Extended Abstracts of the 2018 International Conference on Solid State Devices and Materials, Tokyo, 2018, pp1071-1072

Electrodeposition of thin-film Ni-Si composite for application as anode-materials in lithium-ion-battery

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Abstract

Ni-Si composite has been obtained through electrochemical reduction of SiO_2 powder on nickel-substrate for application as anodes in lithium-ion-battery. Formation of Ni-Si composite has been confirmed through Raman spectroscopy and X-ray diffraction method. Photoluminescence measurement primarily suggest formation of Ni-Si nanocrystalline films on nickel-substrate.

1. Introduction

Recently silicon (Si)-based materials with highest-known theoretical charge capacity of 4200 mAh/g, have been considered as promising anode materials in lithium-ion battery (LiB) [1]. Specially, Si-metal based (Carbon, nickel, iron etc.) nanocomposites have drawn particular attention as it can effectively suppress volume expansion due to free space surrounding the nano-structures, and thus improve life cycle of the LiB [2]. However, obtaining nanocrystalline-Si (nc-Si) based composites involves re-crystallization of amorphous-Si, or direct deposition of nc-Si using plasma enhanced chemical vapor deposition (CVD), hot wire CVD, glow-discharge CVD, sputtering etc. Application of toxic materials in CVD process, various complex-steps, requirement of higher process-temperature, high vacuum system in the deposition process limit its potential applications for large scale production at commercial stage. In this paper, we have reported a low-cost electrochemical route for the formation of Ni-Si based alloys on Ni-substrates obtained through electrochemical reduction of silica powder (SiO_2). SiO_2 source material is the most abundant material after oxygen and therefore very cheap. In addition, electrodeposition is a low cost, non-vacuum method with simple process parameters. Thus, the process will allow large scale production of Si-Ni composites on to be used as anodes in LiB.

2. Experimental

Electrodeposition of Ni-silicides has been carried out in a Al_2O_3- crucible containing SiO_2 powder and placed inside a quartz electrochemical cell equipped with three-electrode system [3]. Graphite has been used as counter electrode (CE) as well as reference electrode (RE), while Ni-sheet was used as the working electrode (WE) as well as substrate for the electrodeposition. Electrochemical analysis has been done under Ar-gas at 860^{O} Celsius using $CaCl_2$ molten salt as electrolytes. Chronoamperograms (CA) has been done at constant potential (E) applied between Ag-substrate (WE) and graphite reference electrode. Cyclic voltammetry (CV), and all the constant potential electrolysis were carried out with an HSV-110 potentiometer (Hokuto Denko, Japan). Several Ni-Silicides films were electrodeposited by changing the reduction potential applied between Ni-working electrode and graphite RE.

Characterization of the electrodeposited Ni-Si layers was performed using Raman spectroscopy, Photoluminescence (PL), scanning electron microscope (SEM), and X-ray diffraction (XRD) technique. A 532-nm excitation line from a Nd:YAG laser source has been used for the PL and Raman spectroscopy. All the characterization of the Si layer has been done at room temperature (RT).

3. Results and discussions

To investigate the reduction potential of the SiO_2 powder in $CaCl_2$ melt, we have performed CV on the Ni-substrate (WE) in the $CaCl_2$ melt containing SiO_2 at 855^{o} C. Fig. 1(a) shows the CV curve on the Ni-substrate (WE), obtained with applying potential, E vs. graphite-RE at a scan rate of 10 mV/s. During cycle-1, a reduction current seems to be appeared after 0.7 V (negative potential) vs graphite-RE. Current continues to increase until reduction of Ca^{+2} appeared around 1.4 V at cycle-1. With further scanning in the reverse direction (-1.6 V to -0.2 V), an oxidation peak appears around 0.45 V negative potential vs. graphite RE

(Cycle-1). Thus, after analyzing the CV curve in the Fig. 1, it is clear that reduction of the SiO_2 is possible from – 0.7 V to more negative potential vs. graphite RE.

