Nanostructures generated during SiC graphitization process

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Introduction: *Surface morphological features and nanostructures generated during SiC graphitization process can significantly affect the fabrication of high-quality epitaxial graphene on semiconductor substrate. The research is still lacking about the generation and morphological evolution of various nanostructures all through the thermal decomposition of SiC, as well as the understanding of their thermodynamics and kinetics. Therefore, the detailed information about the localized surface morphologies and individual growth features is worth examining during SiC graphitization process, and further exploration is warranted in order to know if annealing procedures either promote or hinder graphitization.*

Experimental methods

■ Omicron UHV STM system @ RT ------

Base pressure better than 2.0×10^{-10}*mbar*

STM data processing system — Matrix 3.1

Direct-current heating and infrared pyrometer measurement

Bias voltage at +3 ~ -3V, tunneling current at 0.2 ~ 1.5nA

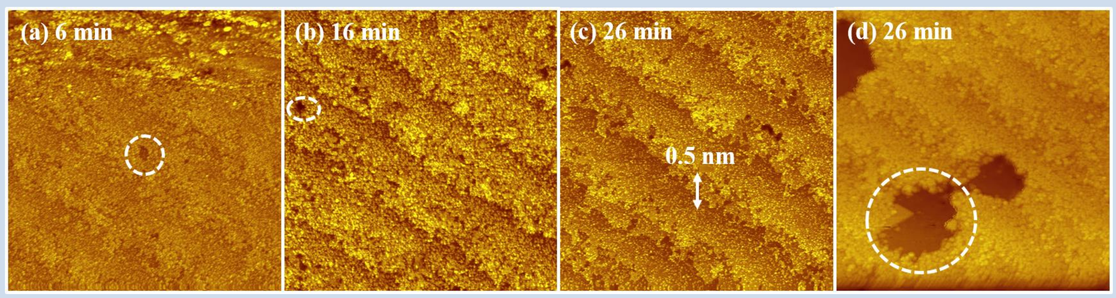
■ Substrate and methods ------

4H-SiC(0001), with N doping density of $10^{18\text{-}19}$ cm^{-3}

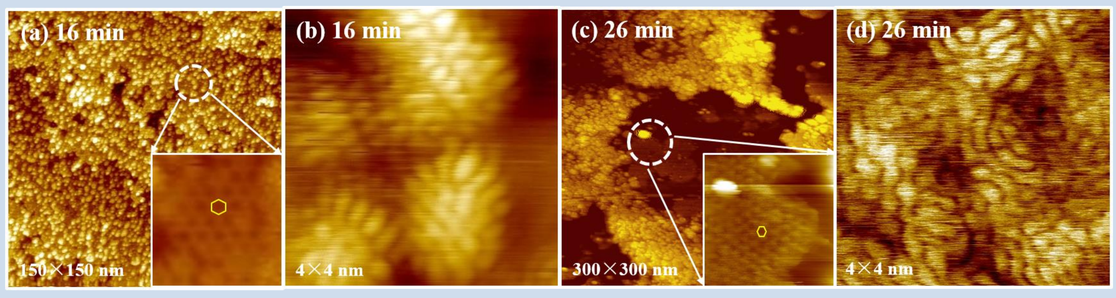
degassed at 950℃ for 30min, annealed at 1100 ℃ for 6 ~ 26min

the clean substrate annealed at 900 ℃, 1000 ℃, 1100 ℃, 1200 ℃ and 1300 ℃ for 10min, respectively.

Surface morphologies of clean SiC

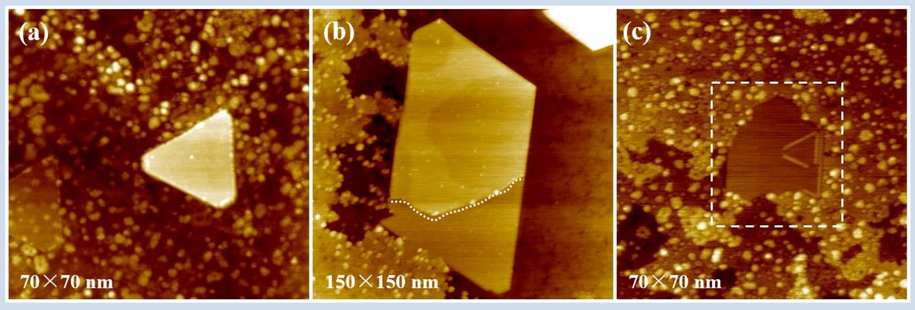


- With the increasing of annealing time, the surface terraces and steps become more and more defined.
- Steps in (c) are composed of two Si-C bilayers along the (0001) direction and the whole height of steps is 0.50 ± 0.03nm.
- Pits of size 10-50nm appear on the step, expanding to 200nm, and nanostructures inside.



