Nanoscale Silicon Particles in Sandwich Structures Fabricated on Silicon Wafers: HRTEM Techniques as Imaging Tool

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Introduction

The use of silicon in optical applications is limited by its small and indirect band gap. However, recent observations of photoluminescence in porous Si and in Si ultra fine particles suggest that Si nanoclusters may become a promising material for optical applications. The nanometric Si-clusters have a band gap enlarged into the visible range and quantum confinement effect is believed to be the mechanism for light emission. The ability to control the size and structure of nanoparticles would allow the fabrication of structures with desired electrical and optoelectronic properties for device applications.
We present the structural characterization and composition studies of the fabricated nanostructures using high resolution transmission electron microcopy (HRTEM).

Materials studied

We synthesized quantum dot-type specimen by ultra-high vacuum nano-cluster deposition. The specimen as presented are nanosized silicon particles with controlled diameter, deposited on silicon wafers, cut in (400).
For the sample fabrication we used a ultra-high vacuum nano-cluster deposition apparatus from Oxford Applied Research (Nanodep60), with a DC magnetron sputtering unit for sputtering particles into aggregation regions. Sputtered particles form clusters passing a quadruple mass filter (QMF) to allow pre-selection of the size-distribution of nano-particles.

Sample preparation for HRTEM

Adequate sample preparation is crucial for HRTEM studies in order to obtain artifact-free structure information. In this study, cross-sectional sample preparation technique (XTEM) was used to prepare nano-sized Si particles. To prepare the XTEM specimen, a series of rectangular Si-wafers, whereon the Si nano-size particles were deposited, were cut into 4 mm x 5 mm slabs. These slabs were glued together allowing the interfaces with Si-particles to be glued to a 3-mm thick sandwich structure. The sandwich was cut by a slow-speed diamond saw along the transitional orientation to obtain a 1-mm thick slice. The 1-mm thick slice was used to cut out 3-mm discs in perpendicular direction using an ultrasonic disc cutter. Following this, the disc was mounted, ground to a thickness of around 200 µm, and then dimpled to around 20 µm using a Southbay D500i Dimpler, and finally polished to achieve electron transparency using a Fischione 1010 low angle ion mill. A schematic illustration of this procedure is provided in Figure 2. The appearance of the prepared samples is shown in Figure 3. Some samples do not survive sample preparation in one piece and break after ion milling. The achieved sample thickness in the center of the a disc was around 90-100 nm. One broken sample piece was placed on the copper grid for further TEM investigations (Figure 3 (a)). A XTEM sample after performing ion milling is displayed in Figure 3 (b), showing the characteristic perforation in the center of the XTEM sample disc.

HRTEM imaging and analysis

A state-of-the-art transmission electron microscope (TEM), TECNAI-Supertwin-F30 TEM, was utilized to examine the prepared specimens. The specimens were well prepared and large electron-transparent areas were available. Therefore, the well-defined nano Si-particles and their substrate can be distinguished due to the difference in the electron contrast caused by the difference in the individual structure.

Figure 4 shows the distribution of Si-particle on the Si-substrate at low magnification (x 7,100) as well as the atomic arrangement of silicon atoms within the Si-nano-clusters at high magnification (x 1,000,000) and high resolution (0.22 nm).

Figure 5 shows the distribution of Si-particles at intermediate magnification (x 200,000) and at high resolution (x 1,000,000). The HRTEM image displays a quantum dot single crystal and the arrangement of Si-atoms within the quantum dot single crystal is resolved. The lattice plane distance (ca. 35 nm) confirms the $d_{(111)}$ lattice plane of silicon (silicon $d_{(111)} = 0.31355$ nm).

The size of the Si-nano particles is around 15-18 nm.

Conclusion

Crystallized and single-crystal Si-nano particles were produced by ultra-high vacuum nano-cluster deposition. A XTEM sample preparation technique was developed and quantum dot-type nano-sized Si-particles could be imaged at magnifications up to 1,000,000 times with point resolutions of 0.22 nm. The arrangement of Si-atoms within the Si-nano-particles was resolved and the measured lattice plane distance of 35 nm confirms the $d_{(111)}$ lattice plane of silicon (0.31355 nm). The size of the Si-nano-clusters was 15-18 nm.

