture strength follows Weibull's theory based on the side-wall surface area of the specimen [6]. Recently, DelRio et al. reported that near-theoretical fracture strength (≈22 GPa) was observed in a silicon nanowire with a diameter of ≈30 nm [7]. Fabrication processes and surface treatment effects have also been investigated and reported since the 1990 s, specifically since early studies on the effect of surface roughness [8,9] and polysilicon grain size [10]. In more recent research, it has been discovered that atomic-scale flaws in native oxides can decrease their fracture strengths [7,11].

Materials testing at the micro- and nanoscale has been an active area of research since the 1980 s. Simple tensile test methods with specimen manipulation using external grippers that utilize mechanical fixtures [2,6,12–14] or electrostatic forces [1,15] have been proposed and widely employed. Many other different methods have also been proposed, including bending tests [3,4,11], on-chip tensile-testing mechanisms [16], thermally actu-

* Corresponding author.
E-mail address: ozaki@mosk.tytlabs.co.jp (T. Ozaki).

https://doi.org/10.1016/j.sna.2018.05.034
0924-4247/

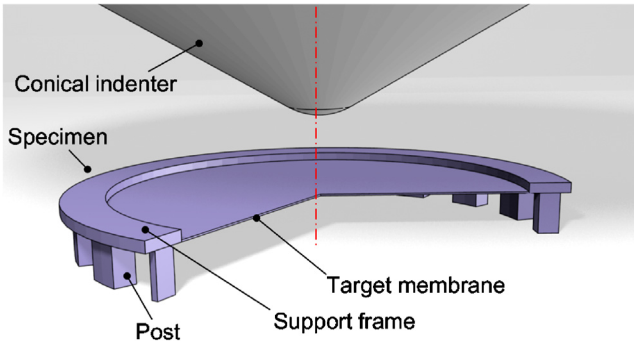

Fig. 1. Specimen structure and NI tip. The specimen is cut away to show its cross section.

ated testers [17], tensile tests driven by internal stress in the specimen [18], tensile testing by Θ-shape-compliant mechanisms [19], cyclic loading by external shakers [20], and electrostatic comb-drive actuators [21]. Most of these previous studies involve uniaxial tests in which a uniaxial tensile or bending mode is utilized for loading stress. However, uniaxial strength alone is insufficient for predicting fractures in complex stress fields; both the uniaxial and biaxial strengths are required. Bulge testing is a common and well-established method for applying a biaxial load, and it is suitable for evaluating the biaxial elastic moduli of thin rectangular [22,23] and circular [24] membranes. Although it can also measure fracture strengths, the values obtained do not represent solely biaxial fracture strengths in most cases because fractures are often observed near the membrane edges where the stress field is nearly uniaxial. It is possible to find cases of biaxial tensile fracture at the membrane center from cases of edge failure, but the selection can affect the estimation of the fracture probability parameters and decrease the throughput of the test. Another method to measure biaxial strength is plane biaxial tensile testing, which is based on cross-shaped specimens stretched in two directions [25]. An attractive feature of this method is that the tensile forces can be varied independently. However, a disadvantage of this method is the increased complexity of specimen handling compared to those of uniaxial and bulge testing. Thus, an easy-operation and reproducible method for microscale biaxial testing with a low risk of fractures at unintended positions is demanded.

Accordingly, we herein propose a new technique for on-chip testing of biaxial flexural strength. The concept was inspired by the ball-on-ring biaxial test commonly employed in macroscopic flexure testing of ceramics [26,27]. In our technique, the specimen is tested as a circular membrane fixed to a substrate along its circumference. The center of the membrane is pushed by a conical nanoindenter (NI) until the membrane fractures on the reverse side of the pushing point owing to the induced biaxial tensile stress. Since the testing system is axis-symmetric, an ideal biaxial tensile stress can be induced at the center of the reverse side of a membrane. This test is performed with an on-chip system, and it eliminates the need to grip and manipulate microscale specimens, allowing a high degree of simplicity and reproducibility. Furthermore, the fracture is most likely to occur at the center owing to higher concentrated stress at the indented point. Another feature of this technique is that there are no corners, edges, or fixtures near the fracture point, which means that the method is less affected by patterning processes, such as photolithography and etching. In addition, the load and displacement results obtained by the NI are of high resolution, enabling precise strength evaluation and application to very weak next-generation structures, i.e., nanoscale membranes.