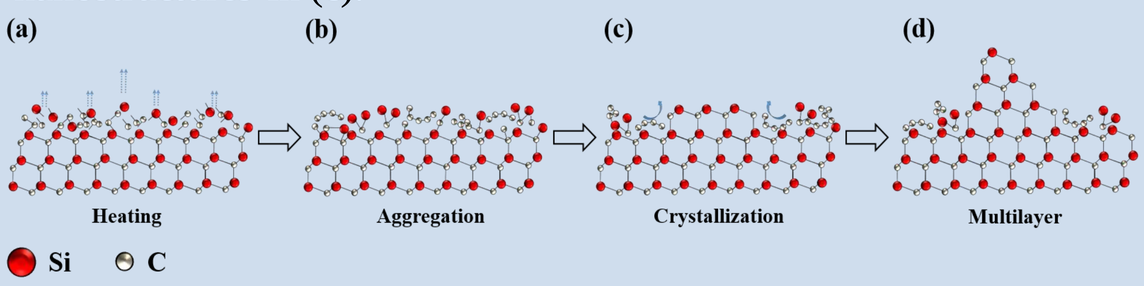
- ➢ Typical $(\sqrt{3}\times\sqrt{3})$ and (6×6) reconstruction on clean surface of SiC.
- ➢ The dome features magnified in (b) corresponds to a hexagonal network with C-C bonds of the so-called "carbon nanocaps".
- ➢ Carbon atoms are aggregated into carbon nanocaps which subsequently join together to form a buffer layer.

Nanostructures generated during graphitization process



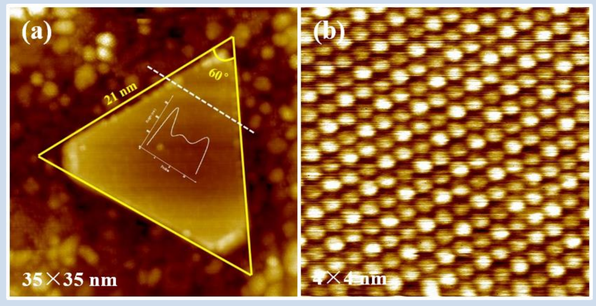
Nanostructures on the clean surface of SiC after 1100℃ annealing for 26min.

- Non-accidentally and randomly distributed in terrace, with 10-100nm in size & 0.1-0.4nm in height.
- Multilayer configuration, with a step height of 0.20 ± 0.02nm between the upper and lower terraces in (b).
- A small number of nanowires on the smooth surface of nanostructures in (c).



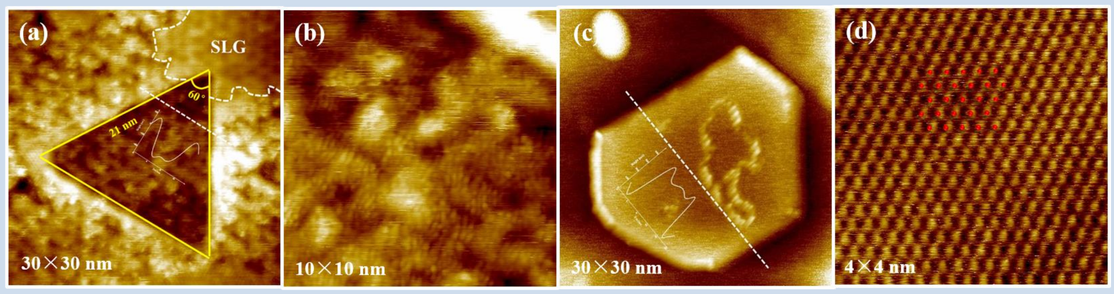
Schematic diagram for the SiC (1×1) nanostructure formation and growth.

- (a) Heating causes the Si atoms to desorb from the substrate and the surface atoms become active.
- (b) $(\sqrt{3}\times\sqrt{3})$ structure exists among the nanocaps, and (6×6) configurations appear randomly distributed on the surface.
- (c) When C atoms migrate to a suitable region and the ratio with Si atoms is exactly 1:1, recrystallize into SiC nanostructures.
- (d) Appearance of nanowires or multilayers on the surface of nanostructures.



A triangular nano-island after annealed at 1100℃ for 26 min

- ➢ The triangular nano-island shows an ordered structure with a lattice consistent with that of 4H-SiC.
- ➢ Graphitization process: $(3\times3)_{SiC}\rightarrow(1\times1)_{SiC}\rightarrow(\sqrt{3}\times\sqrt{3})_{SiC}\rightarrow(6\sqrt{3}\times6\sqrt{3})_{SiC}+(1\times1)_{graphene}\rightarrow(1\times1)_{graphene}$.
- ➢ The nanostructure is identified as SiC(1×1) crystalline structure, called 'SiC nanostructure'.



Two typical nanostructures on the surface after annealing at 1300℃ for 10 min

- ➢ Reduction in the number of nanostructures due to high temperature decomposition of SiC.
- ➢ An equilateral triangular nanohole surrounded by graphene, with (6×6) construction interior.
- ➢ Elliptical nanostructure covered by graphene with ring defects on the graphene surface in (c).
- ➢ Multilayer graphene on the surface of nano-island due to that only three of the six C atoms in the benzene ring in (d).

Conclusion:

- Exhibiting the appearance and gradual developments of SiC(1×1) nanostructures after 1100℃ annealing treatments, irregularly distributed among carbon nanocaps and $(\sqrt{3}\times\sqrt{3})$ reconstruction domains.
- A model for the formation and growth progression of SiC(1×1) nanostructures has been proposed.
- Triangular nano-pits and nano-islands on the surface due to thermal decomposition of SiC, with multilayer graphene covering on the nano-islands.

References:

1. *K. S. Novoselov, A. K. Geim, et al. Science 2004, 306: 666-669.*
2. *D. Geng, J. Hu, et al. ACS Appl. Mater. Inter 2019, 11: 39109-39115.*

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