# Arc Plasma Synthesis of Silicon Nanoribbons and Their Electrochemical Properties for Lithium Storage Postprint

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## Abstract

Silicon nanoribbons (Si NRs) were prepared by direct current arc plasma method in a hydrogen and helium mixed atmosphere. The morphology, structure, and composition of the Si NRs were characterized using transmission electron microscopy (TEM), atomic force microscopy (AFM), X-ray diffraction (XRD), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), and other techniques. The results demonstrate that these silicon nanoribbons (Si NRs) are ribbon-like nanostructures formed by the stacking of fine flake-like Si crystals along a specific crystallographic direction, with a coalescence growth mechanism being dominant. The Si NRs exhibit a width of approximately 28 nm, length greater than 200 nm, thickness of 6.2 nm, and a specific surface area of approximately 164 $m^2 \cdot g^{1}$. The formation mechanism of Si NRs is thoroughly investigated in this work. This method for preparing Si NRs is low-cost and scalable for commercial mass production, with a yield of approximately 18.6 $g \cdot h^{1}$. Lithium-ion batteries were fabricated using these Si NRs as the anode active material, and their lithiation/delithiation electrochemical performance was evaluated. Such lithium-ion batteries deliver an initial discharge specific capacity of 2460 $mAh \cdot g^{1}$, maintaining a capacity of 316 $mAh \cdot g^{1}$ after 40 cycles, demonstrating favorable lithium storage electrochemical activity.

## Full Text

## Preamble

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## Abstract

Silicon nanoribbons (Si-NRs) were successfully synthesized by direct-current (DC) arc-discharge plasma in a mixed atmosphere of hydrogen and helium, and subsequently characterized by transmission electron microscopy (TEM), atomic force microscopy (AFM), X-ray diffraction (XRD), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS). This work provides a low-cost preparation method for Si-NRs that could be commercially produced with a production rate of 18.6 g · h $^{1}$. The Si-NRs consist of fine sheets approximately 28 nm in width, over 200 nm in length, and about 6.2 nm in thickness, with a specific surface area of 164 m$^{2}$ · g $^{1}$. The measured electrochemical performance of the Si-NRs as an anode material for lithium-ion batteries reveals a first discharge specific capacity of 2460 mAh · g $^{1}$, which reaches 316 mAh · g $^{1}$ after 40 cycles, exhibiting high activity for Li ion insertion/extraction and potential for further improvement of cycle stability.

**Keywords:** inorganic non-metallic materials, silicon nanoribbon, direct-current arc-discharge, electrochemical reaction, lithium-ion battery, anode material

## Introduction

Silicon is one of the most abundant elements in nature, second only to oxygen, and is environmentally benign. Silicon can react with lithium to form various lithium silicide phases such as Li Si , Li Si , Li Si , and Li Si [1]. As an anode material for lithium-ion batteries, silicon offers a theoretical specific capacity of up to 4200 mAh · g $^{1}$, the highest among known lithium storage materials. Additionally, silicon exhibits a low lithiation potential below 0.4 V and low reactivity with electrolytes, making it a promising candidate for high-performance batteries. However, silicon undergoes substantial volume expansion (exceeding 300% [2]) during lithiation, leading to material fracture and pulverization [3,4], which causes loss of electrical contact and deactivation of active material. This severe limitation has hindered the practical application of silicon in lithium-ion batteries.

Nanostructuring represents an effective approach to improve the electrochemical performance of silicon. Nanostructured materials have smaller dimensions, which reduces the absolute volume change during lithiation/delithiation within a confined space, thereby enhancing cycling stability. Nanostructures also provide abundant interfaces and active sites, improve electrode reaction kinetics, shorten lithium ion and electron transport pathways, and mitigate stress and strain from non-uniform lithium diffusion [5,6]. The high density of grain boundaries in nanomaterials, with lower packing density than perfect crystals, helps accommodate overall volume expansion during cycling. Furthermore, reduced grain size makes formation and movement of perfect dislocations difficult or even impossible, while nanostructured metals and alloys more readily form partial dislocations, enabling nanocomposite electrodes to withstand higher stress before pulverization [7,8]. The enhanced plasticity and deformability of nanomaterials [9] help absorb stresses generated by volume changes, improving cycling stability. The absolute volume change of individual nanoparticles during lithiation/delithiation is significantly smaller than that of microparticles, resulting in more stable structures during cycling [10].

