TENSILE PROPERITIES OF SINGLE-CRYSTAL-SILICON FULLY COATED WITH SUBMICROMETER-THICK PECVD DLC

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ABSTRACT

The paper reports the improvement of tensile strength of single-crystal silicon (SCS) microstructures fully coated with sub-micrometer thick diamond like carbon (DLC) film using plasma enhanced chemical vapor deposition (PECVD). To minimize the deformations or damages caused by non-uniform coating of DLC, which has high residual stress, the released SCS specimens with the dimensions of 120 µm long, 4 µm wide and 5 µm thick were coated from the top and bottom side simultaneously. Three kinds of different deposition bias voltages were adopted with the thickness around 150 nm. The tensile strength improved up to 53.5% by the full coatings depending on deposition bias voltage. In addition, the deviation in strength reduced significantly compared to bare SCS specimen.

INTRODUCTION

DLC film is a kind of amorphous coating material with high hardness, toughness, good tribology properties, chemical stability and biocompatibility [1], and now is widely used as an surface enhancing material in traditional industry [2]. Furthermore, within adjusting the chemical composition or doping technology, DLC film would exhibit different excellent properties as a function material besides only protecting material, which attracted many attentions from the surface material researchers [3].

DLC film would be promising for improving the reliability of silicon-based MEMS components as well as a potential function material for more complex usage. Thus, understanding the mechanical properties, including the tensile properties by adopting DLC coating, is required for improved design of DLC coated MEMS structures. However, due to its very high residual stress, it is difficult to coat DLC on MEMS for a reliable structure. Isono et al. [4] coated DLC film only on the top surface of SCS tensile specimens but the result showed a 25-50% strength decrease, which may be caused by deformation of silicon microstructures induced by unbalanced residual stress.

Within present study, released free-standing tensile specimens were subjected to PECVD DLC coating, to cover the whole surfaces and to avoid non-uniform coating causing deformation or fracture of SCS microstructures. Three different bias voltages were chosen with the thickness of about 150 nm. The evaluation experiments using Raman spectroscopy, surface profilemeter and nanoindenter were carried out to evaluate the chemical composition, residual stress and hardness of the film. Then, the tensile strength between bare silicon specimen and DLC coated specimens were compared using a quasi-static tensile tester and fracture mechanism difference was briefly discussed.

EXPERIMENTAL METHOD

The SCS mirco-scaled structure for tensile testing was fabricated from silicon on insulator (SOI) wafer of 5-µm device layer using a standard MEMS manufacturing technique. The surface orientation of the device layer was (100). First, photoresist (OFPR-800LB, Tokyo Ohka Kogyo) was applied on the front side and the tensile test sample pattern was formed using UV lithography machine (PEM-800, Union Optics). The device layer was etched by Bosch process using inductive coupled plasma reactive ion etching (ICP-RIE) (RIE-iPB, Samco) with notch free process. Then, the same photoresist was applied on the backside and patterned to make substrate opening using backside alignment of the lithography machine. Then the handle layer was etched using Bosch process. After the resist layers on the both surface were removed using oxygen etching. The buried oxide layer was etched using buffered hydrofluoric acid (BHF) and the specimens were released from the handle wafer. The specimen free end including the gauge part was released and its underlying substrate was removed for realizing coating from backside on the next step. (Figure 1)

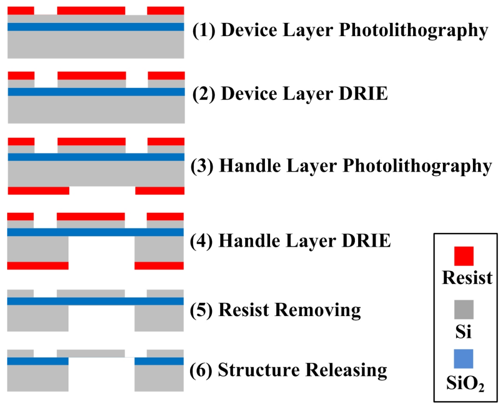

Figure 1: SCS mirco-scaled structure fabrication process.

DLC film was coated on the fabricated chips using a PECVD machine (ACV-1060, Shinkoseiki) for machine parts coatings. The chip carrying “bare” SCS specimens were put in the chamber (Figure 2). The chip was attached on a jig with an opening, and the jig revolves both on its own axis and the center axis of deposition chamber. In this machine, plasma is generated at the center of the chamber and film is deposited by the negative bias voltage applied on the holder. The chip is exposed from the both sides to the plasma by the revolutions, which realized a conformal deposition. Acetylene with flow rate of 150 sccm was used as the reaction gas with a working pressure of 0.04 Pa. Three bias voltages at −200 V, −300 V and −400 V were used at the same deposition time of 90 s with the thickness of 138 nm, 150 nm and 132 nm, respectively.

