X-ray Micro-Tomosynthesis System Coupled with Optical Lens for Battery Materials Research

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1. Introduction

As the demand of electric vehicles increases, the importance of lithium-ion batteries (LIBs) with high energy density is increasing exponentially [1]. Accordingly, silicon based anode materials have high theoretical capacity (≈ 4200 mAh g-1), low cost due to abundant reserves, and a reduction potential close to lithium, which can be applied as high energy density LIBs [2]. However, silicon still exhibits disadvantages of continuous consumption of electrolyte due to anode pulverization due to large volume expansion and contraction, destruction and reformation of the SEI (solid electrolyte interphase) layer, and rapid deterioration. [3] In order to solve this problem, silicon nanoparticles are used to minimize volume expansion and contraction which causing performance degradation. [4] However, silicon nanoparticles do not maximize the advantages because the aggregation occurs during manufacturing of anode. Therefore, it is important to observe the aggregation of silicon nanoparticles after fabrication of the anode material.

Tomosynthesis can reconstruct a sliced image of an object or a three-dimensional (3D) image of an object by intergrating multiple two-dimensional (2D) projected images at different angles [5]. Compared to conventional computed tomography (CT) techniques, it has the advantage of a small rotation angle. Therefore, it is advantageous for short exposure time. In recent years, X-ray tomosynthesis technology has become one of the most important methods in field of X-ray imaging.

The X-ray imaging system coupled with the optical lens is composed of a scintillator and an optical detector coupled with optical lens. This system can develop high-resolution X-ray image. [6] When this optical detector with optical lens has an effective pixel size from nano to sub-micro meter, X-ray images with micron spatial resolution can be acquired. Since the geometric magnification method is not used, there is no need to use a nano- or micro-focused X-ray tube, so the price is very low and the volume of the X-ray system is small.

In this study, X-ray micro-tomosynthesis system coupled with the optical lens was developed for acquiring sliced micro X-ray images of coin-cell battery. X-ray micro-tomosynthesis of coin-cell battery was used for observing the aggregation of silicon nanoparticles.

2. Methods and Results

2.1 Developed System

Micro-tomosynthesis system was designed in a high-resolution X-ray imaging system coupled with optical lens. This X-ray system consists of a micro-focus X-ray tube (P030-24-12F100W, Petrick GmbH, Bad Blankenburg, Germay), an optical lens, and a scientific complementary metal-oxide-semiconductors (sCMOS) detector (pico.edge 4.2, PCO, Kelheim, Germany). The operating voltage and current of micro-focus X-ray tube are 50 kVp and 1 mA. The focal spot size is 30–55 µm. A 10x infinity corrected objective lens (f = 200 mm) and a tube lens (f = 160 mm) are used to magnify the X-ray image on scintillator film, and a sCMOS detector is used to develop this image. The sCMOS detector has 6.5 µm suqaure pixel size. The effective pixel size is 812.5 nm. The X-ray tube The X-ray tube rotates about the center of the scintillator. After acquiring 2D X-ray images at various angles, slice images of an object were acquired using the shift and add tomosynthesis method. (Fig. 1.a) The principle of shift-and-add tomosynthesis is that multi projection images were taken when the x-ray tube was moved. The acquired images are appropriately shifted and added to bring micro-structures in objects in focus, while structure outside the plane of focus blurred across the image. The finally developed system is shown in Fig. 1.b.

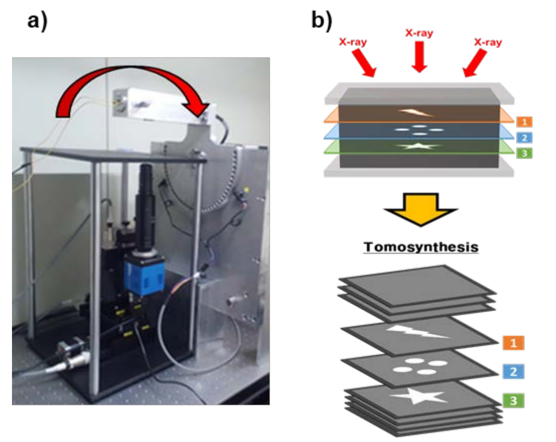

Fig. 1. a) Mechanism of X-ray micro-tomosynthesis, b) Developed X-ray micro-tomosynthesis system coupled with optical lens

2.2 Resolution of Sliced X-ray Image

The resolution of X-ray imaging system coupled with optical lens was measured. The Jima RT RC-04 resolution chart was used to measure the resolution of the sliced X-ray image. (Fig.3) T resolutions of 1.5 - 2 μm were measured.