Computational studies by Kulish et al. [11] have shown that two-dimensional silicon nanosheets can serve as lithium-ion battery anode materials. Yan et al. [12] prepared two-dimensional silicon nanosheets using graphene oxide as a template and demonstrated that after 100 cycles, the electrode maintained high specific capacity with significantly better cycling performance than Si nanoparticles. As an anode material, two-dimensional silicon nanostructures offer greater structural advantages and application prospects compared to zero-dimensional structures.

In this work, we employ DC arc-discharge plasma—a method distinct from conventional chemical vapor deposition—to synthesize silicon nanoribbons (Si NRs) in a hydrogen and helium mixed atmosphere, and investigate their formation mechanism in detail. The Si NRs were then used as the active material for lithium-ion battery anodes, and their electrochemical lithium storage performance was evaluated.

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## 1. Experimental Methods

### 1.1 Synthesis of Silicon Nanoribbons

Industrial-grade bulk silicon (99.99% purity) was used as the raw material and served as the anode in the DC arc plasma apparatus, with a tungsten electrode as the cathode. The reaction chamber was first evacuated to the required vacuum level, then flushed with a small amount of helium and re-evacuated to remove any residual air. A mixed reaction gas consisting of 0.01 MPa hydrogen and 0.02 MPa helium was introduced. After initiating the arc, the current was set to 90 A with voltage ranging from 20–40 V (depending on the distance between cathode and anode). The evaporated silicon underwent nucleation and growth processes to form nanoparticles that deposited on the water-cooled chamber walls. After cooling to room temperature, a small amount of air was introduced for passivation, forming a thin silicon oxide layer on the nanoparticle surfaces to prevent further oxidation upon exposure to atmosphere. The silicon nanopowder was collected, weighed, and used for structural characterization and electrochemical testing.

### 1.2 Morphological and Structural Characterization

The morphology of the silicon nanopowder was examined using transmission electron microscopy (TEM, Tecnai$^2$ 20 S-TWIN, 200 keV) and atomic force microscopy (AFM, Agilent PicoPlus II). For AFM measurements, samples were prepared on mica substrates and tested in tapping mode. X-ray diffraction patterns were obtained using an Empyrean diffractometer with Cu K radiation ( = 0.15416 nm). Raman spectra were collected on an InVia spectrometer with an excitation wavelength of 632.8 nm. X-ray photoelectron spectroscopy (XPS) was performed using a ThermoEscalab 250Xi spectrometer with monochromatic Al K radiation (h = 1486.6 eV), calibrated using the C1s emission peak of hydrocarbon compounds at 284.8 eV. Specific surface area was measured by BET method using an Autosorb-1 MP instrument at 77.3 K.

### 1.3 Electrochemical Performance Characterization

Electrodes were prepared by mixing active material (Si NRs powder), conductive agent (Super P carbon black), and binder (polyvinylidene fluoride, PVDF) in a 5:4:1 mass ratio with an appropriate amount of N-methyl-2-pyrrolidone (NMP) solvent. The slurry was coated onto copper foil and vacuum-dried at 120°C for 24 h. CR2025 coin cells were assembled in an argon-filled glove box with H O and O levels below 0.1 ppm, using 1 mol·L$^{1}$ LiPF in ethylene carbonate (EC) and diethyl carbonate (DEC) (1:1 v/v) as electrolyte, lithium metal foil as counter electrode, and polypropylene (PP) membrane as separator. Cyclic voltammetry was performed on a CHI660D electrochemical workstation between 0.01-2.5 V at 0.1 mV·s$^{1}$. Electrochemical impedance spectroscopy (EIS) was measured from 0.01 Hz to 100 kHz with 5 mV amplitude. Charge/discharge capacity, Coulombic efficiency, and lithium storage potential were tested using a LAND CT2001A battery system at 100 mA·g$^{1}$ current density between 0.01-2.0 V (vs. Li/Li ).

