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Fabrication of single-crystal silicon micro pillars on copper foils by nanosecond pulsed laser irradiation

Jiwang Yan (2)[a,*], Jun Noguchi [a], Yoshitake Terashi [b]

[a] Department of Mechanical Engineering, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan
[b] Central Research Institute, Kyocera Corporation, Hebimizo-cho 1166-6, Higashiōmi, Shiga 527-8555, Japan

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ABSTRACT

A nanosecond pulsed Nd:YAG laser was used to irradiate a mixture of silicon powders, polyimide and carbon black deposited onto a copper foil. Silicon micro pillars with a single-crystalline structure were successfully created through laser-induced material self-organization. The shape, orientation and growth rate of the pillars were controllable by varying the fluence, incident angle and scan speed of the laser. The resulting pillar-on-foil structures were applied as negative electrodes of lithium ion batteries, and excellent electrical capacities as well as charge/discharge cycle characteristics could be obtained. The applicability of the proposed method to the manufacture of three-dimensional microstructures was demonstrated.

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1. Introduction

Silicon (Si) micro pillars are important surface structures having unique properties, such as antireflective ability and controllable wettability, and thus are interesting for applications in many fields, e.g., for energy harvesting, super hydrophobicity, biomolecule analysis, and so on [1–3]. Currently, Si micro pillars are fabricated on Si wafers by chemical etching or reactive-ion etching [4]. However, there is minimal literature on fabricating Si micro pillars on metal substrates, even though they have a wide range of potential applications. Like Si-based porous nanocomposites [5–8], Si micro pillars on metal foils are expected as new materials for fabricating lithium ion battery electrodes, which have a theoretical capacity ten times higher than that of a conventional carbon electrode.

In this study, a novel method for fabricating Si micro pillars on a copper (Cu) foil was developed based on nanosecond pulsed laser irradiation of a mixture of Si powders, polyimide (as a binder), and carbon black (as a sacrificial agent) deposited on the Cu foil. The shape, orientation, crystallinity and growth rate of the pillars were investigated under various conditions. The performance of the fabricated pillar-on-foil structure as an electrode for a lithium ion battery was evaluated and the results thereof strongly demonstrate the effectiveness of the proposed method.

2. Mechanism for laser-induced pillar formation

Si pillars were fabricated through an additive manufacturing approach [9–11] by laser-induced material self-organization. As illustrated in Fig. 1, Si grains at the top layer are directly heated by the laser and melt (a–b). The melted Si descends/flows to cover Si grains at the lower layer. With further laser heating, the lower Si grains are also melted due to thermal conduction from the upper layer (c). After Si has been completely melted, it spreads and wets the Cu foil (c). Due to surface tension, liquid Si grains merge to form big Si islands on the Cu foil (e). Under laser irradiation, carbon black, which is used as a sacrificial agent, undergoes plasma gasification and partial ignition in air, generating a high-pressure plasma and carbon oxide gas around the liquid Si islands (f). Due to the plasma-induced pressure, the liquid Si islands are stretched along the laser beam direction, forming long and thin micro pillars after resolidification (g). The polyimide binder has a very high thermal stability, and thus is less influenced by laser irradiation. It remains at the pillar/foil boundary through solid state and/or partial melt bonding [12], and strengthens the Si/Cu interface. This pillar fabrication method has never been reported before.

As shown in Fig. 2(a), when Si bulk is used as a lithium ion battery electrode, ion storage causes Si to expand by a factor of three to four, which causes fracture of the electrode. In contrast, Si pillars can accommodate the volume expansion due to the available space around the pillars, as shown in Fig. 2(b). As a

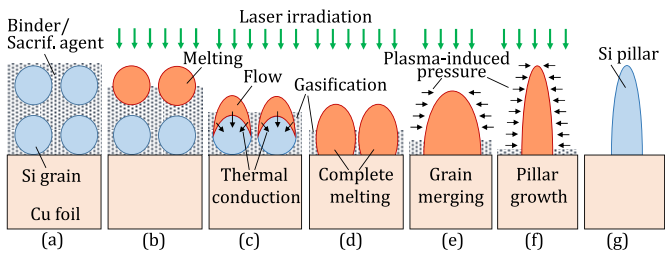

Fig. 1. Model for laser-induced micro pillar formation.

