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# *In situ* observation of tensile behavior in a single silicon nano-helix grown by glancing angle deposition

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ABSTRACT

In order to investigate the mechanical properties of amorphous silicon helical nanoelements (nano-helices), grown by the glancing angle deposition (GLAD) technique, we conducted tensile testing experiments on single nano-helices, which focused on the proof-of-principle. Three isolated nano-helices were prepared from the same GLAD layer by the removal of adjacent nano-helices using a nano-probe, and the top end was adhered to the loading tip with an electron beam curing adhesive. *In situ* observation by scanning electron microscopy revealed that the single nano-helix underwent a large longitudinal elastic elongation until fracture (about 40% of the fracture displacement) owing to its helical shape. In the early stage of deformation, there was a jump in the load-displacement relationship, due to partial breaking of the connecting leg at the root of the nano-helix. The tension induced plastic deformation and fracture in the bottom zone where the wire diameter was smaller. Examination based on element geometry at local zones indicated that deformation in the elastic stage was governed by the element shape. The yield stress of the amorphous silicon for the nano-helix was estimated to be approximately 0.8 GPa.

## 1. Introduction

Recent advances in deposition techniques have enabled the fabrication of nanometer-sized elements, typified by nanowires [1–4] and nanotubes [5]. Among the various fabrication techniques used, glancing angle deposition (GLAD) is simple and efficient for the formation of thin films with shape-controlled nanoelements on a substrate [6–14]. The GLAD method has been reported to produce nanostructured films with variously shaped (slanted, zigzag, and helical) nanoelements [15–19]. In particular, films of helical nanoelements (nano-helices) have drawn the attention of researchers and engineers, owing to their mechanical properties such as large reversible deformation [20–23]. For example, by inserting such a thin film into an interface between dissimilar materials, stress singularities at interface cracks or edges due to deformation mismatch can be eliminated [22,23].

A few experimental techniques have been reported for compressive testing of GLAD films [24–28], using atomic force microscopy (AFM) and nano-indentation devices. However, since an indentation tip is typically much larger than a single nanoelement, the load is applied to numerous elements. Thus, the test results represent the average despite nano-elements, which are formed on the substrate by GLAD at the same time, might differ in shape and mechanical properties. While there have been reports in which the load was applied on individual nano-helices, these experiments were conducted under compression in the elastic region [27,28]. Tensile tests conducted on individual elements can reveal details of the mechanisms involved in the elasto-plastic deformation and fracture of an individual element. However, there have been no such reports to date. In addition, while *in situ* observation by electron microscopy would provide details of the deformation and fracture processes for the element material, the task of applying the load correctly under high magnification remains a challenge.

In this study, a tensile testing method was developed for a single silicon nano-helix prepared by the GLAD technique under *in situ* observation using scanning electron microscopy (SEM). In particular, we focused on a proof-of-principle development of the tensile measurement technique. Experiments were conducted on several elements to determine the differences in deformation and fracture properties between individual nanoelements extracted from the same film and to elucidate the mechanisms involved.

## 2. Experimental

### 2.1. Material and specimen

A thin film composed of silicon nano-helices was formed on a 525 μm-thick Si(100) substrate using the GLAD technique. Deposition was performed under a vacuum of $1.0 \times 10^{-3}$ Pa using electron-beam evaporation (supply power: 3 kV). In the evaporator, the crucible diameter was 30 mm. The distance between the evaporation source and the

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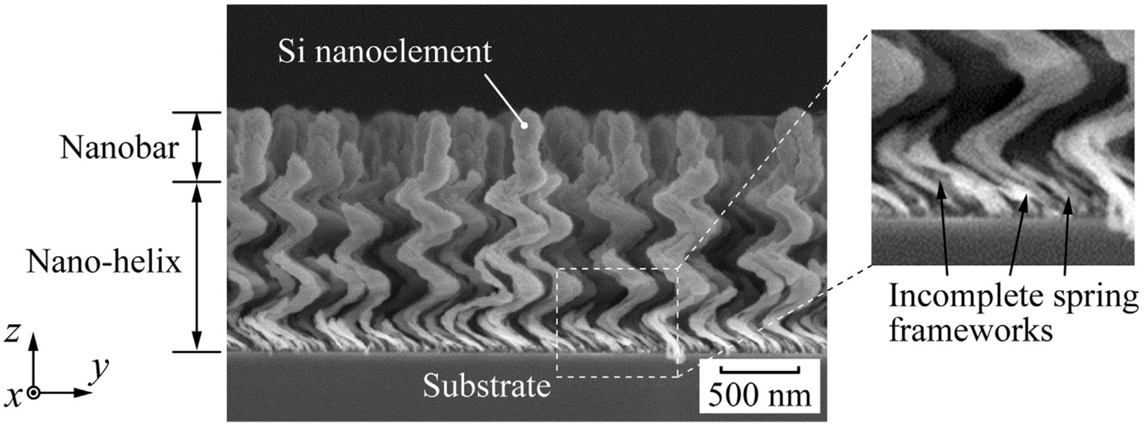
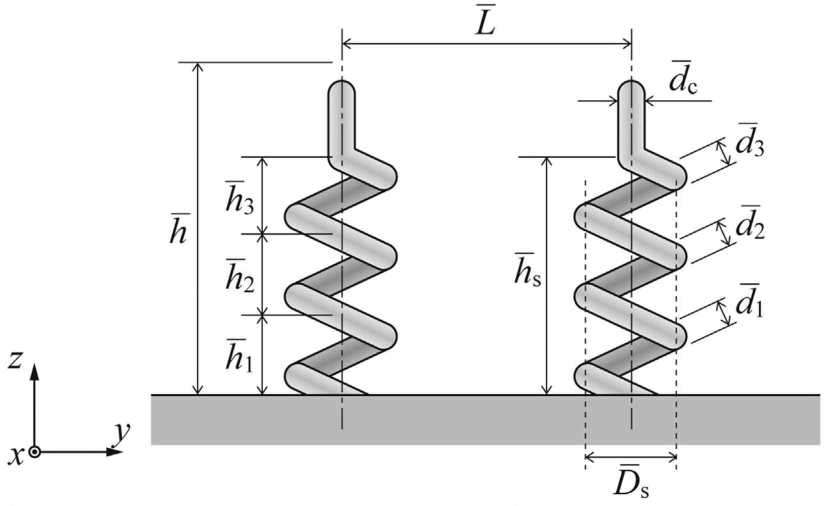