## 2. Results and Discussion

### 2.1 Morphology of Silicon Nanoribbons

Figure 1 [Figure 1: see original paper] presents TEM, HRTEM, and AFM images of the silicon nanopowder synthesized in the hydrogen-helium mixed atmosphere. The silicon nanostructures exhibit ribbon-like morphology with widths of approximately 28 nm, lengths exceeding 200 nm, and thicknesses of about 6.2 nm, corresponding to an aspect ratio of ~4.5. These dimensions justify classification as silicon nanoribbons (Si NRs) [13]. The specific surface area of these Si NRs is approximately 164 m$^{2}$·g$^{1}$, with the N adsorption-desorption isotherm shown in Figure 2d [Figure 2: see original paper].

Detailed analysis of Figure 1b reveals that the Si NRs result from anisotropic growth of silicon, forming a ribbon structure composed of stacked polycrystalline sheets. The lattice spacing of ~0.31 nm corresponds to the Si (111) crystal plane, while the ribbon surface is terminated by (110) facets. Clear interfaces observed in the image confirm that the nanoribbons are formed by oriented stacking of smaller nanosheets. The growth direction of the Si NRs forms a ~20°

angle ( ) with the <111> crystal direction. Since the angle between <111> and <112> directions is approximately 18°, close to the measured value, the growth direction can be identified as <112> [14].

A thin amorphous silicon oxide layer is observed on the Si NR surface, resulting from the passivation treatment during synthesis. The thickness of this oxide layer depends on the surface activity of the Si nanostructures, with the number of active sites and surface energy determining the oxide content. AFM analysis (Figure 1c) and the corresponding cross-sectional profile (Figure 1d) confirm an average thickness of ~6.2 nm and width of 13 nm for the Si NRs, similar to the 7.5 nm thickness reported for silicon nanosheets [15]. Based on the collected powder mass, the synthesis efficiency is calculated to be approximately 18.6 g · h $^{1}$.

## 2.2 Structure of Silicon Nanoribbons

Figure 2a shows XRD patterns of bulk silicon and Si NRs, with characteristic diffraction peaks confirming the cubic crystal system (JCPDS 05-0565). Two key features are evident: first, the diffraction peaks of Si NRs are significantly broadened compared to bulk silicon, indicating that the nanostructures consist of fine crystallites; second, while the strongest peak for bulk silicon is the (220) plane, Si NRs exhibit the strongest peak for the (111) plane, demonstrating anisotropic crystal growth consistent with HRTEM analysis. The anisotropic growth can be quantified using the intensity ratio I(111)/I(220), which is 0.83 for bulk silicon and 2.08 for Si NRs, confirming the pronounced anisotropic characteristics resulting from nucleation and growth in the hydrogen-helium atmosphere.

Raman spectroscopy further characterized the Si NRs across 100–1200 cm $^{1}$ (Figure 2b). Characteristic silicon vibrational modes are observed, including the first-order transverse optical (TO) phonon peak, the second-order transverse acoustic (2TA) phonon peak, and the second-order transverse optical (2TO) phonon peak. Bulk silicon shows a strong Raman peak at 520 cm $^{1}$ with a full width at half maximum (FWHM) of 10 cm $^{1}$, corresponding to Si-Si bond stretching vibrations. For Si NRs, the TO peak shifts to 498 cm $^{1}$ with an FWHM of 22 cm $^{1}$, indicating significant changes in phonon scattering characteristics due to the nanoscale sheet structure.