In this paper, we describe the design and characteristics of the test, as derived using finite element analysis (FEA). We particularly focus on the effect of misalignment between the conical indenter and the specimen, which is the most probable geometrical error source in this test. To demonstrate the technique, we performed experimental testing on polysilicon specimens. The procedure and results of these tests are then discussed. Since the experimental results obtained are compatible with the FEA and published strengths, we confirm the validity of the technique.

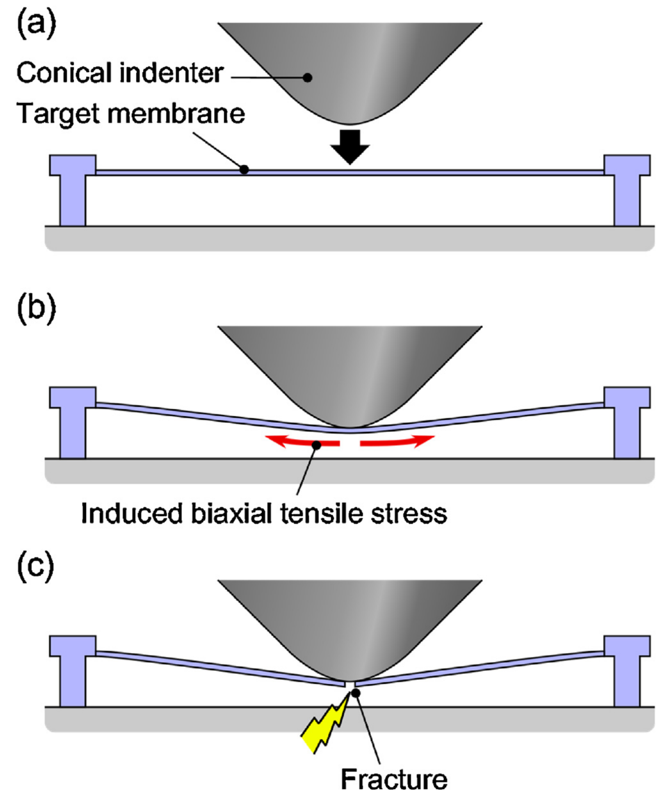

Fig. 2. Procedure for biaxial flexure testing using NI. (a) The conical indenter is aligned and moved to the center of the target membrane; (b) biaxial tensile stress is induced by pushing the indenter into the membrane; and (c) fracture occurs when the stress exceeds the biaxial strength of the membrane.

2. Design

2.1. Specimen structure and test method

Fig. 1 is a schematic diagram of the testing system. A circular membrane is surrounded and supported by a rigid frame, and they are fixed to a substrate via posts. The testing system employs a conical diamond indenter with rounded tip to avoid sharp stress concentration. Fig. 2 depicts the test procedure. First, the conical indenter and the center of the target membrane are aligned using the optical microscopy (OM) and scanning probe microscopy (SPM) modes of the NI for coarse and fine positioning, respectively. After alignment, the NI tip is moved into contact with the membrane surface. When the conical indenter is pushed into the membrane, a biaxial tensile stress is induced on the bottom surface of the membrane. When the stress exceeds the biaxial strength of the membrane it fractures.

To demonstrate this concept we fabricated and tested polysilicon specimens with a thicknesses and diameters of 80 nm and 20 μm, respectively. Although we used polysilicon, the method is applicable to most materials compatible with surface-micromachining processes.