* Corresponding author.
E-mail address: yan@mech.keio.ac.jp (J. Yan).

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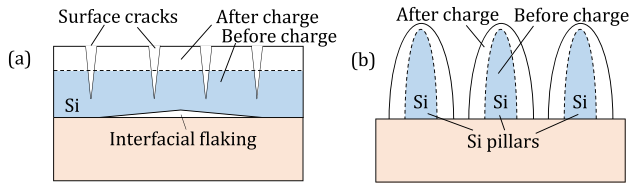

Fig. 2. Model of charge-induced expansion for (a) bulk Si and (b) Si pillars.

consequence, the pillar-on-foil structure may be a breakthrough in the fabrication of Si-based lithium ion batteries.

3. Experimental procedures

Laser irradiation experiments were carried out using a Nd:YAG laser with a wavelength of 532 nm. At this wavelength, the laser light can penetrate into Si up to a depth of ~1 μm, which matched the average grain size of the Si powders. The laser pulse duration was 48.4 ns. The laser beam had a Gaussian profile with a diameter of 85 μm. The laser fluence was changed in a range of 529–2643 mJ/cm^2, while the scan speed was varied from 1 to 10 mm/s. The laser beam was scanned over an area of 300 × 300 mm^2 by a galvanometer mirror. The workpiece was placed on a stage, which was adjustable in vertical direction to change the focus position of the laser beam.

A mixture of Si powders, polyimide, and carbon black was used as a specimen. Rather than using expensive pure Si nanoparticles, we used waste Si powder from a wire saw slicing process for Si wafers in the manufacturing of solar cells [6,7]. The Si grains (melting point 1414 °C) ranged in size from a few sub-microns to several microns. Carbon black (ignition point 520 °C), which contained carbon nanoparticles with sizes ranging from a few nanometer to hundreds of nanometer, was used as a sacrificial agent. Polyimide, which is an infusible polymer of imide monomers, was used as an interfacial binder. The carbon black and the polyimide were added into the Si powders at a mass ratio of 10:15:75. The mixture was agitated by a ball mill to obtain a slurry into which *N*-methylpyrrolidone was added as an organic solvent. Then, a 10–200 μm thick layer of the slurry was deposited onto the Cu foil and dried at 100 °C. The Cu foil thickness was 50 μm. The laser irradiation experiments were conducted in air.

4. Results and discussion

4.1. Effect of laser fluence and scan speed

Fig. 3 displays scanning electron microscopy (SEM) images of an irradiated mixture of Si, polyimide and carbon black at various laser fluences (slurry thickness 10 μm, laser scan speed 10 mm/s). As the laser fluence increased, micro pillars appeared and grew continuously in height. At a laser fluence of 2115 mJ/cm^2, individual conical micro pillars were formed with a height of ~15 μm, as shown in Fig. 3(d). Fig. 4 displays SEM images of irradiated samples at a laser scan speed of 1 mm/s, i.e., lower than that for the samples shown in Fig. 3. As laser fluence increased, micro pillars grew faster and were thinner than those shown in Fig. 3. At a laser fluence of 2115 mJ/cm^2 (Fig. 4(d)), nearly cylindrical micro pillars were formed. The height of the tallest one was over 25 μm. In this case, the formation of micro pillars looked similar to the formation of Si micro wires observed in infrared light sintering of Si-based composites [7], indicating the possibility of a vapor-phase pillar growth, i.e., Si is partially evaporated and deposited on the top of the pillars.