Silicon has a conventional face-centered cubic structure where each Si atom is covalently bonded to four neighbors with long-range order in bond length and angle [16]. The 22 cm $^{1}$ shift of the Raman TO peak to lower frequency in Si NRs likely originates from size effects. As AFM and HRTEM reveal a limiting dimension (thickness) of 6.2 nm, the periodic boundary conditions of the silicon lattice are constrained, causing breakdown of translational symmetry and reduction of the correlation length, which affects first-order optical phonon behavior. In Raman scattering, when the correlation length becomes less than one-tenth of the optical wavelength, silicon phonons transition from vibrational to localized modes [16], explaining the significant peak shift and broadening, as theoretically demonstrated by Faraci et al. [17]. In two-dimensional silicon nanocrystals, low-frequency Raman shifts may also be attributed to quantum confinement effects [12,18,19], where phonons are confined in potential wells and electrons with two-dimensional subband energies are frozen in the z-direction, moving only in-plane [16].

XPS analysis of the Si NR surface composition is shown in Figure 2c for Si 2p binding energies. A peak at 99.6 eV corresponds to elemental Si (Si ) from the Si NR core, while peaks at 102.9 eV and 103.4 eV indicate slight surface oxidation. The 103.4 eV peak corresponds to stoichiometric SiO  [20], while the SiO  peak at 102.9 eV represents oxygen-excess doped silicon oxide with an O:Si atomic ratio of approximately 5.6:1. The N  adsorption-desorption isotherm for Si NRs (Figure 2d) shows a Type V isotherm [21] with sharp capillary condensation at high pressure, indicating low micropore content and predominant mesoporous structure likely arising from inter-ribbon stacking. The measured specific surface area is ~164 $m^2 \cdot g^{1}$. The small thickness of Si NRs and high surface oxygen content revealed by TEM, AFM, and XPS indicate abundant lattice defects, with the small dimensions and defect density contributing to the high specific surface area.

### 2.3 Formation Mechanism

The Si NR nanostructures result from nucleation and anisotropic growth of Si atoms under plasma conditions containing high-energy H  and He  ions. During arc discharge, the arc flame can be divided into inner, middle, and outer zones [22,23]. The inner zone has high silicon atom concentration where gas-phase nucleation occurs, followed by growth in the middle and outer zones [22-24]. H  ions promote silicon atom evaporation, while He  ions facilitate condensation and modify the surface energy state of nuclei.

Crystalline silicon has a face-centered cubic (FCC) structure with different atomic densities on various crystallographic surfaces: $r(100) = 2/a^2$, $r(110) = 4/(2^1/^2a^2)$, and $r(111) = 4/(3^1/^2a^2)$. The surface energy of each plane can be expressed as   = ½N_b r_a [25], where N_b is the number of broken bonds,   is half the bond strength, and r_a is surface atomic density. With an atomic coordination number of 12 in FCC silicon, the {100}, {110}, and {111} surfaces have N_b values of 4, 5, and 3, respectively. This yields surface energies of  {100} = 4 $/a^2$,  {110}   3.46 $/a^2$, and  {111}   2.45 $/a^2$, showing that {111} surfaces have the lowest energy and are most stable, making <111> the slowest growth direction. Under H  and He  ion bombardment, however, the surface energy states are altered, making <111> one of the favorable growth directions [26].

The periodic bond chain (PBC) theory also predicts preferred growth directions [27,28]. According to this principle, {100} surfaces are flat (F-faces) with one PBC crossing the surface, {110} surfaces are stepped (S-faces) with two PBCs, and {111} surfaces are kinked (K-faces) with three PBCs. In simple cubic crystals, {110} and {111} faces grow faster than {100} faces [27]. These analyses indicate that under H  and He  ion influence, Si nuclei possess the conditions for anisotropic growth, forming fine sheet-like cluster nuclei.

The growth of Si NRs proceeds via coalescence of these sheet-like cluster nuclei. In the hydrogen-helium mixed atmosphere, He  provides efficient condensation and energy exchange, producing smaller nuclei than those formed under Ar  condensation, which favors growth by cluster accumulation (coalescence growth). Additionally, helium' s low diffusion coefficient [29,30] sustains the condensation effect during stacking of fine sheet-like nuclei, promoting secondary nucleation (coalescence) and forming ribbon-like nanostructures with ordered sheet arrangement, as shown in Figure 1b. The Si NR growth direction (ribbon axis) forms a ~20° angle with the <111> direction of the sheet-like nuclei, allowing calculation of the growth direction as <112>.