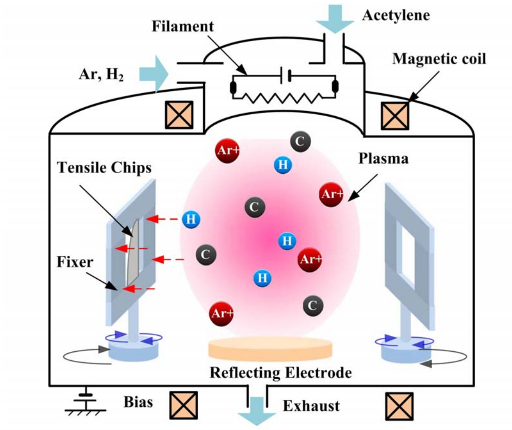

Figure 2: DLC coating by PECVD deposition.

Figure 3(a) shows the shape of tensile test sample, with a gauge part of 120 µm long and 4 µm wide. One end of the gauge part connected to a large paddle which was gripped by electrostatic force [5]. The loading axis of the specimen was <110>. Four beams attached on the paddle were used to support the released test sample and were cut by a laser cutting system just before the tensile test.

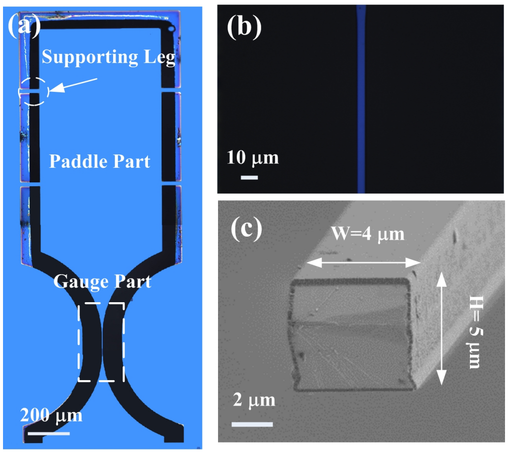

Figure 3: DLC coated micro-scaled tensile specimen. (a) Specimen coated with DLC at bias of −400 V; (b) Partial enlarged drawing of gauge part; (c) Cross section of SCS specimen coated with DLC at bias of −400 V.

The chemical composition of DLC film was analyzed by Raman spectra with an excitation laser wavelength of 488 nm (LabRAM HR-800, Horiba). The thickness of DLC film and the radius of curvature of the substrate were measured using a stylus profilometer (DekTak XT-S, Veeco), and the residual stress was calculated through Stoney's formula [6]. The elastic modulus and hardness of the DLC films were measured by a nanoindenter (TI-950, Hysitron) with a tip radius of 100 nm, maximum indentation depth of 40 nm and calculated by Oliver–Pharr method.

The tensile properties of bare and DLC-coated SCS samples were measured by a custom made quasi-static tensile tester which was reported before [5]. (Figure 4) Tensile testing was conducted at room temperature with 20 tensile samples for each type. The loading rate was controlled at the stage speed of 0.75 µm/s. The fractured surfaces were observed using field emission scanning electron microscope (FESEM) (SU-8020, Hitachi) and nominal tensile stress was calculated by dividing the tensile force by the cross sectional area measured by the FESEM of each sample.

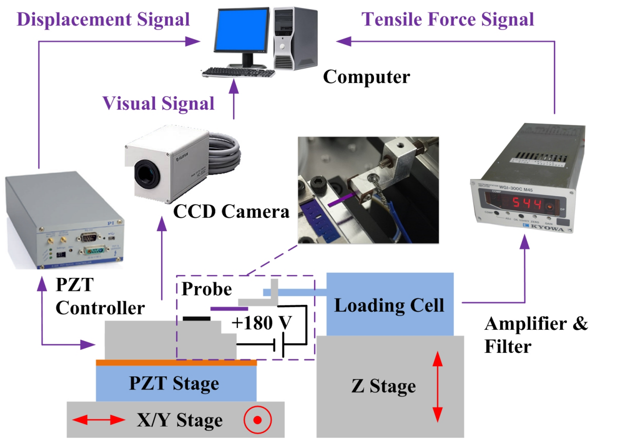

Figure 4: Sketch of tensile testing system.

