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Study of Si-layers on graphite-substrates obtained through electrochemical reduction of silica powder

Muhammad Monirul Islam[1], Hajer Said[2], Hichem H. Ahmed[2], Katsuhiro Akimoto[1], Takeaki Sakurai[1]

[1]Univ. of Tsukuba
1-1-1 Tennodai, Tsukuba, Ibaraki 305-8573, Japan
[2]Univ. Tunis-El-Manner
B.P. n° 94 - Rommana, Tunis 1068, Tunisia
E-mail: islam.monir.ke@u.tsukuba.ac.jp

Abstract

Si-layers on graphite-substrates were formed through electrochemical reduction of silica powder (SiO_2). Structural and optical properties of the Si-layers were studied in relation to the reduction potentials applied during electrochemical experiment.

1. Introduction

Silicon (Si) is a key semiconductor used in electronics, energy generation, and energy storage devices. Application of the Si largely depends on the structure and dimension of its crystallinity. For example, Si in the form of amorphous phase, or crystalline phase in thin films, nanowires, or nanoparticles, *etc.* has their particular application. Nevertheless, present commercial production of high purity Si through carbothermic reduction of the SiO_2 involves multiple steps at high temperature of ~1700 °C [1].

Here, we have reported a low-cost process for the formation of Si-layers on graphite-substrates obtained through electrochemical reduction of silica powder (SiO_2). Structural properties, including morphology, and Optical properties of the electrodeposited Si-layers were studied in relation to the reduction potentials applied during electrochemical process.

2. Experimental

Electrodeposition of Si-layer has been carried out in a Al_2O_3-crucible placed inside a quartz three-electrode electrochemical-cell (Fig. 1) [2].

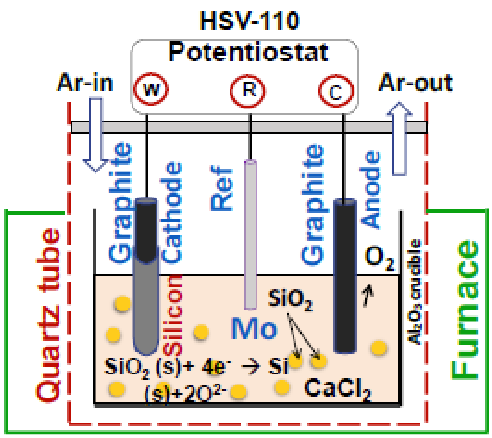

Fig. 1 Three-electrode based electrochemical cells for electrochemical reduction.

Electrochemical reduction of the SiO_2 occurs at high temperature of 850 °C in $CaCl_2$ molten salt following the equation:

$$SiO_2\,(s) + 4e^- \rightarrow Si(s) + 2O^{2-} \qquad (1)$$

Graphite was used as both the counter electrode (CE) and the working electrode (WE), while a molybdemum (Mo) wire was used as a reference electrode (RE). Electrochemical reduction was done under Ar-gas at 860^{O} Celsius using $CaCl_2$ molten salt. Chronoamperograms (CA) was done at constant potential (E) applied between graphite-substrate (WE) and Mo-RE. Cyclic voltammetry (CV), and all the constant potential electrolysis were carried out with an HSV-110 potentiometer (Hokuto Denko, Japan). Several samples were deposited by varying the applied reduction potential ranging from E= -1.15 to -1.25 volt with respect to Mo-RE.

Structural properties were studied using scanning electron microscope (SEM) and X-ray diffraction (XRD) techniques, while optical properties were investigated using Raman spectroscopy and steady state micro-photoluminescence (PL) method using a 532-nm excitation line from a Nd:YAG laser source, with CCD detector at RT.

3. Results and Discussions

To confirm the formation of any Si-layer, at first, we have performed Raman measurement.

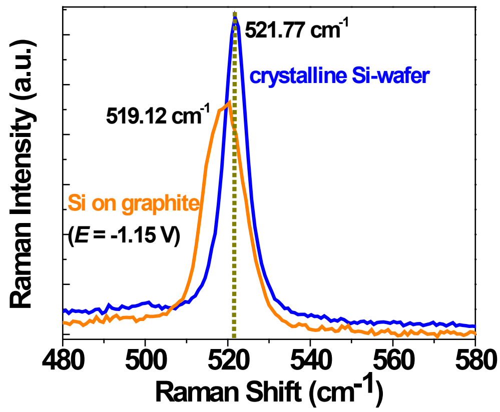

Fig. 2 Raman active mode for a Si-film along with a crystalline Si-wafer as reference.

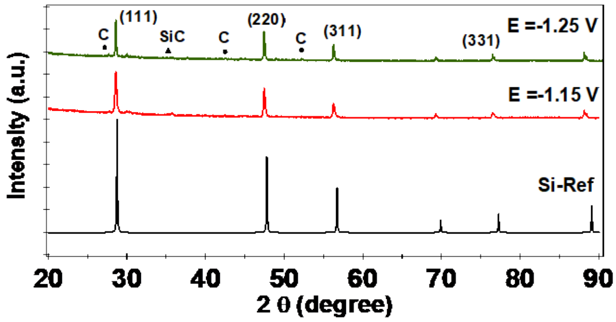

Fig. 2 XRD pattern of electrodeposited Si samples obtained at various reduction potentials.

Fig. 2 shows Raman spectra of an electrodeposited Si-layer on graphite-substrate obtained with a reduction potential, E = -1.15 Volt, applied between the graphite-substrate and Mo-RE. A peak around ~ 519 cm^{-1} with a slight downshift compared to the Si optical phonon peak of single crystal Si-wafer suggests formation of crystalline Si-layer on the graphite substrates [3].

Shown in Fig. 3 is the XRD pattern of Si samples electrodeposited with potential, E= −1.15 and -1.25 volts, respectively vs. Mo-RE. To avoid the strong XRD-peaks from graphite substrate, we have etched electrodeposited Si-layers from the substrate before taking XRD pattern. XRD supports the formation of mainly Si layer using the electrodeposition technique on the graphite-substrate. Very weak peaks related to carbon came from the substrates during etching.

Later to investigate morphology of the obtained Si-layer, we have performed SEM on the surface of the sample electrodeposited with potential, E= −1.15 V as shown in Fig. 4. As seen from the figure, electrodeposition on graphite with E= −1.15 V basically associated with formation of Si nanowires on the substrates. With an increase in the reduction potential, along with the existence of nanowires, cluster of nano-agglomerates of Si were observed in SEM images (not shown). Thus, morphology along with particles size were found to be controlled by varying the reduction potential applied during electrochemical reduction process.

4. Conclusions

Optical and structural properties of the electrodeposited Si-layers have been studied in relation to the various reduction potential applied during electrochemical reduction of SiO_2. Morphology and crystalline size of the Si nanowire structures has been found to be controllable using the applied reduction potential.

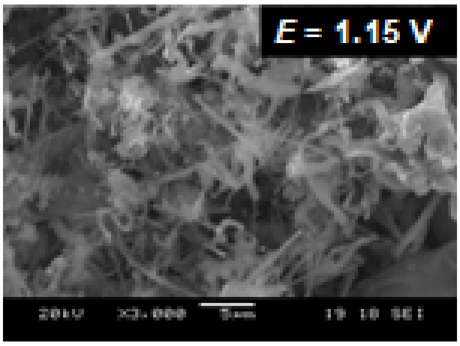

Fig. 3 SEM surface image of a Si-layer on graphite substrates obtained at reduction potential of E = -1.15 V.

References

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