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Full Length Article

Effect of substrate bias voltage on tensile properties of single crystal silicon microstructure fully coated with plasma CVD diamond-like carbon film

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ABSTRACT

Tensile strength and strength distribution in a microstructure of single crystal silicon (SCS) were improved significantly by coating the surface with a diamond-like carbon (DLC) film. To explore the influence of coating parameters and the mechanism of film fracture, SCS microstructure surfaces ($120 \times 4 \times 5$ μm^3) were fully coated by plasma enhanced chemical vapor deposition (PECVD) of a DLC at five different bias voltages. After the depositions, Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), thermal desorption spectrometry (TDS), surface profilometry, atomic force microscope (AFM) measurement, and nanoindentation methods were used to study the chemical and mechanical properties of the deposited DLC films. Tensile test indicated that the average strength of coated samples was 13.2–29.6% higher than that of the SCS sample, and samples fabricated with a −400 V bias voltage were strongest. The fracture toughness of the DLC film was the dominant factor in the observed tensile strength. Deviations in strength were reduced with increasingly negative bias voltage. The effect of residual stress on the tensile properties is discussed in detail.

1. Introduction

Diamond-like carbon (DLC) is a kind of amorphous film with outstanding mechanical properties, such as high elastic modulus, hardness, fracture toughness, and reliable tribological properties [1]. Its good chemical stability and biocompatibility [2] make DLC films attractive in various coating applications [3]. Furthermore, due to reliable adhesion on silicon surfaces [4], the use of DLC films is promising in micro electro-mechanical systems (MEMS) fabricated as silicon microstructures as the coatings enhance mechanical reliability and improve wear resistance. However, exploitation of these outstanding features, especially for the optimum design of DLC coated structures depends on understanding the mechanical properties of DLC-coated silicon microstructures, including their tensile strength and fracture mechanism.

On the other hand, due to the brittleness of their constituent material, silicon microstructures can undergo catastrophic fracture, due to stress concentrations at unavoidable surface microdefects [5]. Thus, various surface modifications, including laser treatment, KOH etching, hydrogen treatment [6,7] and coating methods aiming to minimize fractures resulting from such defects have been examined, to enhance the strength of silicon microstructures. Still, due to the difficulties in making specimens for mechanical tests given the high residual stress inherent in DLC films, only a few studies have focused on the characterization of DLC coated silicon microstructure. Isono et al. [8] deposited DLC film on the top surface of silicon microstructures and found a 25–50% drop in strength after DLC application. The authors concluded that residual stress was responsible for the observed negative effects, manifested as a fracture mechanism driven by film-substrate spalling and sample deformation. In our previous research [9], we developed a full coating process for free-standing silicon microstructures, employed an electrostatic gripping system for tensile tests and observed significant improvements in tensile strength after DLC applications. However, correlations between coating parameter and improvement in tensile strength and strength deviation remained unclear.

In the present work, to examine the effects of substrate bias voltage on tensile strength and reliability enhancement, single crystal silicon (SCS) microstructures were fully coated with DLC film via a plasma enhanced chemical vapor deposition (PECVD) technique. Five deposition bias voltages, ranging from −200 V to −600 V, were adopted to explore the influence of bias voltage on the mechanical properties of deposited DLC films. Tensile tests were conducted with a quasi-static thin film tensile tester with

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an electrostatic gripping system. The measured tensile properties of the microstructures were correlated with the chemical composition and mechanical properties of the DLC films.

2. Experiments

2.1. Microstructure fabrication

The SCS microstructures were fabricated from a single piece of silicon on insulator (SOI) wafer, surface orientation (100), by a standard MEMS manufacturing technique (Fig. 1(a)). First, photoresist (Tokyo Ohka Kogyo THMR-iP1800) was applied to the surface of the device layer and the microstructure pattern was formed with a stepper (Nikon NSR2205i11D) to achieve smooth sidewalls. The device layer was then etched by a Bosch process using inductively coupled plasma reactive ion etching (ICP-RIE, Samco RIE-iPB800) at an etching rate of 118 nm/cycle. Next, a different positive photoresist (Tokyo Ohka Kogyo OFPR-800LB) was applied on the backside and patterned using a double-sided mask aligner (Union Optics PEM-800). The ICP-RIE was used to open windows on the handle layer at an etching rate of 3.5 μm/cycle. The resist layers on both surfaces were removed using an oxygen plasma assisted ashing process. Buried oxide layers were etched using buffered hydrofluoric acid and the structures were released from the handle wafer.