RESULTS AND DISCUSSION

Chemical Composition

Micro Raman spectroscopy analysis was conducted to exam the chemical composition difference between the different bias voltages. Figure 5 (b) depicts the Raman spectra of DLC film deposited at the bias voltage of -400 V. The spectra exhibited a typical composite band for DLC film centered on 1500 cm^{-1} which could be divided into two peaks, which correspond to D Peak at around 1350 cm^{-1} and G Peak at around 1530 cm^{-1}.

The effect of the bias voltage on chemical composition could be inferred through the ratio of integrated intensities value of D peak (I(D)) to that of G peak (I(G)) according to Robertson`s work. The greater I(D)/I(G) was, the larger *sp2/sp3* ratio it had [7]. Therefore, it could be suggested that the *sp2/sp3* ratio in the deposited DLC film also decreased with the increasing of negative bias voltage. (Figure 5(a))

The change of residual stress inside the film would cause a shift in the position of G Peak. With higher compressive residual stress, with the position transport to a higher Raman Shift [8]. Figure 5(a) shows the value of G Peak position and it could be inferred the residual stress also increased with the increasing of negative bias voltage in this work.

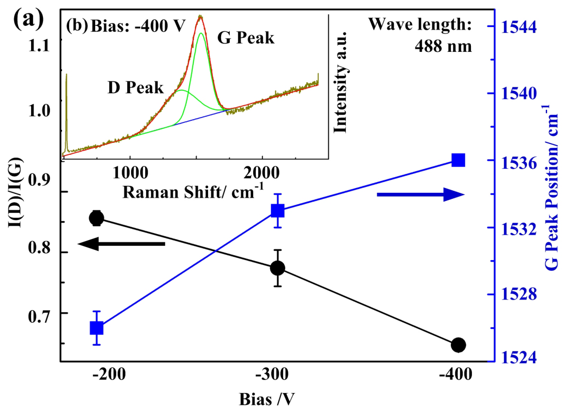

Figure 5: Properties of DLC films. (a) I(D)I(G) and G Peak position value; (b) Raman spectra of DLC -400 V

Elastic Modulus, Hardness and Residual Stress

Figure 6 displays the nanoindentation curve and elastic modulus, hardness values of DLC film with different bias voltage. For the DLC samples, elastic modulus and hardness increased as deposition bias voltage increased, which were in agreement with the result of Raman spectroscopy analyzes. Since the maximum penetration depth of indenter went beyond the 20% of the film thickness, the value of hardness and elastic modulus were considered as a mixture of film and substrate [9].

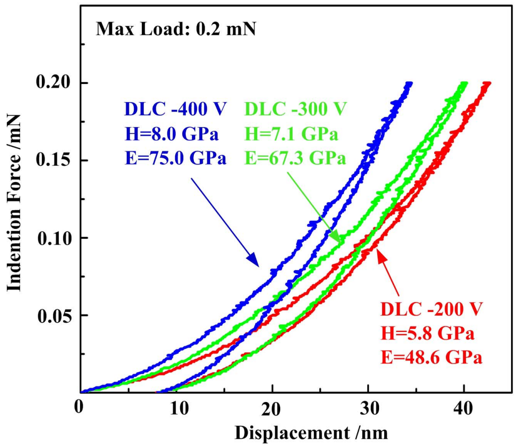

Figure 6: nano-indentation curve of DLC coating with different bias voltages. Elastic modulus (E) and hardness (H) are shown in plot.

The compressive residual stress at bias voltages of -200 V, -300 V and -400 V were -0.48 GPa, -0.91 GPa and -1.13 GPa respectively. The stress increased by increasing the bias voltage. The value of both the hardness and residual stress were smaller than that of film coated by sputtering method [10], which may due to the lower coating energy in PECVD method.

Tensile Strength

The tensile stress-stage displacement curves of all samples are shown in Figure 7. Both bare and coated samples exhibited a prefect linear plots and sudden drops of tensile stress, which indicates the brittle fracture of silicon and DLC films.