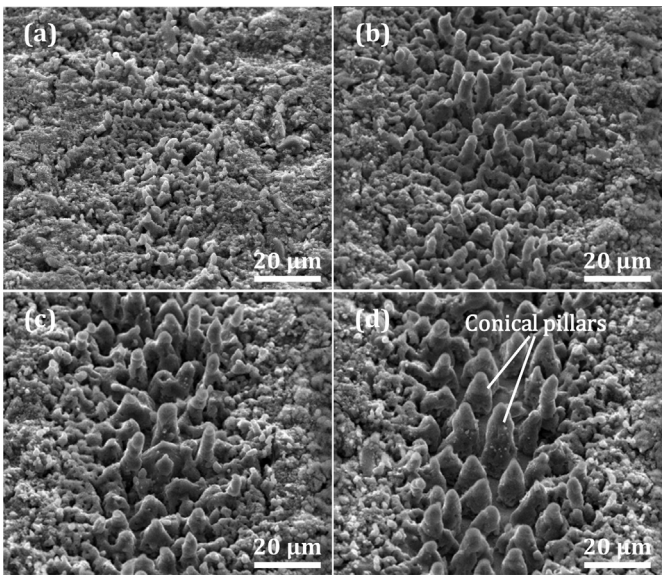

Fig. 3. Micro pillar growth at various laser fluences: (a) 529, (b) 1057, (c) 1586, (d) 2115 mJ/cm^2, and a scan speed of 10 mm/s.

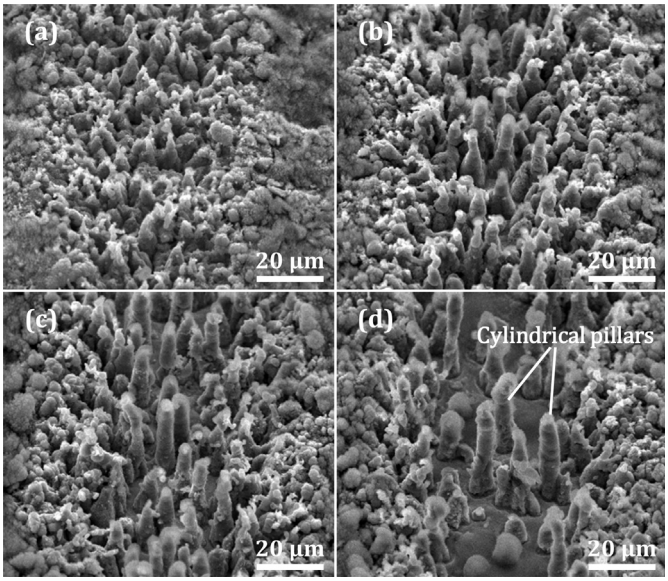

Fig. 4. Micro pillar growth at various laser fluences: (a) 529, (b) 1057, (c) 1586, and (d) 2115 mJ/cm^2 and a scan speed of 1 mm/s.

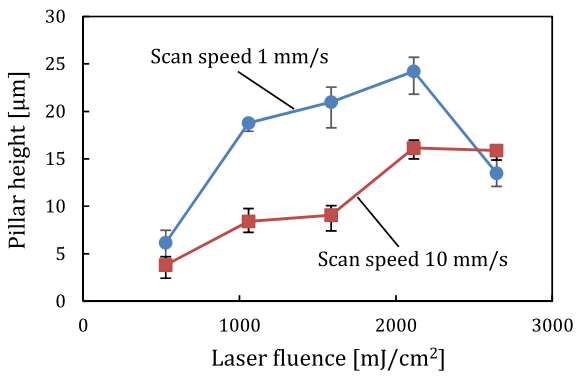

Fig. 5. Change of pillar height with laser fluence and scan speed.

Fig. 5 is a plot of the change of pillar height with laser fluence and scan speed. In the figure, the pillar height is defined as the average height of all identifiable pillars over a laser scan distance of 500 μm. As a general trend, pillar height increased with laser fluence, but decreased with laser scan speed. It should be noted that, at a very high laser fluence (2643 mJ/cm^2) and a low scan speed (1 mm/s), the number of pillars was reduced due to successive laser irradiation, leading to a sharp drop in the curve.

4.2. Effect of laser incident angle

To investigate the relationship between pillar growth and the incident direction of the laser, experiments were conducted at various laser incident angles. Fig. 6 shows SEM photographs of micro pillars formed at a laser incident angle of 60° for various laser fluences. It is clear that the pillars are orientated at an angle of ~60° away from the vertical direction. This observation indicates

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