### 2.4 Electrochemical Performance of Silicon Nanoribbons

Electrodes using Si NRs as the active material were prepared and assembled into CR2025 coin half-cells for electrochemical testing, with results shown in Figure 3 [Figure 3: see original paper]. Figure 3a displays the first three cyclic voltammograms of the Si NR electrode. During the first cathodic scan, a broad peak appears at 0.52 V that disappears in the second cycle, corresponding to formation of the solid electrolyte interphase (SEI) layer. A prominent broad peak between 0.25–0.01 V corresponds to Li  insertion and transformation of amorphous LixSi to crystalline Li  Si  [31]. During the first anodic scan, two broad peaks at 0.36 V and 0.55 V represent Li  extraction from Si-Li alloys, with peak currents gradually increasing in subsequent cycles, indicating that Li  extraction requires progressively more energy with cycling [32]. The phase transformation sequence is: Si (crystalline) → LixSi (amorphous) → Li  Si  (crystalline) → Si (amorphous) [33]. No redox peaks from the Super P conductive additive are observed.

Figure 3b shows the initial two charge/discharge curves, revealing first-cycle discharge and charge capacities of 2460 and 1508 mAh·g$^{1}$, respectively, with a reversible capacity of 1493 mAh·g$^{1}$ in the second cycle and a Coulombic efficiency of 83%. Figure 3c illustrates capacity evolution over 40 cycles, with reversible capacity stabilizing at 316 mAh·g$^{1}$ and Coulombic efficiency reaching ~93%, exceeding the practical capacity of conventional carbon materials. The large irreversible capacity in the first cycle likely originates from Li  consumption during SEI film formation [34,35]. These results demonstrate high initial electrochemical activity of Si NR-based electrodes, with the surface composition of Si nanostructures facilitating lithiation/delithiation and delivering high lithium storage capacity. However, the large surface area also promotes SEI formation, causing significant irreversible capacity loss. The cycling performance indicates that some Li  becomes irreversibly trapped in the Si lattice [36], and that single two-dimensional nanostructures alone cannot adequately accommodate volume changes, leading to deactivation or loss of electrical contact for many active components.

To further investigate interfacial changes, electrochemical impedance spectra were analyzed before cycling and after three cycles (Figure 3d). The depressed semicircles in the high-to-medium frequency region correspond to SEI film formation during initial cycling and charge transfer at the interface after cycling. The semicircle diameter increases significantly after three cycles because repeated lithiation/delithiation causes volume changes in Si active units, loosening contact between active material and conductive additive [37] and gradually weakening electrical contact and lithium capture capability. New SEI formation on fresh surfaces also increases system impedance. The low-frequency line corresponds to Li diffusion impedance in the electrode [38], with the slope increasing after three cycles, indicating enhanced Li diffusion [39]. These results demonstrate that Si NRs exhibit promising lithium storage electrochemical activity and are potential candidates for lithium-ion battery anodes.

## Conclusion

Using DC arc-discharge plasma—a method distinct from conventional chemical and vapor-phase approaches—Si nanoribbons were synthesized in a hydrogen-helium mixed atmosphere. These Si nanoribbons have widths of 28 nm, lengths exceeding 200 nm, thicknesses of approximately 6.2 nm, and specific surface areas of about 164 $\mathrm{m^{2}\cdot g^{1}}$. This work provides a low-cost synthesis route for silicon nanoribbons. The formation of Si nanoribbons originates from anisotropic growth under the combined influence of H and He ions, with fine sheet-like cluster nuclei stacking along specific crystallographic directions through a coalescence-dominated growth mechanism. Due to the small dimensions and altered lattice periodicity, the XRD patterns and Raman spectra of Si nanoribbons differ significantly from bulk silicon, showing peak broadening and Raman shift. A passivation layer forms on the Si nanoribbon surface, composed of stoichiometric SiO and non-stoichiometric oxygen-doped silicon oxide. The Si NRs demonstrate favorable electrochemical lithium storage activity.

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