Sample Preparation

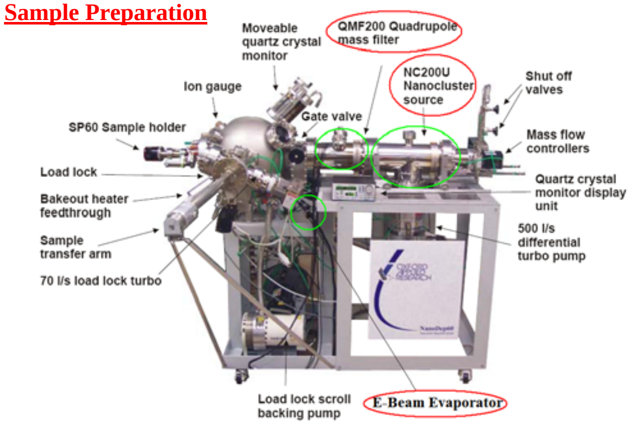


Figure 1: Ultra-high vacuum nano-cluster deposition apparatus from Oxford Applied Research (Nanodep60)

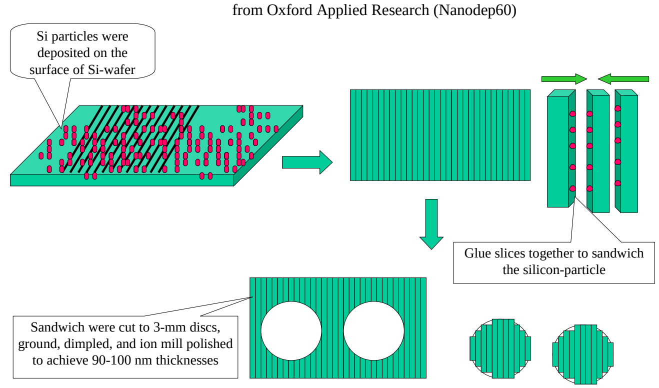


Figure 2: XTEM sample preparation procedure (schematic)

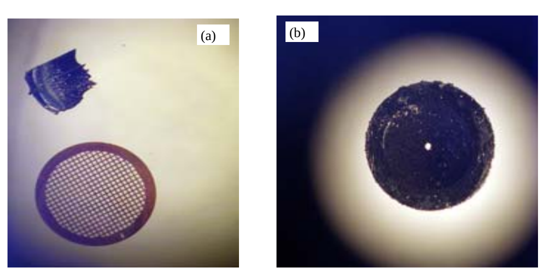


Figure 3: Appearance of prepared XTEM sample

Results

Click image to enlarge

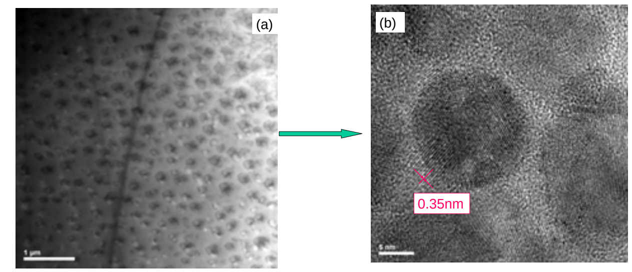
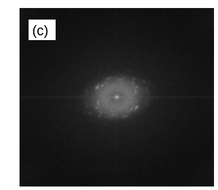


Figure 4: TEM image of Si-particles

(a) Low magnification (x 7,100) image is displaying the Si-nano-particles with darker electron contrast relatively to the Si-wafer with brighter electron contrast.
(b) High magnification (x 1,000,000) image is resolving the atomic arrangement within the individual quantum dot, measuring 16-18 nm in diameter. The lattice plane distance can be measured to approximately 0.35 nm, confirming the lattice plane $d_{(111)}$ of silicon (0.31355 nm).
(c) Fast Fourier Transformation (FFT) of (b) indicating the crystallinity of Si-nano particles.



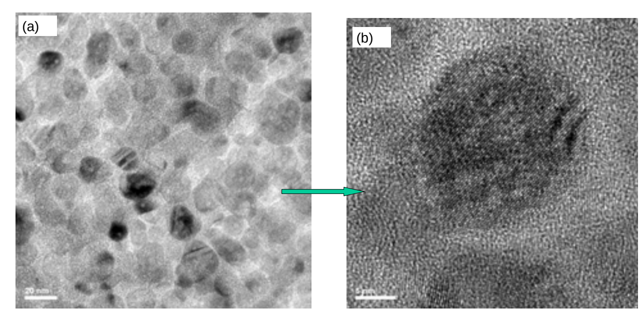
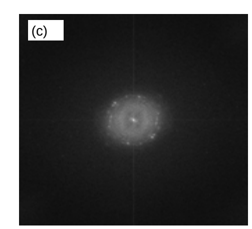


Figure 5: TEM image of Si-particles in different areas

(a) Intermediate magnification (x 200,000) image is showing the quantum dot distribution.

(b) High magnification (x 1,000,000) image is showing a quantum dot single crystal. The arrangement of Si-atoms within the quantum dot single crystal is resolved. The lattice plane distance (ca. 35 nm) confirms the $d_{(111)}$ lattice plane of silicon (Si $d_{(111)} = 0.31355$ nm).

(c) Fast Fourier Transformation (FFT) of (b) indicating higher crystallinity of the Si-nano clusters compare to Figure 4.



Acknowledgements

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