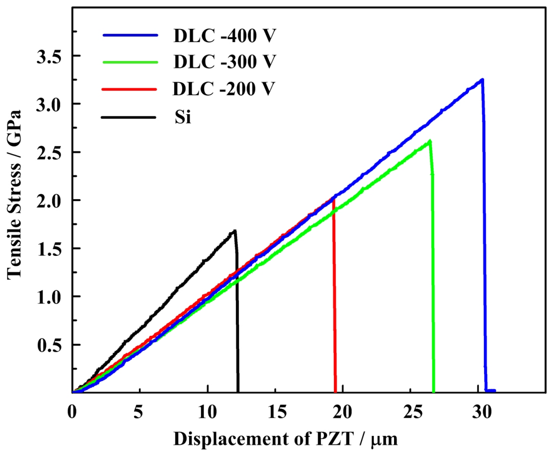

Figure 7: Typical displacement-stress curve of tensile test.

Figure 8 summarizes the average tensile strength of the specimens with and without DLC coatings. The as-fabricated specimen has the average tensile strength of 1.86 GPa. Comparing with bare silicon samples, the tensile strength increased by 13.4%-53.5% by adopting DLC film. Furthermore, with the increasing of negative bias voltage, the tensile strength increased obviously.

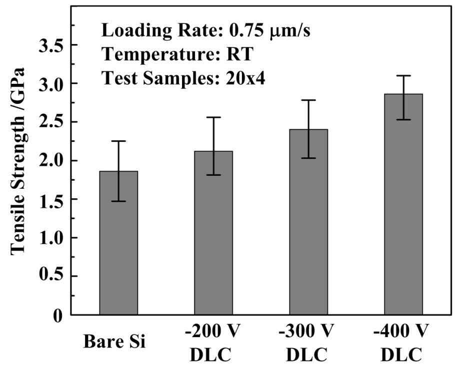

Figure 8: Average tensile strength with error bars indicates standard deviations.

Since both the DLC and silicon were brittle materials, the tensile strength had large scatterings. The two-parameter Weibull analysis was conducted for evaluating the strength deviation. Figure 9 shows the Weibull plot with the scale parameter (η) and shape parameter (Weibull Modulus, β). It could be concluded that the DLC coated silicon microstructures had smaller scatterings and also, with increasing the bias voltage, the deviation in tensile strength became smaller.

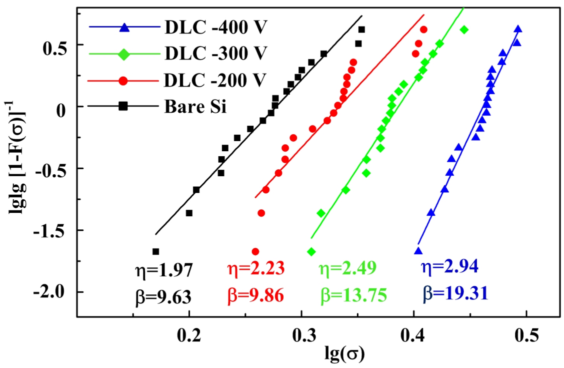

Figure 9: Weibull plot of tensile strength of SCS specimen with and without DLC film, β is fitted Weibull Modulus.

In this research, the tensile strength of silicon mirco-scaled structure increased obviously by adopting fully coated DLC film which may due to the improved surface condition. Since most of the fracture of bare SCS specimen happened at the quite rough side wall of the specimen induced by DRIE process [11], the DLC film may cover the notch of side wall as well as fill the defects on the silicon surface by using PECVD method. Moreover, the DLC coating presented a relatively high fracture toughness compared with that of silicon according to Li`s study [12] and may result in a better surface fracture resistance by DLC coating.

These improved tensile properties show a good correlation with the coating bias. The balanced compressive residual stress of DLC would seal the crack from becoming serious to fracture, and suppress the defects activation. Thus the average tensile strength and its deviation were improved by adopting DLC film and increasing bias voltage. The result reported here agreed with M. Tojek`s theoretical analyses about the influence of compressive residual stress on tensile strength and deviation [13].

CONCLUSIONS

The SCS micro-scaled structures were fully coated with around 150 nm thick PECVD DLC film from the top and bottom side simultaneously. The bias voltage of -200 V, -300 V and -400 V were used for the coating. Both of the hardness and compressive residual stress of DLC film rose with raising the negative bias voltage. The tensile strength was improved due to DLC coating up to 53.5% and increased with increasing the bias voltage. Furthermore, the Weibull analysis showed the coated microstructures had smaller scatterings and also, with increasing the bias voltage, the deviation in tensile strength became smaller. The proposed new coating methods and improved reliability of SCS microstructures will open new applications of SCS microstructures especially in corrosive or harsh environments.

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