**Fig. 1.** Cross-sectional SEM image of thin film composed of silicon nanoelements.



| $\bar{h}$ | $\bar{L}$ | $\bar{d}_c$ | $\bar{h}_s$ | $\bar{D}_s$ | $\bar{d}_1$ | $\bar{d}_2$ | $\bar{d}_3$ | $\bar{h}_1$ | $\bar{h}_2$ | $\bar{h}_3$ |
|---|---|---|---|---|---|---|---|---|---|---|
| 1139 | 107 | 126 | 829 | 311 | 56 | 89 | 111 | 296 | 292 | 241 |

**Fig. 2.** Average dimensions of a nano-helix (units: nm).

substrate was 400 mm. High purity silicon (99.999%) pieces (Nilaco Corp.) were used as an evaporant. The deposition angle and rotation speed of the substrate were 86° and 0.3°/s, and 88° and 3.0°/s, respectively, for the nano-helix and the upper straight part (nanobar). The evaporation rate was set around 10 Å/s, which was controlled by a feedback system. The total deposition time was about 40 min. Fig. 1 shows a cross-sectional image of the thin film. A nanobar was fabricated on top of each nano-helix in preparation for tensile loading. In this study, the

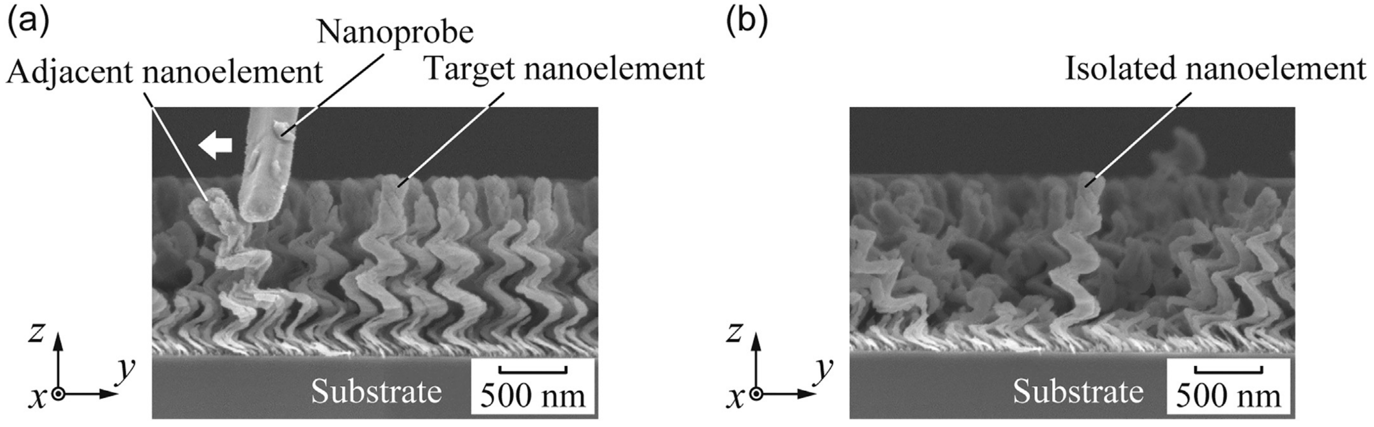


**Fig. 3.** (a) Extraction of a single Si nano-helix by removal of nano-helices adjacent to the target nano-helix using a nanoprobe. (b) Isolated individual Si nano-helix.