DLC film was deposited on the released SCS microstructures with a PECVD machine (Shinkoseiki ACV-1060) whose sample stage rotates around the center of the plasma while spinning axially [9]. Before placing the microstructures in a vacuum chamber, they were cleaned in a concentrated sulfuric acid and hydrogen peroxide bath (piranha solution) for ten minutes and dried in air. After the vacuum chamber was evacuated to a base pressure of 8×10^{-4} Pa, argon gas was introduced and the chips were bombarded for 1 min at a −400 V DC bias. To improve the adhesion strength between the SCS microstructure and DLC film, a 20 nm SiC interlayer was formed prior to DLC film deposition. A mixture of acetylene (150 SCCM) and hydrogen (10 SCCM) was used as a DLC deposition precursor at a working pressure was set to 0.04 Pa. Bias voltages from −200 V to −600 V for every −100 V were used with deposition times maintained at 90 s, for a target film thickness of 150 nm.

A DLC coated microstructure is shown in Fig. 1(b). The size of the gauge part was designed to be 120 μm long, 4 μm wide and 5 μm high. The gauge part provided a connection between the substrate and a paddle that was gripped by electrostatic force during tensile tests [10]. Four beams were designed to support the paddle during fabrication, and were later severed with a laser cutter (Hoya HL-10) prior to tensile test.

2.2. Characterization of DLC films

The deposition bias voltage influenced the chemical composition (relative amounts of sp^2 carbon, sp^3 carbon and hydrogen) of the DLC films and thus affected a number of mechanical properties. The chemical composition was qualitatively investigated by Raman spectroscopy (Horiba LabRAM-HR800) at a laser excitation wavelength of 488 nm, recorded in the range 500–2400 cm^{-1}. The ratio of sp^2 and sp^3 phases (sp^2/sp^3) was quantitatively investigated by X-ray photoelectron spectroscopy (XPS, Ulvac Phi 5000VersaProbeII) using a monochromatic Al Kα X-ray source. Hydrogen content was monitored with a thermal desorption spectrometer (TDS, ESCO TDS1200II). To ensure that hydrogen atoms were released, and to eliminate the influence of substrate silicon melting at the same time, samples were heated gradually up to 1200 °C in 1200 s by infrared radiation and maintained at this temperature for another 300 s.

The surface roughness of the DLC film was measured using an AFM system (Bruker Multimode 8). The residual stress was calculated through Stoney's formula [11]. The curvatures of coated samples and film thicknesses were measured with a profilometer (Veeco DektakXT-S). Elastic modulus and hardness were tested using a nanoindenter (Elionix ENT-2100) with a Berkovich diamond tip (100 nm radius). The maximum indentation force was set at a relatively low value of 0.2 mN to minimize the influence of the silicon substrate. Values were calculated using the Oliver-Pharr method and an average of three indentation results. The fracture toughness K_c of silicon and DLC/silicon system was calculated using Niihara's formula [12,13],

$$K_c = A\left(\frac{E}{H}\right)^{2/5}\frac{P}{L^{3/2}} \quad (1)$$

where E and H are the elastic modulus and hardness respectively. P is the indentation load and L is the crack length measured from the center of the indented area to the crack tip. A is an empirical constant that depends on the indenter. Five maximum loads of 15 mN, 25 mN, 50 mN, 75 mN and 100 mN were used. After nanoindentation, the crack length was measured with a field emission scanning electron microscope (FESEM, Hitachi SU-8020).

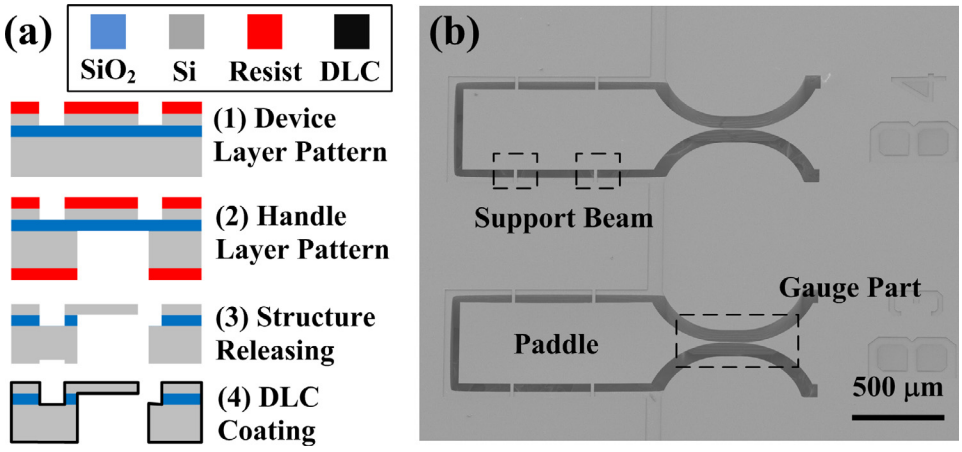

Fig. 1. DLC coated microstructure. (a) Microstructure Fabrication. (b) Microstructure coated with DLC at bias of −200 V.