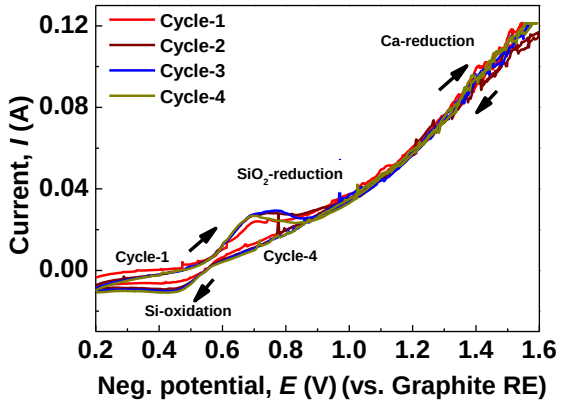

Fig. 1 Cyclic voltammogram of the Ni-electrode (substrate), obtained with potential, *E* vs. graphite-RE applying at a scan rate of 10 mV/s, in the $CaCl_2$-melt containing SiO_2 -powder at 855° C.

To confirm the formation of any Ni-Si based material on the Ni-substrate, we have performed Raman spectroscopy of the electrodeposited Ni-Si layers (not shown). A peak around ~ 318 cm-1 together with appearance of broadened peaks in between 350~ 500 cm-1 suggest formation of Ni-Silicides on the Ni-substrate. X-ray diffraction (XRD) pattern (Fig. 2) also supports formation of Ni-Si alloys on Ni-substrate.

Shown in Fig. 3 is the PL spectrum of the obtained Ni-Si layer electrodeposited on Ni-substrate with $E = 0.7$ V negative potential vs. graphite-RE.

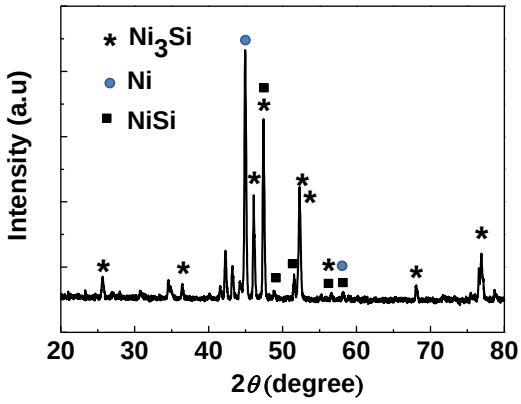

Fig. 2 XRD pattern, taken at θ-2θ mode, of the Ni-Si film deposited on the Ni-substrate.

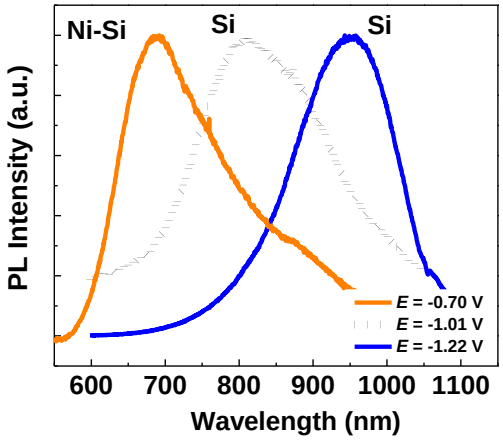

Fig. 3 PL spectrum of electrodeposited Ni-Si layer on Ni-substrate. PL spectra of nanocrystalline Si films electrodeposited on silver-substrate have been shown to show the effect of reduction potential.

PL spectra of the nanocrystalline Si-layers obtained through same electrodeposited technique on the silver-substrates at different reduction potential have been shown on the same figure for comparison. PL-peak of the Ni-Si layer around 680 nm in the wavelength scale primarily suggest formation of Ni-Si nanocomposite on the Ni-substrate. Moreover, size of the nanocrystal may depend on the applied reduction potential during the electrodeposition technique, and independent of the substrate material used.

Finally, other optical and structural properties of the electrodeposited Ni-Si layers have been studied in relation to the effect of various reduction potential applied during electrochemical reduction of SiO_2.

4. Conclusions

Ni-Si layers were electrodeposited on Ni-substrate through electrochemical reduction of SiO_2 powder in $CaCl_2$ molten salt at high temperature. Both Raman spectroscopy and XRD confirm the formation of Ni-Si composite. PL measurement primarily suggests formation of Ni-Si nanocomposites on the Ni-substrate.

Acknowledgements

This work was partly supported by the University of Tsukuba-industry Cooperation project (FY2018).

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