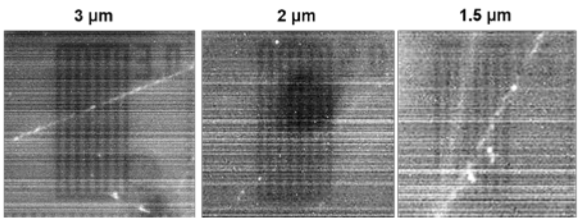

Fig. 3. Micro X-ray images of Jima RT RC-04 resolution chart

2.3 Fabrication of Silicon Nanoparticle Anode

The silicon nanoparticle based anode fabricating method is shown in Fig. 4. Silicon nanoparticles (0.54g) were put into the N-Methyl-2-pyrrolidone (NMP) solution, and 0.18g of polyvinylidene fluoride (PVdF) as a binder and carbon nanoparticles as a conducting material were added. This slurry was mixed by ball milling. After put the prepared slurry on the copper foil, it was spread thinly using a doctor blade. And it was dried at 130^o for 24 hours.

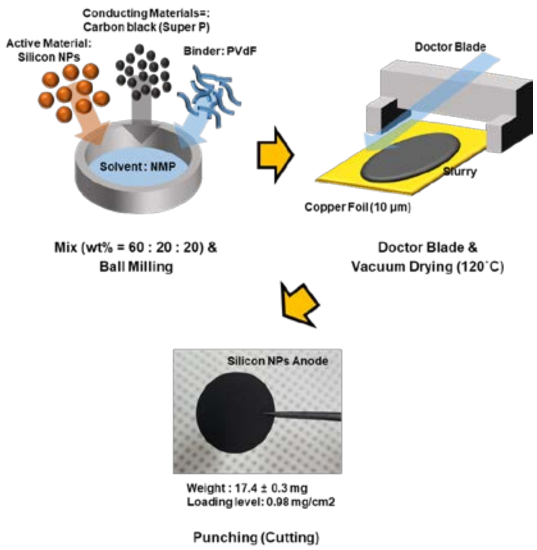

Fig. 4. Fabrication method of Silicon nanoparticle anode

2.3 X-ray Micro-Tomosyntehsis Image

The X-ray image of the manufactured silicon nanoparticle anode is shown in Fig. 5. Fig. 5.a shows a 2D X-ray image, and the resolution of the image is relatively low because of reducing X-ray by copper foil. However, The X-ray micro-tomosynthsis image has the high contrast so the resolution of the image was increased.

Ball milling was performed during the manufacture of the anode material. However the aggregation of silicon nanoparticles was not completely resolved.

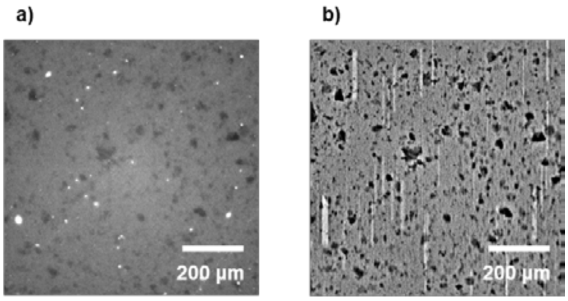

Fig. 5. a) 2D X-ray micro-image, b) Focusing image of X-ray micro-tomosynthesis image of Silicon nanoparticle anode

3. Conclusions

An X-ray micro-tomosynthesis system coupled with optical lens was developed. X-ray micro-tomosynthesis system was designed into this system for acquiring sliced micro X-ray images of coin-cell battery. Through this device, the aggregation of silicon nanoparticles inside the anode material was successfully observed. The method to resolve the aggregation of silicon nanoparticles through this device will be studied in the future.

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